

**Potato peel and Date palm waste char:  
Production techniques and their effects on soil  
properties and plant growth**

**Monica Isukapalli**

**Doctor of Philosophy**

**University of York**

**Department of Environment and Geography**

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## Abstract

In arid regions such as the United Arab Emirates, where 80 % of the land consists of deserts, sandy soils pose significant agricultural challenges due to their low organic carbon content, poor water retention capacity, and high nutrient leaching. This study investigates the potential of potato peel (PP) waste and palm waste (PW) chars, produced through microwave pyrolysis, conventional pyrolysis and microwave hydrothermal carbonisation, to improve soil properties, nutrient retention, and plant growth in sandy soil. Three wheat-pot experiments were conducted under controlled conditions over 12 weeks.

Char characterisation revealed that production temperature and feedstock composition significantly influenced biochar physiochemical properties. The initial lab experiments revealed that higher pyrolysis temperatures reduced biochar yield but increased surface area and porosity. Chars with high cationic exchange capacity (CEC), particularly potato peel biochars produced via conventional (PP-CB) and microwave pyrolysis (PP-PB), and palm waste biochar produced via conventional pyrolysis (PW-CB), significantly reduced water leaching and leaching of nutrient, including calcium ( $p \leq 0.05$ ), magnesium ( $p \leq 0.05$ ), and nitrate ( $p \leq 0.05$ ), while enhancing plant growth. In contrast, chars produced via hydrothermal carbonization (PP-MWH and PW-MWH) were hydrophobic, leading to increase leachate volumes and nutrient losses. Despite PP-CB exhibiting high CEC, excessive potassium leaching ( $p \leq 0.05$ ) was observed, leading to nutrient imbalances and inhibited root development.

Plant growth analysis showed that char amended soils generally improved biomass yield and nutrient uptake, except for hydrothermal chars and PP-CB. Leaf number remained similar across treatments during the initial stages but increased significantly from weeks 6 to 12 ( $p \leq 0.05$ ).

This study compares the effectiveness of different char produced from different feedstocks and production methods on char properties, soil leachate, nutrient retention, and dissolved organic carbon leaching. These findings suggest that PP-PB, PW-CB are the most effective treatments for improving soil fertility and crop productivity in arid environments.

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## List of Abbreviations

<i><b>Abbreviations</b></i>	<i><b>Definitions</b></i>
<i>BC</i>	Biochar
<i>C</i>	Control
<i>Ca</i>	Calcium
<i>CB</i>	Conventional Biochar
<i>Cd</i>	Cadmium
<i>CEC</i>	Cationic exchange capacity
<i>Cl</i>	Chloride
<i>cmol/kg</i>	Centimole of positive charge per kilogram
<i>CR</i>	Crop residue
<i>Cr</i>	Chromium
<i>CRM</i>	Certified reference material
<i>Cu</i>	Copper
<i>DL</i>	Detection limit
<i>DOC</i>	Dissolved organic carbon
<i>Fe</i>	Iron
<i>FW</i>	Food waste
<i>g</i>	Grams
<i>ha</i>	Hectare
<i>hg/ha</i>	Hectogram per hectare
<i>HTC</i>	Hydrothermal carbonisation
<i>IBI</i>	Internation Biochar Initiative
<i>ICP</i>	Inductively couple plasma
<i>K</i>	Potassium
<i>kg</i>	Kilogram
<i>Kt</i>	Kilotonne
<i>m</i>	Meter
<i>mg</i>	Milligram
<i>min</i>	Minutes
<i>Mn</i>	Manganese
<i>mol/kg</i>	Moles per kilogram
<i>Mpa</i>	Mega Pascal
<i>MW</i>	Microwave
<i>MWB</i>	Microwave Biochar
<i>MWH</i>	Microwave Hydrochar
<i>N</i>	Nitrogen
<i>Na</i>	Sodium
<i>NH<sup>+</sup><sub>4</sub></i>	Ammonium
<i>Ni</i>	Nickel

$NO_3^-$	Nitrate
P	Phosphorus
PB	Pure Biochar
Pb	Lead
PCB	Partially Charred Biochar
PP	Potato peel waste
PSI	Pounds per square inch
PW	Palm waste
s	Seconds
S	Sulphur
SEM	Scanning electron microscopy
SSA	Specific Surface Area
TGA	Thermogravimetric Analysis
UAE	United Arab Emirates
W	Watt
WB	Woody Biomass
WDPT	Water drops penetration test
wt	Weight
Zn	Zinc



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To my wonderful and loving family in the UK, Amy, Adrien, Gillian and Ben, thank you for taking care of me and supporting me during this significant chapter of my life. Your kindness and encouragement have made all the difference.

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Lastly, I thank God for providing me with strength, resilience and blessings to pursue and complete the work.

## **Declaration**

I declare that this thesis is a presentation of original work, and I am the sole author. This work has not previously been presented for an award at this, or any other, University. All sources are acknowledged as References.

The Results and the discussion from Chapters 7 and 8 in this thesis are presented for publication as a paper entitled 'Date Palm waste Biochar: Production techniques and their effects on soil properties and plant growth' included as a chapter in 'Sustainable Valorization of Date Palm By-Products and Waste'. I designed the experiments, carried out the practical work, analyzed the data and produced the first draft. Co-authors commented on the initial and subsequent drafts.

Results and discussion from Chapters 5 and 6 has been presented in the British Soil Science Early Career Research's Conference in December 2023. The Literature review from Chapter 2 has been presented internally in the Department of Environment and Geography, University of York, 2022.

## Chapter 1: General introduction

Soils found in arid and semi-arid regions are often characterised by a coarse sandy texture, low organic carbon and poor water retention and a high susceptibility to nutrient leaching (Elie et al., 2024; El-Naggar et al., 2019). This makes the agricultural management of such soils a significant challenge, particularly if the aim is to increase crop yield. One potentially effective method to improve the properties of such soils is the addition of char (Musei et al., 2024; Lehmann and Joseph, 2015; Jeffery et al., 2011).

According to the International Biochar Initiative (IBI), char is defined as a porous solid material that is generated through biomass pyrolysis in an oxygen-limited environment (Bhattacharya et al., 2024). Char is considered suitable for enhancing soil fertility because of its characteristics such as high porosity, alkalinity, and high cationic exchange capacity. Its introduction into soil increases the specific surface area (SSA) of the soil by promoting internal porosity (Baiaumont et al., 2021). Its application to sandy soils improves water and nutrient retention, enhances soil aggregation, and soil organic carbon content (Laghari et al., 2015).

The development of technology over years have provided various processes for the conversion of biomass into value-based products such as char, oil, and gas. Among the known thermochemical processes, the most used to produce char is pyrolysis due to its ability to recover the chemical and calorific value of biomass feedstock (Mutsengerere et al., 2019; Lee et al., 2019). Depending on the temperature and the residence time, there are three types of pyrolysis: slow pyrolysis, fast pyrolysis, and flash pyrolysis (Foong et al., 2020). Hydrothermal carbonization (HTC) is an emerging thermal technique to produce char (Fu et al., 2019; Petrović et al., 2024). It involves heating at a low temperature (180 – 300° C) in the absence of oxygen under pressure (2-6 Mpa) for 5 -20 min, using water as the reacting medium (Kambo and Dutta, 2015; Ischia and Fiori, 2020). Research on the pyrolysis and HC of biomass had traditionally focused on conventional heating methods. However, microwave-assisted pyrolysis and HTC are gaining attention as promising alternatives due to their various advantages (Huang et al., 2016). Unlike conventional techniques, microwave technology directly heats biomass, resulting in faster and more uniform heating. This method had been

shown to significantly reduce energy consumption throughout the production process (Nizamuddin et al., 2018). Additionally, microwave assisted methods enhance process efficiency by facilitating safer chemical reactions, increasing product yield, and shortening reaction times (Siddique et al., 2022).

The properties of char depend on several factors such as the composition of the feedstock, pre-treatment process and temperature (Banik et al., 2018). Different types of feedstocks have been used to produce char such as woody biomass, crop residue and organic waste (Gul et al., 2015). Many studies in recent years, have focused on the use of woody biomass (WB) and crop residue (CR) as feedstocks to produce char. WB and CR upon pyrolysis have low ash content due to the presence of high organic carbon percentage in the feedstock compared to organic waste. One of the main drawbacks of WB and CR is low content in elements such as nitrogen (N), phosphorus (P), potassium (K) and calcium (Ca) making them unsuitable for providing nutrients to the soil (Ji et al., 2022). However, if the aim is to increase the nutrient content in the soil, a recent study on food waste revealed that they have a high source of nutrients present in them making them suitable as char for increasing the nutrient concentration in soil (Ji et al., 2022).

Potatoes are considered as one of the most important crops for human consumption globally. In 2021, the total consumption of potatoes reached around 402 kt in the United Arab Emirates (UAE) with a population of 9.8million people (Helgilibrary, 2023; World Population Review, 2024). Each year, potato peel as a by-product of potato consumption goes to waste. Most peel waste is either sent to landfills or incinerated, methods that are not environmentally suitable due to the various issues they cause (Önal et al., 2012). Over years, with the increase in the population, there had been an increase in the food waste generated. Food wastage is one of the most critical problems in the UAE (Kohli, 2022; DCCE, 2022). Char production is one of the most sustainable pathways that integrate the principles of circular economy, making it a sustainable approach for transforming waste into valuable products. There had been an increase in the studies conducted on the use of food waste as char, but very few studies have investigated the effects of food waste char in sandy or desert soils (O'Connor et al., 2021; Bashir et al., 2017; Bong et al., 2020).

Date palm trees (*Phoenix dactylifera*) are considered as one of the major crops grown in the UAE (Clean Deal Gardens, 2022). During the lifespan of the palm trees, special care is required

for them to grow healthily especially the date palm trees to produce more crop. This includes the removal of offshoot, dead or dried leaves, and dried fruit. In recent years, most of the palm waste generated over the life span of the date palm is collected and usually used as compost or incinerated. Incineration of palm waste can cause environmental problems due to the release of nitrogen compounds, such as nitrous oxide and the substantial amount of smoke produced during the incineration process. Char production is a potentially sustainable alternative that aligns with the principles of circular economy, effectively converting agricultural waste into valuable products. Over the past few years, there have been various studies involved in the use of date palm waste as char (Elie Le Guyader et al., 2024; Al-Wabel et al., 2017; Sizirici et al., 2021; Alotaibi and Schoenau, 2019 and Sait et al., 2022).

The main aim of this study is to investigate the effects of char amendments on soil properties, specifically in sandy soils. The United Arab Emirates has around 80% of its land being desert (Tarantino, 2024). In arid and semi-arid regions like United Arab Emirates, one effective method to improve soil properties is the addition of char. The addition of organic matter in the form of char can help preserve soil fertility and increase nutrients content and water retention capacity (Baiamonte et al., 2020). Numerous studies have investigated the addition of char to improve the porous structure, increase water holding capacity, carbon content and nutrients in the sandy soil but, only a few studies have been conducted on the effect of char in desert soil (Laghari et al., 2015 and Alotaibi and Schoenau, 2019). Baiamonte et al. (2019) showed that the application of char to desert sand modified the soil pore size, increased soil porosity, and improved aggregation stability. Another study by Baiamonte et al. (2020) had conducted research in Abu Dhabi near Al Foah with forest biomass as char. It was seen to improve the structural and aggregation soil properties.

## **1.1 Research Aim and objectives**

The objective of this study is to explore and compare the physical and chemical properties of chars from different feedstocks (date palm waste and potato peel waste) and production techniques, as well as their impact on soil physical and chemical properties, focusing on nutrient retention, cationic exchange capacity, water retention and plant growth. The research initially aimed to identify optimal production parameters to enhance char's effectiveness as a sustainable soil amendment. However, the focus shifted to comparing chars produced using different production methods due to the constraints and scaling issues encountered during the experimental process. Later, the research aimed to compare and contrast the effectiveness of chars derived from two specific feedstocks: potato peel and palm waste, using different production techniques, in improving soil properties and promoting plant growth. This approach was chosen to evaluate how feedstock type and production techniques influence char properties and their potential as soil amendments.

### **Research objectives**

1. To investigate how char properties, change with varying production conditions for potato peel and palm waste feedstocks, providing insights into optimal conditions needed to maximize their effectiveness as soil amendments.
2. To compare char produced through different pyrolysis techniques (microwave pyrolysis, hydrothermal carbonization, and conventional pyrolysis) to assess how production methods influence char properties.
3. To evaluate the effect of char application on soil properties, with a particular focus on sandy soils.
4. To assess the impact of char on nutrient retention and plant growth in sandy soils.

5. To compare the effects of different feedstocks (potato peel waste and date palm waste) on the characteristics of chars and their influence on soil properties and plant growth.

## **1.2 Outline of the thesis**

This PhD consists of nine chapters, and the contents of each chapter are described briefly below:

Chapter 1 provides a brief introduction to the use of char for enhancing sandy soils in arid regions, addressing challenges such as poor water retention, low organic carbon content, and nutrient leaching. It introduces various char production techniques, including pyrolysis and hydrothermal carbonisation, with a focus on the use of microwave technology in these processes. Furthermore, it outlines the structure and objectives of the thesis, setting the context for the research.

Chapter 2 presents a literature review investigating the potential for using char's derived from potato peel waste and palm waste to enhance soil fertility. It explores various char production methods, including pyrolysis and hydrothermal carbonisation, with a focus on utilising microwave technology for these processes. This chapter also reviews the influence of production parameters and feedstock types on physical and chemical properties of char, examining their subsequent impact on soil quality and plant growth. Additionally, existing research on the application of char for soil remediation, as well as its use in the UAE, was reviewed to contextualize these findings within the current body of knowledge. This chapter provides information on the current state of knowledge and identifies relevant knowledge gaps.

Chapter 3 outlines the materials, methods, and analytical techniques used in this study to characterise the physio-chemical properties of char. It also details the methodology used to investigate the influence of different char treatments on soil properties and wheat growth. The chapter is organised into several sections, beginning with an overview of the raw

materials and their preparation. Following the preparation of raw material, the next section details the char production, along with the characterisation of the chars. Soil preparation for plant growth experiments are outlined, emphasising the soil treatment combinations used to evaluate the impact of char amendments on wheat plant growth and soil properties.

Chapter 4 investigates the conversion of potato peel waste and palm waste into char using a laboratory scale microwave reactor. The preliminary study aimed to investigate the properties of the resulting char, optimise pyrolysis parameters, and develop insights for scaling up char production for plant-pot experiments in the later chapters.

Chapter 5 and 6 presents the results of plant-pot experiments using chars derived from potato peel waste through microwave pyrolysis, conventional pyrolysis, and hydrothermal carbonisation. The results provide detailed insights into the characteristics of the derived chars and their effect on soil leachate volume, nutrient leaching and soil properties. The 12-week plant pot experiments further explore the impact of these chars on plant growth and the final yield of above and ground biomass, and their nutrient concentration in the biomass.

Chapter 7 examined the results of the plant-pot experiments using the chars derived from palm waste from microwave pyrolysis, conventional pyrolysis, and hydrothermal carbonisation. Similar analysis to those in Chapters 5 and 6 were applied to understand the effects of palm waste derived chars on soil properties and plant growth.

Chapter 8 provides a detailed discussion of the findings from Chapter 5, 6 and 7. The first section investigates the influence of different production techniques on char properties and examines how these chars affect soil properties and plant growth in both potato peels and palm waste studies. The second section examines the effect of feedstock type (potato peel and palm waste) on char properties, soil properties and plant growth.

Chapter 9 concludes the thesis by summarising the key findings, providing a critique of the research conducted, discussing the environmental implications of using chars as soil amendments, and identifying areas for future research in the context of specific findings of the thesis and in a broader context.



## **Chapter 2: Literature Review**

### **2.1 Introduction**

Char, a carbon rich material derived from biomass through thermochemical processes like pyrolysis and hydrothermal carbonisation, had emerged as a versatile solution for sustainable agriculture and waste management. Its properties such as high porosity, surface area and cationic exchange capacity, make it an effective soil amendment for increasing water and nutrient retention, and mitigating environmental challenges such as soil degradation and nutrient leaching.

This review investigates the production process, properties, and applications of char derived from diverse biomass sources such as food waste and agricultural residue like potato peel and palm waste with a particular focus on production methods like pyrolysis and hydrothermal carbonisation. The review also focuses on the use of sustainable technology such as the microwave technology to produce these chars. In this review, 'sustainable' refers to technologies that minimises environmental impact and enhances resources efficiency. For instance, microwave assisted pyrolysis is considered a sustainable char production method due to its rapid heating, reduced processing time and lower energy consumption compared to conventional techniques. By examining the impact of feedstock types, production conditions and the physiochemical properties of char, this chapter aims to provide a comprehensive understanding of its role in advancing sustainable practices. This literature review synthesises current research, highlights gaps, and identifies opportunities for optimising char production and its applications.

## **2.2 Biomass**

Biomass is defined as biological matter which is directly or indirectly produced from photosynthesis (Bioenergy, 2022). Biomass plays a significant role in the production of char, fuel, electricity and heat. In terms of alternative energy sources, biochar can serve as a potential renewable fuel due to its high carbon content and energy density, making it a strongly desired alternative by the European Union (Tursi, 2019). The world's total biomass is around 4.8 trillion tons. Apart, from being used as an alternative source for energy production, the use of char for agriculture purposes such as to improve soil fertility and the soil remediation is gaining interest which had been further discussed in this literature. Biomass can be derived from various sources.

The most used biomass is woody biomass which involves stems, branches, leaves, barks of different trees. The main source of woody biomass is the forest areas. It can also be derived from various commercial sources such as the wood processing industries in the form of shavings, sawdust, and chips (Tripathi et al., 2016).

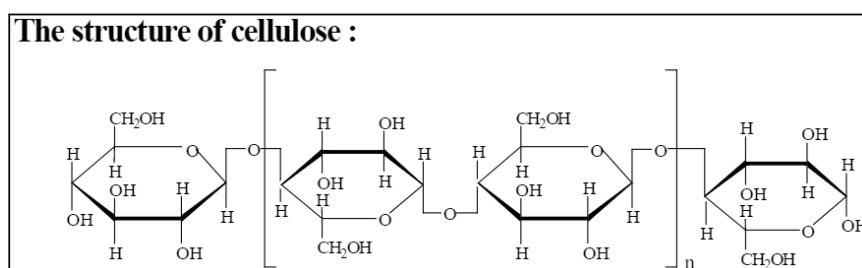
Human and animal waste could be considered as another category for biomass feedstock. Different animal manures, cooked and uncooked food, fruit and vegetable peels, coffee grounds, paper, plastic all fall into this category. Industrial waste such as food waste from food processing industries, paper sludge from paper industries and sewage sludge from municipal can also be considered in this category (Tripathi et al., 2016).

### **2.2.1 Lignocellulosic biomass**

Lignocellulosic biomass is composed of three main structural components: cellulose, hemicellulose, and lignin. It is one of the most abundant natural resources on earth, found in agricultural residues, forestry waste and various plant-based materials. Over years, lignocellulosic biomass had been gaining attention as a raw material to produce biochar due to its renewable properties, easy availability, and low cost (Yaashikaa et al., 2019).

## Cellulose

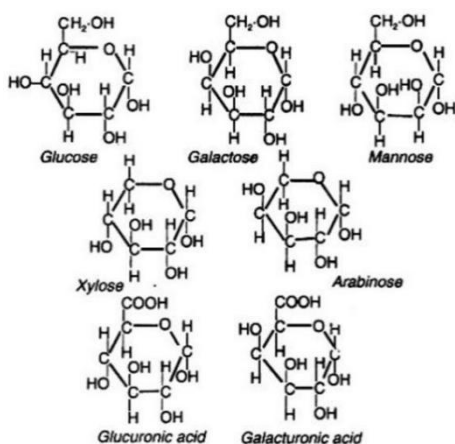
Cellulose is one of the most abundant organic polymers found in nature and it is considered as the fundamental building block of the plant cell wall (Tursi, 2019). Cellulose comprises repeating monomers of D-glucopyranosyl- $\beta$ -1,4-D-glucopyranose (Acharya and Chaudhary, 2012) (Figure 2-1). Upon, thermal decomposition, cellulose undergoes pyrolysis, breaking down into smaller molecules such as levoglucosan, furan, acetaldehyde and volatile compounds (Kumagai et al., 2021; Osatiashtiani et al., 2022). This process is influenced by factors like temperature and heating rate, which can affect the distribution of pyrolysis products.



**Figure 2- 1.** Structure of cellulose (Acharya and Chaudhary, 2012).

## Hemicellulose

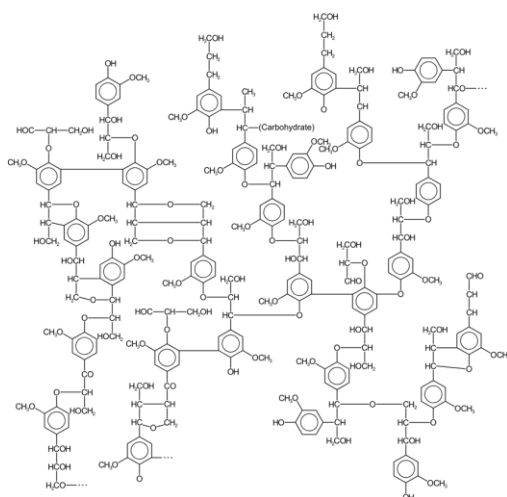
Hemicellulose is the second most abundant component of the plant cell wall. Hemicellulose is a linking material between cellulose and lignin. Hemicellulose structure comprises highly branched heteropolymers of xylose, along with glucose, mannose, galactose and arabinose (Dhyani and Bhaskar, 2018) (Figure 2-2). Hemicellulose undergoes degradation at a lower temperature range compared to cellulose, due to its amorphous structure and lower thermal stability. During this process, hemicellulose breaks down into a variety of volatile compounds, including glycolaldehyde, acetic acid, furfural, along with char and gases like CO and CO<sub>2</sub> (K N et al., 2022; Syguła et al., 2024).



**Figure 2- 2.** Monomer structure of hemicellulose (Dhyani and Bhaskar, 2018).

## Lignin

Lignin is a structural component found in the cell walls of plant, where it acts as natural adhesive, binding cellulose and hemicellulose together. This binding strengthens the cell wall and provides structural integrity, enhancing the plant's ability to withstand environmental stresses. Lignin is a phenolic polymer which is primarily made of three monomers units: a) guaiacyl propane, b) syringyl propane, and c) 4-hydroxyphenylpropane, which are chemically connected by alkyl-ether, carbon-carbon, and aryl-ether bonds (Ahuja et al., 2017) (Figure 2-3). Thermal decomposition of lignin exhibits a complex degradation process due to its highly cross linked and heterogeneous structure. Unlike cellulose and hemicellulose, lignin decomposes over a broader temperature range (300-550°C) and generates a variety of phenolic compounds, including guaiacol, syringol, phenol and other volatile organic compounds (K N et al., 2022; Syguła et al., 2024).



**Figure 2- 3.** Structure of lignin (Dhyani and Bhaskar, 2018).

### 2.2.2 Food waste biomass

Accumulation of waste has been increasing over years with the increase in the global population. It has been estimated that there is around 1.3 billion tons of food waste (FW) generated globally per year. The increase in FW can lead to various environmental problems (Elkhalifa et al., 2019). It has been estimated that FW emits over 20% of the total greenhouse gases such as methane and nitrous oxide from the landfill sites to the atmosphere (O'Connor et al., 2021).

Food wastage is one of the most critical problems in the UAE. An article published in 2022 stated that “In the UAE, an average person wastes around 224kg of food each year according to Food Sustainability Index 2020, where almost one-third of the food produced every year ends in the landfills” (Kohli, 2022). According to Dubai Carbon, roughly 38% of food prepared every day in the emirate is wasted (DCCE, 2022). The government had been discussing various ways to reduce FW and its impact on the environment. If systematic research had been done on FW in UAE, then there would not be a need for research on FW data (El Bilali and Hassen, 2020). The government had initiated a few programs to reduce organic waste and ensure a safe and sustainable future. Dulsco - an environmental organisation in Dubai partnered with EXPO 2020 to contribute to a more sustainable future with new technologies. Dulsco trucks

are now powered by biofuels which will collect all the waste from the EXPO 2020 site and are delivered to the Central Waste Facility, where the waste is further sorted into organic and inorganic waste. Organic waste mainly FW, fruits and vegetable waste were converted into compost and is used as fertiliser (News Details, 2022). There have been various other programs where the FW is converted into compost as mentioned in Hussein et al. (2021) but there have been studies based on the conversion of FW into biochar. Converting FW into value-added products will not only help in reducing landfill materials but also reduce greenhouse gases emission from them. FW can be turned into biochar which helps increase soil fertility and increase soil nutrients.

There is a wide range of methods that are currently being used for the disposal of FW including composting, aerobic fermentation, and thermochemical processes. Compositing is an effective way to recycle FW as it helps to provide an ideal environment for microorganisms and improve soil health. However, composting requires a large amount of space, high cost in the transportation of the waste and a long reaction time. Anaerobic fermentation takes place with the metabolic process conversion of FW into biogas in the absence of oxygen. The drawback for anaerobic fermentation is that it requires large capital investment to construct biogas plants and to purchase equipment and during the process toxic sulphur-containing compounds are produced. So, it is important to find an effective and sustainable process to dispose of FW. The thermochemical process had recently been gaining interest for its use to convert FW into value-based products such as biochar, bio-oil, and syngas. Thermochemical process offers a simple and energy-efficient way to dispose of FW. The different kinds of thermochemical processes used are discussed in further Section 2.4.

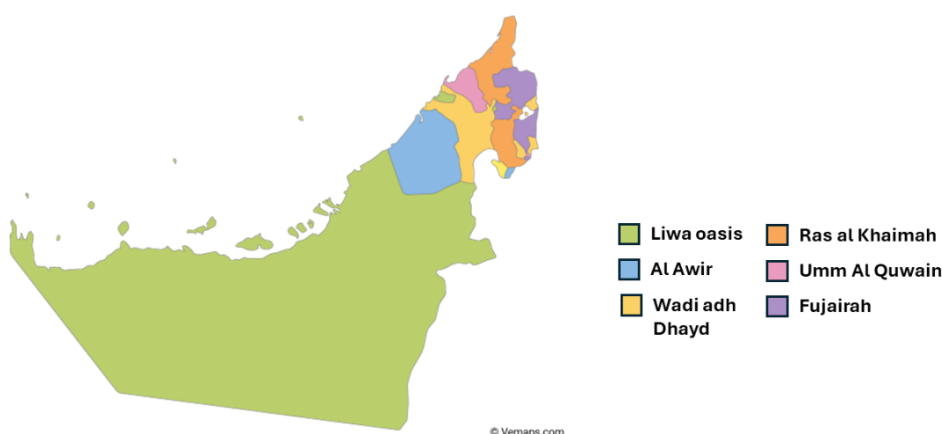
Typically, FW consists of 36% carbohydrate, 26% protein and 15% fat. FW is also rich in elements such as nitrogen (N), phosphorus (P), potassium (K), calcium (Ca), magnesium (Mg), carbon, and other minor elements such as iron and zinc (O'Connor et al., 2021). Thermal decomposition of the FW had a different effect on the yield of biochar compared to lignocellulose biomass. This is because of the stability and thermal degradability of the different polymer structures and the C pools in the FW biomass (Bong et al., 2020).

There have been few studies on the conversion of FW into biochar and its effects on remediation of metal contaminated soil. O'Connor et al. (2021) had shown that the use of orange peel as biochar helps reduce the solubility of lead (Pb) in the soil and enhance the

physical properties of soil. Application of sugarcane bagasse biochar to the soil had been shown to reduce the availability of cadmium (Cd) and Chromium (Cr) by 62.5 and 85% through adsorption and immobilization by the biochar. It was also observed that there was a significant decline in the uptake of Cd from 32% to 28% and Cr from 41% to 33% by the plant (Bashir et al., 2017). A study by Bong et al. (2020) investigated the use of peanut shell biochar. The peanut shell biochar was shown to have a high cationic exchange capacity (CEC) which results in a high ability to adsorb and retain nutrients such as ammonium ( $\text{NH}_4^+$ ) and  $\text{K}^+$ . There have been various studies on the use of FW as digestate and compost to provide nutrients and to immobilize the contaminants, but there is a knowledge gap on the use of FW in the form of char for soil remediation.

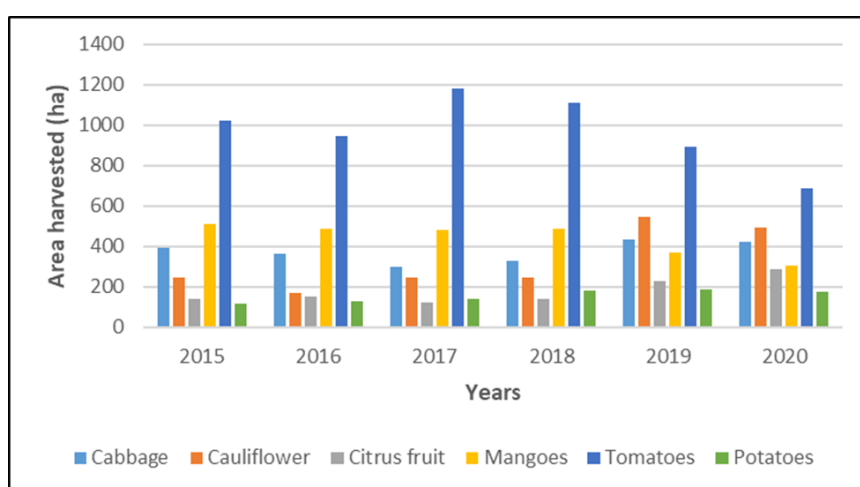
### 2.3 Agriculture in UAE

For many years, among the Emirati people, agriculture had been one of the most common trades. Figure 2-4 shows some of the major areas in UAE for farmers to carry out their cultivation have been Ras al Khaimah, Umm Al Quwain, the coastal areas of Fujairah, Wadi adh Dhayd in Sharjah, Al Awir in Dubai and Liwa Oasis in Abu Dhabi (MyBayut, 2024). Agriculture in UAE had been carried out on a total of 160,000 hectares of cultivable land. Most of the cultivable land is currently being used for date palm cultivation.



**Figure 2- 4.** Geographical distribution of agricultural regions in the United Arab Emirates (MyBayut, 2024).

Over the years, the government had encouraged farmers to increase the production of other crops by providing them with a 50% subsidy on seeds. In recent years, there had been an increase of around 30,000 farms across UAE with an exponential increase from 4,000 farms in the year 2020. Apart from the date palm, there are several other crops grown in UAE including tomatoes, potatoes, cabbage, cauliflower, and squash. Other than vegetables, a few citrus fruits and mangoes are also grown (MyBayut, 2022). Figure 2-5 shows the data on the amount of area harvested for various crops from the year 2015 to 2020. After date palm, tomatoes are considered to have the highest production rate in UAE (FAOSTAT, 2022).



**Figure 2- 5.** Various crops harvested in United Arab Emirates from 2015 to 2020 (FAOSTAT, 2022).

### 2.3.1 Potato peel waste in UAE

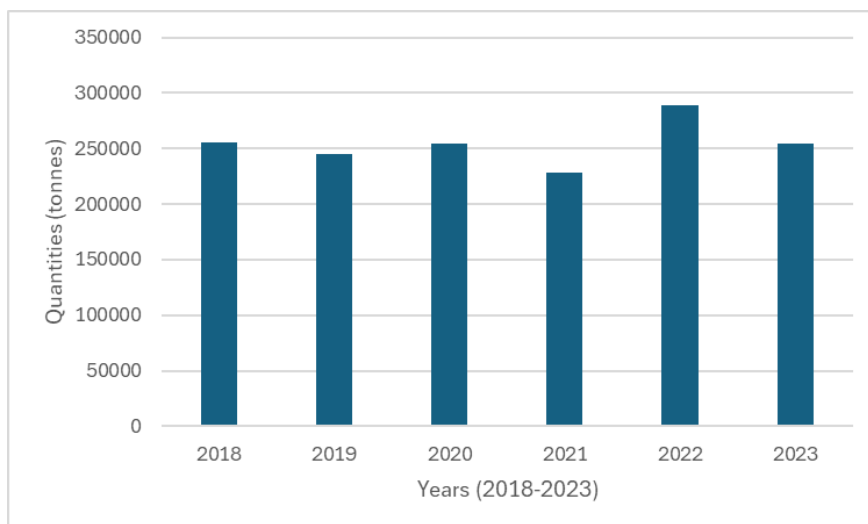
A wide variety of crops are grown and harvested around the world, but potatoes are among the most abundant crops after sugarcane, maize, rice, and wheat (Table 2-1) (Statista, 2022). Potato peel accounts for around 4% of world crop production along with oil palm fruit (Agricultural production statistics, 2022).

**Table 2- 1.** Production volume of most produced food worldwide in 2019 (Statista, 2022).



<b>Crops</b>	<b>Volume in million metric tonnes</b>
Sugarcane	11949.3
Wheat	1148.5
Rice, paddy	755
oil palm fruit	410.7
Potatoes	370.4
Cassava	303.6
Tomatoes	180.8
Barley	159
Bananas	116.8
Watermelons	100.4

In 2020, around 359 million metric tons of potatoes were produced globally (Gholami and Rahimi, 2022). Potatoes are considered one of the most important crops for human consumption globally. In 2019, the total consumption of potatoes reached around 163kt in the United Arab Emirates (UAE) with a population of 9.771 million people (Potato Consumption (Total) in the United Arab Emirates, 2022). Most of the potatoes consumed in UAE are imported from various countries such as India, Pakistan, and United States (Volza - Grow Global, 2022). Around 200,000 tonnes of potatoes are imported to UAE from various countries every year. The total potatoes import was the highest in the year 2022 with 280,000 tonnes of potatoes and the lowest in 2021 as deduced from the available data (Figure 2-6) (FAOSTAT, 2023). UAE had been ranked the top 1 import market for potatoes with around 14,830 shipments every year (Volza - Grow Global, 2022).



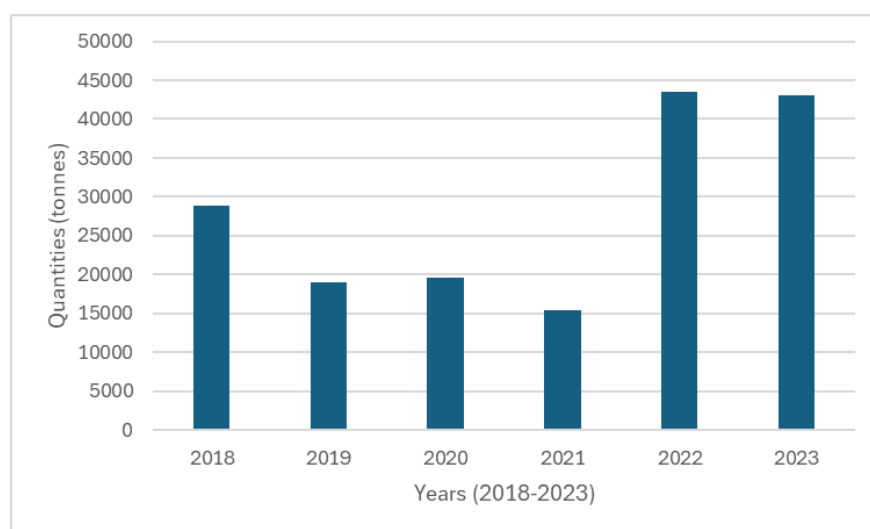
**Figure 2- 6.** Annual import quantities of potatoes to United Arab Emirates from 2018 to 2023, measured in tonnes (FAOSTAT, 2023).

In addition to relying on potato imports for consumption, UAE had also begun harvesting its own potato crops. Over the years, the farmers have gained more experience and improved their methods taking into consideration the harsh weather conditions of UAE and increasing the production of potatoes over the years despite this the production and the area of planting have decreased consistently. Table 2-2 summarises the area harvested, yield and production of potatoes from 2018 to 2023 in UAE (FAOSTAT, 2023).

**Table 2- 2.** Area harvested, yield of the crop and the production volume of the potato peel in United Arab Emirates from 2018-2023 (FAOSTAT, 2023).

	2018	2019	2020	2021	2022	2023
<b>Area Harvested (ha)</b>	141 (not accurate)	186	177	74	50	49
<b>Yield (hg/ha)</b>	250556	225430	239435	25003	25820	26111
<b>Production (tonnes)</b>	451	4193	4238	1853	1291	1288

Apart from being the leading import market for potatoes, UAE is also the leading export market for potatoes to Oman, Bahrain, Kuwait, Pakistan, and Afghanistan (Wamucii, 2022). The total potato export quantities from UAE to other countries from 2018 to 2023 have been summarised below in Figure 2-7 (FAOSTAT, 2023).



**Figure 2- 7.** Annual export quantities of potato peels from United Arab Emirates from 2018 to 2023, measured in tonnes (FAOSTAT, 2023).

Each year, significant quantities of potato peel by products from potato consumption are discarded as waste. Based on the 2019 national consumption of approximately 163kt of potatoes, and assuming the peels contribute 10% of the total potatoes weight, it is estimated that UAE generates roughly 16,300 tonnes of potato peel waste annually (Almeida et al., 2023). A large portion of this waste ends up in landfills or is incinerated, that causes several environmental challenges. Landfilling is unsustainable due to limited landfill capacity and the potential for chemical leachates from accumulated waste to contaminated groundwater, leading to environmental pollution. Incineration of organic waste, including potato peel, can release harmful pollutants such as nitrous oxide, sulphur oxides, particulate matter, and dioxins into the atmosphere, contributing to air quality degradation and associated health risks (Önal et al., 2012).

Potato peel waste from household waste, restaurant and even food processing industry waste makes it a suitable raw material to produce biochar as it is easily available and also cheap raw product (Singh et al., 2022). Potato peel waste can be considered a promising biomass

resource for the large-scale production of biochar, bio-oil, and various chemicals.

In recent years, there have been various studies based on the pyrolysis of potato peel. Most of the literature found focused on the use of potato peel biochar as an adsorbent and there have been very few studies focused on the use of potato peel biochar to improve soil fertility. Table 2-3 summarises the recent literature on the use of potato peel as biochar, focusing primarily on the pyrolytic conditions employed in these studies. This table highlights typical parameters such as type of reactor, temperature ranges and residence time, that influences biochar production.

**Table 2- 3.** Summaries of studies on various pyrolysis techniques used to produce potato peel waste biochar and their applications.

Aim of the study	Production parameters	Main findings	References
Potato peel biochar as adsorbent for chlorpyrifos removal	<ul style="list-style-type: none"> <li>• Slow pyrolysis in a muffle furnace at 350°C for 2hr.</li> </ul>	<ul style="list-style-type: none"> <li>• SEM images showed that slow pyrolysis led to the release of volatile matters and increased the biochar porosity.</li> <li>• Biochar activity in the soil</li> <li>• Maximum adsorption of the pesticide occurred at a biochar dosage of 1.04 g/L.</li> <li>• Maximum pesticide removal was achieved by the biochar when the solution pH was maintained at 5.04.</li> <li>• Biochar increases the chlorophyll content, phenolic compound, and antioxidants level of plants.</li> </ul>	Singh et al., 2022

Potato peels derived biochar: production, characterisation and potential applications	<ul style="list-style-type: none"> <li>Pyrolysis took place in a muffle furnace at 250, 300, 450 and 600°C.</li> </ul>	<ul style="list-style-type: none"> <li>Potato peel biochar at 600°C had high ash content, rich in elemental components suitable as a bio-fertiliser.</li> <li>The biochar yield decreased with the increase in the temperature.</li> </ul>	Najdi, et al., 2020
Potato peel biochar for the adsorption and immobilisation of heavy metals in soil	<ul style="list-style-type: none"> <li>Pyrolysis took place in a muffle furnace under an N<sub>2</sub> environment at 550°C.</li> </ul>	<ul style="list-style-type: none"> <li>An increase in the soil pH, CEC, and organic carbon content upon the application of biochar.</li> <li>The maximum adsorption of the Cd, nickel (Ni) and Pb was best at 8% of biochar amended soil.</li> </ul>	Gholami and Rahimi, 2022
Potato peels as valueless waste material for nitrate removal from water supplies	<ul style="list-style-type: none"> <li>Potato peels were heated at 500°C for 3h under an N<sub>2</sub> flow.</li> </ul>	<ul style="list-style-type: none"> <li>Potato peel biochar had a highly porous structure and large specific surface area.</li> <li>pH and point of zero charges of the biochar have an influence on the adsorption of the nitrate ions.</li> </ul>	El-Nahas et al., 2018

		<ul style="list-style-type: none"> <li>The biochar when applied to real water samples, high nitrate adsorption rate was observed in a short time.</li> </ul>	
Potato peel into an eco-friendly bio-organic NPK fertiliser	<ul style="list-style-type: none"> <li>Potato peel was carbonised in an electric furnace at 450°C.</li> </ul>	<ul style="list-style-type: none"> <li>Addition of potato peel biochar provided high amounts of N, K, P, and trace amounts of Mg.</li> <li>Addition of biochar improved the plant growth, with a 12-14 % increase in the fruit size and 62-68 % extra chlorophyll in the leaves compared to the control.</li> </ul>	Majee et al., 2021
Production and characterisation of potato peel biochar and bio-oil	<ul style="list-style-type: none"> <li>Pyrolysis was conducted in an auger reactor at 450°C with N<sub>2</sub> purge.</li> <li>The bio-oil was fractionated into water-soluble and water-insoluble fractions.</li> </ul>	<ul style="list-style-type: none"> <li>Had high yields of biochar than bio-oil</li> <li>A significant number of alkanes and alkenes in bio-oil were derived.</li> <li>The carbohydrate-derived compounds in bio-oil water-soluble components can be fermented into chemicals.</li> </ul>	Liang et al., 2014

Preparation of high-performance H <sub>2</sub> S removal biochar using potato peel waste	<ul style="list-style-type: none"> <li>The biochar was prepared using a fluidised bed at 500 °C for 5 min with 8000 (Lmin<sup>-1</sup>kg<sup>-1</sup>)</li> </ul>	<ul style="list-style-type: none"> <li>The biochar produced observed a high surface area of 62 m<sup>2</sup>/g and was alkaline in nature.</li> <li>The increased porosity and the alkaline nature of the biochar increased H<sub>2</sub>S removal capacity.</li> <li>The presence of moisture improved H<sub>2</sub>S adsorption performance of the biochar.</li> </ul>	Sun et al., 2017
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### 2.3.2 Palm Tree waste in UAE

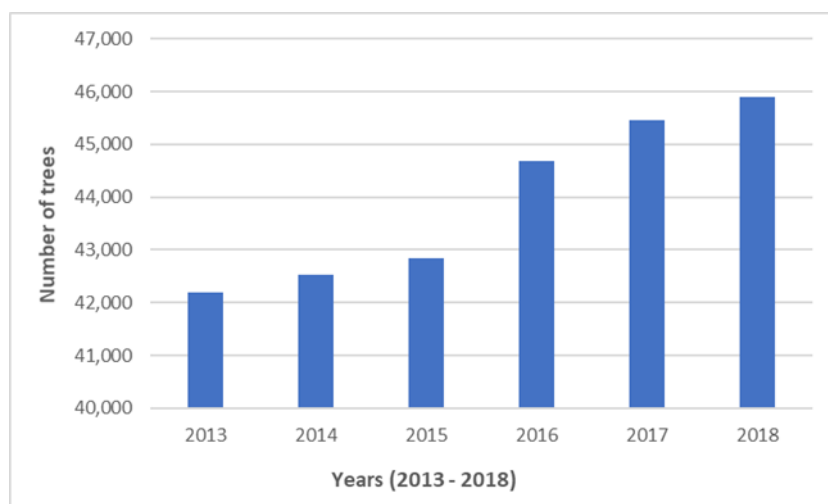
Palm trees are evergreen plants belonging to the Arecaceae family. Palm trees are recognised by either their fan-shaped or feather-like leaves (fronds) and fibre-covered trunks or stems. There are over 2600 species of palm trees around the world that are grouped into over 200 genera (Faiad et al., 2022). In the past few years, there had been an increase in palm tree cultivation in the green areas of Dubai as seen in Figure 2-8.

Date palm trees (*Phoenix dactylifera*) are one of the most cultivated palm trees in UAE along with other kinds of palm trees (Table 2-4) (Clean Deal Gardens, 2022). Date palm is considered one of the major crops grown in the UAE. Around 6 % of the world's date production is accounted for by UAE (MyBayut, 2022). The area harvested, yield and production of date crops in UAE from the year 2017 to 2022 has been summarised below in Table 2-5 (FAOSTAT, 2023).

**Table 2- 4.** Types of palm trees cultivated in UAE (Clean Deal Gardens, 2022).

Type of palm	Scientific name	Height (m)
Areca Palm	Chrysalidocarpus Lutescens	9
Bismarck palm	Bismarckia Nobilis	7
Bottle palm	Hyophorbe Lagenicaulis	3-6
Coconut palm	Cocos Nucifera	20-30
Canary Island palm	Phoenix Canariensis	8-10
California fan palm	Washingtonia Filifera	12-18
Fiji fan palm	Pritchardia Pacifica	6-9
Foxtail palm	Wodyetia Bifurcata	7-19
Date palm	Phoenix Dactylifera	10-15

Fountain palm	<i>Livistona Chinensis</i>	6-15
Fishtail palm	<i>Caryota Mitis</i>	10
Lady palm	<i>Raphis Excelsa</i>	2
Mexican fan palm	<i>Washingtonia Robusta</i>	18-27
Mediterranean fan palm	<i>Chamaerops Humilis</i>	4-7
Pigmy date palm	<i>Phoenix Roebelenii</i>	2-4
Royal palm	<i>Roystonea Regia</i>	18-30
Sabal palm	<i>Sabal Palmetto</i>	3-7.5
Sago palm	<i>Cycas Revoluta</i>	3
Triangle palm	<i>Dypsis Decaryi</i>	4-7



**Figure 2- 8.** General count of palm trees cultivation in green areas of Dubai from 2013- 2018 (Faia et al., 2022).

**Table 2- 5.** Area harvested, yield of the crop and the production volume of commercial production of date palm in the United Arab Emirates from 2018-2023 (FAOSTAT, 2023).

	<b>2018</b>	<b>2019</b>	<b>2020</b>	<b>2021</b>	<b>2022</b>	<b>2023</b>
<b>Area Harvested (ha)</b>	38117	39020	38422	40615	40738	39771
<b>Yield (hg/ha)</b>	90542	87454	87542	94298	97531	8284
<b>Production (tonnes)</b>	345119	341246	328669	382988	397329	329447

Palm trees usually grow in arid and semi-arid regions and can live up to one hundred years under good soil and weather conditions. Among all the palm trees, Mexican Fan Palm is considered to have the maximum average lifespan. Along with Mexican Fan Palm, Date Palms are also considered to have a long lifespan (Palm Tree Lifespan: How Long Do Palm Trees Live, 2022). The average economic lifespan of date palm trees is around 40 to 50 years (Faia et al., 2022). During the lifespan of the palm trees, special care is required for them to grow healthily especially the date palm trees to produce more crop. This includes the removal of offshoot, dead or dried leaves, and dried fruit which generates around 20 kg of waste per tree annually (Faia et al., 2022) (Figure. 2-9). Apart from the waste generated while grooming the trees, fronds of the palm trees fall onto the ground when they are dead are also considered as waste. These dead fronds are considered to be a fire hazard, especially during the summers and also, they serve as a bedding roost for rodents, because of this they have to be removed by law in some areas (Terraforma Dubai, UAE, 2022).



**Figure 2- 9.** Palm waste including a) off shoot, b) empty fruit bunch and c) dried leaves (Faiad et al., 2022).

Years ago, the waste generated from the date palm trees were used in many ways such as building boats, shelter, basket, mats, rope etc. (Tahir et al., 2020). But in recent years, due to the advancement of technology alternative products have replaced the use of palm waste products. In recent years, according to two local farmers and one garden maintenance company in the UAE that were personally consulted over the phone the waste generated over the life span of the date palm is collected and usually used as compost or incinerated. Incineration of palm waste can cause environmental problems due to the N concentration and the amount of smoke that gets released during the incineration process. The waste generated by the date palm tree is considered the biggest type of lignocellulose solid waste in the world with around 2million metric ton per year (Faiad et al., 2022).

Effective management had been an issue in the palm industry, especially the oil palm and date palm industries. In this current study, we will be focusing on the use of palm waste as a biomass feedstock to produce biochar. Over the past few years, there have been various studies on the use of date palm waste as biochar (Table 2-6). However, there have been limited studies on the use of date palm biochar to improve soil fertility and most of the studies have examined the use of conventional pyrolysis to produce date palm biochar.

According to the one garden centre contacted, apart from the date palm, which is considered the major palm waste contributor, there are other palm trees in UAE which produce waste as well that needs to be effectively managed. This study will focus on the use of Mexican Fan palm provided by the Royal Botanical Gardens of Edinburgh as the biomass feedstock to produce biochar through microwave pyrolysis and hydrothermal carbonisation on lab and commercial scales as well. The char produced through the MW process will be analysed and

characterised through various analysis processes. In the final part of the study, we will be exploring the effect of the char produced on the properties of the sandy soil and as well the effect on plant growth.

**Table 2- 6.** Summaries of studies on various pyrolysis techniques used to produce palm waste biochar and their applications.

Aim of study	Type of date palm feedstock and reactor used	Main Findings	References
Use of date palm waste to alter soil respiration, microbial biomass C and heavy metal mobility in contaminated mine soil	<ul style="list-style-type: none"> <li>• Date palm waste</li> <li>• Electrical muffle furnace at 300, 500 and 700 °C</li> </ul>	<ul style="list-style-type: none"> <li>• With the increase in the pyrolysis temperature there was: <ul style="list-style-type: none"> <li>○ a decrease in yield and volatile content of the biochar.</li> <li>○ an increase in the carbon content of the biochar.</li> </ul> </li> <li>• Biochar produced at 300 °C decreased the mobility of Cd, copper (Cu), Pb and zinc (Zn) in contaminated soil.</li> <li>• Additionally, biochar produced at 700 °C decreased mobility of iron (Fe) in soil.</li> </ul>	Al-Wabel et al., 2017

Ability of date palm biochar to remove single and multiple metals in an aqueous solution	<ul style="list-style-type: none"> <li>• Date palm leaves and fronds</li> <li>• Electrical muffle furnace at 400, 500 and 600 °C at a rate of 8 °C min<sup>-1</sup></li> </ul>	<ul style="list-style-type: none"> <li>• Increase in temperature, led to increase in pore size and surface area of biochar.</li> <li>• An increase in the pH was observed in the aqueous solution with the addition of biochar, which resulted in increase in the adsorption of Cu, Zn, Ni and Fe.</li> <li>• Biochar produced at 600 °C showed increased adsorption capacities for both single and multiple metals from contaminated water.</li> </ul>	Sizirici et al., 2021
Addition of biochar to sandy desert soil and its effect on crop growth, water retention and physical and chemical properties of soil	<ul style="list-style-type: none"> <li>• Date palm Midrib and frond base</li> <li>• Electrical muffle furnace at 300, 400, 500 and 600 °C</li> </ul>	<ul style="list-style-type: none"> <li>• Soil amended with biochar produced at 300 and 400 °C had an increased water retention compared to higher temperature biochars and the water retention increased with the incubation period.</li> <li>• Soil amended with biochar produced at 300 and 400 °C had the highest wheat</li> </ul>	Alotaibi and Schoenau, 2019

		<p>yield and as well as high N and P along with the NPK fertiliser.</p> <ul style="list-style-type: none"> <li>• The addition of the biochar increases the soil's ability to retain nutrients due to high CEC and had no effect on soil pH.</li> </ul>	
<p>Hydrogen-rich syngas and biochar production by non-catalytic valorisation, and evaluating process efficiency and potential applications in renewable energy and agriculture</p>	<ul style="list-style-type: none"> <li>• Date palm seeds.</li> <li>• Fixed bed pyrolyser at 400, 600, 800 and 1000 °C</li> </ul>	<ul style="list-style-type: none"> <li>• Carbon content in biochar increased from 400 to 600 °C to 800 °C but decreased at 1000 °C.</li> <li>• The use of date seed biochar had resulted in 47 % adsorption of boron boron from seawater.</li> <li>• High carbon content makes it a suitable low-cost absorbent.</li> <li>• With the increase in the temperature O, Mg, K and P decreased.</li> </ul>	<p>Sait et al., 2022</p>



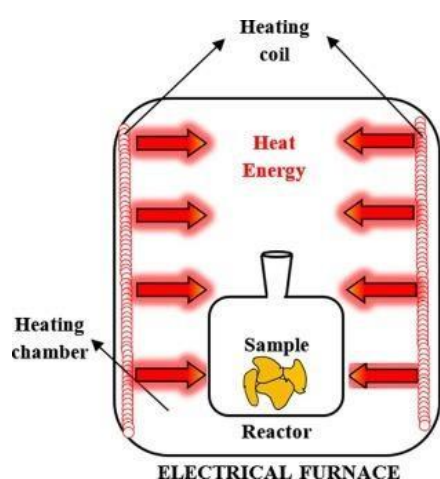
Applying biochar from date palm waste to increase the organic matter content, nutrient retention and water retention in sandy oasis soil	<ul style="list-style-type: none"> <li>• Date palm waste.</li> <li>• Continuous flow kiln pyrolysis at temperatures up to 500 °C for 30 to 40 min.</li> </ul>	<ul style="list-style-type: none"> <li>• Addition of biochar to the soil increased: <ul style="list-style-type: none"> <li>○ Soil organic content from 0.17 to 1.40 %</li> <li>○ No significant change in soil pH</li> </ul> </li> <li>• Increase in the N and K levels in plants with high levels of biochar (20 tha<sup>-1</sup>) in the soil.</li> <li>• Increase in the incubation time of the biochar in the soil decreased the soil P.</li> </ul>	Karbout et al., 2019
Quality indicators for a sandy textured soil treatment with biochar produced from fronds of date palm	<ul style="list-style-type: none"> <li>• Date palm fronds.</li> <li>• Muffle furnace pyrolysis at 400 °C for 30 min.</li> </ul>	<p>With the addition of biochar to the soil:</p> <ul style="list-style-type: none"> <li>• soil pH decreased from 8.4 to 7.9.</li> <li>• soil CEC increased from 2.5 to 6.7 g kg<sup>-1</sup></li> <li>• the sodium adsorption ratio of the soil decreased to below sodic levels (&lt; 13).</li> </ul>	Khalifa and Yousef, 2015

## 2.4 Production of biochar

Over the past years, a wide range of processes have been developed for the production of biochar. Among these, thermochemical methods such as torrefaction, combustion, pyrolysis and hydrothermal carbonization are commonly employed due to their efficiency in transforming organic materials into stable carbon-rich products (Mukherjee et al., 2022).

### 2.4.1 Pyrolysis

Pyrolysis of biomass is a complex endothermic thermochemical process in which the biomass is decomposed at high temperatures in absence of oxygen. It produces three main products: char (solid), bio-oil (aqueous) and gas (Lee et al., 2019). The heating mechanism of the pyrolysis is based on the thermal energy that is supplied externally from the heating coil to the reactor (Figure 2-10) (Foong et al., 2020). The heat transfers to the sample inside the vessel through conduction and convection, which is slow and energy inefficient due to non-selective heating. It requires a high energy gradient for the sample present in the reactor to reach the required temperature for pyrolysis (Foong et al., 2020).



**Figure 2- 10.** Mechanism of conventional heating in an electrical furnace (Foong et al., 2020).

Pyrolysis of biomass comprises of three stages of reaction: 1) evaporation of moisture in the biomass, 2) devolatilisation of biomass into different carboxyl, carbonyl and hydroxyl groups referred to as primary pyrolysis and 3) the thermal cracking process of heavy compounds into char and gaseous products such as  $C_2H_4$ , CO,  $CH_4$  and  $CO_2$  (Jerzak et al., 2024) referred to as the secondary pyrolysis. Some of the volatile compounds released during the secondary pyrolysis condense to form bio-oil (Tripathi et al., 2016; Foong et al., 2020). Depending on the temperature and the residence time, pyrolysis is categorized into three types: slow pyrolysis, fast pyrolysis and flash pyrolysis. The product yield of biomass can be varied based on the kind of pyrolysis process the biomass undergoes (Foong et al., 2020).

### **Slow pyrolysis**

Slow pyrolysis occurs at relatively low temperatures (400 – 700 °C) and a heating rate of <2 °C/s (Lee et al., 2019). Higher temperatures are generally avoided in slow pyrolysis because they promote thermal cracking and secondary reactions, resulting in the formation of bio-oil and reducing the yield of char yield (Mukherjee et al., 2022). Slow pyrolysis produces a high biochar yield (around 50 wt%) with small quantities of liquid and gaseous products (Bhatta et al., 2018; Foong et al., 2020). The yield of biochar decreases with increasing temperature. Additionally, different pyrolysis temperatures influence the physical properties of the biochar (Section 2.5).

### **Fast pyrolysis**

Fast pyrolysis involves the rapid thermal decomposition of the biomass in the absence of oxygen, at moderate to high temperatures (500 to 1000°C) and high heating rate (10-200°C/s) (Gul et al., 2015; Foong et al., 2020; Mukherjee et al., 2022). Due to the fast- heating rate, re-condensation of volatiles is minimised, leading to the recovery of volatiles as bio-oil (30-60 wt%) and bio-gas (15-35 wt%), but less biochar is produced (Bhatta et al., 2018; Foong et al., 2020).

## **Flash pyrolysis**

Flash pyrolysis involves thermal decomposition at a very high temperature (500 to 600 °C) and extremely high heating rates (>1000 °C/s) (Ighalo et al., 2022). The short residence time and higher heating rates result in higher bio-oil yields of about 65-75 wt%.

### **2.4.2 Hydrothermal Carbonisation**

Hydrothermal carbonisation (HTC) had gained significant attention for the conversion of biomass into hydrochar (Petrović et al., 2024). Hydrochar is similar to biochar but is produced using a different pretreatment process and condition. Hydrochar is produced as a slurry (a mixture of solid and liquid) using HTC process (Kambo and Dutta, 2015).

The HTC process is a thermochemical process occurring in subcritical water, at temperatures ranging from 180 to 260 °C, with pressures of 2 to 6 MPa, and durations of 5 to 240 minutes (Ischia and Fiori, 2020). HTC of biomass results in a high yield of hydrochar (45 -70 %) and a liquid by-product (5-25 %). This liquid fraction, characterised by a high-water content, includes valuable chemicals such as 5-hydroxymethylfurfural, phenol, formic acid, lactic acid, levulinic acid, acetic acid and other organic acid. Further research into extracting these chemicals from the liquid by-product is warranted, given their commercial value. HTC also produces a small percentage of gas; typically comprising 90 % CO<sub>2</sub>, along with other minor gases such as CH<sub>4</sub>, H<sub>2</sub> and CO (González-Arias et al., 2021). The relative yields of the end products (hydrochar, liquid product and biogas) vary depending on the types of feedstocks as seen in Table 2-7. The total mass recovery generally exceeds 90 %, but variability arises mainly from challenges in accurately measuring produced water due to errors introduced during multiple rinsing and drying steps.

**Table 2- 7.** % yield of HTC products from different biomass feedstocks, biomass: water and operating conditions.

Biomass feedstock	Biomass: Water ratio	Temp (°C)	Duration of hydrolysis (min)	Hydrochar %	Liquid product %	Biogas %	Reference
Sugarcane bagasse	1:8	255	30	45	30	10	Hoekman et al., 2012
Rice hulls	1:8	255	30	55	19	8	Hoekman et al., 2012
Corn stove	1:8	255	30	40	42	13	Hoekman et al., 2012
Fan palm leaves	8:150	240	60	45	15	40	Yao & Ma, 2019
Olive husk	50:1000	250	180	24	65	11	González-Arias et al., 2020

### Role of water in HTC

The critical point of HTC is 374 °C at 22.1 MPa (Peterson et al., 2008). Water below the critical point is referred to as sub-critical water and above the point is referred to as super-critical water. In HTC, due to the presence of water in subcritical conditions, the organic compounds are easily miscible with the water. Lignocellulose material consists of solid and polymeric organic compounds. During the HTC of lignocellulose biomass, oxygen containing functional groups such as hydroxyl (-OH) and carboxyl (-COOH) groups are cleaved from the biomass structure. This process begins with the hydrolytic breakdown of major polymeric components into low molecular weight organic compounds. These intermediates then undergo secondary reactions including dehydration, decarboxylation, and polymerization which lead to the formation of solid carbonaceous material (hydrochar) with high carbon content as the by-product (Khan et al., 2019).

## **2.5 Impacts of pyrolysis parameters and feedstocks on the properties of biochar**

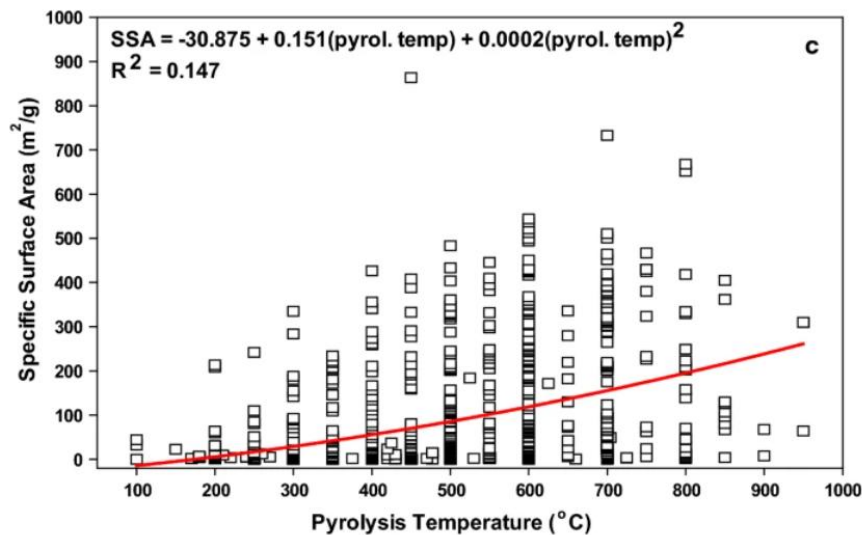
### **2.5.1 Effect of production temperature on yield**

Temperature is a key factor influencing both the yield and properties of biochar. As the temperature increases, the yields of oil and gaseous products increases, while the yield of char decreases. This reduction in char yield is attributed to the enhanced thermal cracking of high molecular weight hydrocarbons present in biomass (Al-Haj Ibrahim, 2020). For instance, at 200°C, wheat straw undergoes thermal decomposition, forming a char precursor through dehydration and initial breakdown of hemicellulose. This results in a relatively high yield of 84.95 %, as limited volatilisation occurs at this stage. As the temperature increases upto 600°C, continued devolatilisation and thermal cracking removes more water and light organics, reducing the biochar yield by 52 % (Zhang et al (a)., 2015).

Similarly, in FW studies, Zhang et al (b). (2017) reported that pyrolysis of peanut shells at 350 °C produced a maximum yield of 43.16 %. As the temperature increased to 850 °C, the yield decreased to 29.48 %. However, when the temperature exceeded 900 °C, a slight increase in the biochar yield to 30.14 % was observed. Comparatively, peanut shells exhibited higher biochar yields at low temperatures than other feedstocks such as pine, oak, and sugarcane (Zhang et al (b). 2017). Therefore, along with temperature the type of feedstock also effects the yield of biochar being produced. In addition to yield, pyrolysis temperature also significantly affects the physical and the chemical composition of the biochar such as the pore structure, surface area, pH, CEC and the elemental composition which is discussed in Section 2.5.5.

### **2.5.2 Effect of production temperature on pore structure and surface area**

A study by Pallarés et al. (2018) investigated the effect of pyrolysis temperature on the pore size and surface area. When the pyrolysis temperature was less than 400 °C, no significant change in the pore size and surface area of biochar was observed. This was because, at low temperature, volatile organic compounds (lower hydrocarbons, aldehydes, ketones) are not fully released from the biomass. These retained volatile undergo secondary polymerization reactions, leading to deposition of tar like substances within the pore structure. This process can block existing pores or inhibit the formation of new pores, thereby limiting the development of surface area and porosity (Pallarés et al., 2018). A meta analysis by Li et al. (2022) showed that high pyrolysis temperatures ( $> 500^{\circ}\text{C}$ ) increased biochar surface area by an average of 3.8 times compared to low temperatures ( $\leq 500^{\circ}\text{C}$ ) (Figure 2-11). This is primarily attributed to the thermal decomposition of lignin and cellulose, which promotes the development of tube like structures and increase porosity and surface area (Tomczyk et al., 2020; Ahmed et al., 2016; Handiso et al., 2024 and Ippolito et al., 2020). A study by Zhang et al. (2019) had also observed a high surface area of biochar when the cow manure was pyrolysed at a high temperature (700 °C). This was because the release of the volatile compounds resulted in the formation of pore structures. The increase in the surface area and the development of micro-pore at high temperatures (400 °C), promotes an increase in the water and nutrient holding capacity.

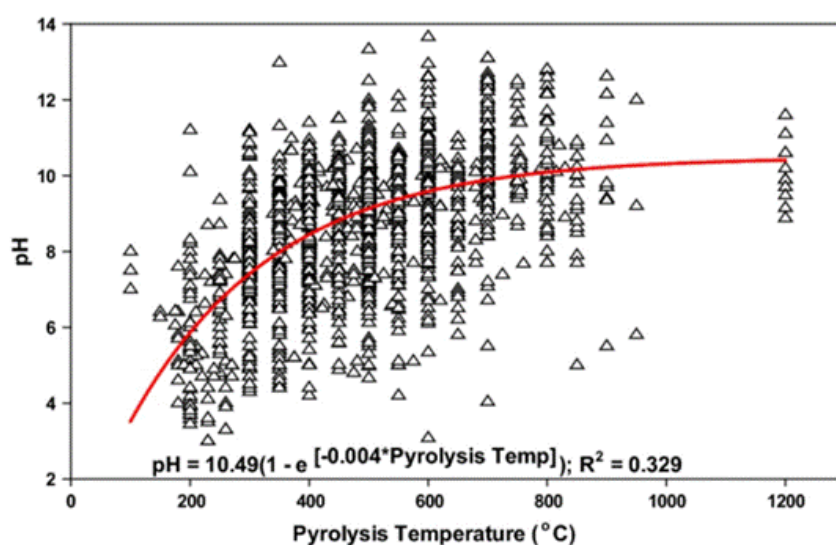


**Figure 2- 11.** Relationship between pyrolysis temperature and specific surface area (SSA) of biochar based on meta-analysis data. Each data point represents an individual observation from various studies (Ippolito et al., 2020).

### 2.5.3 Effect of production temperature on pH, cationic exchange capacity (CEC) and N-content in biochar

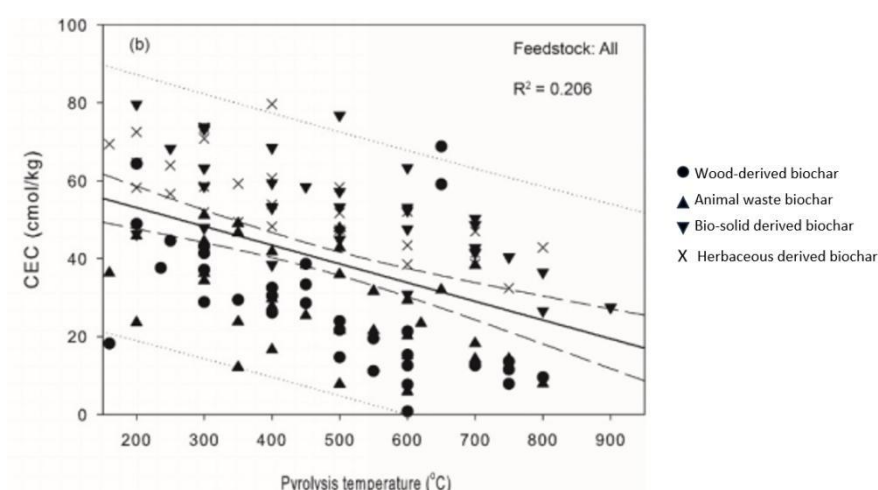
Multiple meta-analyses have confirmed that pyrolysis temperature significantly influences the pH of the biochar. Li et al. (2022) and Ippolito et al. (2020) reported that biochars produced at higher temperatures (average pH  $9.32 \pm 0.20$ ) had higher pH than those produced at lower temperatures (average pH  $8.25 \pm 0.14$ ), due to the reduction of the acidic functional groups and formation of alkali metals such as oxides and carbonates (Ding et al., 2014; Yuan et al., 2011; Shinogi and Kanri, 2003) (Figure 2-12). This trend is also further supported by Roshan et al. (2023), found that while the overall correlations between the pyrolysis temperature and biochar pH is not statistically significant, 67% of the studies still reported a positive correlation between the pyrolysis temperature and biochar pH. The variability is due to the heterogenous study designs and feedstock types, but the main trend that links higher temperature with increase in pH is due to the volatilisation of acidic groups and accumulation of alkaline salts.





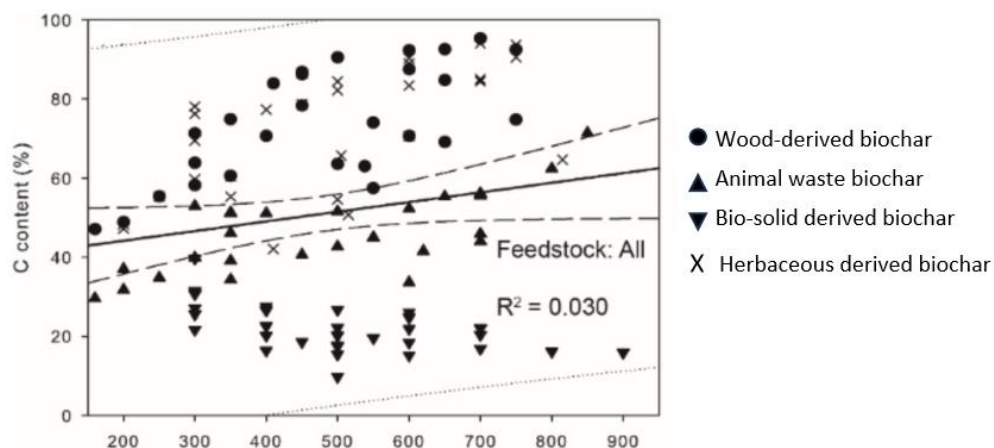
**Figure 2- 12.** Relationship between pyrolysis temperature and pH of biochar based on meta-analysis data. Each data point represents an individual observation from various studies (Ippolito et al., 2020).

Multiple studies have demonstrated that the CEC of a biochar decreases as pyrolysis temperature increases (Mukherjee et al., 2011; Song and Guo, 2012; Yao et al., 2012). Meta analyses carried out by Li et al. (2019) and Roshan et al. (2023) also show that the CEC of biochars decrease with increasing pyrolysis temperature (Figure 2-13). The negative correlation ( $R^2 = -0.336$ ) is attributed to the thermal degradation of oxygen containing functional groups, which are key contributors to CEC (Banik et al., 2018). For example, Kloss et al. (2012) reported a decline in CEC from 9.2 cmol kg<sup>-1</sup> at 400°C to 8.29 mmol kg<sup>-1</sup> at 525°C. Low temperature biochars (<450°C) had higher CEC values up to 22.8 mmol kg<sup>-1</sup>. Carboxylate and phenolate groups are responsible for the negative charge sites on biochar surfaces, while positive charges are due to oxonium groups in aromatic rings (Tomczyk et al., 2020; Banik et al., 2018). While higher pyrolysis temperatures enhance carbon stability and pH, they also reduce the number of reactive surface sites responsible for cation retention. As a result, biochars produced at lower temperatures tend to have higher CEC values and are more effective at retaining nutrients in soil (Li et al., 2019).



**Figure 2- 13.** Relationship between pyrolysis temperature and cationic exchange capacity (CEC) of biochar based on meta-analysis data from multiple feedstocks. Each point represents a reported CEC value from an individual study (Li et al., 2019).

Pyrolysis temperatures plays an important role in determining the elemental composition of biochar particularly, its carbon and nitrogen contents. Meta analyses show that increasing pyrolysis temperature increases carbon content across various feedstocks due to greater thermal decomposition and carbonisation. According to Li et al. (2019), this trends results from volatilisation of weakly bonded functional groups such as -OH and -NH<sub>2</sub>, which reduces the content of labile elements while retaining more aromatic carbon. As a result, the relative proportion of carbon increases, except in biosolid derived biochars where carbon is more thermally labile and tends to decrease with temperature (Figure 2-14). Li et al. (2022) similarly reported that higher pyrolysis temperatures significantly increased the carbon content of wood biochar, from  $70.65 \pm 1.36$  at  $\leq 500^{\circ}\text{C}$  to  $79.32 \pm 1.60$  at  $> 500^{\circ}\text{C}$ , although this effect was not significant for corn stover or switchgrass biochars. Roshan et al. (2023) also observed a general but statistically non-significant positive correlation between temperature and biochar carbon content, with high pyrolysis temperatures associated with carbon content ranges as high as 97.3%.



**Figure 2- 14.** Relationship between pyrolysis temperature and carbon content (%) of biochar derived from various feedstocks, based on meta analysis data. Each symbol represents an individual observation from a specific feedstock type (Li et al., 2019).

Meta analysis findings from Roshan et al. (2023) indicates that pyrolysis temperature had a minimal and statistically non significant effect on total N content of biochar (Figure 2-18). However, in some studies by De et al. (2024) and Yang et al. (2015) has observed at higher pyrolysis temperatures the total N content in biochar decreased significantly due to volatilisation. This decrease is attributed to the loss of N in the forms of  $\text{NH}_4^+$  and nitrate ( $\text{NO}_3^-$ ) as the charring process intensifies (De et al., 2024).

However, the concentrations of P, K, Ca and Mg often increase with higher temperatures because these elements are more stable and concentrate in the residual biochar (Gunes et al., 2015 and Mercl et al., 2020). A meta-analysis assessing the effect of pyrolysis temperature on nutrient concentration in biochars found a general trend of increasing Ca, Mg, and K concentration with increasing temperature, though none of the correlations were statistically significant. As temperature increases, it further breaks down more organic matter, stable inorganic nutrients become more concentrated, leading to higher nutrient concentration in the biochars (Roshan et al., 2023). While higher pyrolysis temperatures can concentrate certain nutrients in biochar, it can also lead to the formation of more stable and less soluble forms potentially reducing their immediate availability to plants (Gunes et al., 2015; Bruun et al., 2017).



#### **2.5.4 Effect of feedstock composition**

The size of the biomass particle influences the temperature gradient from the surface to the core of the particle during the pyrolysis. When the particles size of the biomass is large, the heat supplied during the pyrolysis process on the outer surface area of the biomass will not be able to pass rapidly and more time will be required to reach the required pyrolysis temperature which means it will slow down the heating rate. In smaller particles, heat transfer occurs more rapidly, resulting in a higher heating rate. As mentioned before, slow heating rates promote a high yield of biochar. Demirbas (2004) investigated the effect of particle size on the yield of biochar during pyrolysis at 677 °C. It was found when the particles size of corn cob increased from 0.5 to 2.2 mm, there was an increase in the char yield from 5.7 % to 16.6 %. However, a study by Onay and Kockar (2003) observed that with an increase in the size particles of rapeseed from 0.425 to 0.85 mm there was a decrease in the char yield approximately from 23 % to 16 %. But when the particle size exceeded 10 mm to 1.8 mm there was an increase in the char yield to 21 %. Different observations related to the particle size on the biochar yield suggest that the role of particle size had not been understood, and further research would be required in this area.

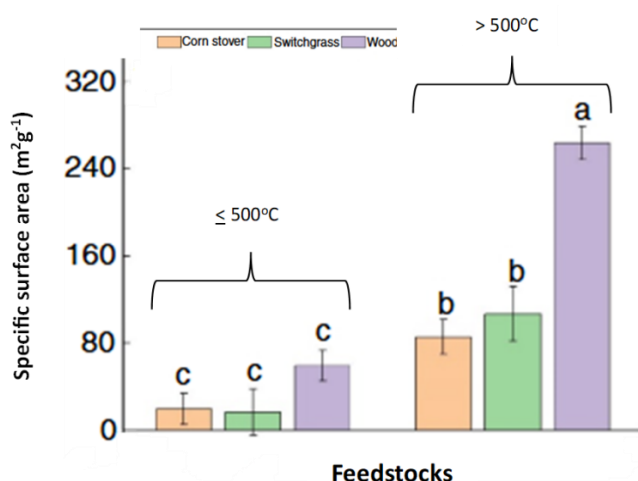
#### **2.5.5 Effect of different types of biomass feedstock on the properties of biochar**

Biochar properties are influenced by several factors such as feedstock composition, pre-treatment processes and pyrolysis temperature (Banik et al., 2018). Various feedstocks, such as woody biomass, crop residue and organic waste have been used in biochar production, with a predominant focus on woody biomass (WB) and crop residue (CR) due to their cost effectiveness and the potential to address waste disposal issues (Gul et al., 2015).

The pyrolysis process has a different effect on the decomposition of the biomass components depending on the temperature, reaction rate, residence time and the type of reactors being used. The first component to decompose during pyrolysis is hemicellulose at 250 – 350 °C, then cellulose at 325 – 400 °C. The decomposition of the lignin occurs at a much higher temperature (300 – 550 °C) because it is the most stable component than cellulose and hemicellulose. Stefanidis et al. (2014) had shown upon pyrolysis of each component

(cellulose, hemicellulose, and lignin) separately, cellulose has a high yield of oil and a lower yield of solid residue, hemicellulose resulted in a high yield of gas and oil with poor carbon content and high ash percentage. It was also shown that hemicellulose decomposed at a lower temp (200 °C) than the required pyrolysis temperature (500 °C). The pyrolysis of lignin has shown a high yield of solid residue. They also performed pyrolysis on mixtures. Mix 1 contained cellulose and hemicellulose whereas mix 2 contained cellulose, hemicellulose, and lignin. The results showed that there was an increase in the yield of char and gas for both the mixtures. The composition of the biomass feedstock also affects various physical and chemical properties of biochar.

The composition of biomass feedstock, mainly the non-combustible components content significantly influences the surface area and porosity of biochar (Wang et al., 2015). The non-combustible component includes moisture and ash content (Tomczyk et al., 2020). Lignocellulosic biomass, particularly WB, is especially effective in this regard (Leng et al., 2020), because the non-combustible components were found to be higher in herbaceous biochar than woody biochar. Ronsse et al. (2012) observed that WB had a higher surface area compared to algae biomass, attributing this to the lower ash content in WB. Similarly, another study found that among 12 feedstocks, biochar produced from sawdust had the highest surface area, while that produced from cow manure and waterweeds had the lowest. This is because organic waste contains a higher percentage of inorganic material (ash content) than WB, that partially fills or blocks pores (Zhao et al., 2013). According to a meta analysis by Li et al. (2022), wood derived biochars exhibited higher surface area than those produced from corn stover or switchgrass (Fig. 2-15). This trend is due to the higher lignin and cellulose, which enhances pore development during pyrolysis. Furthermore, woody feedstocks also contains lower ash content upto five times less than corn stover and switchgrass biochars (Tomczyk et al., 2020 and Wang et al., 2015). Lower ash content prevents pore blockage, thus contributing to increased surface area.

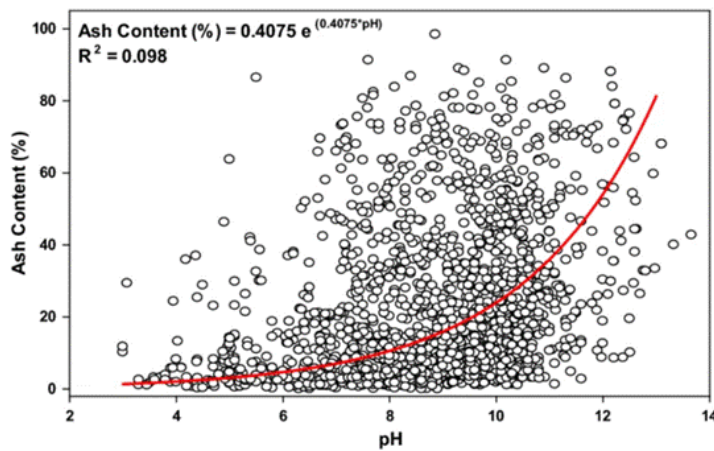


**Figure 2- 15.** Effect of pyrolysis temperature (< 500°C vs > 500°C) on the specific area of biochar derived from different feedstocks (corn stover, switchgrass, and wood), based on meta-analysis data. Bars represent mean values, and error bars indicate standard deviations. Different letters above the bars indicate statistically significant differences between groups ( $p < 0.05$ ) (Li et al., 2022).

Although pyrolysis temperature plays a role in determining biochar CEC, the meta analysis evidence suggests that feedstock selection also influences the CEC, as seen in meta analysis (Li et al., 2019 and Roshan et al., 2023). Biochars derived from nutrient rich feedstocks particularly biosolids and herbaceous materials consistently exhibited higher CEC values than those from woody biomass (Figure 2-13). This is attributed to their higher concentration of alkali metals such as potassium, calcium, magnesium, and phosphorus, which promote the formation of oxygen containing functional groups during pyrolysis (Tomczyk et al., 2020). These functional groups enhance electrostatic retention of positively charged nutrients, contributing to improve nutrient retention (Ji et al., 2022; Khare et al., 2019).

Meta analysis review by Li et al. (2022) and Ippolito et al. (2020) revealed that type of feedstock significantly influences the pH of biochar. Among the studied biomass types, corn stover had the highest pH ( $10.13 \pm 0.22$ ), followed by switchgrass ( $9.00 \pm 0.52$ ) and wood biochar ( $8.83 \pm 0.19$ ). The elevated pH in corn stover biochar is due to its higher content alkali metals, which are retained during the pyrolysis process and contribute to its more alkaline nature. In contrast, woody biomass generally contains lower levels of these alkali metals, resulting in more neutral or slightly alkaline nature biochars. The presence of ash content is directly proportional to the biochar pH (Figure 2-16). Biomass feedstocks such as corn stover and switchgrass has more ash content that resulted in higher biochar pH compare to the low

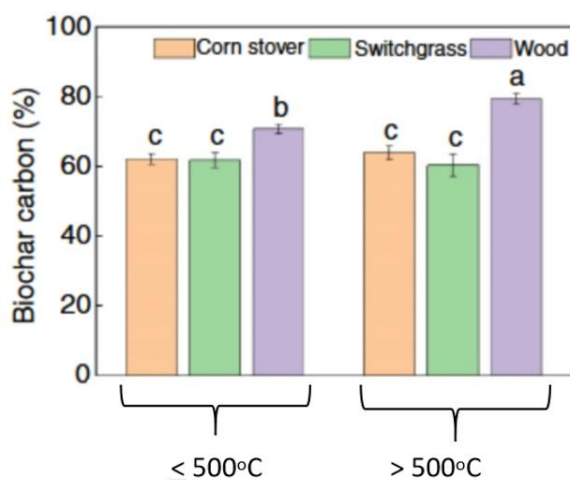
ash content in wood biochar (Tomczyk et al., 2020; Tag et al., 2016).



**Figure 2- 16.** Relationship between biochar pH and ash content (%) derived from various feedstocks, based on meta-analysis data. Each data point represents an individual observation from various studies (Ippolito et al., 2020).

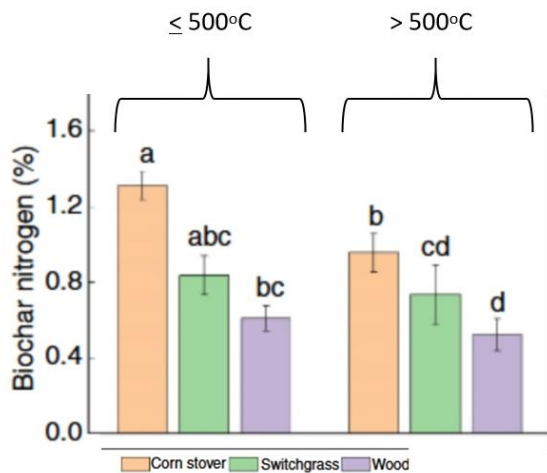
According to Li et al. (2022), wood derived biochars consistently showed higher carbon content than those from herbaceous feedstocks, due to their higher lignin content and low ash concentrations (Figure 2-17). In terms of carbon content, feedstocks like manure and grass contain more inorganic components that, upon pyrolysis, result in higher ash content and lower carbon levels. In contrast, WB had fewer inorganic components, leading to lower ash content and a higher carbon concentration after pyrolysis (Ji et al., 2022 and Li et al., 2019). If the purpose of using biomass is for carbon sequestration, WB is more suitable because it generates char with a high aromatic carbon content (Al-Wabel et al., 2017).





**Figure 2- 17.** Effect of pyrolysis temperature (< 500°C vs > 500°C) on the carbon content (%) of biochar derived from different feedstocks (corn stover, switchgrass, and wood), based on meta analysis data. Bars represent mean values, and error bars indicate standard deviations. Different letters above the bars indicate statistically significant differences between groups ( $p < 0.05$ ) (Li et al., 2022).

According to the meta analysis review by Li et al. (2022) and Roshan et al. (2023) biochar nitrogen content significantly differed among feedstocks ( $p < 0.0001$ ), with wood derived biochar exhibiting the lowest nitrogen content ( $0.075 \pm 0.05$ ), followed by switchgrass ( $0.79 \pm 0.09$ ) and corn stover ( $1.13 \pm 0.06$ ) (Figure 2-18). These differences are attributed to the initial nitrogen content. Therefore, selecting nitrogen rich feedstocks is important to produce biochar with higher nitrogen content.



**Figure 2- 18.** Effect of pyrolysis temperature (< 500°C vs > 500°C on the nitrogen content (%) of biochar dervied from different feedstocks (corn stover, switchgrass, and wood), based on the meta-analysis data. Bars represent mean values, and error bars indicate standard deviations. Different letters above te bars indicare statistically significant differences between groups ( $p < 0.05$ ) (Li et al., 2022).

Feedstock type plays an important role in determining the nutrient compoisiton. Meta analysis data by Ippolito et al. (2020) show that manure and biosolid based biochars typically contain the highest concentration of P, Ca, Mg, while grass based biochars also exhibit high levels of K and Ca. In contrast, crop residue and wood derived biochars contain lower concentration of these nutrients. Additionally, Singh et al. (2022) found that herbaceous biochars significantly enhanced crop productivity compared to wood derived biochars. This improvement is attributed to the higher nutrient availability in herbaceous feedstocks, whereas wood biochars, rich in lignin, can immobilise nutrients and reduce their immediate bioavailability (Kloss et al., 2012). Therefore, selecting the appropriate feedstock for biochar production depends on the intended purpose, whether for carbon sequestration or enhancing soil fertility through nutrient addition.

## 2.6 Microwave technology

Microwave (MW) technology, which was initially developed in the 1930s for communication purposes, saw a significant breakthrough in the 1940s when its heating effect was discovered. This discovery accelerated the development of MW technology, and it was eventually adopted for industrial production in the 1960s (Tian et al., 2022). MW chemistry had evolved to comprise diverse applications such as synthesising chemicals, organometallics, and nanoparticles. This technology enables more efficient and safer chemical reactions, with increased yield and reduced response time (Siddique et al., 2022).

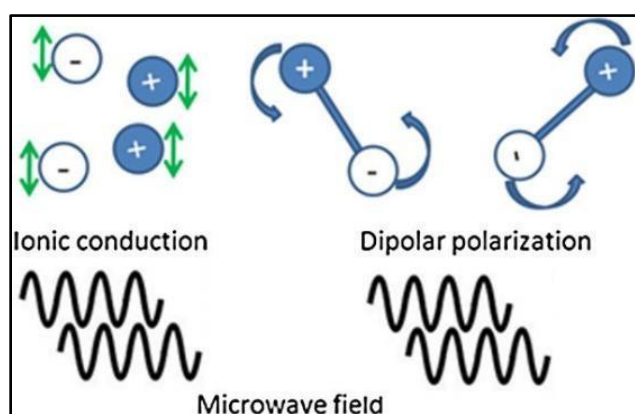
### 2.6.1 Dielectric heating

MW heating operates on the principle of dielectric heating, where a material's ability to convert electromagnetic energy into heat is determined by its loss tangent (Foong et al., 2020). Materials with high loss tangent values, such as carbon materials, heat quickly under MW radiation. Conversely, materials with low loss tangent values can be made more MW absorbent by incorporating additives or catalysts with higher loss tangents. For instance, carbon-based materials like graphite and activated charcoal, with loss tangent values ranging from 0.1 to 0.8, exhibit strong MW energy absorption (Foong et al., 2020; Synthesis, 2022; Huang et al., 2016). As a result, when combined with materials with lower loss tangent such as biomass and other organic wastes, it increases the microwave absorbancy to achieve higher temperatures in order to for extensive pyrolysis to occur (Lam and Chase, 2012)

Biomass, inherently having low MW absorption, can increase its MW energy uptake with higher moisture content. This characteristic is particularly advantageous for wet biomass, such as FW, which typically poses challenges during the charring process due to the presence of water. During pyrolysis, biomass undergoes a conversion process in which water content is removed subsequently increasing MW absorbance. The resulting char, enriched in carbon and with an altered structure, absorbs MW energy more effectively than raw biomass (Macquarrie et al., 2012). However, most solid wastes, in general, have relatively low MW absorption, an issue that can be mitigated by incorporation of MW absorbing materials (Li et al., 2016).

The conversion of electromagnetic energy into heat is dependent on two main mechanisms

dipole polarisation and ionic conduction. Dipole polarisation takes place when a substance possesses a molecular structure that is partly negative and partly positive charged. Dipolar polarisation is a process by which heat is generated in polar molecules. When the molecules are exposed to an oscillating electromagnetic field of appropriate frequency, the polar molecules try to follow and tend to align themselves with respect to the field. This alignment causes rotation, which results in a collision of the molecules. This generates friction which produces heat (Figure 2-19) (Kappe et al., 2012). In Ionic Conduction, the dissolved charged particles usually oscillate back and forth due to the influence of microwave radiation. This leads to the collision of the particles with the surrounded molecules or atoms, which in result creates heat energy (Gude et al., 2013).



**Figure 2- 19.** Movement of molecules under dipole polarisation and Ionic conduction in the presence of microwave (MW) field (Kappe et al., 2012).

### 2.6.2 Penetration Depth

It was seen that the penetration depth differs for material types, microstructure properties and temperature. For example, at 2.45GHz water with temperature 25.1 °C has a penetration depth of 14mm and at 90.1 °C the penetration depth increases to 57mm. Different biomass materials show different penetration depth of MW frequencies depending on the density and the water content (Li et al., 2016) (Table 2-8). Biomass loading in the vessel can also affect the penetration depth. If the biomass loading is too high, then it is hard for the MW radiation to penetrate (Siddique et al., 2022) (Table 2-8).

**Table 2- 8.** Key factors influencing microwave (MW) penetration depth in various materials (Siddique et

al., 2022; Li et al., 2016).

<b>Factor</b>	<b>Example</b>	<b>Penetration Depth</b>	<b>Main findings</b>
<b>Temperature</b>	Water at 25.1°C	14 mm	Penetration depth increases with temperature.
<b>Material Composition</b>	Different types of materials	Variable	Dielectric properties influence penetration depth.
<b>Moisture Content</b>	Biomass with high water content	Increased	Higher moisture content increases MW absorption.
<b>Density</b>	Dense vs. less dense biomass	Variable	Denser materials generally have shorter penetration depths.
<b>Biomass Loading</b>	High biomass loading	Reduced	Excessive biomass loading hinders microwave radiation penetration.
<b>Microstructure Properties</b>	General materials	Variable	Composition and microstructure affect penetration depth.
<b>Frequency of MW Radiation</b>	Different MW frequencies	Inversely proportional	With the decrease in the MW frequency the penetration depth increases.

### 2.6.3 Advantages of microwave-assisted technology

MW pyrolysis has several advantages over conventional pyrolysis such as selective heating and rapid volumetric heating (Li et al., 2016). Some of the advantages of using MW technology over conventional heating are:

- **Rapid heating:** MW heating allows for rapid and uniform heating of biomass particles, leading to faster pyrolysis reactions. This can significantly reduce processing times compared to conventional heating methods, which often rely on slower heat transfer mechanisms through conduction or convection (Zhang et al., 2018). However, in the current study during the microwave pyrolysis of potato peel waste using a large-scale MW reactor, the formation of hotspots was evident. These hotspots were a result of uneven thermal distribution within the reactor, resulting in the formation of two distinct types of char with different physical and chemical properties. The variation in char characteristics subsequently influenced soil properties and plant growth.
- **Energy efficiency:** Microwave pyrolysis is generally more energy-efficient because it directly heats the biomass material, minimizing heat losses to the surroundings (Salema and Ani, 2011). This results in lower energy consumption and operating costs. For example, conventional pyrolysis has been reported to use approximately 50% more energy than microwave pyrolysis under comparable conditions (Serio et al., 2023).
- **Improved biochar properties:** The rapid and uniform heating provided by microwaves can lead to biochar and bio-oil products with enhanced properties, such as higher surface area, improved pore structure, and lower levels of contaminants or tar compared to products from conventional pyrolysis (Brickler et al., 2021).
- **Environmental benefits:** Microwave assisted pyrolysis has been proposed as a more efficient alternative to conventional pyrolysis under certain conditions particularly when using dry, low moisture feedstock's, and with optimised reactor design that enables uniform energy absorption and precise temperature control. In such cases

microwave assisted pyrolysis can result in shorter processing times, reduced energy losses and lower energy emission (Muhammad et al., 2018). However, at certain cases microwave pyrolysis can undergo uneven heating, especially with heterogenous or moist biomass, leading to inconsistent char properties and lower yield. In this study, the efficiency of microwave assisted process varied, highlighting the importance of feedstock's characteristics in determining the overall performance and environmental benefit.

#### **2.6.4 Microwave hydrothermal carbonisation**

There have been recent studies focusing on the use of microwave HTC over conventional HTC because it is considered to be innovative, energy efficient and environmentally friendly. As microwave HTC reaction takes place in the presence of water, it makes the overall process effective because the presence of the water in the reaction makes it an effective absorber of microwave energy. Water is considered renewable, non-flammable, readily available, a good absorber of microwave energy and a green solvent making water suitable for microwave HTC (Nizamuddin et al., 2018).

Different operating parameters affect the microwave HTC such as the reaction temperature, residence time, feedstock type and microwave power. Reaction temperature is one of the significant parameters in deciding the composition of the final product. Nizamuddin et al. (2018) reported a reduction in hydrochar yield as temperature increased. Their study investigated the microwave HTC of sugarcane bagasse at temperature of 210 °C and 290 °C, at 210 °C, a high hydrochar yield was achieved, while at 290 °C, the yield decreased from 93.2 % to 52.6 % respectively (Chen et al., 2012). This is because at high temperature gasification takes place resulting in the loss of carbon in the form of volatile compounds.

## **2.7 Application of biochar for soil improvement**

Soil degradation poses a significant challenge to sustainable agriculture, affecting soil fertility and nutrient availability. Biochar a carbon rich material derived from biomass pyrolysis, has emerged as a soil amendment due to its ability to improve soil properties and enhance plant growth. The high surface area, porosity, and CEC contribute to better nutrient retention and increased water holding capacity. This section explores how biochar can contribute to soil improvement by reducing soil salinity, increasing nutrient availability, and highlighting its role in improving agricultural productivity in sandy soils.

### **2.7.1 Reduction of soil salinity**

Soils with high salt content that negatively impact plant growth are referred to as saline soils (Drainage of Salty Soils, 1973). These salt-affected soils have elevated concentrations of chlorides, sulfates, carbonates, and bicarbonates of  $\text{Na}^+$  and  $\text{Cl}^-$  (Singh et al., 2019). High salinity affects plant growth by increasing concentrations of ions such as  $\text{Na}^+$  and  $\text{Cl}^-$ , which can become toxic at high levels, limit water uptake, and cause nutritional imbalances (Nguyen et al., 2022). Soil salinity is a major global challenge, particularly in arid and semi-arid regions, where it significantly impacts agricultural production. Currently, soil salinity affects 20 % of the total cultivated and 33 % of irrigated agricultural lands worldwide, and this rate is expected to increase rapidly by 2050 due to factors such as climate change, over-irrigation, and poor drainage management (Mukhopadhyay et al., 2021).

Recently, the use of biochar for amending salt-affected soils has gained more attention. Due to its high adsorption and ion exchange capacity, biochar is effective for improving saline soils (Huang et al., 2019). In arid and semi-arid areas, salt accumulates, forming saline soils. The  $\text{Na}^+$  in these soils causes slaking, swelling, and dispersion of clay, which leads to poor soil particle cohesion. Biochar can mitigate salt stress by adsorbing sodium ions and releasing inorganic nutrients such



as  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  into the soil. The release of these nutrients improves soil quality and enhances crop yields. By reducing the availability of  $\text{Na}^+$  ions, soil aggregation and water adsorption capabilities are enhanced.

A study by Huang et al. (2019a) demonstrated an increase in  $\text{K}^+$ ,  $\text{Ca}^{2+}$ , and  $\text{Mg}^{2+}$  levels upon the addition of woodchip biochar to saline soil. The  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  released by the biochar displace  $\text{Na}^+$  from the exchange sites, leading to higher leaching of  $\text{Na}^+$  ions and lower soil salinity. Another study by Huang et al. (2019b) showed that adding biochar to saline soil reduced the uptake of  $\text{Na}^+$  ions and increased the uptake of  $\text{K}^+$  ions in sweet corn. This is because the biochar's adsorption ability immobilizes sodium ions, improving the ionic balance in the soil by releasing elemental nutrients such as  $\text{K}^+$  ions.

It is important to note that careful cultivation methods are needed to maintain this balance, considering irrigation and evaporation rates. While saline soils may have high concentrations of  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  the additional  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  from biochar help displace  $\text{Na}^+$  ions, thus improving soil structure and reducing salinity effects. This results in improved growth and physiology of the sweet corn crop under saline conditions.

### **2.7.2 Increase in the nutrients in the soil**

Soil is the most important source for supplying nutrients to enhance plant growth. Plants require a few nutrients like N, P, K for their growth, but the nutrients in the soil decrease over time after crop harvesting. The extensive use of fertilisers and over cropping could be considered as the reason to lower the nutrient composition in the crops and degrade the soil fertility over the long run (Biochar - An Imperative Amendment for Soil and the Environment, 2019).

Biochar as a soil amendment, improves the soil quality and plant growth by supplying and retaining nutrients in the soil and hence can be a suitable way to promote sustainable agriculture. Biochar can supply nutrients such as N, P and K

present in the original feedstock used to produce biochar. The nutrient content in the biochar depends on the type of feedstock material and the pyrolytic conditions as discussed in the above section. Biochar also helps in improving the nutrient retention capacity of the soil due to its large surface area, porosity, and CEC. High CEC biochar reduces nutrient leaching and increase nutrient retention (Tomczyk, Sokołowska and Boguta, 2020). Its addition also increases soil CEC, which corresponds with higher N content (Moradi et al., 2019).

Nitrogen is one of the most limiting nutrients in the soil due to the leaching process. Continuous application of the N-fertiliser is a necessary step for many soils to maintain the production of crops depending on how extensively the land is being used. Different feedstocks has different amounts of nitrogen content present in them. Biochar derives from sewage sludge, poultry litter, grass waste has high N content compared to WB (Ding et al., 2016). Furthermore, the N content in the biochar also decreases with an increase in the pyrolytic temperature due to the loss of  $\text{NH}_4^+$  as ammonia ( $\text{NH}_3$ ) during pyrolysis. For instance, a study by Song et al. (2018) had seen a reduction in the nitrogen content of maize biochar from  $12.46 \text{ g kg}^{-1}$  ( $300^\circ\text{C}$ ) to  $12.05 \text{ g kg}^{-1}$  ( $600^\circ\text{C}$ ). Upon pyrolysis, the N containing compounds such as nitrates and ammonium salts can be present on the surface of the biochar or inside the pores. In another study, it was noticed that among all the feedstock the N concentration was the highest in corn crop when grown with FW biochar (Rajkovich et al., 2011).

Like the N content, the P and K contents of the biochar also depends on the feedstock type used for the biomass and the temperature of pyrolysis. Similar to the N content, the P content is higher in sewage sludge and poultry litter than manure, CR and WB. Unlike the N content, the P and K content increases with the increase in temperature (Ding et al., 2016). A study by Moradi et al. (2019) noticed an increase in the K content in the soil after treating the soil with biochar. It was noted that the increase in the K content in the soil is due to the presence of high concentration of K in grape biochar along with the increase in the soil CEC. Table 2-9 provides a synthesis of key studies highlighting the impact of different types of biochar on soil nutrient content and crop performance. The table groups biochars by feedstock type and summarises common patterns in soil improvement

and plant growth outcomes, helping to generalise trends across studies.

**Table 2- 9.** Summary of biochar effects on soil nutrient concentration and crop performance across different feedstocks and pyrolysis conditions.

<b>Biomass Feedstocks</b>	<b>Effects on soil nutrient concentration</b>	<b>Crop Performance</b>	<b>References</b>
Herbaceous feedstock (Rice straw, Maize, Corn stover, Peanut hulls, Grape biochar)	Increase in soil N, P, K concentration; increase in soil CEC and pH; improves nutrient retention.	Increases the crop yield and the effect increases with the application rate.	Khadem et al., 2021; Yao et al., 2021; Khan et al., 2024; Moradi et al.,2019; Thapa et al., 2024
Woody feedstock (Pine chips, wood biochar, Pine bark, Eucalyptus sawdust)	Minimal effect on soil N and P; slight increase in K and Ca; increase in soil pH.	Limited or no immediate yield response.	Gaskin et al., 2010; Ji et al., 2022; Domingues et al., 2017
Manure/Biosoild-based feedstock (Poultry litter, Chicken manure)	Increases soil P, Ca, Mg concentration, concentration of K was found to increase with temperature, increases soil pH.	Increases the crop yield and also increases nutrient concentration in the crops.	Subedi et al., 2016; Xiao et al.,2018
Hydrochar (Cattle manure)	Addition of biochar improves available P and K	Increases crop yield in sandy soil	Ding et al., 2022

### **2.7.3 Application of biochar in sandy soils**

Dryland covers about 72% of the world's total land area, and most of the soils in the dry regions are coarse in texture making it limited to agriculture production due to low water and nutrient retention capacity and low organic carbon content in the soil (Laghari et al., 2015). Specific surface area is a fundamental property in soil that is connected to water holding capacity, CEC and release of various elements. In sandy soils, the presence of low organic matter, low nutrient content and low CEC affects the plant growth (Baiaamonte et al., 2019). Biochar is a carbon-rich organic material which has recently been used in arid soils for improving the physical and chemical properties of the soil. The addition of biochar to the sandy soils helps in improving the water and nutrient retention capacity, improving the soil aggregation, and increasing the organic carbon content in the soil (Laghari et al., 2015). The addition of biochar to the soil is expected to increase the surface area of the soil due to the development of internal porosity (Baiaamonte et al., 2019). Several studies have evaluated the effects of different biochar feedstocks, pyrolysis temperatures, and application rates on sandy soils. These studies report variations in soil nutrient dynamics, water retention, pH, and crop responses on the type of biochar used. A synthesis of key findings is presented in Table 2-10. There have been various studies on the use of WB and agriculture as biochar in sandy soil as discussed in Table 2-10 but there have been limited studies based on the use of FW as biochar which will be focused on later during this research.

**Table 2- 10.** Summary of biochar effects of sandy soils across different feedstocks and pyrolysis conditions.

<b>Feedstock/Pyrolysis type</b>	<b>Soil Effects</b>	<b>Crop Effects</b>	<b>General Trends observed by the addition of biochar</b>	<b>References</b>
Pine derived biochars (400°C - 600C)	Increase soil surface area and porosity, C, CEC, P and K, decrease in pH, no significant change in N.	Increase in plant height at 1% application but decreased with the increase in application rate (2%).	Improve soil structure and porosity, Moderate nutrient improvement, optimal performance at medium rates.	Laghari et al., 2015; Baiamonte et al., 2019
Date palm waste (300-600°C)	BC300 increased N and P uptake, BC at lower temperature retained water compared to BC at higher temperature (BC300 > BC400 > BC500 > BC600), BC600 increased soil porosity.	Wheat yield increase with BC + NPK, no effect was observed with the application of BC alone.	Low temperature biochar improves nutrient uptake and water retention; high temperature increases porosity.	Alotaibi and Schoenau, 2019

Rice straw (300 and 700°C)	Increase in water holding capacity at BC300, increase in soil pH, C, surface area, bulk density at BC700, addition of BC700 increased aggregate formation early but decline over time.	N/A	Low temperature improves water retention; high temperature increases structural and chemical stability.	Zhang et al., 2019
Olive tree pruning (300-350°C)	Increase in soil pH, N, P and K at low application rates.	Increased tomato and bell pepper yield at lower application rate and decreased yield at higher application rate.	Optimal application rate needed, excessive rate reduced yield.	Mohawesh et al., 2021
Orange peel (300 - 600°C)	BC300 increases soil micro pores, CEC, microbial activity.	N/A	Low temperature enhances CEC and microbial activity, effect on water holding capacity inconsistent.	Abdelaal et al., 2020

## 2.8 Conclusion

This review emphasises the remediation effect of biochar from different feedstock on various types of soil properties such as the pH, CEC, porosity, surface area, water, and nutrient retention capacity. Although there have been various studies involved on the use of various techniques to produce biochar for soil application, this review focuses on two main processes namely pyrolysis and hydrothermal carbonization (HTC). It was also reviewed the HTC could be considered as a more suitable process than pyrolysis if the feedstock used has a high moisture content, considering the HTC process takes place in the presence of water. The review also focuses on the effect of various process conditions on the biochar in terms of physical and chemical properties.

The temperature of the process and the type of feedstock are two main factors that influence the properties of the biochar. Depending on the type of properties required in the biochar for soil improvement purposes, the process parameters and the feedstock type could be chosen. The review also investigates recent trends into sustainable production of biochar which is the use of MW technology for pyrolysis and HTC of biomass into biochar. Comparing MW heating with conventional heating, it was shown that the MW process is more suitable to produce a high yield biochar, requires less temperature and time to carry out the process. In the last section of the review, various applications of biochar from individual studies have been summarised.

Based on the above literature review, some of the knowledge gaps that were highlighted and explored further in the research are:

1. Most research on potato peel biochar reported in the literature focuses on its absorbent properties for pesticides and metals. Fewer studies explored its role in improving soil nutrients and plant growth.
  - In this study, the use of potato peel char (produced via conventional pyrolysis, microwave pyrolysis and microwave hydrothermal carbonisation) to enhance soil water retention, reduce nutrient leaching losses, and support plant growth was evaluated through plant pot experiments and soil analyses.



2. Few studies investigated the impact of palm waste biochar on soil fertility, water retention and crop yields.
  - This study assessed palm waste derived biochars under different production methods to determine their influence on soil nutrient availability, water retention, soil chemical properties and plant growth indicators.
3. Most of the literature covered above used conventional pyrolysis techniques to produce biochar and analyse its characteristics. The impact of microwave pyrolysis produced chars on nutrient retention, soil properties and plant yield have been limited.
  - This current study included chars produced via microwave pyrolysis and microwave hydrothermal carbonization. The chars were characterised and compared with each other along with conventional pyrolysis chars. They were evaluated for their influence on water retention, nutrient leaching, soil chemical properties, and plant growth, providing comparative insights into the effectiveness of different production methods.
4. Most studies on hydrothermal liquefaction focus on the production and characterisation of bio-oil and other soluble organic compounds rather than on the potential agricultural applications of the solid residue (hydrochar).
  - This study included hydrochars from palm waste and potato peel and examined their effects on water and nutrient retention and plant performance in soil, thereby contributing to understanding their agronomic potential.
5. Most research focuses on biochar's impact on clay and loamy soils, with limited studies on sandy soils. There is limited information on how different biochar types affect soil properties and plant yield in sandy soils.
  - While this study was limited to two biomass types (potato peel and palm waste), it broadened the scope by including three different production techniques – conventional pyrolysis, microwave pyrolysis and microwave hydrothermal carbonization. These chars were tested for their impact on sandy soils, with particular attention to soil nutrient retention, water-holding capacity, and plant response, thereby providing insights specifically relevant to sandy soil contexts.

## **Chapter 3: Methodology**

### **3.1 Introduction**

This chapter describes the materials, methods, and analytical techniques used in the study to investigate the influence of different char treatments on soil properties and wheat growth. The materials and the characterisation methods mentioned in this chapter are also used to carry out the initial laboratory scale microwave pyrolysis experiments. The chapter is organized into several sections, beginning with an overview of the raw materials and their preparation. Following the preparation of raw materials, the next section details the char production processes, which include microwave pyrolysis, conventional pyrolysis and microwave hydrothermal carbonization. The chars produced were characterised to evaluate properties such as carbon and nitrogen content, pH, cationic exchange capacity (CEC), nutrient concentration and hydrophobicity/hydrophilicity. Soil preparation for plant-pot experiments is outlined, emphasising the soil treatment combinations used to evaluate the impact of char amendments on plant growth and soil properties. The final section concludes with an overview of the statistical methods employed to analyse the data. Statistical assumptions were tested, and appropriate post hoc tests were applied where necessary.

### **3.2 Raw materials**

In this study, two kinds of raw materials were used to produce char: potato peel (PP) and palm waste (PW).

The potato peel waste (PP) used in this study was provided by Troy Foods Ltd, Leeds and McCain's, Scarborough. In these industries, a steam peeling process is used to remove potato skins, generating a sludge of potato peel waste. Although the companies provided details about the PP processing method, the age of the supplied PP was unknown. The PP sludge was washed with deionised

water and sieved to remove impurities, such as dirt, then dried in an oven at 105 °C to achieve constant dryness. The dried PP sample was cut into small pieces using a small-scale industrial shredder and sieved to achieve particle size of <10mm. The sample was stored in an airtight bag at room temperature prior to undergoing the pyrolysis and hydrothermal carbonisation processes.

Palm waste (PW), including leaves, branches, and stem bark from the species *Sabal Mexicana* (Texas palm), was provided by the Royal Botanical Garden, Edinburgh. The waste was processed into small pieces using a small-scale industrial shredder and sieved to achieve a particle size of <10 mm and then mixed thoroughly. The shredded particles were dried in an oven at 105 °C until a constant weight was achieved and stored in an airtight bag at room temperature before pyrolysis and hydrothermal carbonisation.

Lufa 2.1 soil, purchased from Lufa Speyer (Table 3-1), was chosen for its similarity to Torripsamment soils, which are extensively found in the UAE and are considered suitable for agriculture with proper irrigation and management practices (Shahid et al., 2008). The soil was air-dried and sieved to particle size <2 mm before use in the experiments.

**Table 3- 1.** Reported properties of the Lufa 2.1 soil (Lufa-speyer.de, 2024). Data are presented as the mean ± standard deviation.

	Soil texture	C (%)	N (%)	pH	CEC (cmol/kg)
<b>Lufa 2.1 soil</b>	86.2% sand, 10.4% silt and 3.4% clay	0.83 ± 0.29	0.08 ± 0.02	4.90 ± 0.24	4.10 ± 0.67

To match the calcium carbonate (CaCO<sub>3</sub>) content typical of torripsamment soils, limestone boulders from the White Chalk subgroup from the Campanian stage of the Cretaceous period were collected from Danes Dyke Beach, Bridlington (TA 2165 6917) (Ukge, 2023). The limestone was crushed to particles <2 mm and thoroughly mixed into the sieved Lufa 2.1 soil in a mass ratio of 1.2:1 Lufa soil to limestone to approximate the CaCO<sub>3</sub> content of typical torripsamment soils (Table 3-2).

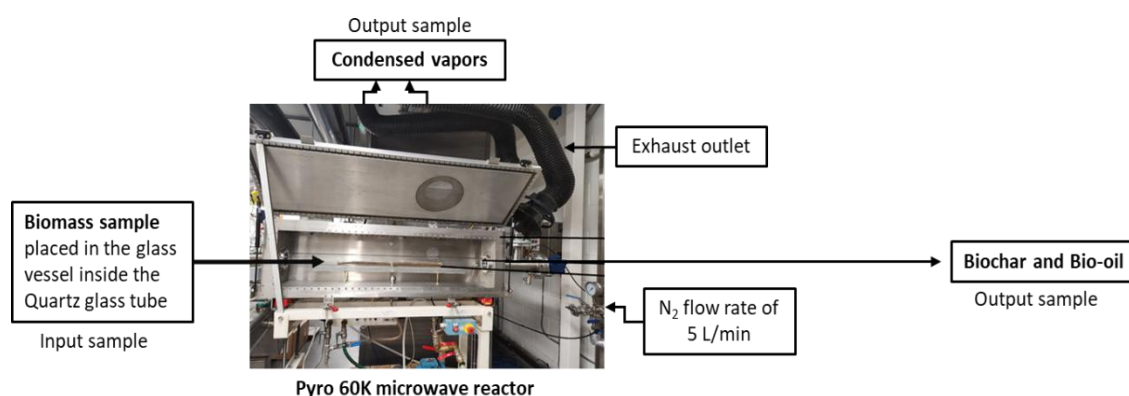
**Table 3- 2.** Measured properties of the Lufa 2.1 soil mixed with 46% CaCO<sub>3</sub>, as used for the plant-pot experiments. Data are presented as the mean  $\pm$  standard errors, n=3. The methods used for these measurements are detailed in section 3.4.

Parameters	Soil mixture
C (%)	1.68 $\pm$ 0.09
N (%)	0.15 $\pm$ 0.01
pH	8.17 $\pm$ 0.08
CEC (cmol/kg)	4.7 $\pm$ 0.72
Concentration of potassium on soil exchange sites (mg/g)	0.104 $\pm$ 2.48*10 <sup>-16</sup>
Concentration of magnesium on soil exchange sites (mg/g)	0.004 $\pm$ 0.002
Concentration of calcium on soil exchange sites (mg/g)	0.083 $\pm$ 0.013

### 3.3 Char production

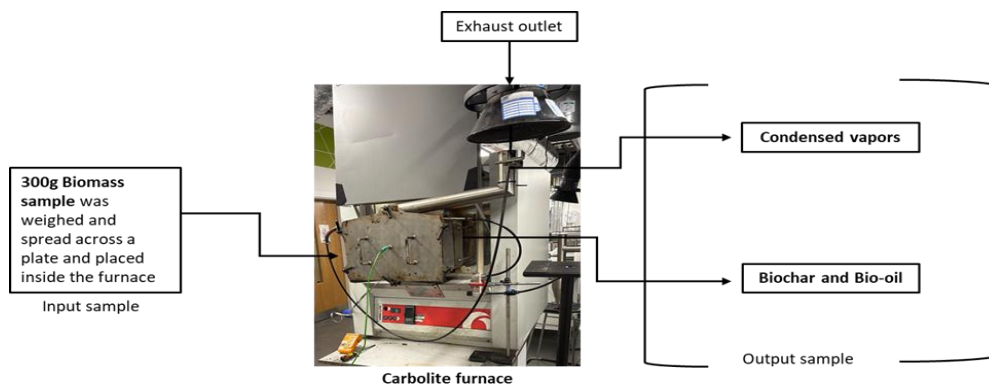
The chars were produced using three different methods: microwave pyrolysis, conventional pyrolysis, and microwave hydrothermal carbonisation.

Microwave pyrolysis was conducted using a Pyro 60K microwave at the Bio Renewables Development Centre (BDC), York, YO19 5SN, UK. The large-scale microwave reactor at BDC, was modified for batch operation by removing the auger screw and using glass vessels (Figure 3-1). In this study, the pyrolysis process was performed with a microwave power of 2 kW for approximately 1 h under a continuous N<sub>2</sub> flow rate of 5 L/min to maintain an inert atmosphere. The operating conditions for the large scale microwave reactor were determined through a trial and error approach, in consultation with experienced technical staff. The parameters of the microwave reactor were optimised based on the reactor's design limitations, safety considerations, and the desired yield of the char. Each experimental run involved either approximately 150 g of PP or 70 g of PW, which was evenly distributed in two glass vessels (300 x 65 x 40 mm) within a quartz tube (80 mm od, 70 mm id, length 1200 mm) in the microwave chamber. The biomass sample within the vessel remained static within the microwave cavity, without stirring or mechanical agitation.



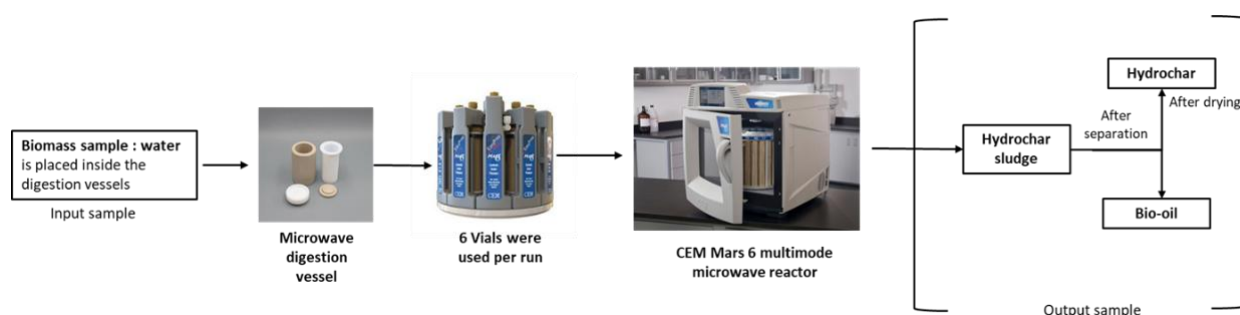
**Figure 3- 1.** Workflow of a large-scale microwave pyrolysis reactor for the production of char from potato peel (PP) and palm waste (PW) biomass. The system includes a stainless-steel microwave chamber, a quartz glass tube reactor, and a glass vessel containing the the biomass sample. Microwave energy is applied to heat the feedstock under continuous nitrogen ( $N_2$ ) flow.

Conventional pyrolysis was conducted using a Carbolite chamber furnace (model GPC 12/65) equipped with a gas tight A105 Inconel front loading retort with an inert  $N_2$  gas flow. The pyrolysis was carried out at 500 °C with a heating rate of 5 °C/min requiring a total of 100 min to reach the desired temperature. Once 500 °C was achieved, it was maintained for an additional 240 min. The final temperature for this process was selected based on the thermogravimetric analysis (TGA) data of the PP and PW biomass. TGA curves provided insight into the decomposition temperatures of the major components, allowing for the identification of the optimal pyrolysis temperature (Figure 3-2). The heating rate and the residence time were chosen based on values commonly reported in the literature, as well as recommendations from technical experts to ensure efficient carbonisation. Each run involved pyrolysis of 300 g of biomass feedstock.



**Figure 3- 2.** Workflow of conventional pyrolysis of potato peel (PP) and palm waste (PW) biomass using a Carbolite furace. 300g of biomass is spread evenly on a plate and placed inside the furnace chamber. The sample is pyrolysed under controlled temperature condition, leading to formation of biochar.

Microwave hydrothermal carbonisation was conducted using a CEM MARS 6 multimode microwave reactor (Figure 3-3). The reaction was performed at 200 °C, with a holding time of 20 min. Each run included six vials with a volume of 100ml, each containing approximately 10 g of biomass feedstock and 50 ml of water. The biomass sample within the vial remained static within the microwave cavity, without stirring or mechanical agitation.



**Figure 3- 3.** Workflow of CEM Mars microwave hydrothermal carbonisation of potato peel (PP) and palm waste (PW) biomass. The biomass slurry is loaded into the vessels, placed into a microwave digestion rotor system, and processes under elevated temperature and pressure. After completion, six reaction vessels are cooled, and hydrochars are collected.

During the microwave pyrolysis of PP, hot spots formed within the char as a result of irregular heating patterns. These hot spots cause localised, more extensive carbonisation of the feedstock compared to neighbouring regions. The severity of hotspots formation during microwave pyrolysis was moderate but noticeable, and it was observed specifically during the pyrolysis of PP biomass. In PP, hotspots led to visibly heterogeneous biochar with variations in texture and colour, reflecting uneven carbonisation. Although, the issue did not prevent biochar production, this non-uniformity resulted in differences in physiochemical properties of the char. The formation of these hotspots arises from the uneven distribution of the microwave field, influenced by biomass characteristics such as particle size and moisture content (Lin et al., 2022; Horikoshi et al., 2011). Biomass with uneven moisture distribution absorbs microwave energy inconsistently, as water, with its high dielectric loss factor, converts microwave energy into heat effectively. Consequently, moist regions heat faster, creating localised hotspots (Chen et al., 2024). Additionally, irregular particle sizes affect the microwave field's distribution, as areas with larger particles absorb more energy and form hotspots (Lei et al., 2009). The highly carbonised biochar formed at these hot spots was designated pure biochar (PB), while the surrounding char was classified as partially charred biochar (PCB).

To further separate PB from PCB particles, a water flotation method was used based on density differences. PCB was added to a 500 ml beaker containing 500 ml of deionised water, thoroughly mixed, and left to stand for 2 hours. The less dense PB particles floated to the surface, allowing separation from the denser PCB particles.

These density differences arise from varying levels of carbonisation. The hotspots absorbed more heat, resulting in greater pyrolysis in those regions. This process led to the release of water and volatile organic compounds in the form of oil or gas, leaving behind a highly porous carbon structure characteristic of PB biochar (Zhang et al., 2015; Khanmohammadi et al., 2015). The lower porosity of PCB biochar (Figure 5-1a) further contributed towards its higher density. Whereas the increased porosity (Figure 5-1b) reduced PB biochar's density, making it float in water (Brewer et al., 2014). In contrast, regions surrounding the hotspots underwent less extensive pyrolysis, retaining more water and organic compounds, which increased their density and caused PCB biochar to sink.

From PP feedstock, chars were classified based on production techniques: microwave partially

charred biochar (PCB), microwave pure biochar (PB), conventional biochar (CB), and microwave hydrochar (MWH). Similarly, PW derived chars were classified as microwave biochar (MWB), conventional biochar (CB), and microwave hydrochar (MWH).

All char samples underwent washing with 100ml of acetone followed by vacuum filtration to remove residual bio-oil. The level of bio-oil contamination was moderate in both microwave and conventional pyrolysis, as most volatiles were released and partially/completely removed during the high temperature carbonisation process. However, in the case of microwave hydrothermal carbonisation, a significant higher amounts of bio-oil were retained in the final product. This is because, during microwave hydrothermal carbonisation produces hydrochar sludge under subcritical water conditions, where the lower reaction temperatures and the aqueous medium inhibit complete devolatilisation. As a result, hydrochar contains more tarry residues and soluble organic compounds. Washing the chars was essential to reduce potential phytotoxicity and also helped to eliminate the influence of residual pyrolysis by-products on soil properties thereby, allowing accurate assessment of char effects on soil properties and plant growth. The washed chars were air-dried overnight in a fume hood to allow the volatilisation of excess acetone. Once acetone removal was confirmed by the absence of odour, the chars were further dried in an oven at 105 °C to a constant mass and stored in airtight containers prior to analysis. All analyses were conducted in triplicate.

### **3.4 Char characterization**

#### **a) Thermogravimetric analysis (TGA)**

TGA is a technique used to measure the weight loss of a sample as temperature increases. TGA was conducted using a NETZSCH STA449 instrument to evaluate the thermal decomposition behaviour of the char samples for further differentiation. Approximately 20 mg of each sample was heated to 800 °C under nitrogen atmosphere at a rate of 10 °C/min.



## **b) Carbon (C) and Nitrogen (N) characterisation**

Total carbon (C) and nitrogen (N) contents were measured using a Vario MacroCube Elemental Analyser. Prior to analysis, all char samples of approximately 30mg of the dried material were weighed into tin capsules and sealed into compact pellets. Each run included blanks, glutamic acid standards, and a certified reference material (CRM) to ensure accuracy. Samples were combusted at high temperatures in an oxygen-rich environment. The resulting gases were analysed, and results were corrected using a daily calibration factor derived from glutamic acid standards. Final C and N values were reported as a percentage of dry weight.

## **c) Water drops penetration time (WDPT) test**

The hydrophobicity of the char samples was assessed using the WDPT test (Usevičiūtė and Baltrėnaitė, 2019). To evaluate their wettability, 5 g of homogenised char was placed in 12 ml vials. Using a pipette, three drops of water were carefully applied to the surface of the char from a height of less than 10 mm to ensure consistent droplet formation. The water drop penetration time was measured using a stopwatch. The categories of hydrophobicity with corresponding WDPTs were referenced from Usevičiūtė and Baltrėnaitė (2019).

## **c) pH characterisation**

The pH was determined after shaking a suspension of char and soil samples in deionised water in a 1:20 solid (mass) to-water (volume) ratio for 1.5 h using a rotary shaker at 200 rpm (Arwenyo et al., 2023). The char and soil samples were allowed to settle for five minutes before pH measurement using a Thermal Orion pH meter. Before measuring the pH of the char samples, the equipment was calibrated using a three-point calibration method (pH 4, pH 7, pH 10) to ensure high accuracy.

#### d) Cationic exchange capacity (CEC)

To determine the CEC of the char samples, the AOAC method 973.09 was used (Rippy and Nelson, 2007). Since CEC is pH dependant, char samples were pre-treated by adjusting the pH to 7. A 0.2 g of oven-dried char was mixed with 4 mL of deionized water and shaken for 4 h at 200 rpm to ensure complete wetting. The pH of the slurry was adjusted to 7 using Sodium hydroxide (NaOH) and monitored periodically over 48 hours to ensure stabilisation. The water was then removed by filtration through 2 µm filter paper (Whatman Nucleopore Track-Etch membrane). The pH-adjusted char sample was combined with 4 mL of 1 M Ammonium acetate (NH<sub>4</sub>OAc) in a Buchner funnel, swirled, and allowed to stand for 2 h before applying vacuum to remove the NH<sub>4</sub>OAc. Repeated additions of 4 mL NH<sub>4</sub>OAc were performed for a total of 4 times. Cations in these extracts were quantified using an inductively couple plasma (ICP) spectrometer using the Thermo iCAP 7200 ICP-OES to identify individual elements on the exchange sites in the char and soil samples. Subsequently, non-complexed NH<sub>4</sub><sup>+</sup> was removed by four washes of 4 mL of ethanol (EtOH 100%). The EtOH wash was performed 4 times. Finally, complexed NH<sub>4</sub><sup>+</sup> was displaced with potassium chloride (KCl): 4 mL of 2 M KCl was added to the char sample, swirled, and allowed to stand for 2 h, and then vacuum was applied to collect the filtrate in an Erlenmeyer flask. A total of four extractions were done, producing 16 mL of extracted volume. Lastly, the ammonium was measured using the Seal Analytical Autoanalyzer 3 and CEC was calculated as follows (Kharel et al., 2019):

$$CEC (cmol\ kg^{-1}) = \frac{NH_4^+ conc (mgL^{-1}) \times V(L) \times 100}{molecular\ mass\ of\ NH_4 \times W(g)} \quad (1)$$

Where NH<sub>4</sub><sup>+</sup> conc is the concentration of NH<sub>4</sub><sup>+</sup> in the extract, V is the volume of the extract and W is the mass of the char.

#### **e) Surface area and Porosimetry**

Prior to analysis, all char samples of approximately 150mg of the dried material was accurately weighed and subjected to degassing at 200°C overnight under vacuum to remove any adsorbed moisture or volatile impurities. After degassing, the samples were cooled to room temperature under inert conditions before analysis.

The nitrogen adsorption–desorption isotherms were recorded at liquid nitrogen temperature (77K) on a Micromeritics TriStar II porosimeter (or Micromeritics ASAP 2020 porosimeter) using the Barrett- Joyner-Halenda (BJH) and Brunauer-Emmett-Teller (BET) method. All measurements were performed in triplicate, and results are reported as mean  $\pm$  standard deviation.

#### **f) Proximate analysis**

Proximate analysis was performed to determine the moisture content, volatile matter, and ash content of the biomass samples (Aller et al., 2017).

- a) Moisture content: 1g of oven dried biomass weighed and heated at 105°C for 24 hours in a drying oven. The sample was cooled and reweighed. Moisture content was calculated as the percentage weight loss relative to the original sample.
- b) Volatile matter: A separate 1g sample of the dried biomass was placed in a covered crucible and heated at 950°C for 10min in muffle furnace. The crucible was cooled and reweighed. The volatile matter was expressed as the percentage of mass lost during heating, excluding moisture.
- c) Ash content: To determine, ash content, another 1g sample was heated in an uncovered crucible at 750°C for 6 hours in a muffle furnace to allow combustion of organic matter. After cooling, the remaining inorganic residue was weighed, and ash content was calculated as a percentage of the original dry weight.

### **3.5 Soil preparation for the wheat pot trial**

Three separate plant-pot experiments were conducted to examine the impact of chars on plant growth and soil properties.

The first plant-pot examined the impact of a control (C) and two treatments using potato peel waste: 5 % of PCB and 5 % of PB of mass added to the soil.

A study by Kim et al. (2016) observed that the application of 5 % biochar to soil resulted in an increase in soil pH, CEC, nutrient concentration and an increase in the plants dry yield. Lower application rates of 1 % and 2 % did not show significant effects compared to the control. However, increasing the application rate of biochar beyond 5 % led to decrease in plant biomass yield, primarily attributed to an excessive increase in soil pH. The elevated soil pH can induce nutrient deficiencies, which become less available in alkaline conditions, thereby negatively affecting plant growth (Mohawesh et al., 2018). Considering the amount of raw biomass available for this study, a 5 % application rate of char appeared to be the most suitable, as it balanced the benefits of improved soil properties and plant yield without causing adverse effects due to excessive alkalinity.

80 g of oven-dried char was added to 1.6 kg of air-dried soil-limestone mixture and then moistened with 400 g of deionised water to achieve a water content of 25 % before being mixed and left overnight. 400 g of moist soil mixture was added to 1 L plant pots that were 9.2 cm high and had a 10 cm diameter at their top. Four replicates were set up per treatment.

The second plant-pot examined the impact of control (C) and two treatments using potato peel waste: 5 % MWH and 5 % CB of mass added to the soil. 60 g of oven-dried char was added to 1.2 kg of air-dried soil-limestone mixture and then moistened with 300 g of deionised water to achieve a water content of 25 % before being mixed and left overnight. 400 g of moist soil mixture was added to 1 L of plant pots that were 9.2 cm high and had a 10 cm diameter at their top. Three replicates were set up per treatment for this experiment due to the shortage of the raw material provided.

The third plant-pot experiment examined the impact of control (C) and three treatments using palm waste: 5 % MWB, 5 % CB and 5 % MWH if mass added to the soil. 80 g of oven-dried char

was added to 1.6 kg of air-dried soil-limestone mixture and then moistened with 400 g of deionised water to achieve a water content of 25 % before being mixed and left overnight. 400 g of moist soil mixture was added to 1 L plant pots that were 9.2 cm high and had a 10 cm diameter at their top. Four replicates were set up per treatment.

### **3.6 Wheat pot trial**

Prior to sowing, the winter wheat seeds (*Triticum aestivum* L.) were germinated on wet paper towel in petri dishes for a week. After the emergence of roots and a small sprout, the seeds were sown to a depth of approximately 20 mm in each pot.

All the three plant-pot plant experiments were conducted in the Department of Environment and Geography at the University of York. In the first and the second plant-pot experiment, plants were grown next to a window under ambient laboratory conditions with an average temperature of 18 °C. The first experiment ran from February 2023 to May 2023, and the second experiment from August 2023 to October 2023. The pots were arranged in a randomized block design, with each block containing a control and two treatments.

To maintain the plant growth for the third pot experiment at the same temperature during the winter-time, the third plant-pot experiment plants were grown in a controlled room at 18 °C at the Department of Environment and Geography, University of York from November 2023 –February 2024. The pots were arranged in a randomized block design, with each block containing a control and three treatments. Each block was positioned under a LED growth light, providing light with intensity of 200  $\mu\text{mol}/\text{m}^2/\text{s}$ , as measured by a Photosynthetically Active Radiation (PAR) meter.

In all the experiments, the seedlings were watered with 50 mL of deionised water on Mondays, Wednesdays and Fridays. From the 5<sup>th</sup> week onward Hoagland solution (Hoagland et al., 1938) was used instead of de-ionised water to ensure adequate nutrient supply.

The number of leaves was recorded weekly, and water leachate volume was measured at the same intervals to determine soil leachate volume. The collected leachates were filtered using grade 2 Whatman qualitative filter paper. Elemental concentrations in the leachates were analysed using a Thermo iCAP 7200 ICP-OES spectrometer, while pH was measured with a

Thermal Orion pH meter. Dissolved organic carbon (DOC) concentrations were analysed using a Vario cube TOC analyser. The quality control of these analysis have been mention below in section 3.8.

After 12 weeks of plant growth, the above ground plant material, comprising leaves and stems, were cut 1 cm above the soil, oven dried at 105 °C overnight, and then weighed. The total soil porosity  $\phi$  ( $\text{cm}^3 \text{ cm}^{-3}$ ) was calculated by the following equation (Alghamdi et al., 2020):

$$\phi = 1 - \frac{\rho_b}{\rho_s} \quad (2)$$

Where  $\rho_b$  is the soil bulk density ( $\text{g cm}^{-3}$ ), and  $\rho_s$  is the soil particle density (taken as  $1.43 \text{ g cm}^{-3}$ ). The soil bulk density ( $\text{g cm}^{-3}$ ) was calculated by the following equation (Al-Shammary et al.,2018):

$$\rho_b = \frac{\text{Dry soil weight (g)}}{\text{Volume of the pot (cm}^3\text{)}} \quad (3)$$

And the volume of the pot ( $\text{cm}^3$ ) was calculated by the following equation:

$$V = \frac{\pi H}{3} (R^2 + Rr + r^2) \quad (4)$$

Where H is the height of the pot (cm), R is the radius of the outer circle (cm) and r is the radius of the inner circle (cm).

Wheat biomass was digested for nutrient analysis using Kjeldahl digestion (Alexander et al.,2006). About 0.5 g of dried, ground plant material is treated with concentrated nitric acid and left overnight. The sample is then heated in a digestion block at 60 °C for 3 hours, followed by 110 °C for 6 hours. After cooling, the digest is filtered using prewashed filter papers into 100 ml volumetric flask, with thorough rinsing to ensure complete transfer. The final volume is adjusted to 100 ml with ultra-pure water, and the extract analysed by ICP-OES.

At the end of the experiment approximately 3g of moist soil was extracted to determine available  $\text{NO}_3^-$  and  $\text{NH}_4^+$  following the methods of Jackson and Bloom (1994). Soil extraction for  $\text{NO}_3^-$  and  $\text{NH}_4^+$  analysis involves preparing 0.5M KCl, adding 30 ml of it to 3 g of fresh soil or blank tubes, and shaking for 1 hour. The mixture is then filtered, and the filtrated solution are analysed using auto analyser.

The remaining soil was air dried, and pH, C-N content, available exchangeable cations and CEC were measured using the same methods as those used for char characterisation detailed above (Section 3.4).

### **3.7 Statistical Analysis**

All the data were analyzed using IBM SPSS Statistics 27. Statistical assumptions were tested, and appropriate post hoc tests were applied where necessary. An ANOVA repeated measures test was conducted to determine the significance of the treatments and time on the number of leaves, water leachate volume and composition. When the nutrient concentration in the leachates were below detection limit they were set to a value equal to the BDL divided by the square root of two when used in statistical analysis (Croghan and Egeghy, 2003).

A one-way ANOVA test was conducted to determine the effect of treatments on plant-available  $\text{NO}_3^-$  and  $\text{NH}_4^+$  in the soil, soil pH, and CEC. The Shapiro-Wilik test was used to assess whether the data was subject to normal distribution. The homogeneity of variance was tested using Levene's Test. When the assumption of the homogenous variance for ANOVA was violated, a non-parametric Kruskal-Wallis test was conducted (Table 3-3). Based on the variance assumed, Tukey's and Games-Howell post hoc tests were used. Pearson's correlation was used to look for correlations between soil  $\text{NO}_3^-$  and  $\text{NH}_4^+$ , soil CEC and pH across the treatments.

**Table 3- 3.** Summary of non-parametric analyses performed across studies on the effect of different char treatments on soil properties and nutrient concentration in plants.

Study	Analyses that were non-parametric
Potato peel: PCB and PB study (Chapter 5)	Soil porosity, soil nitrate, soil pH, Soil potassium, soil calcium, soil magnesium and soil zinc concentration in wheat biomass
Potato peel: MWH and CB study (Chapter 6)	Phosphorus concentration in wheat biomass
Palm waste: MWB, CB and MWH (Chapter 7)	Soil nitrate, soil potassium, soil phosphorus and concentration of phosphorus in wheat biomass.

### 3.8 Quality Control

All the leachates Chapter 5, 6 and 7 were analysed for DOC concentration was analysed using TOC analyser; the elemental nutrient concentration (potassium, magnesium, calcium, iron and zinc) was analysed using ICP-OES. The wheat biomass digestion in Chapter 5,6 and 7 was also analysed using ICP-OES, and their quality control data were calculated and represented in Table 3-4 a-e.

#### a) Certified reference material (CRM)

The CRM for measuring potassium, magnesium, calcium, iron and zinc in leachates and wheat biomass digestion used in house 0.5 mg/L standard. The CRM for measuring dissolved organic carbon in leachates used in house 25mg/L 90326 TOC standard. The CRM for measuring CN in the char samples used EMA birch leaf standard.



## **b) Accuracy**

Accuracy was calculated by dividing the measured CRM concentration by the reported CRM concentration and multiplying by 100. The calculated accuracy for nutrient concentration in the potato peel (PCB and PB) and palm waste char leachates are represented as  $n = 4 \pm$  standard deviation and in the potato peel (MWH and CB) are represented as  $n = 3 \pm$  standard deviation (Table 3-4 a-e). The accuracy of CN measurements for soil and char samples are shown in Table 3-4f. The accuracy of DOC concentration in the leachates are represented as  $n = 4 \pm$  standard deviation (Table 3-4f).

## **c) Precision**

Precision was evaluated by analysing paired samples and expressed as the coefficient of variation (Gill and Ramsey, 1997). To ensure accuracy, duplicate measurements should be conducted on 10 % of the samples, with values at least 100 times higher than the detection limits. For each duplicate pair, the mean should be calculated, followed by the absolute difference between the two values in each pair. This difference is then expressed as a percentage of the mean. The median percentage provides a reliable estimate of the coefficient of variation. The calculated precision in the potato peel (PCB and PB) and palm waste char leachates are represented as  $n = 4 \pm$  standard deviation and in the potato peel (MWH and CB) are represented as  $n = 3 \pm$  standard deviation (Table 3-4 a-e).

#### d) Detection limit (DL)

DL was calculated using the mean plus six times the standard deviation of blank analyses (Walsh, 1997). The calculated DL in the potato peel (PCB and PB) and palm waste char leachates are represented as  $n = 4 \pm$  standard deviation and in the potato peel (MWH and CB) are represented as  $n = 3 \pm$  standard deviation (Table 3-4 a-e).

The values that were below the DL were set to DL divided by the square root of 2, and then these converted values were used in further statistical analysis (Croghan and Egeghy, 2003).

**Table 3- 4a.** Quality control for potassium analysis of leachates and wheat biomass digestion.

Measurement	Accuracy (%)	Measured CRM	Precision (%)	Detection limit
Leached potassium concentration in control soil	109.07	In house multi-elements 0.5 mg/L standard	0.98	0.227 mg/L
Leached potassium concentration in potato peel – PCB and PB leachates	$94.9 \pm 4.61$	In house multi-elements $0.46 \pm 0.02$ mg/L standard	$0.76 \pm 0.12$	$0.374 \pm 0.15$ mg/L
Leached potassium concentration in potato peel – MWH and CB leachates	$101.09 \pm 4.21$	In house multi-elements $0.51 \pm 0.02$ mg/L standard	$0.47 \pm 0.44$	$0.818 \pm 0.17$ mg/L
Leached potassium concentration in palm waste – MWB, CB and MWH	$106.87 \pm 4.85$	In house multi-elements $0.53 \pm 0.03$ mg/L standard	$1.44 \pm 0.93$	$0.237 \pm 0.13$ mg/L

Concentration of potassium in wheat biomass digestion	106.422	In house multi-elements 0.53 mg/L standard	2.65	0.435 mg/L
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**Table 3-4b.** Quality control for calcium analysis of leachates and wheat biomass digestion.

<b>Measurement</b>	<b>Accuracy (%)</b>	<b>CRM</b>	<b>Precision (%)</b>	<b>Detection limit</b>
Leached calcium concentration in control soil	86.112	In house multi-elements 0.5 mg/L standard	0.64	0.128 mg/L
Leached calcium concentration in potato peel – PCB and PB leachates	111.5 ± 0.44	In house multi-elements 0.55 ± 0.005 mg/L standard	0.71 ± 0.202	0.44 ± 0.08 mg/L
Leached calcium concentration in potato peel – MWH and CB leachates	110.91 ± 4.42	In house multi-elements 0.55 ± 0.03 mg/L standard	0.51 ± 0.16	0.94 ± 0.58 mg/L
Leached calcium concentration in palm waste – MWB, CB and MWH	110.37 ± 2.76	In house multi-elements 0.55 ± 0.02 mg/L standard	1.55 ± 0.91	0.48 ± 0.35 mg/L
Concentration of calcium in wheat biomass digestion	110.16	In house multi-elements 0.55 mg/L standard	0.89	0.029 mg/L

**Table 3-4c.** Quality control for magnesium analysis of leachates and wheat biomass digestion.

Measurement	Accuracy (%)	CRM	Precision (%)	Detection limit
Leached magnesium concentration in control soil	115.64	In house multi-elements 0.57 standard	4.96	0.076
Magnesium concentration in potato peel – PCB and PB leachates	113.45 ± 7.61	In house multi-elements 0.56 ± 0.04 mg/L standard	5.41 ± 3.47	0.008 ± 0.006 mg/L
Magnesium concentration in potato peel – MWH and CB leachates	112.25 ± 5.82	In house multi-elements 0.55 ± 0.02 mg/L standard	0.32 ± 0.36	0.083 ± 0.025 mg/L
Magnesium concentration in palm waste – MWB, CB and MWH	109.5 ± 6.42	In house multi-elements 0.54 ± 0.04 mg/L standard	1.72 ± 0.65	0.004 ± 0.002 mg/L

**Table 3-4d.** Quality control for iron analysis of leachates and wheat biomass digestion.

Measurement	Accuracy (%)	CRM	Precision (%)	Detection limit
Leached iron concentration in control soil	117.81	In house multi-elements 0.58 mg/L standard	0.87	0.006 mg/L
Iron concentration in potato peel – PCB and PB leachates	110.83 ± 9.46	In house multi-elements 0.55 ± 0.04 mg/L standard	9.76 ± 4.13	0.005 ± 0.002 mg/L
Iron concentration in potato peel – MWH and CB leachates	112.74 ± 0.21	In house multi-elements 0.56 ± 0.003 mg/L standard	1.945 ± 0.98	0.008 ± 0.0007 mg/L
Iron concentration in palm waste – MWB, CB and MWH	113.17 ± 3.57	In house multi-elements 0.56 ± 0.017 mg/L standard	2.015 ± 2.09	0.005 ± 0.003 mg/L
Concentration of iron in wheat biomass digestion	109.32	In house multi-elements 0.55 mg/L standard	2.88	0.0026 mg/L

**Table 3-4e.** Quality control for zinc analysis of leachates and wheat biomass digestion.

Measurement	Accuracy (%)	CRM	Precision (%)	Detection limit
Leached zinc concentration in control soil	116.61	In house multi-elements 0.58 mg/L standard	2.49	0.005 mg/L
Zinc concentration in potato peel – PCB and PB leachates	113.53 ± 4.58	In house multi-elements 0.55 ± 0.009 mg/L standard	2.16 ± 0.31	0.0025 ± 0.001 mg/L

Zinc concentration in potato peel – MWH and CB leachates	108.12 ± 0.14	In house multi-elements 0.53 ± 0.007 mg/L standard	3.7 ± 1.72	0.003 ± 0.002 mg/L
Zinc concentration in palm waste – MWB, CB and MWH	109.99 ± 0.74	In house multi-elements 0.53 ± 0.04 mg/L standard	1.61 ± 0.604	0.0029 ± 0.002 mg/L
Concentration of zinc in wheat biomass digestion	105.94	In house multi-elements 0.53 mg/L	2.48	0.0041 mg/L

**Table 3-4f.** Quality control for CN measurements of soil and char samples and DOC concentration of the leachates.

Measurements	Accuracy (%)	CRM
CN measurement for char and soil samples	C: 98.96 N: 79.08	EMA birch leaf standard: C: 48.09 % N: 6.55 %
DOC concentration in potato peel – PCB and PB	107.59 ± 0.25	In house 25mg/L TOC standard
DOC concentration in potato peel – MWH and CB	106.72 ± 0.30	In house 25mg/L TOC standard
DOC concentration in palm waste – MWB, CB and MWH	103.92 ± 0.12	In house 25mg/L TOC standard

## **Chapter 4: Production of Potato Peel Waste and Palm Waste Biochar Using Laboratory-Scale Microwave Reactor**

### **4.1 Introduction**

This chapter investigates the production of biochar from potato peel waste (PP) and palm waste (PW) using a laboratory scale microwave reactor. Microwave assisted pyrolysis was conducted at varying temperatures to evaluate biochar yield, surface area, and pH for its potential applications. The study aims to optimise pyrolysis parameters for these feedstocks, providing insights into the effect of temperature on biochar characteristics.

This preliminary investigation not only contributes to the understanding of biochar properties derived from different biomass sources but also enhances the understanding for scaling up biochar production. By identifying the conditions that maximise yield and desired properties, this research provides a basis for future studies focused on enhancing biochar's utility in agricultural applications.

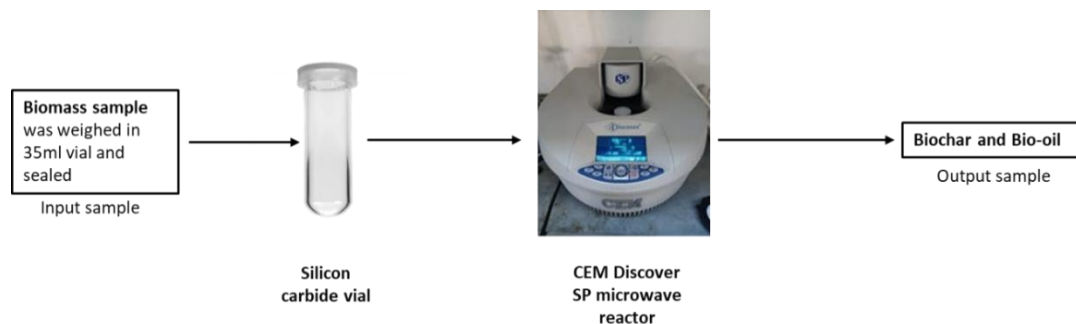
### **4.2 Experimental Procedure**

#### **4.2.1 Raw Material**

Refer to Chapter 3, Section 3.2

### 4.2.2 MW Pyrolysis Experiments

The microwave-assisted pyrolysis was carried out in a CEM Discover SP microwave working at 2.4 GHz (Figure 4-1).



**Figure 4- 1.** Workflow diagram of the laboratory scale microwave pyrolysis reactor used for the production of biochars from potato peel and palm waste biomass. Biomass samples were loaded into sealed glass vials and subjected to pyrolysis in a closed-vessel microwave reactor.

Pyrolysis experiments were conducted at four different temperatures 150, 180, 200 and 250 °C for PP and the pyrolysis experiments for PW were conducted at two different temperatures 150 and 180 °C. The selection of these temperatures for the pyrolysis experiments was based on the TGA results of the biomass feedstock (Section 4.3.1). Based on the TGA graph, major devolatilisation phase occurred between approximately 188-346°C for PP and 172-346°C for PW. Based on this, the target pyrolysis temperatures were selected to ensure maximum breakdown of volatile components and sufficient carbonisation. For conventional pyrolysis, temperatures in the range of 280-430°C for PP and 340-420°C for PW are seemed to suitable to capture the peak degradation of cellulose and hemicellulose, while initiating lignin breakdown. However, for microwave pyrolysis, where the heat transfer is



more efficient, the selected temperature range was reduced approximately 100°C (Budarin et al., 2010). As a result, the temperature range to carry out microwave pyrolysis of PP and PW biomass will be at 180-330°C and 240-320°C respectively.

Although the initial experimental setup for PW included four different temperatures, only two temperatures (150 and 180 °C) produced valid data for analysis, as the pyrolysis process failed under the other two conditions. All trials were carried out at a power setting of 100 W and a maximum pressure limit of 300 PSI. At higher temperatures, the pyrolysis process failed due to excessive internal pressure buildup, which exceeded the reactor's safety threshold and caused the system to stop the reaction.

For each experiment, 1 gram of PP or PW sample was weighed separately in a 35 ml silicon carbide vial and sealed. The biomass sample in the sealed 35 ml vial was subjected to the microwave heating process in the reactor at the required temperature for 90 s. After reaching the desired temperature, the reactor automatically stops and cools down.

The biochar derived from PP and PW was vacuum filtered using a porous crucible with a pore size filter of 8 µm and washed twice with 20 ml of acetone to recover the bio-oil. The biochar was then dried in an oven at 105 °C overnight. Once dried, the biochar samples were stored in sealed containers for subsequent analysis. Biochar samples were named as following "Biomass Name-Temperature" (e.g. PP-150 means pyrolysis of potato peel at 150 °C). All the experiments were conducted three times to determine the variability of the results and to assess any experimental errors.

The yield of the biochar was calculated by first recording the weight of the empty crucible prior to the experiment. The initial weight of the biomass was then measured and recorded. After pyrolysis, the crucible containing the biochar was weighed, and the recorded values were used to determine the biochar yield. After pyrolysis, the biochar sample was dried to remove any residual moisture, and the crucible with the dried biochar was weighed again. The difference in weight between the crucible with the dried biochar and the empty crucible provided the mass of the biochar. This mass was then expressed as a percentage of the initial weight of the feedstock to determine the biochar yield.

#### **4.2.3 Biochar Characterisation**

This study measured the surface area and pH of PP and PW biochars produced at different temperatures. PP was analysed at four temperatures (150, 180, 200 and 250°C). while PW was analysed at two temperatures (150 and 180°C), all at a microwave power of 100 W power. A detailed explanation of the analytical methods used can be found in Section 3.4.

#### **4.2.4 Statistical Analysis**

Data collected for the yield and pH are means of triplicate measurements. All the data were analysed using IBM SPSS Statistics 27. A one-way ANOVA test was conducted to determine the significance of temperature on the biochar yield and pH of the PP biochar. The Shapiro-Wilk test was used to assess whether the data was subject to normal distribution. The homogeneity of variance was tested using Levene's Test. The assumption of the homogenous variance for ANOVA was not violated and based on the variance assumed, Tukey's post hoc tests were used. To determine the significance of temperature on the biochar yield and pH of PW biochar's, a one sample t-test was conducted.

### **4.3 Results and Discussion**

#### **4.3.1 Biomass Characterization**

The biomass feedstocks used in this study exhibit significant differences in composition. Component analysis was performed using thermogravimetric analysis (TGA), which identifies the major components of PP and PW biomass but may not account for minor or undetected components (Table 4-1). The proximate analysis method was used to determine the chemical composition of biochar samples by measuring moisture content, volatile matters and ash content, as these factors can influence the behaviour of the material during pyrolysis and biochar production (Table 4-2). As shown in Table 4-1, the TGA derived composition of PP includes 15% hemicellulose, 35.85% cellulose, and only 0.15% lignin, together accounting for approximately 51% of the total biomass. The remaining 49% likely comprises other components such as starch and carbohydrates, which are abundant in potato peel that may not be clearly distinguishable from cellulose in TGA analysis (Călin et al., 2024; Branca and Di Blasi, 2023). Therefore, the TGA based data reflects only the major lignocellulosic components, and further analysis would be required to fully characterize the remaining biomass composition.

**Table 4- 1.** Chemical composition of potato peel (PP) and palm waste (PW) derived from thermogravimetric analysis (TGA), showing the percentage of hemicellulose, cellulose, and lignin by weight (wt. %).

Component	Hemicellulose (wt. %)	Cellulose (wt. %)	Lignin (wt. %)
PP	15	35.85	0.15
PW	31.60	65.24	3.44

**Table 4- 2.** Proximate analysis of potato peel (PP) and palm waste (PW), showing moisture content, volatile matter, and ash content by weight (wt. %). Values are averages  $\pm$  standard errors, n=3.

Proximate Analysis Parameters	Moisture Content (wt. %)	Volatile Matter (wt. %)	Ash Content (wt. %)
PP	8.90 $\pm$ 0.11	86.12 $\pm$ 0.78	8.45 $\pm$ 0.10
PW	3.98 $\pm$ 0.05	87.57 $\pm$ 0.81	5.48 $\pm$ 0.08

## Decomposition Stages of Biomass

The TGA curves reveal that there are four phases of weight loss:

### Moisture Loss

The first weight loss is attributed to the moisture loss from the biomass. For PP, this stage happened at the higher temperature of 57 °C to 143 °C, but in the case of PW, the range was lower between 45 °C and 102 °C (Figure 4-2). This moisture loss corresponds with the results obtained by (Alias et al. 2014) which stated that, moisture evaporation in biomass is usually in the range of 30 °C to 150 °C.

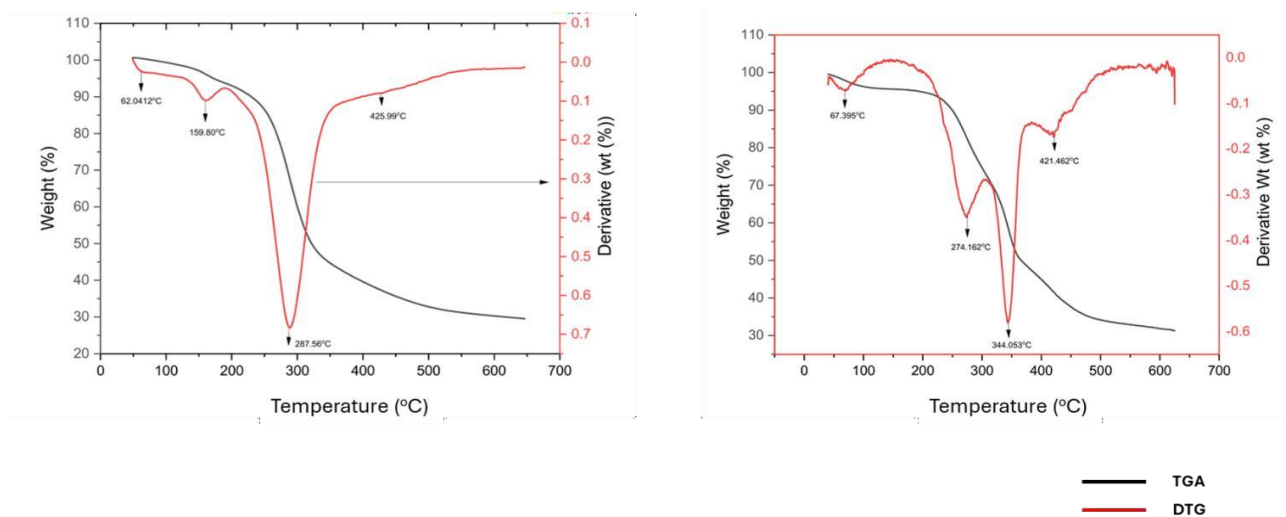
### Devolatilization of Hemicellulose, Cellulose, and lignin

The second and the third phase of weight loss is associated with the breakdown of hemicellulose and cellulose respectively, which typically begins immediately after the moisture loss phase. Decomposition of hemicellulose occurs between 180 °C to 310 °C, while cellulose decomposition occurs between 320 °C to 410 °C (Alias et al. 2014). For PP, weight loss occurred between 188 °C – 346 °C, with a total mass loss of approximately 49 %. For PW, weight loss starts at 172 °C and ends at 346 °C, with a total mass loss of around 45 % (Figure

4-2). The variation in weight loss can be explained due to the presence of high proportion of volatile compounds or other thermally labile organics in PP compared to PW. Lignin, the most resistant component of biomass feedstock to decomposition, undergoes gradual degradation within the temperature range of 380 °C to 700 °C (Alias et al. 2014). The third stage of decomposition, involving the devolatilisation of lignin, occurs at the temperature peaks 426 °C for PP and 421 °C for PW.

### TGA Results and Implications for Pyrolysis

Considering these TGA results for PP and PW, to achieve biochar formation along with release of most of the volatile matter present in the biochar as liquid and gaseous products, temperature range for pyrolysis can be chosen from 280 to 430 °C for PP and 340 to 420 °C for PW (Figure 4-2). According to the study by Budarin et al. (2010) compared to conventional pyrolysis, the biochar is produced at temperatures 100 °C lower in microwave pyrolysis. This suggests that based on the above data, for microwave reactors the temperature range for pyrolysis can be chosen from 180 to 330 °C for PP and 240 to 320 °C and PW.

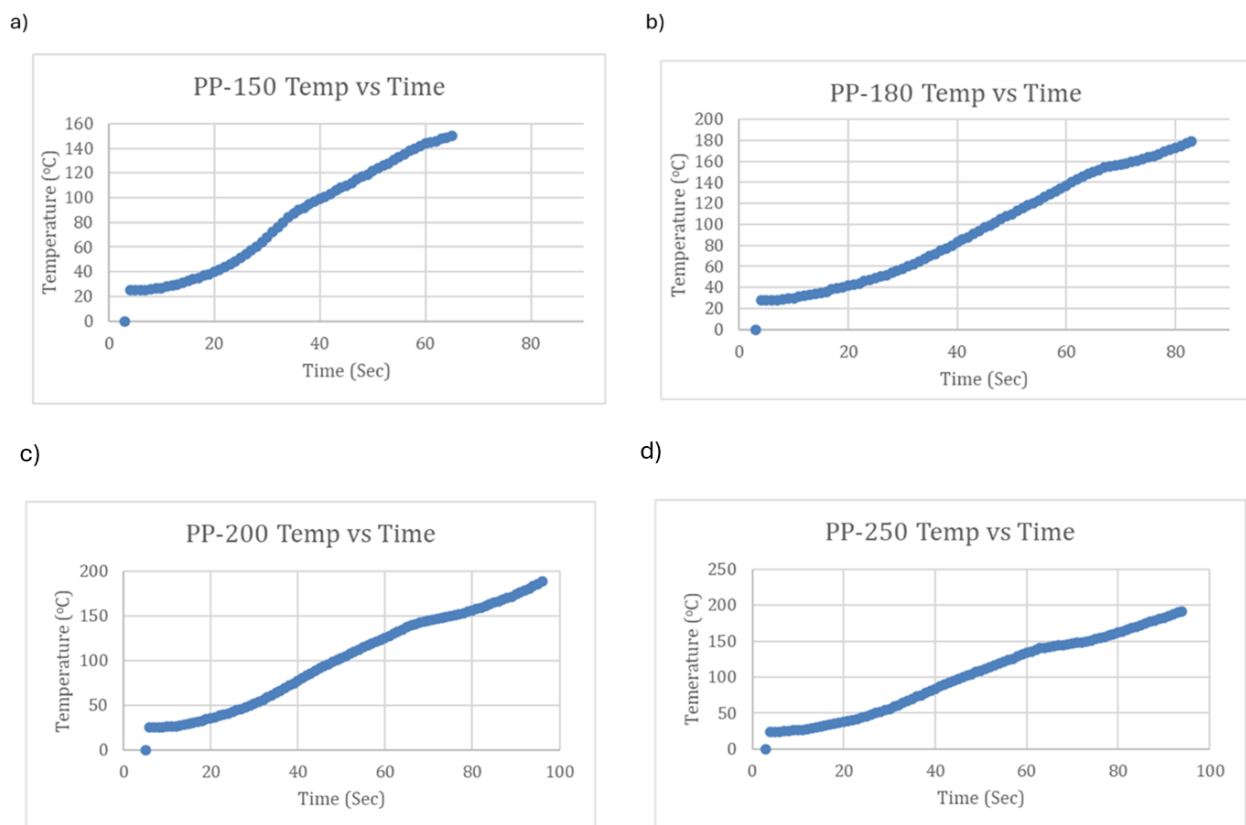


**Figure 4- 2.** TG and DTG graph of a) potato peel and b) palm leaves. The thermogravimetric (TG) curves (black line, left y-axis) represent the weight loss (%) of the biomass samples as a function of temperature (°C), while the derivative thermogravimetric (DTG) curves (red line, right y-axis) indicate the rate of weight loss (%) during thermal decomposition. Key decomposition temperatures are marked with arrows to highlight major thermal points including moisture loss, hemicellulose decomposition, cellulose decomposition, and lignin decomposition.

### 4.3.2 Effect of process parameters on MW-assisted pyrolysis experiments

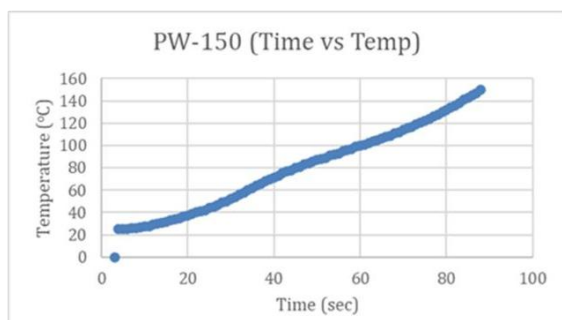
#### a) Reaction Time

During the laboratory-based experiments, the pyrolysis of PP and PW were carried out at a fixed time of 90 s, with varying temperatures and constant power at 100 W (Figure 4-3 and 4-4).

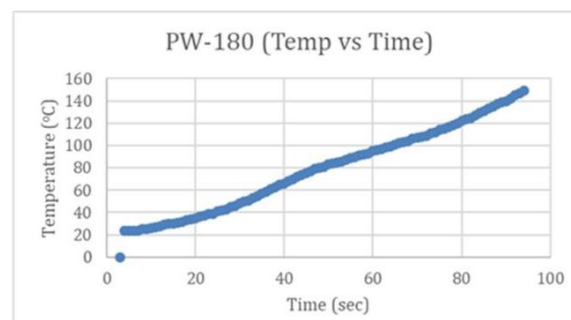


**Figure 4- 3.** Temperature-time profiles during microwave pyrolysis of potato peel (PP) at different temperature profiles: a) PP-150, b) PP-180, c) PP-200, d) PP-250. The pyrolysis was being conducted under a constant microwave power of 100 W.

a)



b)



**Figure 4- 4.** Temperature-time profiles during microwave pyrolysis of palm waste (PW) at different temperature profiles: a) PW-150 and b) PW-180. The pyrolysis was conducted under a constant microwave power of 100 W.

However, the pyrolysis process did not proceed as expected within the given 90 sec timeframes for either PP or PW (Figure. 4-3 and 4-4). Specifically, the expected temperature rise was not achieved within the given time, and the biomass would not have undergone complete decomposition. These irregularities observed during the pyrolysis experiments were likely due to the limitations of the microwave reactor (CEM Discover) and the fixed power method used. The reactor's capacity appears to have been insufficient to maintain consistent performance under specific conditions.

Reaction time is a significant factor influencing both the yield and composition of products in microwave pyrolysis experiments. Short reaction time favours higher yields due to incomplete volatilisation, as less time is available for the release of volatile compounds. In contrast, as reaction time increases, secondary reactions intensify, involving the breaking of long chain molecular bonds and the release of most volatile matter as gas. Longer reaction times lead to a decrease in biochar yield and an increase in gas production (Wang et al., 2020).

Short reaction time results in biochar with lower pH and reduced aromaticity, as the material retains much of its original biomass structure. On the other hand, longer reaction times promote the formation of highly carbonised, aromatic structures, enhancing biochar stability and adsorption capacity (Suman and Gautam, 2017; Leng and Huang, 2018).

## **b) Pressure**

The laboratory-based pyrolysis experiments were conducted using a small-scale microwave reactor with a maximum pressure limit of 300 PSI. This pressure limit restricted the pyrolysis conditions for the feedstocks. For PP, pyrolysis was carried out at temperatures ranging from 150 °C to 250 °C at a power setting of 100W. However, attempts to increase the temperature to 300 °C resulted in rapid pressure buildup, exceeding the reactor's safety limit and stopping the process. Similarly, for PW, pyrolysis was only feasible at 150 °C and 180 °C under the same power setting. Temperatures beyond 180 °C caused a significant increase in internal pressure, likely due to further decomposition of biomass components such as hemicellulose, cellulose and lignin, which release volatile gases during pyrolysis. These gases accumulate within the microwave reactor, causing a rapid rise in pressure. This excessive pressure buildup ultimately led to cracking of the sealed reaction vial, highlighting the limitation of using closed vessel reactor's for higher temperature pyrolysis. While running the reactor with an open vessel could be a potential solution, however due to safety concerns and design constraints it is not possible. Open vessel could compromise microwave absorption efficiency, increase the risk of volatile gases and lead to uncontrolled reaction conditions. Therefore, any modifications to allow high temperature pyrolysis would require pressure resistant vessels or improved gas venting equipment compatible with microwave reactor.

### **4.3.3 Impact of pyrolysis temperature on biochar yield**

The pyrolysis temperature significantly influenced biochar yields for both PP and PW. As the temperature increased, biochar yield decreased. For PP biochar, the highest yield was observed in the PP-150 treatment, which was significantly higher than the other treatments ( $p \leq 0.05$ , one-way ANOVA) (Table 4-3). Similarly, for PW biochar, a significant reduction in yield was seen in PW-180 compared to PW-150 ( $p \leq 0.05$ , t-test) (Table 4-4).



**Table 4- 3.** Yield of PP biochar in relation to different temperatures (150, 180, 200 and 250 °C) and power = 100 W. Values are averages  $\pm$  standard errors, n=3. Values within the biochar yield column with different superscripts <sup>a</sup> and <sup>b</sup> are significantly different from one another according to Tukey's test ( $p < 0.05$ ).

Sample	Sample weight (g)	Biochar yield (%)
PP - 150	1.0042	49.38 $\pm$ 5.101 <sup>a</sup>
PP - 180	1.0031	38.73 $\pm$ 2.147 <sup>b</sup>
PP - 200	1.0017	35.50 $\pm$ 0.633 <sup>b</sup>
PP - 250	1.0036	34.62 $\pm$ 1.439 <sup>b</sup>

**Table 4- 4.** Yield of PW biochar in relation to different temperatures (150, 180, 200 and 250 °C) and power = 100 W. Values are averages  $\pm$  standard errors, n=3. Values within the biochar yield column with different superscripts <sup>a</sup> and <sup>b</sup> are significantly different from one another according to t-test ( $p < 0.05$ ).

Sample	Sample weight (g)	Biochar yield (%)
PW - 150	1.0043	69.78 $\pm$ 0.809 <sup>a</sup>
PW - 180	1.0046	65.71 $\pm$ 2.532 <sup>b</sup>

These findings suggest that lower pyrolysis temperatures promote partial decomposition, allowing biomass to retain substantial amounts of its original mass in the biochar. Biochars produced at lower temperatures tends to have higher yield and lower carbon content. Conversely, as pyrolysis temperature increases, enhanced carbonisation occurs, with more organic material converted into gaseous by products, resulting in reduced biochar yield. Biochars produced at higher temperatures exhibit higher carbon content, well developed pore structures, and increased nutrient concentrations (Altıkat et al., 2024; Alperay et al., 2024; Mašek et al., 2013; Shafie et al., 2012). Both pyrolysis temperature and biomass feedstock significantly influence biochar yield (Das et al., 2021). When comparing yields at 150 °C and 180 °C, PW yielded more biochar than PP. The composition of the feedstock plays

a critical role, as variations in organic components, particularly lignin, impact biochar production. Feedstocks with higher lignin content, like PW, produce more biochar due to lignin's resistance to thermal degradation (Amutairi et al., 2023; Li et al., 2022; Stefanidis et al., 2014). TGA analysis confirmed that PW has a higher lignin content than PP (Table 4-1), which accounts for its greater biochar yield.

#### 4.3.4 Effect of pyrolysis temperature on biochar physiochemical properties

Tables 4-5 and 4-6 summarise the surface area and the pH of the PP-150, PP-180, PP-200, PP-250, PW-150, and PW-180.

**Table 4- 5.** Effect of pyrolysis temperature (150, 180, 200 and 250 °C) on the surface area and the pH of PP biochar at power = 100 W. Values are averages  $\pm$  standard errors, n=3. Values within the biochar yield column with different superscripts <sup>a</sup> and <sup>b</sup> are significantly different from one another according to Tukey's test ( $p < 0.05$ ).

Sample	BET surface area (m <sup>2</sup> /g)	Pore size (nm)	Pore volume (cm <sup>3</sup> /g)	pH
PP - 150	<2	6.504	0.00038	6.2 $\pm$ 0.05 <sup>a</sup>
PP - 180	<2	3.28	0.00275	7.0 $\pm$ 0.606 <sup>a</sup>
PP-200	<2	35.26	0.00485	6.9 $\pm$ 0.445 <sup>a</sup>
PP-250	<2	47.57	0.00189	6.5 $\pm$ 0.605 <sup>a</sup>

**Table 4- 6.** Effect of pyrolysis temperature (150 and 180 °C) on the surface area and the pH of PW biochar at power = 100 W. Values are averages  $\pm$  standard errors, n=3. Values within the biochar yield column with different superscripts <sup>a</sup> and <sup>b</sup> are significantly different superscripts a and b are significantly different from one another according to t-test ( $p < 0.05$ ).

Sample	BET surface area (m <sup>2</sup> /g)	Pore size (nm)	Pore volume (cm <sup>3</sup> /g)	pH
PW-150	<2	6.49	0.00047	6.4 $\pm$ 0.12 <sup>a</sup>
PW-180	<2	33.19	0.00072	7.1 $\pm$ 0.208 <sup>a</sup>

### **c) Surface area of biochar**

The BET surface area, average pore size, and pore volume of PP and PW chars produced at different at different pyrolysis temperatures are summarized in Table 4-5 and 4-6. All samples exhibited very low surface areas ( $<2 \text{ m}^2/\text{g}$ ), which is attributed to the low temperature microwave pyrolysis conditions used. Previous studies have shown that pyrolysis temperatures strongly influence biochar porosity and surface area. For example, Zhang et al. (2013) reported an increase in surface area from  $24 \text{ m}^2/\text{g}$  to  $32 \text{ m}^2/\text{g}$  as the pyrolysis temperature increase from  $350$  to  $700^\circ\text{C}$ . Similarly, Clarke et al. (2024), Supriya et al. (2021), Zhang et al. (2022) observed enhanced surface area with increasing pyrolysis temperature. This trend is due to the extensive decomposition of cellulose, hemicellulose, and lignin at higher temperatures, which releases volatile compounds and leaves behind more carbonized, porous biochar structure (Syguła et al., 2024; Deng et al., 2016; Leng et al., 2021). Conversely, at lower temperatures, incomplete devolatilisation can lead to pore blockage by residual volatiles, limiting the development of surface area (Clarke et al., 2024; Leng et al., 2021). Therefore, the low BET surface areas observed in this study likely result from incomplete carbonization and poor pore development under low temperature pyrolysis.

Despite the low surface area, pore size and pore volume varied with pyrolysis temperature. Although some samples exhibited relatively larger pore volumes (Table 4-5 and 4-6), these wide mesopores contribute less to total surface area compared to higher number of micropores, which have higher surface to volume ratio. Therefore, larger pores can increase volume without significantly increasing surface area (Sparavigna, 2023, Fang et al., 2010 and Leng et al., 2021)

The biomass feedstock significantly influences the surface area and porosity of biochar. Feedstocks with higher lignin content and low ash content produce biochar with higher surface area (Leng et al., 2021; Lu and Gu, 2022; Tomczyk et al., 2020; Li et al., 2022). PW exhibited higher lignin content (Table 4-1), and lower ash content (Table 4-2) compared to PP. Lignin, being rich in aromatic compounds, enhances carbonisation during pyrolysis, leading to the formation of a highly interconnected, microporous structure, which increases surface area (Lu and Gu, 2022). The higher ash content observed in PP biomass, as compared to PW biomass (Table 4-2), likely inhibited surface area development, as the minerals present

can fill or block pores during pyrolysis (Tomczyk et al., 2020; Li et al., 2022). Although, all biochar samples were washed with acetone to remove surface bound residual bio-oil and tars, the biochars produced at low temperature still exhibited significantly lower surface area compared to those produced at higher temperatures. This is primarily because lower temperature pyrolysis result in incomplete decomposition of biomass components, leading to limited pore development, and a less porous structure (Edeh et al., 2023). The most pore development occurs between 350-450°C, with porosity increasing up to 550°C (Edeh et al., 2023). Furthermore, some of these volatiles may be trapped internally that are more difficult to remove with solvent (Sygula et al., 2024). Therefore, even after washing, the limitations of low temperature pyrolysis such as poor devolatilisation and incomplete carbonization are the dominant factors that result in low surface area.

#### **d) pH of biochar**

There was no significant effect of temperature on the pH of PP ( $p > 0.05$ , one-way ANOVA) (Table 4-5) and PW biochar ( $p > 0.05$ , t-test) (Table 4-6). The pH derived was mostly on the neutral/slightly acidic side. The pH of the biochar could be determined by the presence of salts, particularly the carbonates and chlorides of K and Ca, which are produced during pyrolysis and form part of the ash residue (Tomczyk et al., 2020; Luo et al., 2015). In the case of PP and PW biochar's pyrolysis at 150°C (PP-150: 2.24 % and PW-150: 2.27 %) and 180°C (PP-180: 3.97 % and PW-180: 3.69%), the low ash content suggests that there was a minimal formation of these salts. Although the concentration of alkaline compounds in the biochar was not measured in this experiment, its pH of the biochar at these temperatures is likely neutral or slightly acidic, as fewer alkaline compounds would be present to raise the pH. The slightly acidic nature of the low temperature biochar is primarily attributed to the presence of oxygen containing functional groups, which partially degrade at lower temperatures (Wu et al., 2022, Tusar et al., 2023). Additionally, the limited thermal decomposition at lower temperatures reduced the release and the concentration of alkaine elements such as K, Ca, Mg oxides and carbonates, that are also typically responsible for the alkaline nature of the biochar pH at higher temperatures (Tomczyk et al., 2020; Pariyar et al., 2020).

## 4.4 Conclusion

The chapter explored the transformation of PP and PW into biochar using laboratory-scale microwave reactor. Through detailed experimental procedures it was observed that pyrolysis parameters, such as temperature significantly influenced the yield and characteristics of the resulting biochar. The results demonstrated that higher pyrolysis temperatures promote carbonisation and porosity while reducing biochar yield, owing to greater volatile matter release. Conversely, lower temperatures favour higher yields with less porous structures.

The study highlighted the role of feedstock composition, particularly lignin content, in determining biochar yield and surface area. The high lignin and low ash content in the PW feedstock observed a higher yield and high surface area compared to PP feedstock. The comparison between PP and PW feedstocks highlights the importance of selecting suitable biomass for specific biochar applications. For example, biochar derived from PW exhibited a relatively higher surface area (Table 7-1). The higher surface area of PW biochar can enhance soil nutrient and water retention or act as effective adsorbent in environmental applications (Section 7).

While the findings provided insights into optimising microwave pyrolysis conditions, challenges such as pressure limitations and incomplete reactions at fixed power emphasise the need for further investigation. Future research should focus on scaling up the process, exploring alternative reactor designs, and improving operational parameters to enhance char yield, and energy efficiency and product consistency.

## **Chapter 5: Wheat-pot experiments using potato peel biochar produced using large-scale microwave pyrolysis: Results**

### **5.1 Introduction**

This chapter presents a detailed analysis and characterisation of biochars derived from potato peels using microwave pyrolysis and their influence of the physiochemical properties of soil and subsequent effects on plant growth. Two types of biochar were produced: microwave partially charred biochar (PCB) and pure biochar (PB) (Section 3.3).

Initially, the chapter focuses on the characterization of the biochar's, examining their elemental composition, structure, hydrophobic properties, and thermal stability. The chapter further explores the impact of control (C) and biochar treatments (PCB and PB) on soil leachate dynamics over time. Various analyses are conducted to evaluate how these biochar treatments affect soil nutrient dynamics, dissolved organic carbon, cation exchange capacity, pH levels and plant growth.

### **5.2 Biochar characterisation**

Table 5-1 summarizes the elemental composition, including carbon and nitrogen contents, pH, cationic exchange capacity (CEC), nutrient concentration, surfacer area and hydrophobicity/hydrophilicity of partially charred biochar (PCB) and pure biochar (PB) char samples.

**Table 5- 1.** Properties of char samples: PCB (partially charred biochar) and PB (pure biochar). Data are presented as the mean  $\pm$  standard error, n=3. Values across each column with different superscripts <sup>a</sup> and <sup>b</sup> were significantly different from one another from on according to t-test analysis.

Parameters	PCB	PB
Carbon (%)	46.6 $\pm$ 0.32 <sup>a</sup>	56.2 $\pm$ 1.99 <sup>b</sup>
Nitrogen (%)	2.70 $\pm$ 0.58 <sup>a</sup>	3.26 $\pm$ 0.50 <sup>b</sup>
pH	4.87 $\pm$ 0.06 <sup>a</sup>	8.24 $\pm$ 0.01 <sup>b</sup>
CEC (cmol/kg)	118.3 $\pm$ 30.75 <sup>a</sup>	184.3 $\pm$ 13.06 <sup>b</sup>
Concentration of potassium on biochar exchange sites (mol/kg)	0.191 $\pm$ 0.089 <sup>a</sup>	0.593 $\pm$ 0.089 <sup>b</sup>
Concentration of magnesium on biochar exchange sites (mol/kg)	0.272 $\pm$ 0.089 <sup>a</sup>	0.501 $\pm$ 0.089 <sup>b</sup>
Concentration of calcium on biochar exchange sites (mol/kg)	1.49 $\pm$ 0.089 <sup>a</sup>	2.21 $\pm$ 0.089 <sup>b</sup>
Concentration of iron on biochar exchange sites (mol/kg)	0.004 $\pm$ 0.089 <sup>a</sup>	0.004 $\pm$ 0.089 <sup>a</sup>
Concentration of zinc on biochar exchange sites(mol/kg)	0.079 $\pm$ 0.089 <sup>a</sup>	0.093 $\pm$ 0.089 <sup>a</sup>
Phosphate (mol/kg)	0.012 $\pm$ 0.089 <sup>a</sup>	0.046 $\pm$ 0.089 <sup>a</sup>
BET surface area (m <sup>2</sup> /g)	2	11
WDPT (s)	$\leq$ 1	1-60
Category of hydrophobicity	Slightly hydrophobic	Hydrophillic

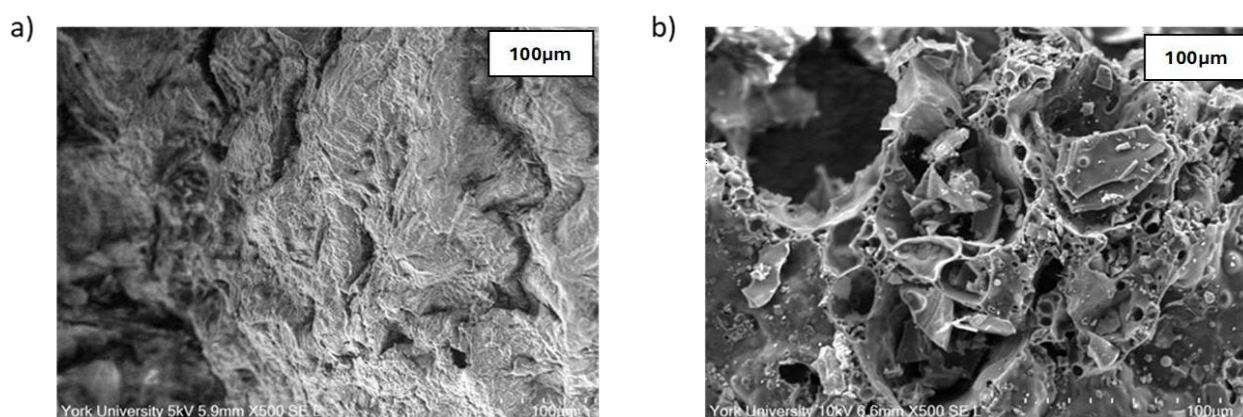
The carbon and nitrogen percentages were significantly higher in the PB sample compared to the PCB sample ( $p \leq 0.05$ , t-test analysis) (Table 5-1).

The pH and CEC were observed to be significantly higher in the PB sample compared to the PCB sample ( $p \leq 0.05$ , t-test analysis) (Table 5-1).

When examining the concentration of elemental ions presenting on the exchange sites between the biochar samples, the PB sample exhibited significantly higher concentrations of potassium, calcium and magnesium present on the exchange sites compared to the PCB sample ( $p \leq 0.05$ , t-test analysis) (Table 5-1). No significant differences were observed between the PCB and PB samples for the concentration of other elemental ions on the exchange sites ( $p > 0.05$ , t-test analysis).

A hydrophobicity test was conducted using the Water Drop Penetration Time (WDPT) method on the PCB and PB char samples (Section 3-3c). The results indicated that the PCB sample was slightly hydrophobic in nature (Table 5-1), suggesting that when added to soil, it could repel water, leading to an increase in leachate. In contrast, the PB sample were hydrophilic, indicating their potential to improve water retention in the soil (Table 5-1).

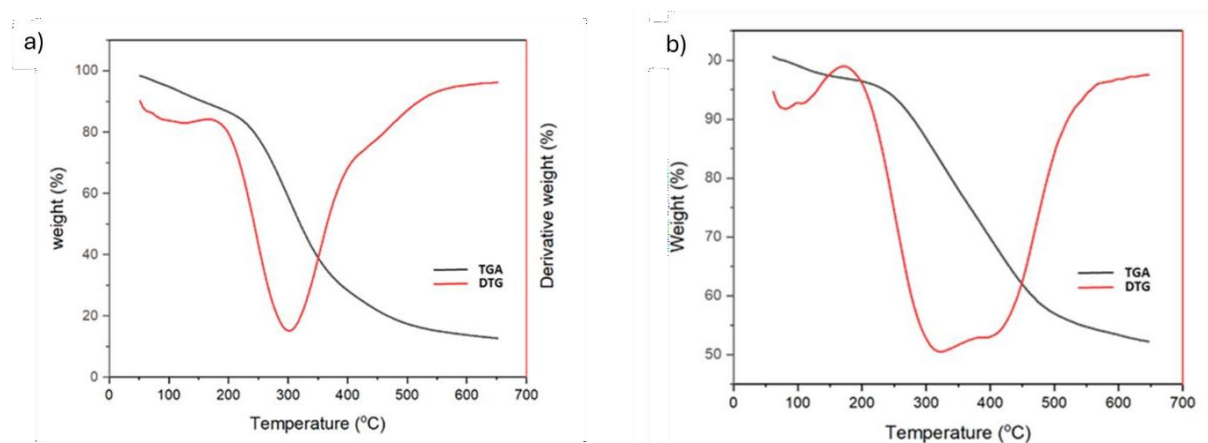
SEM images of PCB and PB illustrated the surface morphology of biochar's produced by microwave pyrolysis (Figure 5-1a and 5-1b). The SEM image of PCB depict a rough granular structure, with less defined and uniform pores compared to PB structure (Figure 5-1a). The PB structure revealed a tubular and well-defined pore structure, with presence of micro porosity (Figure 5.1b) compared to PCB structure. (Figure 5-1a).



**Figure 5- 1.** SEM images at x500 magnification of a) PCB (partially charred biochar) and b) PB (pure biochar).



Thermogravimetric analysis (TGA) provided an insight into the thermal degradation characteristics of these biochar (Figure 5-2a and 5-2b). The DTG curve for both PCB and PB exhibits a sharp peak at 300 °C, indicating the primary phase of decomposition, where the maximum weight loss occurred (Figure 5-2a and 5-2b). This mass loss is likely due to the devolatilisation and combustion of volatiles and biochar (Yi et al., 2012). The major weight loss observed at 300 °C was found to be lower in PB char (36.67 %) compared to PCB char (68.19 %) (Figure 5.2), suggesting the presence of stable residues in PB compared to PCB (Das et al., 2018). The significant mass loss observed, particularly in PCB, indicates that a large proportion of volatile compounds remained in the partially charred biochar during the production process. This suggests the PCB underwent incomplete devolatilisation and lower carbonization. As a result, PCB has lower thermal stability, fixed carbon content and also reduces its effectiveness in long term soil applications. In contrast, the lower mass in PB reflects a more complete thermal decomposition during production, resulting in biochar that is richer in stable carbon structures, more thermally resistant, and likely to have longer soil persistence. Thus the mass loss patterns provide valuable insights on the quality and stability of the resulting biochars.

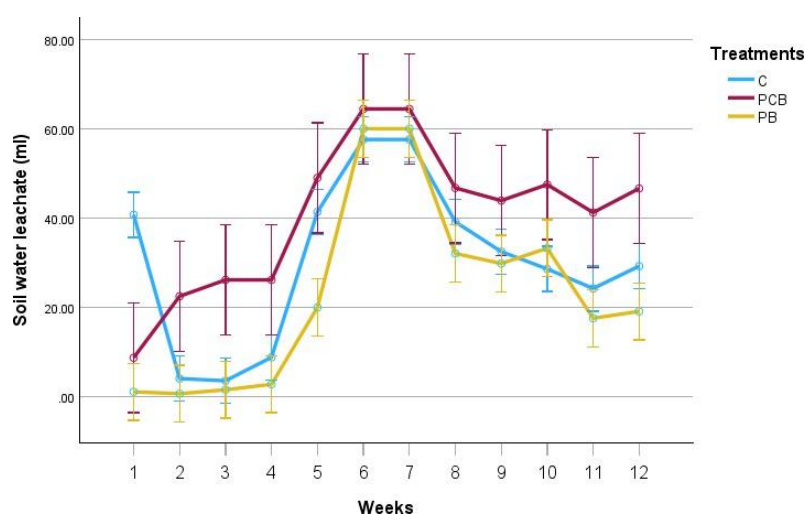


**Figure 5- 2.** TG and DTG graph of a) partially charred biochar (PCB) and b) pure biochar (PB) The thermogravimetric (TG) curves (black line, left y-axis) represent the weight loss (%) of the biomass samples as a function of temperature (°C), while the derivative thermogravimetric (DTG) curves (red line, right y-axis) indicate the rate of weight loss (%) during thermal decomposition.

### 5.3 Effect of biochar treatments on soil leachate

This section reports how different char treatments influenced soil leachate over time. After a detailed description of the trends, there is a summary of the key points at the end of this section.

A significant influence of biochar treatments, incubation periods and their interaction on soil leachate volume was observed ( $p \leq 0.001$ , Repeated measures two-way ANOVA) (Figure 5-3).



**Figure 5- 3.** Average weekly volume (ml) of soil leachate from control (C), partially char biochar (PCB), and pure biochar (PB) treated soils. Values are averages  $\pm$  standard errors,  $n=4$ . The bars in the graph represent standard errors.

In week 1, the volume of soil leachate was significantly higher in the C compared to both PCB and PB treatments ( $p \leq 0.05$ , Bonferroni test), with no significant difference between PCB and PB treatments ( $p > 0.05$ , Bonferroni test). During weeks 2, 3, 4 and 11, the PCB treatment resulted in significantly greater soil leachate compared to PB and C ( $p \leq 0.05$ , Bonferroni test), with no significant differences observed between the PB treatment and C ( $p > 0.05$ , Bonferroni test). In week 5, soil leachate was significantly lower in the PB treatment compared to PCB and C ( $p \leq 0.05$ , Bonferroni test), with no significant difference between PCB and C ( $p > 0.05$ , Bonferroni test). From week 6 to 9, no significant differences in soil leachate were observed between the treatments ( $p > 0.05$ , Bonferroni test). In week 10, soil leachate was significantly greater in the PCB treatment compared to C ( $p \leq 0.05$ , Bonferroni test), with no

other significant difference among treatments ( $p > 0.05$ , Bonferroni test). By week 12, the PCB treatment exhibited the highest soil leachate volume, while the PB treatment exhibited the lowest ( $p \leq 0.05$ , Bonferroni test) (Figure 5-3).

In addition to the treatments, the incubation period significantly influenced soil leachate volume ( $p \leq 0.001$ , Repeated measures two-way ANOVA). For C, soil leachate was significantly higher in week 1 compared to weeks 2 to 4 and week 11 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) but lower than in weeks 6 and 7 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant differences were observed between weeks 1, 5, 8, 9, 10 and 12. Similarly, no significant differences were observed between weeks 2 to 4 ( $p > 0.05$ , Repeated measures two-way ANOVA), which had significantly lower leachate volumes compared to weeks 5 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). During weeks 6 and 7, no significant differences were observed between these two weeks ( $p > 0.05$ , Repeated measures two-way ANOVA), however, the leachate volume was significantly higher than those in all other weeks ( $p \leq 0.05$ , Repeated measures two-way ANOVA). A significant decrease was observed from weeks 7 to 8 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), followed by no significant differences observed until week 10 ( $p > 0.05$ , Repeated measures two-way ANOVA). A significant decrease occurred in week 11 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with leachate volumes remaining constant through week 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 5-3).

For the PCB treatment, no significant difference was observed in leachate volume between weeks 1 to 4 ( $p > 0.05$ , Repeated measures two-way ANOVA). However, leachate was significantly lower in week 1 compared to weeks 5 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). The leachate volume in week 2 to 4 was significantly lower compared to weeks 6 and 7 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant differences observed between weeks 8 to 12 and weeks 2 to 4 ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 5, leachate volume was significantly higher than in week 1 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), but no significant difference was observed between week 5 and remaining weeks ( $p \leq 0.05$ , Repeated measures two-way ANOVA). Leachate volume was significantly higher in weeks 6 and 7 compared to weeks 1 to 4 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant differences observed between weeks 6 and 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 5-3).

For the PB treatment, no significant difference was observed in leachate volumes between weeks 1 to 4 ( $p > 0.05$ , Repeated measures two-way ANOVA). However, the leachate volume was significantly lower than those collected in week 5 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). A significant increase in leachate volume was observed from week 5 to week 6 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with volumes remaining through week 7 ( $p > 0.05$ , Repeated measures two-way ANOVA). From week 8 onward, a significant decrease was observed compared to week 7 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant differences observed between weeks 9 to 12 compared to week 8 ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 10, leachate volume was significantly higher than in week 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant difference observed compared to week 9 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant differences were observed between weeks 9, 11 and 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 5-3).

Summarising the general trends observed in soil water leachate volume from weeks 1 to 12:

- a) The soil leachate was generally significantly greater in PCB and lower in PB treated soils ( $p \leq 0.05$ , Bonferroni test).
- b) For all treatments, the soil leachate volume was low in the initial weeks (1 to 4), increased after week 4 to a plateau in weeks 6 and 7 and then gradually decreased to the end of the experiment.

## 5.4 Effect of biochar treatments on nutrient concentration in soil leachate

In this section the concentrations of primary macro-nutrients (nitrogen, phosphorus, potassium), secondary plant macro-nutrients (calcium and magnesium) and micronutrients (iron and zinc) in the soil leachates over time are presented.

Among the nutrients measured, the leaching behaviour of calcium, magnesium and phosphate was consistent across the treatments, whereas potassium and nitrate showed different patterns across the treatments (Figure 5-4 and 5-5). For iron and zinc leachates, most of the leachate concentrations were below detection level ( $BDL \leq 0.5 \text{ mg/L}$ ) (Figure 5- 4d and 5-4e). The incubation period during plant growth also influenced nutrient leaching. In PCB and

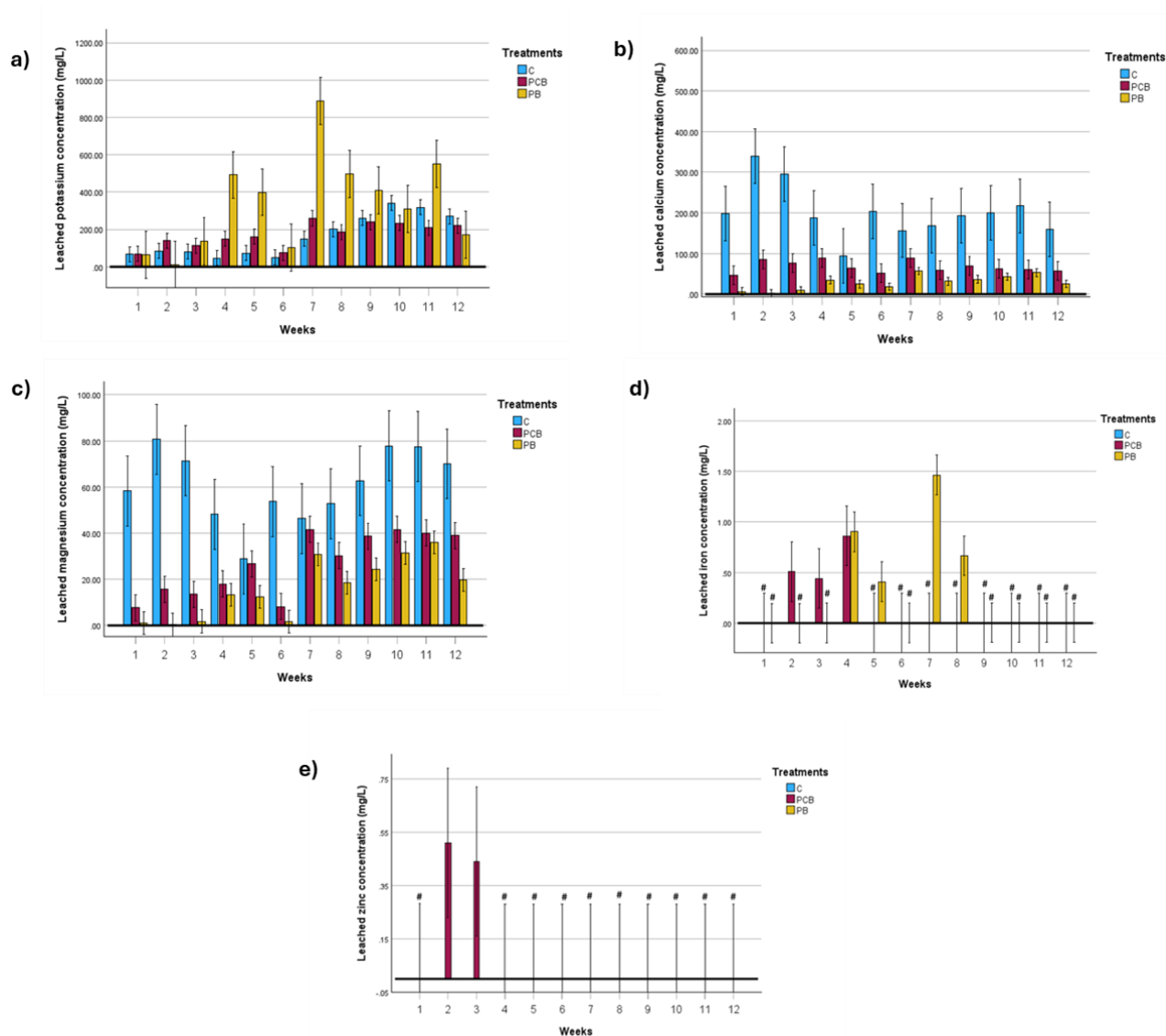
PB treatments, the concentration of leached calcium, magnesium, phosphate and nitrate decreased by weeks 5 or 6 compared to weeks 1 to 4, followed by an increase in week 7 (Figure 5-4b, 5-4c, 5-5a and 5-5b). Whereas, leached potassium in PB and PCB varied across the incubation period (Figure 5-4a). In C, the concentration of leached magnesium and phosphate remained relatively stable throughout the incubation period (Figure 5-4c and 5-

5a), while, for leached potassium, calcium and nitrate varied differently throughout the incubation period (Figure 5-4a, 5-4b and 5-5b). The variations in nutrient concentrations due to the treatments and incubation period are discussed in more detail in the following sections.

After a detailed description of the trends, there is a summary of the key points at the end of each section.

#### **5.4.1 Leached potassium concentration**

There was a significant difference in leached potassium concentration across biochar treatments, incubation times, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA) (Figure 5-4a).



**Figure 5- 4.** Average weekly concentration of leached cations a) potassium, b) calcium, c) magnesium, d) iron and e) zinc in control (C), partially charred biochar (PCB) and pure biochar (PB) treated soils. Values are averages  $\pm$  standard errors,  $n=4$ . The bars in the graph represent standard errors. The # along the error bars for d) iron and e) zinc are below detection level (BDL < 0.5 mg/L) are expressed as the detection limit divided by the square root of two (Croghan & Egeghy, 2003).

Between weeks 1 to 3, 6 and 12, no significant differences were found among the treatments ( $p > 0.05$ , Bonferroni test). In weeks 4, 5 and 7, potassium concentration was significantly higher in PB treated soil and lower in the C ( $p \leq 0.05$ , Bonferroni test). During weeks 8 and 9, potassium concentration was significantly higher in PB treated soil compared to both C and PCB treated soil ( $p \leq 0.05$ , Bonferroni test), with no significant differences observed between C and PCB treated soil ( $p > 0.05$ , Bonferroni test). In week 10, leached potassium was significantly higher in C compared to PCB ( $p \leq 0.05$ , Bonferroni test), with no significant differences observed between C and PB, and between PCB and PB treatments ( $p > 0.05$ , Bonferroni test). In week 11, potassium concentration was significantly higher in PB treated soil and lower in PCB treated soil ( $p \leq 0.05$ , Bonferroni test). (Figure 5-4a).

The incubation time also significantly influenced the potassium concentration in the leachate ( $p \leq 0.001$ , Repeated measures two-way ANOVA). For C, no significant difference in leached potassium was observed from weeks 1 to 6 ( $p > 0.05$ , Repeated measures two-way ANOVA). The leached potassium in leachate during weeks 1 to 6 was significantly lower compared to those in weeks 8 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 7, leached potassium was significantly higher than week 4 and 6 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and significantly lower than in weeks 9 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no other significant differences among the other weeks ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed between weeks 8,9 and 12 ( $p > 0.05$ , Repeated measures two-way ANOVA), although potassium concentration in week 8 was significantly lower than in weeks 10 and 11 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed from weeks 9 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 5-4a).

For the PCB treatment, no significant difference in leached potassium concentration was observed from weeks 1 to 6 ( $p > 0.05$ , Repeated measures two-way ANOVA). Potassium leaching in weeks 1, 2 and 6 was significantly lower than in weeks 7 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 3, leached potassium was significantly lower than in weeks 7, 9, 10 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant differences compared to weeks 8 and 11 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant differences were observed between week 4 and 5 compared to the rest of the weeks ( $p > 0.05$ , Repeated measures two-way ANOVA), except for significantly lower levels in

week 7 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). A significant increase was observed in week 7 compared to weeks 1 to 6 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with levels remaining constant till week 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 5-4a).

For the PB treatment, no significant difference in leached potassium was observed between weeks 1 to 3 ( $p > 0.05$ , Repeated measures two-way ANOVA), but the levels were significantly lower than those in weeks 4, 5, 7, 8, 9 and 11 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). Leached potassium was also significantly higher in week 4 compared to weeks 6 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), and significantly lower than in week 7 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No other significant differences were observed between the other weeks compared to week 4 ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 5, leached potassium was significantly higher than week 6, and significantly lower than week 7. In weeks 6, leached potassium was also significantly lower than weeks 7, 8, 9 and 11 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 7, leached potassium was significantly higher compared to the other weeks ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed between week 8 to 11 ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 11, leached potassium was significantly lower compared to week 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) (Figure 5-4a).

Summarising the general trends observed in leached potassium concentration from weeks 1 to 12:

- a) The concentration of leached potassium was found to be significantly greater in PB treatment compared to PCB and C ( $p \leq 0.05$ , Bonferroni test) with no significant difference between PCB and C ( $p > 0.05$ , Bonferroni test).
- b) During the initial watering period of wheat crop (weeks 1 to 4) using de-ionised water: Leached potassium remained constant across all treatments from weeks 1 to 4, except for the PB treatment, which observed an increase in week 4.
- c) From week 5 to 12 with the introduction of the Hoagland solution:
  - For C and PCB treatment, leached potassium concentrations increased during weeks 7 to 12.
  - For PB treatment, leached potassium increased during week 5 to 11 with few fluctuations and then decreased in week 12.



### 5.4.2 Leached calcium concentration

There was a significant difference in leached calcium concentration across biochar treatments, incubation times, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA). During weeks 1 to 4, leached calcium was significantly higher in C, and lower in PB treated soil ( $p \leq 0.05$ , Bonferroni test). In week 5, leached calcium was significantly higher in PCB treated soil compared to PB ( $p \leq 0.05$ , Bonferroni test), with no other significant differences between the treatments ( $p > 0.05$ , Bonferroni test). In weeks 6, 9 and 12, leached calcium remained significantly higher in C, and lower in PB treated soil ( $p \leq 0.05$ , Bonferroni test). In week 7, the leached calcium was significantly lower in PB treated soil compared to both PCB and C ( $p \leq 0.05$ , Bonferroni test), with no significant difference between PCB and C ( $p > 0.05$ , Bonferroni test). During weeks 8, 10 and 11, leached calcium was significantly greater in C compared to both PCB and PB treatments ( $p \leq 0.05$ , Bonferroni test), with no significant difference observed between PCB and PB treated soils ( $p > 0.05$ , Bonferroni test) (Figure 5-4b).

In addition to the treatments, the incubation period significantly influenced the calcium concentration within each treatment ( $p \leq 0.001$ , Repeated measures two-way ANOVA). For C, leached calcium in week 2 was significantly higher compared to weeks 4, 5, 7, 8, 9 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), and in week 3, leached calcium was significantly higher compared to week 5 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No other significant differences were observed between the week for the concentration of leached calcium ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 5-4b).

For the PCB treatment, no significant difference in leached calcium concentration was observed from week 1 to week 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 5-4b).

For the PB treatment, no significant difference was observed in leached calcium concentrations from weeks 1 to 3 ( $p > 0.05$ , Repeated measures two-way ANOVA), which was significantly lower compared to week 4 and weeks 7 to 11 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 4, no significant difference was observed from weeks 5 to 12 ( $p > 0.05$ ,

Repeated measures two-way ANOVA). In weeks 5 and 6, leached calcium was significantly lower than in weeks 7 and 11 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no other significant differences among the remaining weeks ( $p > 0.05$ , Repeated measures two-way ANOVA). Leached calcium increased significantly in week 7 compared to week 6 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and remained constant till week 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 5-4b).

Summarising the general trends observed in leached calcium concentration from weeks 1 to 12:

- a) The concentration of leached calcium was significantly greater in C, and lower in PB treated soil ( $p \leq 0.05$ , Bonferroni test).
- b) The concentration of leached calcium remained constant throughout the incubation period (weeks 1 to 12) in the PCB treatment.
- c) During the initial watering period of wheat crop (weeks 1 to 4) using de-ionised water:
  - In C, leached calcium increased from week 1 to week 2, then decreased in week 4.
  - In PB treatment, leached calcium was lower in the initial weeks (week 1 to 3) and increased in week 4
- d) From week 5 to 12 with the introduction of the Hoagland solution:
  - In C, leached calcium further decreased in week 5, increased again in week 6 and then remained constant until week 12.
  - In PB treatment, leached calcium remained constant from week 4 onwards through week 12.

### 5.4.3 Leached magnesium concentration

There was a significant difference in leached magnesium concentration across biochar treatments, incubation times, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA). During weeks 1, 4, 6 and 11, leached magnesium was significantly higher in the C compared to both PCB and PB treated soils ( $p \leq 0.05$ , Bonferroni test), with no significant differences between PCB and PB treated soils ( $p > 0.05$ , Bonferroni test). In weeks 2 to 3, weeks 8 to 10 and week 12, leached magnesium was significantly greater in the C, and lower in PB treated soils ( $p \leq 0.05$ , Bonferroni test). In weeks 5 and 7, leached magnesium was significantly lower in PB treated soil compared to PCB treated soil ( $p \leq 0.05$ , Bonferroni test), with no other significant differences between other treatments ( $p > 0.05$ , Bonferroni test) (Figure 5-4c).

In addition to the treatments, the incubation period significantly influenced leached magnesium concentrations within the treatments ( $p \leq 0.001$ , Repeated measures two-way ANOVA). For C, leached magnesium was significantly lower in week 5 compared to weeks 2 to 3 and weeks 9 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no other significant difference between other weeks ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 5-4c).

For the PCB treatment, leached magnesium remained constant from week 1 to 4 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower compared to weeks 7 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 5, leached magnesium was significantly higher compared to weeks 1, 3 and 6 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and significantly lower than weeks 9 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No other significant differences were observed between other weeks compared to week 5 ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 6, leached magnesium was also significantly lower than in weeks 7 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant difference between weeks 1 to 4 ( $p > 0.05$ , Repeated measures two-way ANOVA). An increase in leached magnesium was observed in week 7 ( $p \leq 0.05$ , Repeated

measures two-way ANOVA) and remained constant through week 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 5-4c).

For the PB treatment, leached magnesium was constant from week 1 to 3 ( $p > 0.05$ , Repeated measures two-way ANOVA) and significantly lower compared to weeks 4, 5 and 7 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant difference compared to week 6 ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 4, leached magnesium was also significantly lower compared to weeks 7, 9, 10 and 11 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and significantly higher compared to week 6 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No other significant differences were observed between week 4 and other weeks ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 5, leached magnesium was also significantly lower compared to weeks 7, 10 and 11 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no other significant differences between other weeks ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 6, leached magnesium was also significantly lower than in weeks 7 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed from weeks 7 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 5-4c).

Summarising the general trends observed in leached magnesium concentration from weeks 1 to 12:

- a) The concentration of leached magnesium was significantly greater in C, and the lowest in PB treated soil ( $p \leq 0.05$ , Bonferroni test).
- b) During the initial watering period of wheat crop (weeks 1 to 4) using de-ionised water:
  - In both C and PCB treatment, leached magnesium remained relatively constant from weeks 1 to 4.
  - In PB treatment, leached magnesium was initially lower during weeks 1 to 3 and then increased in week 4.
- c) From week 5 to 12 with the introduction of the Hoagland solution:
  - In C, a further decrease in leached magnesium was observed in week 5, followed by an increase in week 6 and remained relatively stable until week 12.

- In PCB and PB treatments, leached magnesium remained constant from weeks 4 to 5, decreased in week 6, and then increased in week 7, remaining relatively stable till week 12.

#### 5.4.4 Leached iron concentration

For C, leached iron was below detection limit ( $BDL \leq 0.5$  mg/L) from week 1 to week 12, hence, no significant difference was found between the weeks ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 5-4d).

There was a significant difference in leached iron concentration across PCB and PB treatments, incubation times, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA). During weeks 1, 6 and 9 to 12, no significant differences in leached iron concentrations were present between the treatments ( $p > 0.05$ , Bonferroni test). In weeks 2 and 3, leached iron concentrations were significantly higher in PCB treated soil compared to C and PB ( $p \leq 0.05$ , Bonferroni test) with no significant difference between C and PCB treated soils ( $p > 0.05$ , Bonferroni test). In week 4, leached iron was significantly lower in C compared to PCB and PB treated soils ( $p \leq 0.05$ , Bonferroni test), with no significant difference between PCB and PB treated soils ( $p > 0.05$ , Bonferroni test). During weeks 5, 7 and 8, leached iron concentrations were significantly higher in PB treated soil compared to C and PCB ( $p \leq 0.05$ , Bonferroni test), with no significant differences between C and PCB treated soils ( $p > 0.05$ , Bonferroni test) (Figure 5-4d).

Along with the treatments, the incubation time also significantly influenced the concentration of leached iron within the treatments ( $p \leq 0.001$ , Repeated measures two-way ANOVA). For the PCB treated soil, in week 4, leached iron was significantly higher compared to weeks 1, 5 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no other significant differences observed between weeks 2 and 3 compared to 4 ( $p > 0.05$ , Repeated measures two-way ANOVA). For weeks 1 and 5 to 12 leached iron was BDL hence, no significant differences were observed between these weeks ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 5-4d). For the PB treated soil, the leached iron was significantly higher in week 7 compared to the rest of the weeks ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 4, leached iron

was significantly higher compared to weeks 1 to 3, 5, 6 and 9 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), and no significant difference was observed in week 8 compared to week 4 ( $p > 0.05$ , Repeated measures two-way ANOVA). For weeks 1 to 3, 6 and 9 to 12, the leached iron was BDL hence, no other significant differences were observed between these weeks ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 5-4d).

Summarising the general trends observed in leached iron concentration from weeks 1 to 12:

- e) The leached iron concentration was BDL in C from week 1 to 12.
- f) The concentration of leached iron was significantly lower in C compared to PCB and PB treatments, with no significant differences between PCB and PB treatments ( $p \leq 0.05$ , Repeated measures two-way ANOVA).
- g) During the initial watering period of wheat crop (weeks 1 to 4) using de-ionised water:
  - In PCB, leached iron was BDL in weeks 1 and increase in weeks 2 to 4.
  - In PB, leached iron was BDL from weeks 1 to 3, followed by an increase in week 4.
- h) From week 5 to 12 with the introduction of the Hoagland solution:
  - In PCB, leached iron decreased to BDL from week 5 onwards till week 12.
  - In PB, leached iron decreased from week 4 to 6, followed by an increased in week 7, and further decrease was followed till week 12.

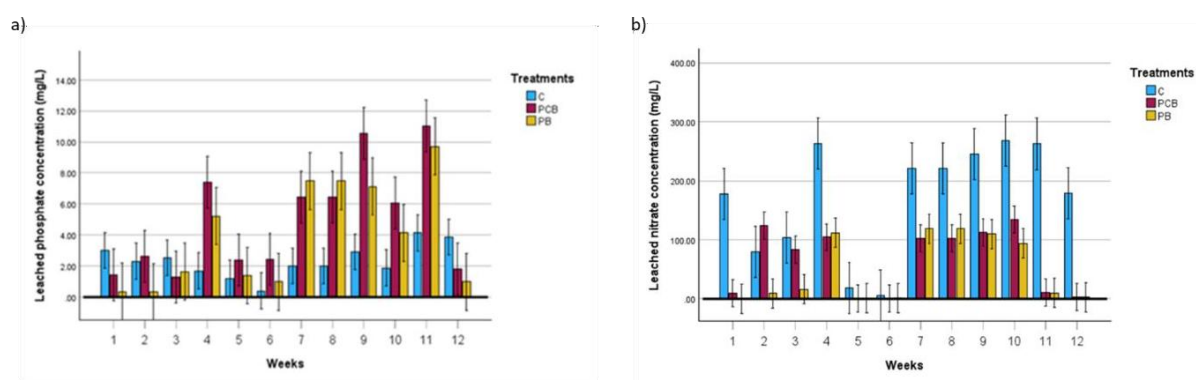
#### 5.4.5 Leached zinc concentration

A repeated measures ANOVA indicated significant differences between biochar treatments ( $p \leq 0.001$ , Repeated measures two-way ANOVA). The incubation time did not have a significant effect on the concentration of the leached zinc in all the treatments ( $p > 0.05$ , Repeated measures two-way ANOVA), as most of the leached zinc in the treatments from week 1 to week 12 have been detected as below detection level ( $BDL \leq 0.5 \text{ mg/L}$ ) (Figure 5- 4e). However, there was a significant influence of treatments on leached zinc ( $p \leq 0.001$ , Repeated measures two-way ANOVA). In weeks 2 and 3, the leached zinc was greater in PCB treated soil compared to PCB and C ( $p \leq 0.05$ , Bonferroni test), with no significant difference between PCB and C ( $p > 0.05$ , Bonferroni test). No other significant differences were observed

between the treatments in week 1 and from weeks 4 to 12 ( $p > 0.05$ , Bonferroni test) (Figure 5-4e).

#### 5.4.6 Leached phosphate concentration

There was a significant difference in leached phosphate concentration across biochar treatments, incubation times, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA) (Figure 5-5a).



**Figure 5- 5.** Average weekly concentration of leached anions a) phosphate and b) nitrate in control (C), partially charred biochar (PCB) and pure biochar (PB) treated soils. Values are averages  $\pm$  standard errors,  $n=4$ . The bars in the graph represents standard errors.

In weeks 1 and 12, leached phosphate concentrations were significantly lower in PB treated soil compared to C ( $p \leq 0.05$ , Bonferroni test), with no significant differences between other treatments ( $p > 0.05$ , Bonferroni test). During weeks 2 to 3 and 5 to 6, no significant differences in leached phosphate concentrations were found among the treatments ( $p > 0.05$ , Bonferroni test). In weeks 4, 7, 8 and 11, leached phosphate was significantly lower in C compared to PCB and PB ( $p \leq 0.05$ , Bonferroni test), with no significant differences between PCB and PB treated soils ( $p > 0.05$ , Bonferroni test). In week 9, leached phosphate was significantly greater in PCB treated soil, and lower in C ( $p \leq 0.05$ , Bonferroni test). In week 10, leached phosphate was significantly greater in PCB treated soil compared to C ( $p \leq 0.05$ , Bonferroni test), with no other significant differences observed between other treatments ( $p > 0.05$ , Bonferroni test) (Figure 5-5a).

Incubation time significantly influenced leached phosphate concentrations ( $p \leq 0.001$ , Repeated measures two-way ANOVA). For C, leached phosphate was significantly lower in week 6 compared to weeks 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant differences observed between other weeks compared to week 6 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 5-5a).

For PCB treated soil, no significant differences were found between week 1 and 3 ( $p > 0.05$ , Repeated measures two-way ANOVA), and leached phosphate was significantly lower in weeks 1 and 3 compared to weeks 4 and 7 to 10 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 2, leached phosphate was significantly lower compared to weeks 4, 7 to 9 and 11 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no other significant differences observed between the remaining weeks ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 4, leached phosphate was significantly higher compared to weeks 5, 6 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant differences from weeks 7 to 11 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant differences were found between weeks 5 and 6 ( $p > 0.05$ , Repeated measures two-way ANOVA), which were significantly lower compared to weeks 7 to 11 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In weeks 7 and 8, leached phosphate was also significantly lower compared to weeks 9 and 11 and significantly higher compared to weeks 10 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant differences between weeks 7 and 8 ( $p > 0.05$ , Repeated measures two-way ANOVA). Leached phosphate was also significantly higher in week 9 compared to week 10 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant differences between weeks 9 and 11 ( $p > 0.05$ , Repeated measures two-way ANOVA). Leached phosphate was also significantly higher in week 11 compared to weeks 10 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) (Figure 5-5a).

For PB treated soil, leached phosphate concentration remained constant from weeks 1 to 3 ( $p > 0.05$ , Repeated measures two-way ANOVA) but was significantly lower compared to weeks 7 to 11 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). Leached phosphate was significantly higher in week 4 compared to weeks 1, 2, 5, 6 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), and significantly lower compared to week 11 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No other significant difference was observed in leached phosphate in weeks 3, 4, 7 to 10 compared to week 4 ( $p > 0.05$ , Repeated measures two-way



ANOVA). For weeks 5 and 6, leached phosphate was significantly lower than in weeks 7 to 9 and 11 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no other significant differences between other weeks ( $p > 0.05$ , Repeated measures two-way ANOVA). Weeks 7 and 8 had significantly higher leached phosphate concentrations compared to week 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 10, leached phosphate was significantly lower than in week 11 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant differences compared to other weeks ( $p > 0.05$ , Repeated measures two-way ANOVA). Leached phosphate significantly decreased in week 12 compared to week 11 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) (Figure 5-5a).

Summarising the general trends observed in leached phosphate concentration from weeks 1 to 12:

- a) The concentration of leached phosphate was significantly lower in C compared to PCB and PB ( $p \leq 0.05$ , Bonferroni test), with no significant differences between PCB and PB ( $p > 0.05$ , Bonferroni test).
- b) During the initial watering period of wheat crop (weeks 1 to 4) using de-ionised water:
  - In C, leached phosphate remained relatively constant from weeks 1 to 4.
  - In PCB and PB treatments, leached phosphate was low in weeks 1 to 3 and increased in week 4.
- c) From week 5 to 12 with the introduction of the Hoagland solution:
  - In C, leached phosphate decreased in week 6, followed by an increase in week 7 and then remained relatively constant till week 12.
  - In PCB and PB treatments, leached phosphate was lower in weeks 5 and 6, increased in week 7, remained constant till week 11, and then decreased further in week 12.

#### 5.4.7 Leached nitrate concentration

There was a significant difference in leached phosphate concentration across biochar treatments, incubation times, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA). In weeks 1, 4, 7 to 9 and 11 to 12, leached nitrate concentration was significantly higher in C compared to PCB and PB treatments ( $p \leq 0.05$ , Bonferroni test), with no significant differences between PCB and PB treated soils ( $p > 0.05$ , Bonferroni test). In weeks 2 and 3, leached nitrate was significantly lower in PB treated soil compared to PCB and C ( $p \leq 0.05$ , Bonferroni test), with no significant differences between PCB and C ( $p > 0.05$ , Bonferroni test). No significant differences were observed between treatments in weeks 5 and 6 ( $p > 0.05$ , Bonferroni test). In week 10, leached nitrate was significantly higher in C, and lower in PB ( $p \leq 0.05$ , Bonferroni test) (Figure 5-5b).

Incubation time also significantly influenced leached nitrate concentration ( $p \leq 0.001$ , Repeated measures two-way ANOVA). For C, leached nitrate was significantly higher in week 1 compared to weeks 2, 5 and 6 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no other significant differences observed other weeks ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant differences were found between weeks 2 and 3 ( $p > 0.05$ , Repeated measures two-way ANOVA), which had significantly lower leached nitrate compared to weeks 4 and 7 to 11 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). Weeks 5, 6, and 12 had no significant differences compared to weeks 2 and 3 ( $p > 0.05$ , Repeated measures two-way ANOVA). Leached nitrate was significantly higher in week 4 compared to weeks 5 and 6 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant difference from weeks 7 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant difference was found between weeks 5 and 6 ( $p > 0.05$ , Repeated measures two-way ANOVA), which were also significantly lower than weeks 7 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant differences were observed from weeks 7 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 5-5b).

For PCB treatment, leached nitrate was significantly lower in week 1 compared to weeks 2 to 4 and 7 to 10 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no other significant differences between weeks 5, 6, 11 and 12 ( $p > 0.05$ , Repeated measures two-way ANOVA).

From weeks 2 to 4, leached nitrate was also significantly higher than in weeks 5, 6, 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no other significant difference between weeks ( $p > 0.05$ , Repeated measures two-way ANOVA). Leached nitrate was significantly lower in weeks 5 and 6 compared to weeks 7 to 10 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant differences between weeks 1, 11 and 12 ( $p > 0.05$ , Repeated measures two-way ANOVA). Leached nitrate significantly increased in week 7 compared to week 6 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and remained constant until week 10 ( $p > 0.05$ , Repeated measures two-way ANOVA). The leached nitrated decreased in week 11 compared to week 10 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and remained constant until week 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 5-5b).

For PB treated soil, no significant difference was observed in the concentration of leached nitrate in weeks 1 to 3 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower than weeks 4 and 7 to 10 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant differences were observed between weeks 5, 6, 11 and 12 ( $p > 0.05$ , Repeated measures two-way ANOVA). Leached nitrate was also significantly higher in week 4 compared to weeks 5, 6, 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no other significant difference between weeks ( $p > 0.05$ , Repeated measures two-way ANOVA). In weeks 5 and 6, leached nitrate was also significantly lower compared to weeks 7 to 10 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant differences between weeks 1 to 3 and 11 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant differences were observed between weeks 7 to 10 ( $p > 0.05$ , Repeated measures two-way ANOVA), with significantly higher concentrations in these weeks compared to weeks 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) (Figure 5-5b).

Summarising the general trends observed in leached nitrate concentration from weeks 1 to 12:

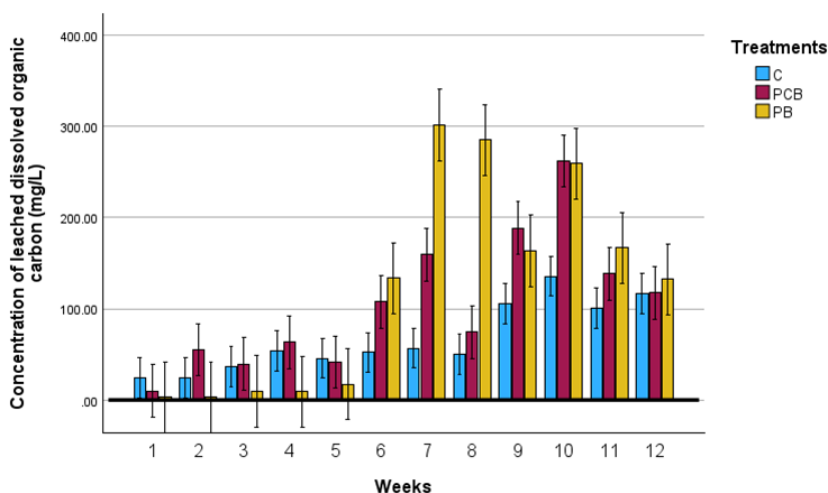
- i) The concentration of leached nitrate was significantly greater in C, and the lowest in PB treatment ( $p \leq 0.05$ , Bonferroni test).
- j) During the initial watering period of wheat crop (weeks 1 to 4) using de-ionised water:
  - o In C, leached nitrate was high in week 1, decreased in weeks 2 and 3, and increased again in week 4

- In PCB treatment, leached nitrated was low in week 1 but increased in weeks 2 to 4.
  - In PB treatment, leached nitrate remained low throughout weeks 1 to 3 and increased in week 4.
- k) From week 5 -12 with the introduction of the Hoagland solution:
- In C, leached nitrate decreased in weeks 5 and 6, followed by an increase in week 7, and then remained constant till week 12.
  - In PCB and PB treatments, leached nitrate decreased in weeks 5 and 6, increased in week 7, remained constant till week 10, and then decreased further in weeks 11 and 12.

## **5.5 Effect of biochar treatments on dissolved organic carbon concentration in soil leachate**

This section explores how different char treatments affect the concentration of dissolved organic carbon (DOC) in the leachate. After a detailed description of the trends, there is a summary of the key points at the end of this section.

There was a significant difference in leached DOC across biochar treatments, incubation times, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA) (Figure 5-6).



**Figure 5- 6.** Average weekly concentration of leached dissolved organic carbon (DOC) in the control (C), partially charred (PCB) and pure biochar (PB) treated soils. Values are averages  $\pm$  standard errors,  $n=4$ . The bars in the graph represents standard errors.

During weeks 1 to 5 and weeks 12, no significant differences were observed in leached DOC concentrations among the treatments ( $p > 0.05$ , Bonferroni test). However, in weeks 6, 9 and 10, leached DOC was significantly lower in C compared to PCB and PB treatments ( $p \leq 0.05$ , Bonferroni test), with no significant difference between PCB and PB treatments ( $p > 0.05$ , Bonferroni test). In weeks 7 and 8, leached DOC was significantly higher in PB compared to PCB and C ( $p \leq 0.05$ , Bonferroni test). Specifically, in week 7, leached DOC was significantly lower in C compared to PCB treatment, while in week 8, no significant difference was observed between C and PCB treatment ( $p > 0.05$ , Bonferroni test). In week 11, leached DOC was significantly lower in C compared to PB ( $p \leq 0.05$ , Bonferroni test), with no other significant difference between other treatments ( $p > 0.05$ , Bonferroni test) (Figure 5-6).

Incubation time also had a significant effect on leached DOC concentration ( $p \leq 0.001$ , Repeated measures two-way ANOVA). For C, no significant difference in DOC concentration was observed from weeks 1 to 8 ( $p > 0.05$ , Repeated measures two-way ANOVA). However, leached DOC was significantly lower in weeks 1 to 8 compared to weeks 9 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant differences observed between weeks 9 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 5-6).

For PCB treatment, no significant difference in DOC concentration was observed from weeks 1 to 5 ( $p > 0.05$ , Repeated measures two-way ANOVA). These weeks had significantly lower DOC concentration compared to weeks 9, 10, 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 6, leached DOC was significantly higher compared to weeks 1 and 5 and significantly lower than weeks 9 and 10 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No other significant differences were observed between other weeks compared to week 6 ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 7, leached DOC was significantly higher than in weeks 1 to 5 and 8 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), and significantly lower than in week 10 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 8, DOC was also significantly lower than in weeks 9 and 10 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no other significant differences between weeks 1 to 6 and 11 compared to week 8 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant differences were observed in DOC concentrations in weeks 7, 10, 11 and 12 compared to week 9 ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 10, leached DOC was also significantly higher than weeks 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed of leached DOC between weeks 11 and 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 5-6).

For PB treatment, no significant difference in DOC concentration was observed from weeks 1 to 5 ( $p > 0.05$ , Repeated measures two-way ANOVA), and these weeks had significantly lower DOC concentrations compared to weeks 6 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 6, leached DOC was significantly lower than in weeks 7, 8 and 10 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant difference between weeks 9, 11 and 12 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed between weeks 7, 8 and 10 ( $p > 0.05$ , Repeated measures two-way ANOVA) and these weeks had significantly higher leached DOC compared to weeks 9, 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant differences were observed in DOC concentrations in weeks 11 and 12 compared to week 9 ( $p > 0.05$ , Repeated measures two-way ANOVA), and no significant differences were observed between week 11 and 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 5-6).

Summarising the general trends observed in leached DOC concentration from weeks 1 to 12:

- a) The concentration of leached DOC was significantly greater in PB and PCB treatment compared to C from weeks 6 to 12.
- b) For C, the leached DOC was constant from week 1 to 8 and increase in week 9 and remained constant till week 12.
- c) For PB and PCB, the leached DOC was low and stable from weeks 1 to 5 and increased significantly from week 6 to week 7. From week 7 onwards the leached DOC fluctuated till week 10 and then observed a significant decrease in week 11. Leached DOC remained constant from week 11 to 12.

## **5.6 Effect of biochar treatments on soil physical structure and chemistry at the end of 12-week experiment period**

Addition of different biochar to the soil can affect soil porosity, soil CEC, pH, and nutrient concentration in the soil exchange sites. This section examines how different char treatments impact physical structure and chemical properties of soil.

### **5.6.1 Effect of biochar treatments on soil porosity**

No significant difference was found in the soil porosity between the C, PCB and the PB treatments ( $p > 0.05$ , Kruskal-Wallis test) (Table 5-2).

**Table 5- 2.** Physical and chemical soil properties for control (C), partially charred biochar (PCB) and pure biochar (PB) treatments. Data are presented as mean  $\pm$  standard error (n=3). Values across each column denoted by different superscripts <sup>a</sup>, <sup>b</sup> and <sup>c</sup> were found to be significantly different from one another according to Tukey's test ( $p \leq 0.05$ , one-way ANOVA).

Properties	C soil	PCB treated soil	PB treated soil
Soil porosity ( $\text{cm}^3 \text{cm}^{-3}$ )	$0.86 \pm 0.011^a$	$0.86 \pm 0.001^a$	$0.86 \pm 0.002^a$
Soil nitrate (mol/kg)	$1.87 \times 10^{-5} \pm 6.5 \times 10^{-6}^a$	$3.66 \times 10^{-5} \pm 1.2 \times 10^{-5}^b$	$9.84 \times 10^{-5} \pm 4.8 \times 10^{-5}^c$
Soil ammonium (mol/kg)	$0.0025 \pm 0.00006^a$	$0.0004 \pm 0.0002^a$	$0.0008 \pm 0.0004^a$
Soil CEC (cmol/kg)	$15.57 \pm 27.59^a$	$17.16 \pm 14.4^a$	$26.89 \pm 27.93^b$
Soil pH	$8.31 \pm 0.28^a$	$7.28 \pm 0.06^a$	$7.90 \pm 0.08^a$
Concentration of potassium on soil exchange sites (mol/kg)	$0.012 \pm 0.0001^a$	$0.18 \pm 0.17^b$	$0.22 \pm 0.07^b$
Concentration of calcium on soil exchange sites (mol/kg)	$0.17 \pm 0.003^a$	$0.17 \pm 0.004^a$	$0.27 \pm 0.52^a$
Concentration of magnesium on soil exchange sites (mol/kg)	$0.004 \pm 0.001^a$	$0.003 \pm 0.002^a$	$0.008 \pm 0.002^a$
Concentration of iron on soil exchange sites (mol/kg)	$0.0001 \pm 0.00005^a$	$0.0001 \pm 0.00005^a$	$0.0001 \pm 0.00008^a$
Concentration of zinc on soil exchange sites (mol/kg)	$0.0011 \pm 0.0002^a$	$0.0009 \pm 0.0004^a$	$0.0009 \pm 0.0003^a$
Concentration of phosphate on soil exchange sites (mol/kg)	$0.0001 \pm 0.00001^a$	$0.0001 \pm 0.00001^a$	$0.0002 \pm 0.00012^a$



### **5.6.2 Effect of biochar treatments on nitrate and ammonium concentrations on soil exchange sites**

The PB treated soil had significantly greater soil nitrate and the lowest in C ( $p \leq 0.05$ , Kruskal-Wallis test) (Table 5-2). No significant difference was observed between the treatments for soil ammonium ( $p > 0.05$ , one-way ANOVA) (Table 5-2). The concentration of the ammonium and nitrate in the soil were positively correlated ( $n = 12$ ,  $r = 0.697$ ,  $p \leq 0.05$ , Pearson correlation analysis) implying that when the concentration of ammonium goes up, nitrate levels also tend to go up and vice-versa.

### **5.6.3 Effect of biochar treatments on soil cationic exchange capacity (CEC) and pH**

The soil CEC was found to be significantly greater in PB treated soil compared to C and PCB treated soil ( $p \leq 0.05$ , one-way ANOVA), with no significant difference between C and PCB treated soil ( $p > 0.05$ , one-way ANOVA) (Table 2, one-way ANOVA) (Table 5-2).

At the end of the experiment, all the soils have a near neutral pH (7 to 8) with no significant difference between them ( $p > 0.05$ , Kruskal-Wallis test) (Table 5-2).

The correlation between pH and CEC was not statistically significant ( $n = 12$ ,  $r = -0.158$ ,  $p > 0.05$ , Pearson correlation analysis), indicating that there is no significant relationship between the CEC and pH of the soils across the treatments.

### **5.6.4 Effect of biochar treatments on soil exchangeable cations**

The concentration of potassium on exchange sites was found to be significantly lower in C compared to PCB and PB treated soils ( $p \leq 0.05$ , Kruskal-Wallis test), with no significant difference between PCB and PB treated soils ( $p > 0.05$ , Kruskal-Wallis test). No other significant differences were observed in the concentration of calcium, magnesium, zinc ( $p >$

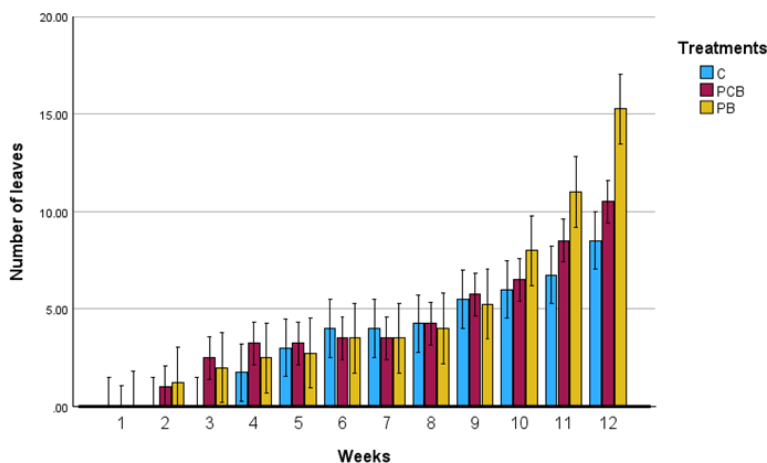
0.05, Kruskal-Wallis test), iron and phosphate ( $p > 0.05$ , one-way ANOVA) on exchange sites in the soil between the treatments (Table 5-2).

Within each treated soil, a significant difference was observed between the concentrations of the elements on exchange sites in the soil ( $p \leq 0.05$ , one-way ANOVA). For C, the concentration of potassium on exchange sites was significantly lower compared to the concentration of calcium on exchange sites in the soil ( $p \leq 0.05$ , one-way ANOVA), and the concentration of potassium and calcium on exchange sites in the soil were significantly greater than magnesium, iron, zinc, and phosphate ( $p \leq 0.05$ , one-way ANOVA). No significant differences were observed between the concentrations of magnesium, iron, zinc, and phosphate on exchange sites in the soil ( $p > 0.05$ , one-way ANOVA). For PCB treated soil, the concentration of potassium and calcium was significantly greater than magnesium, iron, zinc, and phosphate on exchange sites in the soil ( $p > 0.05$ , one-way ANOVA). For PB treated soil, no significant difference was observed between the concentration of potassium and calcium on exchange sites in the soil ( $p > 0.05$ , one-way ANOVA). However, the concentration of potassium and calcium on exchange sites in the soils were significantly greater than the concentration of magnesium, iron, zinc, and phosphate ( $p \leq 0.05$ , one-way ANOVA).

## **5.7 Effect of biochar treatments on leaf number and biomass yield**

This section investigates the impact of different char treatments on plant growth, specifically focusing on the number of leaves and biomass yield. After a detailed description of the trends, there is a summary of the key points at the end of this section.

There was a significant difference in the number of leaves across biochar treatments, incubation times, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA) (Figure 5-7).



**Figure 5- 7.** Average number of leaves on wheat plants in the control (C), partially charred biochar (PCB) and pure biochar (PB) treated soils. Values are averages  $\pm$  standard errors,  $n=4$ . The bars in the graph represents standard errors.

From weeks 1 to 10, there was no significant differences in the number of leaves between the treatments ( $p > 0.05$ , Bonferroni test). However, in week 11, the PB-treated soil had a significantly higher number of leaves compared to C ( $p \leq 0.05$ , Bonferroni test) through no significant differences were found between PB and PCB treated soil and between PCB and C ( $p > 0.05$ , Bonferroni test). By week 12, the number of leaves in the PB treated soil was significantly greater in both PCB and C ( $p \leq 0.05$ , Bonferroni test), with no significant difference between PCB and C ( $p > 0.05$ , Bonferroni test) (Figure 5-7).

Incubation time also significantly affected the number of leaves ( $p \leq 0.001$ , Repeated measures two-way ANOVA). For C, no significant differences were observed in the number of leaves from week 1 to 3 ( $p > 0.05$ , Repeated measures two-way ANOVA), but the number of leaves was significantly lower compared to weeks 6 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). Weeks 4 and 5 did not significantly differ from weeks 1 to 3 ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 4, the number of leaves were significantly lower than weeks 9 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant differences in leaf number between weeks 5 to 9 compared to week 4 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant differences were observed between week 5 to 8 ( $p > 0.05$ , Repeated measures two-way ANOVA), and these weeks had significantly fewer leaves than

week 4 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). From weeks 9 to 12, no significant differences were found ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 5-7).

For PCB treated soil, the number of leaves was significantly lower in week 1 compared to weeks 4 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant differences between weeks 2 and 3 compared to week 1 ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 2, the number of leaves was significantly lower than in weeks 8 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant difference between weeks 3 to 7 ( $p > 0.05$ , Repeated measures two-way ANOVA). Week 3 had significantly fewer leaves than weeks 9 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant difference between weeks 4 to 8 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant differences in leaf number were observed between weeks 4 to 7 ( $p > 0.05$ , Repeated measures two-way ANOVA), although these weeks had significantly fewer leaves than weeks 10 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 8, the number of leaves was significantly lower than in weeks 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant difference between weeks 3 to 7, 9 and 10 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed between weeks 9 and 10 ( $p > 0.05$ , Repeated measures two-way ANOVA), but week 12 had significantly more leaves ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed between weeks 11 and 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 5-7).

For PB treated soil, no significant differences were observed in the number of leaves between weeks 1 to 8 ( $p > 0.05$ , Repeated measures two-way ANOVA). However, these weeks had significantly fewer leaves than weeks 10 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 9, the number of leaves were significantly lower than in week 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant difference compared to week 10 ( $p > 0.05$ , Repeated measures two-way ANOVA). Weeks 10 and 11 had significantly fewer leaves than week 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant difference between weeks 10 and 11 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 5-7).

Summarising the general trends observed in the number of leaves from weeks 1 to 12:

- a) There were no significant differences in the number of leaves between the treatments from weeks 1 to 9 ( $p > 0.05$ , Bonferroni test). However, from week 10 onward, a significant increase in leaf number was observed in the PB treatment ( $p \leq 0.05$ , Bonferroni test).
- b) During the initial watering period of wheat crop (weeks 1 to 4) using de-ionised water:
  - In C, the number of leaves was lower in weeks 1 to 3 and increased in week 4.
  - In PCB treatment, the number of leaves was low in week 1 and gradually increased in week 4.
  - In PB treatment, the number of leaves remained constant from weeks 1 to 4.
- c) From week 5 -12 with the introduction of the Hoagland solution:
  - In C, the number of leaves increased in week 5, remained constant till week 8, with a significant increase till week 12.
  - In PCB and PB treatments, the number of leaves remained constant from weeks 5 to 8 and significantly increased till week 12.

The above ground biomass was significantly higher in PB compared to C and PCB treated soil ( $p \leq 0.05$ , One-way ANOVA) and no significant difference was observed in the above ground biomass between PCB and PB treated soils. The below ground biomass was significantly lower in C compared to PCB and PB treated soils ( $p \leq 0.05$ , One-way ANOVA) and no significant difference was found between PCB and PB treatments ( $p > 0.05$ , Repeated measures two-way ANOVA) (Table 5-3).

**Table 5- 3.** Average aboveground and belowground biomass after the plant-pot experiment for control (C), partially charred biochar (PCB) and pure biochar (PB) treatments. Data are the mean  $\pm$  standard deviation (n=4). Values within each column denoted by different superscripts <sup>a, b</sup> were found to be significantly different from one another according to Tukey's test ( $p < 0.05$ , one-way ANOVA).

Sample	Above ground biomass (g)	Below ground biomass (g)
C	2.44 $\pm$ 0.58 <sup>a</sup>	0.39 $\pm$ 0.19 <sup>a</sup>
PCB	1.41 $\pm$ 0.36 <sup>a</sup>	2.69 $\pm$ 0.85 <sup>b</sup>
PB	3.37 $\pm$ 0.08 <sup>b</sup>	2.99 $\pm$ 0.26 <sup>b</sup>

## 5.8 Effect of biochar treatments on the nutrient concentration at exchange sites in wheat biomass

Biochars are known to influence soil nutrient availability, which in turn can impact the nutrient concentration in the crops. This section explores how different char treatments affects the nutrient concentration on the exchange sites in wheat biomass. The concentration of potassium and calcium on the exchange sites of the wheat biomass was significantly higher in PB treated soil compared to PCB and C ( $p \leq 0.05$ , one-way ANOVA), with no significant difference between PCB and C ( $p > 0.05$ , one-way ANOVA). No significant difference was observed in the concentration of magnesium and iron, phosphorus ( $p > 0.05$ , one-way ANOVA) and zinc ( $p > 0.05$ , Kruskal-Wallis test) on the exchange sites of wheat biomass (Table 5-4).

**Table 5- 4.** Average nutrient concentration in wheat biomass after the plant-pot experiment for control (C), partially charred biochar (PCB) and pure biochar (PB) treatments. Data are the mean values of 4 replicates. Values across each column denoted by different superscripts <sup>a, b</sup> were found to be significantly different from one another according to Tukey's test ( $p < 0.05$ , one-way ANOVA).

Properties	C soil	PCB treated soil	PB treated soil
Potassium (mol/kg)	$3.9 \times 10^{-5} \text{ }^a$	$2.5 \times 10^{-5} \text{ }^a$	$6.2 \times 10^{-5} \text{ }^b$
Calcium (mol/kg)	$2.1 \times 10^{-5} \text{ }^a$	$1.5 \times 10^{-5} \text{ }^a$	$2.9 \times 10^{-5} \text{ }^b$
Magnesium (mol/kg)	$3.1 \times 10^{-5} \text{ }^a$	$2.6 \times 10^{-5} \text{ }^a$	$3.4 \times 10^{-5} \text{ }^a$
Iron (mol/kg)	$5.9 \times 10^{-7} \text{ }^a$	$4.6 \times 10^{-7} \text{ }^a$	$6.3 \times 10^{-7} \text{ }^a$
Zinc (mol/kg)	$1.7 \times 10^{-6} \text{ }^a$	$4.6 \times 10^{-6} \text{ }^a$	$2.5 \times 10^{-6} \text{ }^a$
Phosphorus (mol/kg)	$8.1 \times 10^{-6} \text{ }^a$	$5.3 \times 10^{-6} \text{ }^a$	$8.1 \times 10^{-6} \text{ }^a$

## **Chapter 6: Wheat-pot experiments using potato peel biochar produced using conventional pyrolysis and hydrothermal carbonisation: Results**

### **6.1 Introduction**

This chapter presents a detailed analysis and characterization of chars derived from potato peels using two distinct processes: Microwave hydrochar (MWH) and conventional biochar (CB). The chapter primarily reports how these chars influence the physical and chemical properties of soil and their subsequent effects on plant growth and yield.

Initially, the chapter focuses on the characterization of the chars, examining their elemental composition, hydrophobic properties, and thermal stability. The chapter further explores the impact of control (C) and char treatments (MWH and CB) on soil leachate dynamics over time. Various analyses are conducted to assess how these treatments affect soil nutrient dynamics, dissolved organic carbon, cation exchange capacity, pH levels, and plant growth.

### **6.2 Char characterization**

Table 6-1 summarizes the elemental composition, including carbon and nitrogen contents, pH, cation exchange capacity (CEC), nutrient concentrations, surface area and hydrophobicity/hydrophilicity of microwave hydrochar (MWH) and conventional biochar (CB) char samples.



**Table 6- 1.** Properties of char samples: microwave hydrochar (MWH) and conventional biochar (CB). Values are averages  $\pm$  standard errors, n=3. Values across each column with different superscripts <sup>a</sup> and <sup>b</sup> were significantly different from one another according to t-test analysis.

Parameters	MWH	CB
Carbon (%)	47.8 $\pm$ 0.05 <sup>a</sup>	64.1 $\pm$ 0.14 <sup>b</sup>
Nitrogen (%)	3.04 $\pm$ 0.09 <sup>a</sup>	3.27 $\pm$ 0.15 <sup>b</sup>
pH	5.53 $\pm$ 0.61 <sup>a</sup>	7.34 $\pm$ 0.2 <sup>b</sup>
CEC (cmol/kg)	274.7 $\pm$ 7.86 <sup>a</sup>	301.9 $\pm$ 3.41 <sup>b</sup>
Concentration of potassium on char exchange sites (mol/kg)	0.231 $\pm$ 0.36 <sup>a</sup>	0.957 $\pm$ 0.34 <sup>b</sup>
Concentration of magnesium on char exchange sites (mol/kg)	0.004 $\pm$ 0.36 <sup>a</sup>	0.02 $\pm$ 0.34 <sup>b</sup>
Concentration of calcium on char exchange sites (mol/kg)	0.03 $\pm$ 0.36 <sup>a</sup>	0.064 $\pm$ 0.34 <sup>b</sup>
Concentration of iron on char exchange sites (mol/kg)	0.0002 $\pm$ 0.36 <sup>a</sup>	4.91 $\times 10^{-5}$ $\pm$ 0.34 <sup>a</sup>
Concentration of zinc on char exchange sites (mol/kg)	0.0003 $\pm$ 0.36 <sup>a</sup>	0.0003 $\pm$ 0.34 <sup>a</sup>
Concentration of phosphate on char exchange sites (mol/kg)	0.0002 $\pm$ 0.36 <sup>a</sup>	0.0008 $\pm$ 0.34 <sup>a</sup>
BET surface area (m <sup>2</sup> /g)	<2	8
WDPT (s)	60 - 600	$\leq 1$
Category of hydrophobicity	Strongly Hydrophobic	Hydrophilic

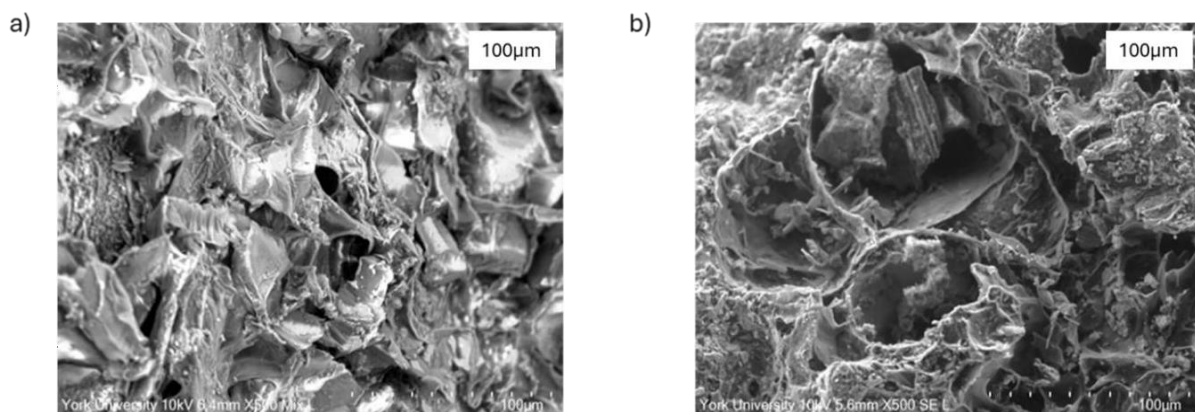
The carbon and the nitrogen percentage was significantly higher in the CB sample compared to the MWH sample ( $p \leq 0.05$ , t-test analysis) (Table 6-1).

The pH and the CEC were found to be significantly higher in CB sample compared to MWH sample ( $p \leq 0.05$ , t-test analysis) (Table 6-1).

When comparing the nutrient concentrations on the exchange sites between the char samples, potassium, magnesium, and calcium concentrations were significantly higher in the CB char compared to the MWH char sample ( $p \leq 0.05$ , t-test analysis) (Table 6-1). No significant differences were observed between CB and MWH samples for other nutrient concentrations on the exchange sites, including iron, zinc, and phosphate ( $p > 0.05$ , t-test analysis).

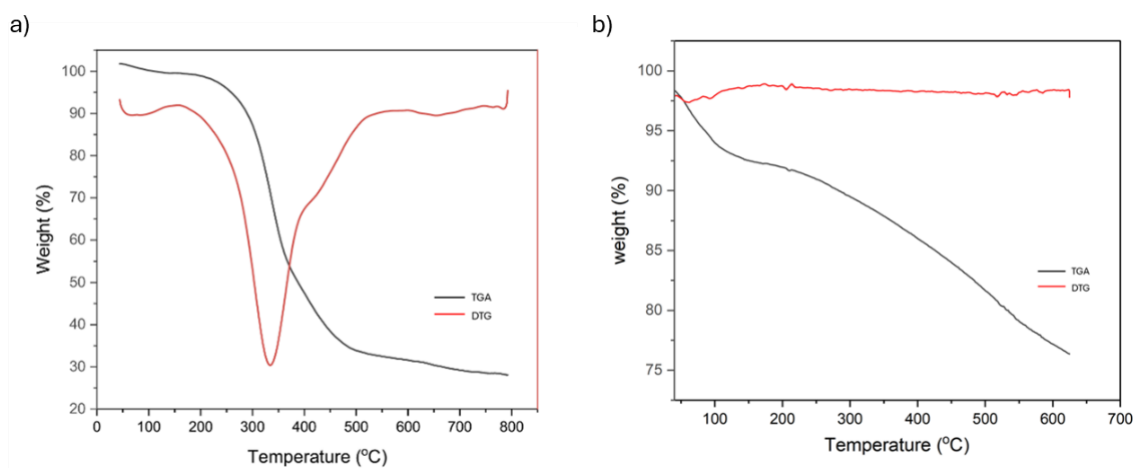
A hydrophobicity test was conducted using the Water Drop Penetration Time (WDPT) method on the MWH and CB char samples (Section 3.4c). The results indicated that the MWH sample was strongly hydrophobic in nature (Table 6-1), suggesting that when added to soil, it could repel water, leading to an increase in leachate. In contrast, the CB sample was hydrophilic, indicating its potential to improve water retention in the soil (Table 6-1).

SEM images of MWH and CB illustrated the surface morphology of chars (Figures 6-1a and 6-1b). The surface of MWH showed a similar structure to that of potato peel biomass with the presence of few pore formations (Figure 6-1a). The surface of CB showed an irregular structure compared to the potato peel biomass with the formation of well define pores on the structure (Figure 6-1b).



**Figure 6- 1.** SEM images at x500 magnification of a) microwave hydrochar (MWH) and b) Conventional biochar (CB).

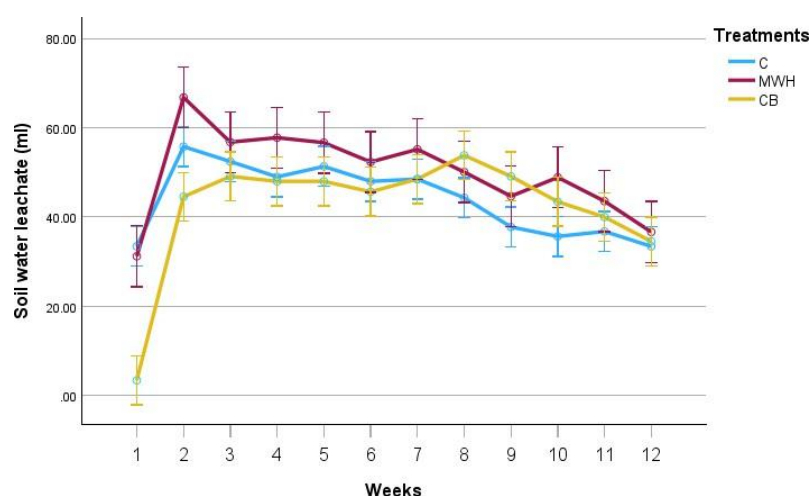
The thermal decomposition of MWH and CB were observed using TGA and DTG (Figure 6-2 and 6-2b). MWH exhibited significant mass loss in three distinct phases in the DTG curve (Figure 6-2a) (Poomsawat and Poomsawat, 2021). The first phase, occurring at 69 °C, is associated with moisture loss. The second phase, with maximum decomposition at 334 °C, is primarily due to the devolatilization of volatile matter leading to char formation. The third phase, occurring between 450 – 650 °C, is attributed to the oxidation of the carbonaceous char material (Figure 6-2a). The DTG curve in CB sample only shows minor fluctuations without any minor peaks, indicating that no rapid weight loss occurred over that temperature range (Figure 6-2b). This indicates that CB decomposes slowly and steadily, making it more thermally stable (Das et al., 2021).



**Figure 6- 2.** TG and DTG graph of a) microwave hydrochar (MWH) and b) conventional biochar (CB). The thermogravimetric (TG) curves (black line, left y-axis) represent the weight loss (%) of the biomass samples as a function of temperature (°C), while the derivative thermogravimetric (DTG) curves (red line, right y-axis) indicate the rate of weight loss (%) during thermal decomposition.

### 6.3 Effect of char treatments on soil leachate

This section reports how different char treatments influenced the volume of soil leachate over time. After a detailed description of the trends, there is a summary of the key points at the end of this section. A significant difference in the volume of soil leachate was observed due to char treatment, incubation time, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA) (Figure 6-3).



**Figure 6- 3.** Average weekly volume (ml) of water leached from control (C), microwave hydrochar (MWH) and conventional biochar (CB) treated soils. Values are averages  $\pm$  standard errors,  $n=3$ . The bars in the graph represent standard errors.

During weeks 1 and 2, soil leachate was significantly lower in CB treated soil compared to C and MWH treated soil ( $p \leq 0.05$ , Bonferroni test). In week 1, there was no significant difference between C and MWH treatments ( $p > 0.05$ , Bonferroni test), but in week 2, soil leachate was significantly lower in C compared to MWH-treated soils ( $p \leq 0.05$ , Bonferroni test). In weeks 3, 5 to 7 and 11 to 12, no significant differences were observed between the treatments ( $p > 0.05$ , Bonferroni test). In week 4, soil leachate was significantly higher in MWH treated soils compared to CB and C treatments ( $p \leq 0.05$ , Bonferroni test), with no significant difference between CB and C treatments ( $p > 0.05$ , Bonferroni test). During weeks 8 and 9, soil leachate was significantly higher in CB treated soils compared to C ( $p \leq 0.05$ , Bonferroni test), with no significant differences observed between MWH and CB, and MWH and C

treatments ( $p > 0.05$ , Bonferroni test). In week 10, soil leachate was significantly higher in MWH treated soil compared to C ( $p \leq 0.05$ , Bonferroni test), with no significant difference between other treatments ( $p > 0.05$ , Bonferroni test) (Figure 6-3).

Along with the treatments, the incubation period also had a significant effect within each treatment ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In C, soil leachate was significantly lower in week 1 compared to weeks 2 to 7 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), and there was no significant difference observed from weeks 8 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in soil leachate between weeks 2 to 7 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower in weeks 9 to 12 compared to weeks 2 to 7 ( $p \leq 0.04$ ). In week 8, soil leachate was significantly higher than weeks 2 and 12 ( $p \leq 0.04$ ), with no significant differences observed with the rest of the weeks ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant differences was observed in soil leachate between weeks 9 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 6-3)

In MWH treatment, soil leachate was significantly lower in week 1 compared to weeks 2 to 8 and 10 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in soil leachate in weeks 9, 11 and 12 compared to week 1 ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 2, soil leachate was higher compared to weeks 8 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and no significant difference was observed compared to weeks 3 to 7 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in soil leachate between weeks 3 to 7 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly higher compared to week 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in soil leachate in weeks 8 to 11 compared to weeks 3 to 7 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed between weeks 8 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 6-3)

In CB treatment, soil leachate was significantly lower in week 1 compared to the rest of the incubation period ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in soil leachate between weeks 2 to 11 ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 12, the soil leachate was significantly lower than in weeks 3, 4, 5, 7, 8 and 9 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), and no other significant differences were

observed with the rest of the weeks ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 6-3).

Summarising the general trends observed in soil leachate from weeks 1 to 12:

- a) The soil leachate was significantly higher in MWH treated soil compared to C and CB ( $p \leq 0.05$ , Bonferroni test), and no significant difference was observed between C and CB ( $p > 0.05$ , Bonferroni test).
- b) In C, the soil leachate was initially lower in week 1, increased in week 2, and remained relatively constant until a decrease was observed in week 8.
- c) In MWH treatment, soil leachate was lower in week 1, increased in week 2 and remained relatively constant until week 8. A further decrease was observed in week 9 and remained relatively constant till week 12.
- d) In CB treatment, soil leachate was lower in week 1, increased in week 2 and remained relatively constant until week 11. A decrease was observed in soil leachate in week 12.

## **6.4 Effect of char treatments on nutrient concentration in soil leachate**

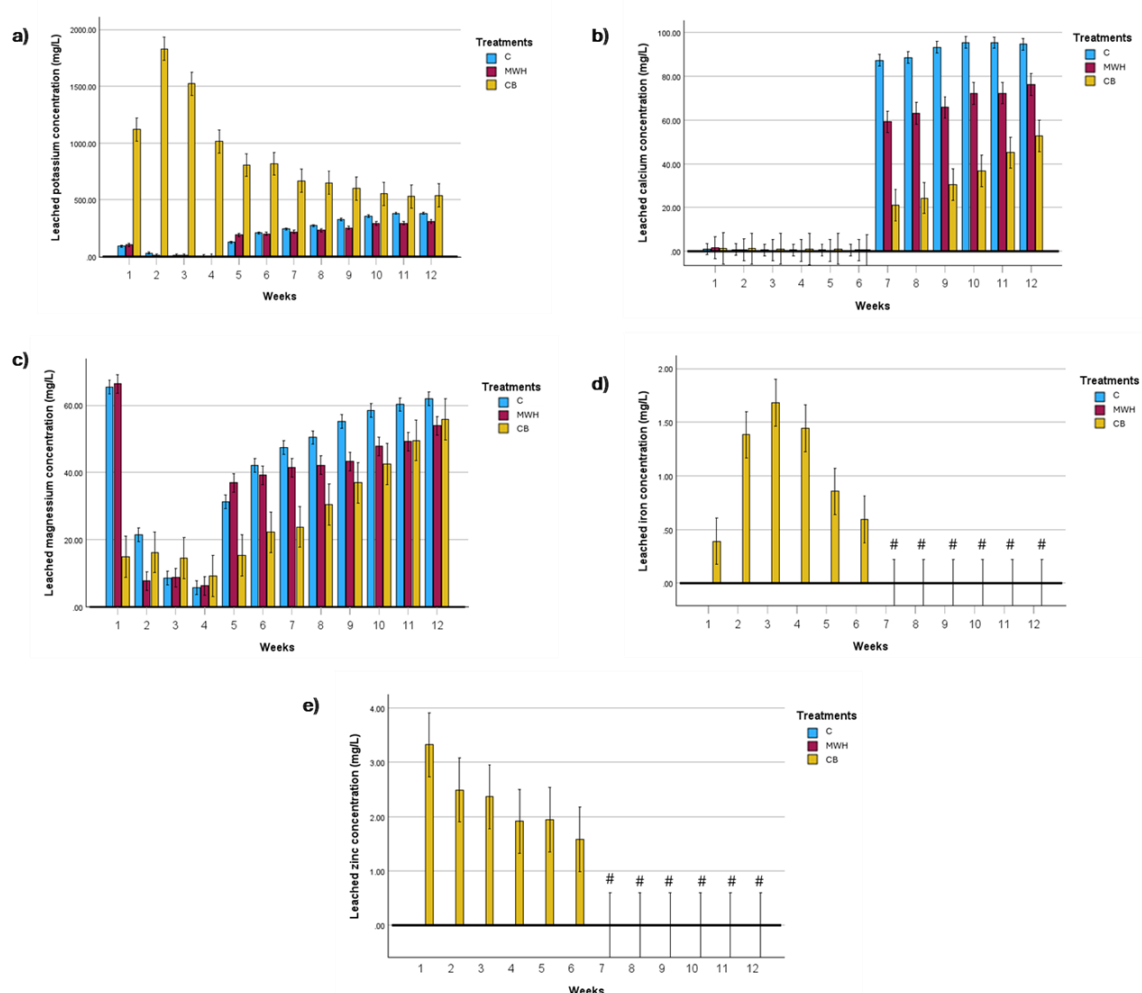
In this section the concentrations of primary macro-nutrients (nitrogen, phosphorus, potassium), secondary plant macro-nutrients (calcium and magnesium) and micronutrients (iron and zinc) in the soil leachates over time are presented.

Among the elements measured, the concentration of iron and zinc in C and MWH leachates were below detection limit ( $BDL \leq 0.5\text{mg/L}$ ) throughout the incubation period, whereas for CB treatment from week 7 onwards the concentration of iron and zinc leachates were BDL (Figure 6-4d and 6-4e). The leaching behaviour of potassium, calcium, magnesium, phosphate, and nitrate was consistent across the treatments, whereas potassium and phosphate showed different patterns. The concentration of these leached element in all the treatments were significantly lower in weeks 1 to 4. The addition of Hoagland solution from week 5 onwards till the end of the incubation period resulted in an increase in the concentration of leached elements from weeks 6 to 12 (Figure 6-4 and 6-5). Variations observed over the incubation period are further described in the following sections.

After a detailed description of the trends, there is a summary of the key points at the end of each section.

### 6.4.1 Leached potassium concentration

A significant difference in leached potassium concentration was observed due to char treatment, incubation time, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA) (Figure 6-4).



**Figure 6- 4.** Average weekly concentrations of leached cations a) potassium, b) calcium, c) magnesium, d) iron and e) zinc in control (C), microwave hydrochar (MWH) and conventional biochar (CB) treated soils. Values are averages  $\pm$  standard errors,  $n=3$ . The bars in the the graph represents standard errors. The # along the error bars for d) iron and e) zinc are below detection level (BDL < 0.5 mg/L) are expressed as the detection limit divided by the square root of two (Croghan & Egeghy, 2003).

From weeks 1 to 4 and in week 6, the CB treatment had significantly higher potassium levels compared to MWH treatment and C ( $p \leq 0.05$ , Bonferroni test), with no significant differences between MWH and C ( $p > 0.05$ , Bonferroni test). In week 5, the CB treatment had the highest, while the C had the lowest potassium levels ( $p \leq 0.05$ , Bonferroni test). From weeks 7 to 12, CB consistently had higher potassium levels, and MWH had the lower levels ( $p \leq 0.05$ , Bonferroni test) (Figure 6-4a).

Incubation time significantly affected leached potassium levels within treatments ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In C and MWH treatments, leached potassium was significantly higher in week 1 compared to weeks 2 to 4 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and significantly lower than in weeks 5 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in leached potassium between weeks 2 to 4 ( $p > 0.05$ , Repeated measures two-way ANOVA) and leached potassium during these weeks was also significantly lower than in weeks 5 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) (Figure 6-4a). In C, leached potassium significantly increased from week 5 to 9 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and was significantly lower in week 5 to 9 compared to weeks 10 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed from weeks 10 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 6-4a). In the MWH treatment, no significant difference was observed in leached potassium between weeks 5 and 6 ( $p > 0.05$ , Repeated measures two-way ANOVA), although leached potassium was significantly lower than in week 9 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in leached potassium between weeks 5 and 6 compared to weeks 7 and 8 ( $p > 0.05$ , Repeated measures two-way ANOVA). Additionally, no significant differences were observed between weeks 7 to 9 ( $p > 0.05$ , Repeated measures two-way ANOVA), but leached potassium was significantly lower than in weeks 10 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in leached potassium between weeks 10 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 6-4a).

In the CB treatment, leached potassium was significantly lower in week 1 compared to weeks 2 and 3, and significantly higher than in weeks 5 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed between weeks 1 and 4 ( $p > 0.05$ , Repeated measures two-way ANOVA). Leached potassium significantly decreased from week 2 to 3 ( $p$



$\leq 0.05$ , Repeated measures two-way ANOVA) and was significantly higher than in weeks 4 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 4, leached potassium was significantly higher than in weeks 7 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant differences observed between weeks 5 and 6 compared to week 4 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in leached potassium between weeks 5 and 6 ( $p > 0.05$ , Repeated measures two-way ANOVA), although leached potassium was significantly higher than in weeks 9 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed between weeks 5 to 8 ( $p > 0.05$ , Repeated measures two-way ANOVA). And no further differences were observed in leached potassium from week 7 onwards to week 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 6-4a).

Summarising the general trends observed in leached potassium concentration from weeks 1 to 12:

- a) The concentration of leached potassium was significantly greater in CB treated soil, and lower in MWH treated soil ( $p \leq 0.05$ , Bonferroni test).
- b) During the initial watering period of wheat crop (weeks 1 to 4) using de-ionised water:
  - In C and MWH treated soils, leached potassium was significantly higher in week 1, then decreased in weeks 2 to 4.
  - In CB treated soil, leached potassium was also higher in week 1, followed by a decreased from week 2 through week 4.
- c) From week 5 -12 with the introduction of the Hoagland solution:
  - In C and MWH treated soils, a significant increase in leached potassium concentration was observed from week 5 until week 10 and then remained constant until week 12.
  - In CB treated soil, leached potassium increased in week 5 and remained relatively constant with few fluctuations until week 12.

#### 6.4.2 Leached calcium concentration

A significant difference in leached calcium concentrations was observed due to char treatment, incubation time, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA). From weeks 1 to 6, no significant interaction between treatment and leached calcium concentration was observed ( $p > 0.05$ , Bonferroni test). However, from weeks 7 to 12, the highest calcium concentration was found in the C treatment and the lowest in the CB treatment ( $p \leq 0.05$ , Bonferroni test) (Figure 6-4b).

Incubation time also significantly influenced calcium levels within treatments ( $p \leq 0.001$ , Repeated measures two-way ANOVA). In the C and MWH treatments, no significant differences were observed in the concentration of leached calcium between weeks 1 and 6 ( $p > 0.05$ , Repeated measures two-way ANOVA), while the leached calcium was significantly lower compared to weeks 7 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In C, leached calcium was significantly lower in week 7 compared to weeks 10, 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant difference observed between 8 and 9 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant differences were observed between weeks 8 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 6-4b). In the MWH treatment, leached calcium was significantly lower in week 7 compared to weeks 10 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant differences between weeks 8 and 9 ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 8, no significant difference was observed in the concentration of leached calcium between weeks 8 to 11 ( $p > 0.05$ , Repeated measures two-way ANOVA), although the leached calcium was significantly lower in week 8 compared to week 12 ( $p \leq 0.04$ ) (Figure 6-4b).

In the CB treatment, no significant difference was observed in the concentration of leached calcium between weeks 1 to 6 ( $p > 0.05$ , Repeated measures two-way ANOVA), while the leached calcium was significantly lower compared to weeks 7 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 7, leached calcium was significantly lower than in weeks 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant differences observed between weeks 8 to 10 ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 8, leached calcium was significantly lower than in weeks 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant difference was observed in weeks 9 and 10 ( $p > 0.05$ ,

Repeated measures two-way ANOVA). No significant difference was observed in leached calcium between 9 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA), except for a significant increase in week 12 compared to week 9 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) (Figure 6-4b).

Summarising the general trends observed in leached calcium concentration from weeks 1 to 12:

- a) The concentration of leached calcium was significantly greater in C, and lower in CB treated soil ( $p \leq 0.05$ , Bonferroni test), with the MWH treatment showing intermediate levels of calcium leaching.
- b) During the initial watering period of wheat crop (weeks 1 to 4) using de-ionised water:
  - In all treatments, the concentration of leached calcium was significantly lower from weeks 1 to 4 compared to weeks 7 to 12.
- c) From week 5-12 with the introduction of the Hoagland solution:
  - In C treatment, leached calcium remained significantly lower in weeks 5 and 6. In week 7, a significant increase was observed, followed by a further increase in week 9, with leached calcium remaining relatively constant till week 12.
  - In MWH treatment, leached calcium remained significantly lower in weeks 5 and 6. A significant increase was observed in week 7, followed by a further increase in week 10, with levels staying relatively constant till week 12.
  - In CB treatment, leached calcium remained significantly lower in weeks 5 and 6. From week 7 onwards, a significant increase was observed, with leached calcium showing a few fluctuations but generally remaining elevated until week 12.

### **6.4.3 Leached magnesium concentration**

A significant difference in leached magnesium concentrations was observed due to char treatment, incubation time, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA). In weeks 1 and 6, leached magnesium was significantly lower in CB-treated soils compared to C and MWH treatments ( $p \leq 0.05$ , Bonferroni test), with no difference observed between C and MWH ( $p > 0.05$ , Bonferroni test). In week 2, MWH-treated soils exhibited

significantly lower magnesium compared to C and CB ( $p \leq 0.05$ , Bonferroni test), with no difference between C and CB ( $p > 0.05$ , Bonferroni test). Weeks 3 and 4 showed no significant differences between treatments ( $p > 0.05$ , Bonferroni test). In week 5, MWH-treated soils had the highest and CB-treated soils the lowest magnesium levels ( $p \leq 0.05$ , Bonferroni test). Weeks 7 and 8 saw highest magnesium levels in C and the lowest in CB-treated soils ( $p \leq 0.05$ , Bonferroni test). From weeks 9 to 11, leached magnesium was higher in C compared to MWH and CB ( $p \leq 0.05$ , Bonferroni test), with no difference between MWH and CB ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 12, leached magnesium was higher in C compared to MWH ( $p \leq 0.05$ , Bonferroni test), with no significant difference between C and CB ( $p > 0.05$ , Bonferroni test) and between MWH and CB ( $p > 0.05$ , Bonferroni test) (Figure 6-4c).

Incubation time had a significant effect on magnesium concentrations within treatments ( $p \leq 0.001$ , Repeated measures two-way ANOVA). In the C treatment, leached magnesium was higher in week 1 compared to weeks 2 to 11 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant difference observed in week 12 ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 2, leached magnesium was also significantly lower compared to weeks 5 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), and significantly higher than in weeks 3 and 4 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in leached magnesium between weeks 3 and 4 ( $p > 0.05$ , Repeated measures two-way ANOVA), although the leached magnesium was significantly lower than in weeks 5 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 5, the leached magnesium was significantly lower compared to weeks 6 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed for leached magnesium between weeks 6 and 7 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower compared to weeks 9 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), except for a significant increase in week 8 compared to week 6 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed between weeks 7 and 8 ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 8, leached magnesium was significantly lower than in weeks 9 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in leached magnesium between weeks 9 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 6-4c).

In MWH treatment, the concentration of leached magnesium was significantly higher in week 1 compared to the rest of the weeks ( $p \leq 0.05$ , Repeated measures two-way ANOVA). The concentration decreased in week 2 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant difference observed in leached magnesium between weeks 2 to 4 ( $p > 0.05$ , Repeated measures two-way ANOVA). From weeks 2 to 4, leached magnesium was significantly lower compared to weeks 5 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in the concentration of leached magnesium from week 5 to 9 ( $p > 0.05$ , Repeated measures two-way ANOVA), although the leached magnesium was significantly lower compared to weeks 10 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in the concentration of leached magnesium from weeks 10 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 6-4c).

In the CB treatment, leached magnesium was significantly lower in weeks 1 to 5 compared to weeks 8 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in weeks 6 and 7 compared to weeks 1 and 5 ( $p > 0.05$ , Repeated measures two-way ANOVA), except for week 4, where leached magnesium was significantly lower than in week 7 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In weeks 6 and 7, no significant difference was observed compared to weeks 8 and 9 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower than in weeks 10 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in leached magnesium between weeks 8 and 9 ( $p > 0.05$ , Repeated measures two-way ANOVA) and were significantly lower than in weeks 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant differences were observed in the concentration of leached magnesium from weeks 10 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 6-4c).

Summarising the general trends observed in leached magnesium concentration from weeks 1 to 12:

- d) The concentration of leached magnesium was significantly greater in C, and lower in CB treated soil ( $p \leq 0.05$ , Bonferroni test).
- e) During the initial watering period of wheat (weeks 1 to 4) using de-ionised water:
  - In C and MWH treatment, leached magnesium was significantly higher in week 1 and decreased in weeks 2 to 4.

- f) In CB treatment, leached magnesium remained relatively constant from weeks 1 to 4.
- g) From week 5-12 with the introduction of the Hoagland solution:
  - In C treatment, from week 5 onwards, leached magnesium generally increased until week 10, where it stabilised.
  - In MWH treatment, an increase in leached magnesium was observed in week 5, which remained constant until week 9, followed by further increase in weeks 10 to 12.
  - In CB treatment, leached magnesium remained constant from weeks 5 to 7. From week 8 onwards, magnesium levels increased, with further increase from weeks 10 to 12.

#### 6.4.4 Leached iron concentration

A significant difference in leached iron concentration was observed due to char treatment, incubation time, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA). From weeks 1 to 6, leached iron was significantly greater in the CB treatment compared to C and MWH ( $p \leq 0.05$ , Bonferroni test), with no significant differences between MWH and C ( $p > 0.05$ , Bonferroni test). From weeks 7 to 12, no significant differences in the concentration of leached iron were observed between the treatments ( $p > 0.05$ , Bonferroni test) (Figure 6-4d).

For both C and MWH treatments, the concentration of leached iron was consistently below detection limits ( $BDL \leq 0.5$  mg/L), from week 1 to 12, with no significant changes over time ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 6-4d).

In CB treatment, the concentration of leached iron was below detection limits ( $BDL \leq 0.5$  mg/L) from weeks 7 to 12. In week 1, leached iron was significantly lower compared to weeks 2, 3 and 4 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant differences observed between week 1 and weeks 5 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA). Additionally, no significant differences were observed between weeks 2 to 4 ( $p > 0.05$ , Repeated measures two-way ANOVA), although the leached iron during these weeks were significantly higher than in weeks 5 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). There were no significant differences in leached iron between weeks 5 and 6 ( $p > 0.05$ , Repeated measures two-way ANOVA), but the leached iron was significantly higher during

these weeks compared to weeks 7 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant differences were observed between weeks 7 and 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 6-4d).

Summarising the general trends observed in leached iron concentration from weeks 1 to 12:

- a) The concentration of iron in C and MWH treatment was below detection limit ( $BDL \leq 0.5$  mg/L) throughout the incubation period from weeks 1 to 12. And, in CB treatment the leached iron was below detection limit ( $BDL \leq 0.5$  mg/L) from weeks 7 to 12.
- b) The concentration of leached iron was significantly greater in CB treated soil compared to C and MWH treated soil ( $p \leq 0.05$ , Bonferroni test), with no significant difference between C and MWH treated soil ( $p > 0.05$ , Bonferroni test)
- c) In CB treatment, from weeks 1 to 4 (in presence of De-ionised water), the leached iron increased from week 1 to 3, then decreased in week 4 and a further decrease was observed in weeks 5 to 6 (with the introduction of the Hoagland solution).

#### 6.4.5 Leached zinc concentration

A significant difference in leached zinc concentration was observed due to char treatment, incubation time, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA). From weeks 1 to 6, CB has significantly higher concentration of leached zinc compared to MWH and C ( $p \leq 0.05$ , Bonferroni test), with no significant difference between MWH and C ( $p > 0.05$ , Bonferroni test). From weeks 7 to 12, no significant differences were observed between the treatments for the concentration of leached zinc ( $p > 0.05$ , Bonferroni test) (Figure 6-4e).

For both C and MWH treatments, the concentration of leached zinc was below detection limits ( $BDL \leq 0.5$  mg/L), which remained constant from week 1 to week 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 6-4e). In CB treatment, the concentration of leached zinc was below detection limit ( $BDL \leq 0.5$  mg/L) from weeks 7 to 12. In week 1, leached zinc was significantly higher compared to weeks 4 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant differences observed between weeks 2 and 3 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed between weeks 2 to 6 ( $p > 0.05$ , Repeated measures two-way ANOVA) although these weeks had higher zinc leachate

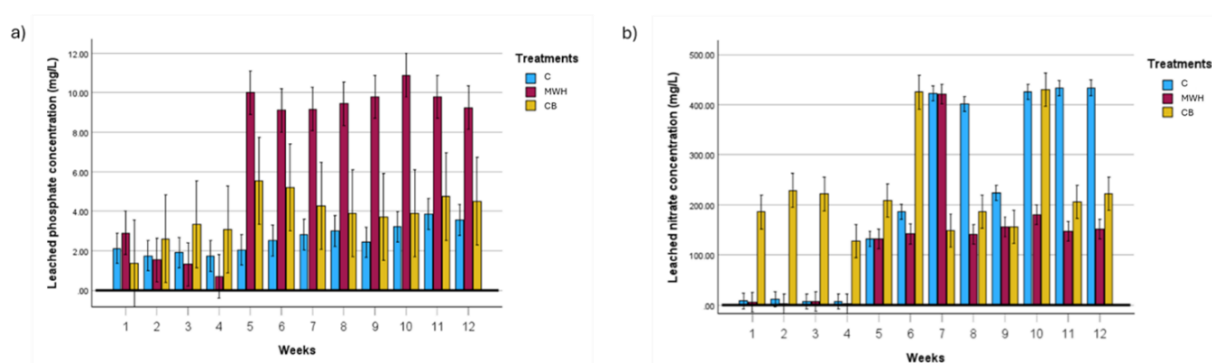
compared to weeks 7 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed between weeks 7 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 6-4e).

Summarising the general trends observed in leached zinc concentration from weeks 1 to 12:

- The concentration of zinc in C and MWH treatment was below detection limit ( $BDL \leq 0.5$  mg/L) throughout the incubation period from weeks 1 to 12 and, in CB treatment the leached iron was below detection limit ( $BDL \leq 0.5$  mg/L) from weeks 7 to 12.
- The concentration of leached zinc was significantly greater in CB treated soil compared to C and MWH treated soil ( $p \leq 0.05$ , Bonferroni test), with no significant difference between C and MWH treated soil ( $p > 0.05$ , Bonferroni test).
- In CB treatment, from weeks 1 to 4 (in presence of de-ionised water), the leached zinc decreased, and a further decrease was observed in weeks 5 to 6 (with the introduction of the Hoagland solution).

#### 6.4.6 Leached phosphate concentration

A significant difference in the concentration of leached phosphate was observed due to char treatment, incubation time, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA) (Figure 6-5a).



**Figure 6- 5.** Average weekly concentration of leached anions a) phosphate and b) nitrate in control (C), microwave hydrochar (MWH) and conventional biochar (CB) treated soils. Values are averages  $\pm$  standard errors,  $n=3$ . The bars in the graphs represent standard errors,  $n=3$ . The bars in the graphs represent standard errors.



From weeks 1 to 4, no significant differences were observed between treatments for leached phosphate concentration ( $p > 0.05$ , Bonferroni test). In weeks 5 and 6, leached phosphate concentration was higher in MWH-treated soil compared to the control and CB treated soil ( $p \leq 0.05$ , Bonferroni test) (Figure 6-5a). From weeks 7 to 12, leached phosphate concentration remained highest in MWH-treated soil compared to C and CB ( $p \leq 0.05$ , Bonferroni test), with no significant difference between C and CB treatments ( $p > 0.05$ , Bonferroni test) (Figure 6-5a).

Incubation time significantly influenced leached phosphate concentrations within treatments ( $p \leq 0.001$ , Repeated measures two-way ANOVA). In C, no significant difference was observed in the concentration of leached phosphate from weeks 1 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA), except for a significant increase in week 11 compared to weeks 2, 3 and 4 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) (Figure 6-5a). In MWH treatments, no significant difference was observed in leached phosphate between weeks 1 to 4 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower than weeks 5 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in leached phosphate from weeks 5 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 6-5a). In CB treatment, no significant differences in leached phosphate concentrations was observed throughout the incubation period from weeks 1 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 6-5a).

Summarising the general trends observed in leached phosphate concentration from weeks 1 to 12:

- h) The concentration of leached phosphate was significantly greater in MWH treated soil, and the lower in C ( $p \leq 0.05$ , Bonferroni test).
- i) During the initial watering period of wheat (weeks 1 to 4) using de-ionised water:
  - In C and CB treatment, the concentration of leached phosphate remained relatively stable from weeks 1 to 4.
- j) In MWH treatment, leached phosphate was lower in weeks 1 to 4 compared to weeks 5 to 12.
- k) From week 5-12 with the introduction of the Hoagland solution:
  - In C treatment, no significant difference was observed in leached phosphate

from week 5 onwards till week 12, except for a significant increase in week 11 compared to week 2, 3 and 4.

- In MWH treatment, an increase in leached phosphate was observed in week 5, which remained relatively constant till week 12.
- In CB treatment, no significant difference was observed in leached phosphate between weeks 5 to 12.

#### **6.4.7 Leached nitrate concentration**

A significant difference in leached nitrate concentrations was observed due to char treatment, incubation time, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA). From weeks 1 to 6, CB-treated soils had significantly higher nitrate concentrations compared to MWH and C ( $p \leq 0.05$ , Bonferroni test). No significant difference was observed between MWH and C from weeks 1 to 5 ( $p > 0.05$ , Bonferroni test); whereas, in week 6, MWH- treated soil had significantly lower nitrate levels compared to C and CB treatments ( $p \leq 0.05$ , Bonferroni test). In week 7, CB-treated soils showed significantly lower nitrate concentrations compared to MWH and C ( $p \leq 0.05$ , Bonferroni test), with no significant difference between MWH and C treatments ( $p > 0.05$ , Bonferroni test). During weeks 8 and 9, nitrate concentrations significantly increased in C compared to MWH and CB treatments ( $p \leq 0.05$ , Bonferroni test), with no significant difference between MWH and CB ( $p > 0.05$ , Bonferroni test). From weeks 10 to 12, leached nitrate concentration was significantly lower in MWH-treated soils compared to CB and C treatments ( $p \leq 0.05$ , Bonferroni test). No significant difference was observed between C and CB in week 10 ( $p > 0.05$ , Repeated measures two-way ANOVA); whereas, in weeks 11 and 12, C had higher nitrate levels than CB ( $p \leq 0.05$ , Bonferroni test) (Figure 6-5b).

The incubation period also had a significant effect on leached nitrate concentrations within treatments ( $p \leq 0.001$ , Repeated measures two-way ANOVA). In C, no significant differences were observed in leached nitrate between weeks 1 to 4 ( $p > 0.05$ , Repeated measures two-way ANOVA), and the leached nitrate was significantly lower compared to weeks 5 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). The concentration of leached nitrate increased significantly from week 5 to week 6 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), and in

both weeks 5 and 6, leached nitrate was also significantly lower compared to weeks 7 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference were observed between weeks 7 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) except in week 9, where leached nitrate was significantly lower than in weeks 7, 8 and 10 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) (Figure 6-5b).

In the MWH treatment, no significant difference was observed in leached nitrate concentrations between weeks 1 to 4 ( $p > 0.05$ , Repeated measures two-way ANOVA), but the concentration was significantly lower compared to weeks 5 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed between weeks 5 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA), except in week 7, where leached nitrate was significantly higher than in weeks 5,6 and 8 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) (Figure 6-5b).

In the CB treatment, leached nitrated concentration was significantly higher in weeks 6 and 10 compared to the rest of the weeks ( $p \leq 0.05$ , Repeated measures two-way ANOVA). Additionally, in weeks 2 and 3, leached nitrate was also significantly higher than in weeks 4, 7 and 9 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 4, leached nitrate was also significantly lower than weeks 5 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No other significant differences were observed between the remaining weeks ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 6-5b).

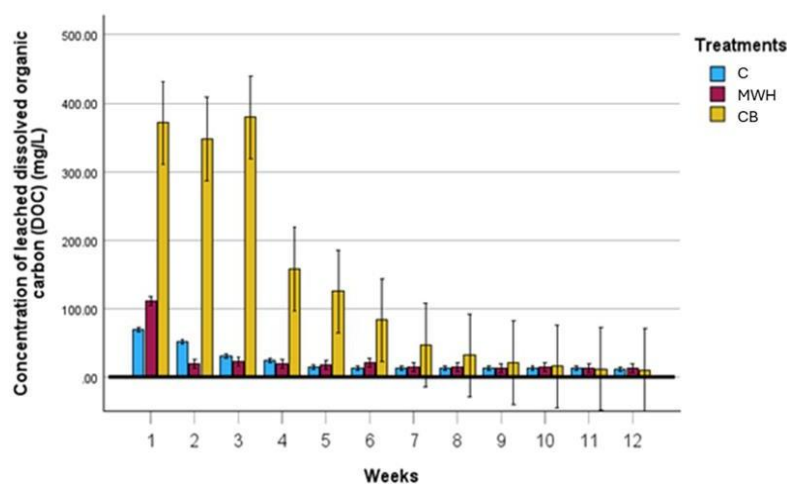
Summarising the general trends observed in leached nitrate concentration from weeks 1 to 12:

- l) The concentration of leached nitrate was significantly lower in MWH compared to C and CB ( $p \leq 0.05$ , Bonferroni test), with no significant differences between C and CB ( $p > 0.05$ , Bonferroni test).
- m) During the initial watering period of wheat (weeks 1 to 4) using de-ionised water:
  - o In C and MWH treatment, leached nitrate was significantly lower in weeks 1 to 4 compared to week 5 to 12.
  - o In CB treatment, leached nitrated remained relatively constant from weeks 1 to 3 and decreased in week 4.
- n) From week 5-12 with the introduction of the Hoagland solution:

- In C treatment, leached nitrate increased significantly from weeks 5 to 7. From week 8 to 12, except for a decrease in week 9, leached nitrate remained relatively constant.
- In MWH treatment, an increase in leached nitrate was observed in week 5, which remained relatively constant except for a significant increase in week 7.
- In CB treatment, leached nitrate was significantly higher in weeks 6 and 10, with the rest of the weeks remaining relatively constant.

## 6.5 Effect of char treatments on dissolved organic carbon concentration in soil leachate

This section reports how different char treatments affect the concentration of dissolved organic carbon (DOC) in the leachate. After a detailed description of the trends, there is a summary of the key points at the end of this section. A significant difference in leached DOC concentrations was observed due to char treatment, incubation time, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA) (Figure 6-6).



**Figure 6- 6.** Average weekly concentration of leached dissolved organic carbon (DOC) in the control (C), microwave hydrochar (MWH) and conventional biochar (CB) treated soils. Values are averages  $\pm$  standard errors,  $n=3$ . The bars in the graph represent standard errors,  $n=3$ . The bars in the graph represent standard errors.

From weeks 1 to 5, leached DOC was significantly higher in CB treated soil compared to C and MWH treated soil ( $p \leq 0.05$ , Bonferroni test). In week 1, C-treated soil had the lowest leached DOC compared to MWH ( $p \leq 0.05$ , Bonferroni test), whereas on week 2, MWH-treated soil had the lowest concentration compared to C ( $p \leq 0.05$ , Bonferroni test). No significant difference was observed between C and MWH-treated soils during weeks 3, 4, and 5 ( $p > 0.05$ , Bonferroni test). From weeks 6 to 12, no significant differences were observed between the treatments regarding leached DOC concentrations ( $p > 0.05$ , Bonferroni test) (Figure 6-6).

Incubation period also significantly affected DOC concentrations within the treatments ( $p \leq 0.001$ , Repeated measures two-way ANOVA). In C, the concentration of leached DOC significantly decreased from week 1 to week 3 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and was significantly higher compared to weeks 5 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 4, leached DOC was significantly lower than weeks 1, 2 and 5 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and no significant difference was observed between weeks 4 and 3 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed between weeks 5 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 6-6). In MWH treatment, DOC concentration was higher in week 1 compared to the rest of the weeks ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed from week 2 onwards till week 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 6-6). In CB treatment, no significant difference was observed in the concentration of leached DOC from weeks 1 to 3 ( $p > 0.05$ , Repeated measures two-way ANOVA), and was significantly higher than weeks 4 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 4, leached DOC was significantly higher than weeks 9 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), and no significant differences were observed in weeks 5 to 8 compared to week 4 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant differences were observed in leached DOC between weeks 5 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 6-6).

Summarising the general trends observed in leached DOC concentration from weeks 1 to 12:

- a) The concentration of leached DOC was significantly greater in CB compared to C and MWH treatment ( $p \leq 0.05$ , Bonferroni test), with no significant difference between C and MWH ( $p > 0.05$ , Bonferroni test).
- b) In C, the decreased DOC decreased from weeks 1 to 5 and remained relatively stable till

week 12.

- c) In MWH treatment, the leached DOC was significantly higher in week 1, decreased in week 2, and then remained constant until week 12.
- d) In CB treatment, no significant difference was observed in leached DOC between weeks 1 to 3. From week 3 onwards, leached DOC relatively decreased until week 12.

## **6.6 Effect of char treatments on soil physical structure and chemistry at the end of 12-week experiment period**

Addition of char to the soil can affect soil porosity, soil CEC, pH, and nutrient concentration in the soil exchange sites. This section reports how different char treatments impact physical structure and chemical properties of soil.

### **6.6.1 Effect of char treatments on soil porosity**

CB treated soil had significantly lower soil porosity compared to MWH treated soil ( $p \leq 0.05$ , one-way ANOVA), with no other significant difference observed between the treatments ( $p > 0.05$ , one-way ANOVA) (Table 6-2).

**Table 6- 2.** Physical and chemical soil properties for control (C), microwave hydrochar (MWH) and conventional biochar (CB) treatments. Data are presented as mean  $\pm$  standard error (n=3). Values within each column denoted by different superscripts <sup>a, b and c</sup> were found to be significantly different from one another according to Tukey's test ( $p < 0.05$ , one-way ANOVA).

Properties	C-treated soil	MWH-treated soil	CB-treated soil
Soil porosity ( $\text{cm}^3 \text{cm}^{-3}$ )	$0.858 \pm 0.001^{ab}$	$0.862 \pm 0.001^b$	$0.857 \pm 0.000^{ac}$
Soil nitrate (mol/kg)	$6.02 \times 10^{-6} \pm 9.35 \times 10^{-7}^a$	$1.28 \times 10^{-5} \pm 1.06 \times 10^{-6}^b$	$2.82 \times 10^{-5} \pm 2.15 \times 10^{-6}^c$
Soil ammonium (mol/kg)	$3.80 \times 10^{-6} \pm 1.72 \times 10^{-6}^a$	$3.55 \times 10^{-6} \pm 7.71 \times 10^{-6}^a$	$1.02 \times 10^{-5} \pm 3.7 \times 10^{-5}^a$
Soil CEC (cmol/kg)	$4.24 \pm 0.04^a$	$3.77 \pm 0.03^b$	$12.5 \pm 0.03^c$
Soil pH	$8.86 \pm 0.09^a$	$9.03 \pm 0.08^a$	$9.08 \pm 0.03^a$
Concentration of potassium on soil exchange sites (mol/kg)	$0.002 \pm 0.004^a$	$0.001 \pm 0.007^b$	$0.042 \pm 0.013^c$
Concentration of calcium on soil exchange sites (mol/kg)	$0.183 \pm 0.004^a$	$0.201 \pm 0.007^a$	$0.260 \pm 0.013^b$
Concentration of magnesium on soil exchange sites (mol/kg)	$0.001 \pm 0.004^a$	$0.002 \pm 0.007^b$	$0.003 \pm 0.013^c$
Concentration of iron on soil exchange sites (mol/kg)	$4.08 \times 10^{-5} \pm 0.004^a$	$7.03 \times 10^{-5} \pm 0.007^a$	$1.74 \times 10^{-4} \pm 0.013^b$
Concentration of phosphate on soil exchange sites (mol/kg)	$1.1 \times 10^{-4} \pm 0.004^a$	$1.01 \times 10^{-4} \pm 0.007^b$	$1.7 \times 10^{-4} \pm 0.013^c$

### **6.6.2 Effect of char treatments on nitrate and ammonium concentration on soil exchange sites**

Between the treatments, the CB-treated soil had the highest, and the C treated soil had the lowest extractable soil nitrate ( $p \leq 0.05$ , one-way ANOVA) (Table 6-2). No significant difference was observed in soil ammonium concentration across all treatments ( $p > 0.05$ , one-way ANOVA) (Table 6-2). The concentration of the ammonium and nitrate in the soil were positively correlated ( $n = 9$ ,  $r = 0.531$ ,  $p \leq 0.05$ , Pearson correlation analysis) implying that when the concentration of ammonium goes up, nitrate levels also tend to go up and vice-versa.

### **6.6.3 Effect of char treatments on soil cationic exchange capacity (CEC) and pH**

CB-treated soil had the highest CEC, while MWH-treated soil had the lowest CEC ( $p \leq 0.05$ , one-way ANOVA) compared to C treated soil (Table 6-2). No significant difference was observed between the treatments for soil pH ( $p > 0.05$ , one-way ANOVA), and the pH value of the soil for all the treatments indicated that the soil was alkaline in nature (Table 6-2). No significant difference was observed in the correlation between soil CEC and pH across all the treatments ( $n=9$ ,  $r= 0.484$ ,  $p > 0.05$ , Pearson's correlation).

### **6.6.4 Effect of char treatments on soil exchangeable cations**

The concentration of potassium, magnesium and phosphate on exchange sites were significantly higher in CB treated soil and significantly lower in MWH treated soil and C ( $p \leq 0.05$ , one-way ANOVA). The concentration of calcium and iron on exchange sites were significantly higher in CB treated soil compared to MWH treated soil and C ( $p \leq 0.05$ , one-



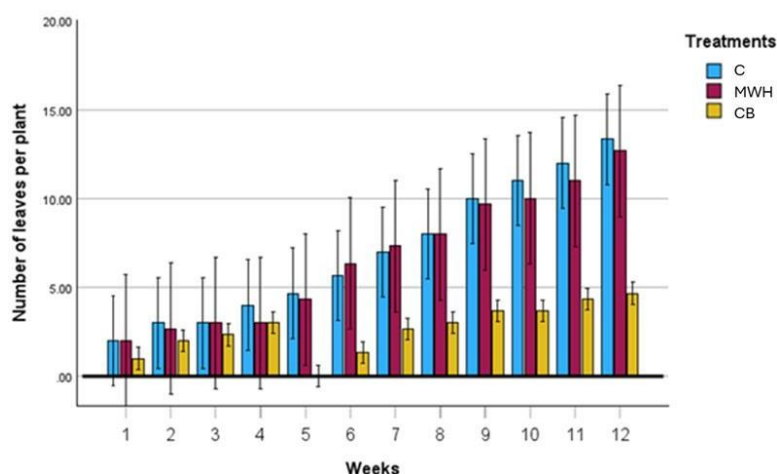
way ANOVA), with no significant difference observed between MWH treated soil and C ( $p > 0.05$ , one-way ANOVA) (Table 6-2).

Within each treated soil, a significant difference was observed between the concentrations of the elements on exchange sites in the soils ( $p \leq 0.001$ , one-way ANOVA). In C treated soil, the concentration of potassium on the exchange sites was found to be significantly higher than iron and phosphate ( $p \leq 0.05$ , one-way ANOVA), whereas significantly lower than calcium ( $p \leq 0.05$ , one-way ANOVA). No significant difference was found between potassium and magnesium on the exchange sites in C treated soil. Whereas the concentration of calcium on the exchange site was found to be significantly higher compared to all the other elements (magnesium, iron, and phosphate). No significant differences were observed between the concentration of iron, magnesium, and phosphate on the exchange sites in C treated soil ( $p > 0.05$ , one-way ANOVA). For MWH treated soil, the concentration of calcium on the exchange sites was significantly higher than other elements (potassium, magnesium, iron, phosphate) ( $p \leq 0.05$ , one-way ANOVA), whereas no other significant differences were observed between potassium, magnesium, iron and phosphate on the exchange sites in MWH treated soil ( $p > 0.05$ , one-way ANOVA). In CB treated soil, the concentration of calcium on exchange sites was significantly greater ( $p \leq 0.05$ , one-way ANOVA) than other elements (potassium, magnesium, iron and phosphate). The concentration of potassium was also significantly greater ( $p \leq 0.05$ , one-way ANOVA) than magnesium, iron, and phosphate on the exchange sites in CB treated soil. No significant differences were observed between the concentration of magnesium, iron, and phosphate on the exchange sites in CB treated soil ( $p > 0.05$ , one-way ANOVA).

## **6.7 Effect of char treatments on leaf number and biomass yield**

This section reports the impact of different char treatments on plant growth, specifically focusing on the number of leaves and biomass yield. After a detailed description of the trends, there is a summary of the key points at the end of this section.

A significant difference in the number of leaves was observed based on char treatment, incubation time, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA) (Figure 6-7).



**Figure 6- 7.** The average number of leaves on wheat plants in the control (C), microwave hydrochar (MWH) and conventional biochar (CB) treated soils. Values are averages  $\pm$  standard errors,  $n=3$ . The bars in the graph represents standard errors,  $n=3$ . The bars in the graph represents standard errors.

From weeks 1 to 4, there were no significant differences in leaf number between treatments ( $p > 0.05$ , Bonferroni test). However, from week 5 onwards until week 12, the CB-treated soil exhibited a significantly lower number of leaves compared to the MWH and C ( $p \leq 0.05$ , Bonferroni test), with no significant differences between MWH and C treatments ( $p > 0.05$ , Bonferroni test) (Figure 6-7).

The incubation period significantly influenced leaf numbers within each treatment ( $p \leq 0.001$ , Repeated measures two-way ANOVA). In C, the number of leaves was not significantly different from weeks 1 to 4 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower compared to weeks 9 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in weeks 5 to 8 compared to weeks 1 to 4 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in the number of leaves between weeks 5 and 6 ( $p > 0.05$ , Repeated measures two-way ANOVA),

and was significantly lower compared to weeks 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in the number of leaves in weeks 7 to 10 compared to weeks 5 and 6 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in the number of leaves from weeks 7 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 6-7).

In MWH treatment, no significant difference was observed in the number of leaves between weeks 1 to 4 ( $p > 0.05$ , Repeated measures two-way ANOVA) and significantly lower than week 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No other significant difference was observed in the number of leaves between the rest of the weeks ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 6-7).

For the CB treatment, in week 1, number of leaves were significantly lower than in weeks 4 and 8 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in weeks 2, 3 and 5 to 7 compared to week 1 ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 2, the number of leaves was significantly higher than week 5, and significantly lower than weeks 9 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant differences were observed in weeks 3,4, 6 to 8 compared to week 2 ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 3, the number of leaves was significantly higher than week 5 and lower than weeks 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in weeks 4, 6 to 10 compared to week 3 ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 4, the number of leaves was also significantly higher than weeks 5, 6 and lower in week 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), and no significant difference was observed in weeks 7 to 11 compared to week 4 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in the number of leaves between weeks 5 and 6 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower than weeks 7 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 7, the number of leaves were significantly lower than weeks 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), and no significant difference was observed in weeks 8 to 10 compared to week 7 ( $p > 0.05$ , Repeated measures two-way ANOVA), In week 8, the number of leaves was significantly lower than week 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), and no significant difference was observed in weeks 9 to 11 compared to

week 8 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant differences were observed in the number of leaves between weeks 9 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 6-7).

Summarising the general trends observed in the number of leaves from weeks 1 to 12:

- a) The number of leaves were significantly lower in CB compared to C and MWH treatment ( $p \leq 0.05$ , Bonferroni test), with no significant difference between C and MWH treatment ( $p \leq 0.05$ , Bonferroni test).
- b) During the initial watering period of wheat (weeks 1 to 4) using de-ionised water:
  - In C and MWH treatment, the number of leaves remained constant from weeks 1 to 4 but was lower compared to weeks 5 to 12.
  - In CB treatment, the number of leaves increased from week 1 and remained constant until week 4.
- c) From week 5-12 with the introduction of the Hoagland solution:
  - In C and MWH treatment, the number of leaves increased relatively from week 5 onwards until week 12.
  - In CB treatment, the number of leaves decreased in week 5 but increased again until week 7. From week 7 onwards until week 12, the number of leaves remained relatively stable.

In comparison between the treatments, the aboveground biomass was significantly lower for CB treatment compared to C and MWH treatment ( $p \leq 0.05$ , one-way ANOVA). No significant difference was observed between C and MWH treatment ( $p > 0.05$ , one-way ANOVA) (Table 6-3). No significant difference was observed between the treatment groups for below ground biomass ( $p > 0.05$ , one-way ANOVA).

**Table 6- 3.** Average aboveground and belowground biomass after the plant-pot experiment for control (C), microwave hydrochar (MWH) and conventional biochar (CB) treatments. Data are the mean  $\pm$  standard deviation (n=3). Values within each column denoted by different superscripts <sup>a, b</sup> were found to be significantly different from one another according to Tukey's test ( $p < 0.05$ , one-way ANOVA).

Treatments	Above-ground biomass (g)	Below-ground biomass (g)
<b>C</b>	3.8 $\pm$ 0.901 <sup>a</sup>	1.1 $\pm$ 0.42 <sup>a</sup>
<b>MWH</b>	3.4 $\pm$ 1.431 <sup>a</sup>	1.1 $\pm$ 0.57 <sup>a</sup>
<b>CB</b>	0.84 $\pm$ 0.334 <sup>b</sup>	0.36 $\pm$ 0.19 <sup>a</sup>

## 6.8 Effect of char treatments on the nutrient concentration at exchange sites in wheat biomass

Chars are known to influence soil nutrient availability, which in turn can impact the nutrient concentration in the crops. This section reports how different char treatments affects the nutrient concentration on the exchange sites in wheat crop. The concentration of potassium, calcium, magnesium and iron was significantly lower on the exchange sites in wheat biomass for CB treatment compared to MWH treatment and C ( $p \leq 0.05$ , one-way ANOVA) (Table 6-4). The concentration of phosphorus on the exchange sites in wheat biomass was significantly lower than CB compared to C and MWH ( $p \leq 0.05$ , Kruskal-Wallis test), with no significant differences between C and MWH ( $p > 0.05$ , Kruskal-Wallis test). No significant difference was observed between the treatments for zinc concentration on the exchange sites in the wheat crop ( $p > 0.05$ , one-way ANOVA) (Table 6-4).

**Table 6- 4.** Average nutrient concentration in wheat biomass after the plant-pot experiment for control (C), microwave hydrochar (MWH) and conventional biochar (CB) treatments. Data are the mean values of 3 replicates. Values across each column denoted by different superscripts <sup>a, b</sup> were found to be significantly different from one another according to Tukey's test ( $p < 0.05$ , one-way ANOVA).

Properties	C soil	MWH treated soil	CB treated soil
Potassium (mol/kg)	$6.39 \times 10^{-4} \text{ }^a$	$5.29 \times 10^{-4} \text{ }^a$	$1.56 \times 10^{-4} \text{ }^b$
Calcium (mol/kg)	$5.27 \times 10^{-5} \text{ }^a$	$4.28 \times 10^{-5} \text{ }^a$	$1.08 \times 10^{-5} \text{ }^b$
Magnesium (mol/kg)	$5.95 \times 10^{-5} \text{ }^a$	$5.53 \times 10^{-5} \text{ }^a$	$2.41 \times 10^{-5} \text{ }^b$
Iron (mol/kg)	$5.42 \times 10^{-7} \text{ }^a$	$5.47 \times 10^{-7} \text{ }^a$	$2.59 \times 10^{-7} \text{ }^b$
Zinc (mol/kg)	$2.17 \times 10^{-7} \text{ }^a$	$2.85 \times 10^{-7} \text{ }^b$	$2.51 \times 10^{-7} \text{ }^a$
Phosphorus (mol/kg)	$6.05 \times 10^{-6} \text{ }^a$	$8.56 \times 10^{-7} \text{ }^a$	$2.67 \times 10^{-8} \text{ }^b$

## **Chapter 7: Wheat-pot experiments using palm waste chars: Results**

### **7.1 Introduction**

This chapter presents a detailed analysis and characterization of chars derived from palm waste using three distinct processes: microwave biochar (MWB) conventional biochar (CB) and microwave hydrochar (MWH). The chapter primarily reports how these chars influence soil leachate and its chemistry, the physical and chemical properties of soil and their subsequent effects on plant growth and yield.

Initially, the chapter focuses on the characterization of the chars, examining their elemental composition, hydrophobic properties, and thermal stability. The chapter further explores the impact of control (C) and char treatments (MWB, CB and MWH) on soil leachate dynamics over time. Various analyses are conducted to assess how these treatments affect soil nutrient dynamics, dissolved organic carbon, cation exchange capacity, pH levels, and plant growth.

### **7.2 Char characterisation**

Table 7-1 summarizes carbon and nitrogen percentage, pH, cationic exchange capacity (CEC), nutrient concentration, surface area and hydrophobicity of microwave biochar (MWB), conventional biochar (CB) and microwave hydrochar (MWH) char samples.

**Table 7- 1.** Properties of char samples: microwave biochar (CB), conventional pyrolysis (CB) and microwave hydrochar (MWH). Values are averages  $\pm$  standard errors, n=3. Values across each column with different superscripts a, b and c were significantly different from one another according to Tukey's test ( $p < 0.05$ ).

Parameters	MWB	CB	MWH
<b>C (%)</b>	60.1 $\pm$ 0.18 <sup>a</sup>	65.7 $\pm$ 0.33 <sup>b</sup>	53.5 $\pm$ 0.24 <sup>c</sup>
<b>N (%)</b>	0.88 $\pm$ 0.02 <sup>a</sup>	1.06 $\pm$ 0.02 <sup>a</sup>	0.67 $\pm$ 0.00 <sup>b</sup>
<b>pH</b>	7.05 $\pm$ 0.35 <sup>a</sup>	9.20 $\pm$ 0.35 <sup>b</sup>	6.84 $\pm$ 0.18 <sup>a</sup>
<b>CEC (cmol/kg)</b>	61.64 $\pm$ 4.71 <sup>a</sup>	186.4 $\pm$ 19.91 <sup>b</sup>	75.77 $\pm$ 9.78 <sup>a</sup>
<b>Concentration of potassium on char exchange sites (mol/kg)</b>	0.005 $\pm$ 0.002 <sup>a</sup>	0.059 $\pm$ 0.005 <sup>b</sup>	0.002 $\pm$ 0.001 <sup>a</sup>
<b>Concentration of calcium on char exchange sites Calcium (mol/kg)</b>	0.029 $\pm$ 0.009 <sup>a</sup>	0.096 $\pm$ 0.005 <sup>b</sup>	0.023 $\pm$ 0.008 <sup>a</sup>
<b>Concentration of magnesium on char exchange sites (mol/kg)</b>	0.006 $\pm$ 0.002 <sup>a</sup>	0.016 $\pm$ 0.001 <sup>b</sup>	0.004 $\pm$ 0.002 <sup>a</sup>
<b>Concentration of iron on char exchange sites (mol/kg)</b>	5.1 $\times 10^{-5} \pm 8.5 \times 10^{-6}$ <sup>a</sup>	2.6 $\times 10^{-5} \pm 2.6 \times 10^{-6}$ <sup>a</sup>	3.8 $\times 10^{-5} \pm 1.5 \times 10^{-6}$ <sup>a</sup>
<b>Concentration of phosphate on char exchange sites (mol/kg)</b>	0.002 $\pm$ 0.0001 <sup>a</sup>	0.006 $\pm$ 0.0003 <sup>b</sup>	0.0003 $\pm$ 0.0002 <sup>c</sup>
<b>BET surface area (m<sup>2</sup>/g)</b>	5	11	<2
<b>WDPT (s)</b>	$\leq 1$	$\leq 1$	60-600
<b>Category of hydrophobicity</b>	Hydrophilic	Hydrophilic	Strongly Hydrophobic



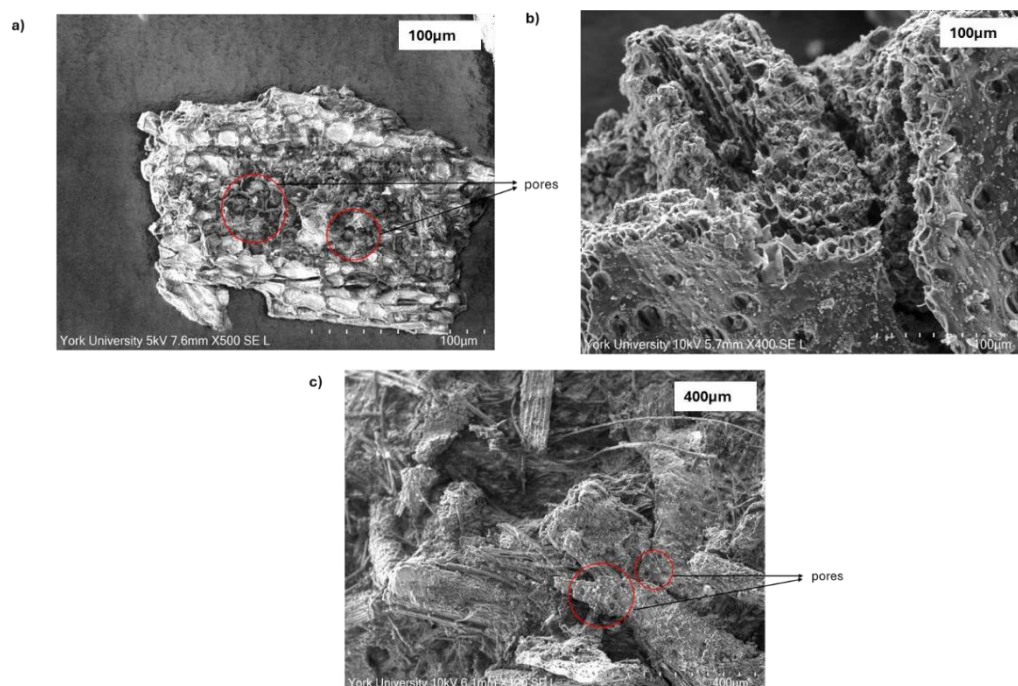
The carbon percentage was significantly greater in CB, and lower in MWH ( $p \leq 0.05$ , one-way ANOVA) and the nitrogen percentage was significantly lower in MWH sample compared to CB ( $p \leq 0.05$ , Kruskal-Wallis test) (Table 7-1).

The pH and the CEC were found to be significantly greater in CB sample compared to MWB and MWH samples ( $p \leq 0.05$ , one-way ANOVA) and no significant difference was observed between MWB and MWH samples ( $p > 0.05$ , one-way ANOVA) (Table 7-1).

When comparing the nutrient concentrations on the exchange sites between the char samples, the concentration of potassium, calcium and magnesium on the exchange sites was significantly higher in the CB char compared to the MWB and MWH char samples ( $p \leq 0.05$ , one-way ANOVA) and no significant difference was observed between MWB and MWH ( $p > 0.05$ , one-way ANOVA) (Table 7-1). No significant difference was observed in the concentration of iron on the exchange sites in the char sample ( $p \leq 0.05$ , one-way ANOVA) (Table 7-1). The concentration of phosphate on the exchange sites was lower in MWH and higher in CB ( $p \leq 0.05$ , one-way ANOVA) (Table 7-1). The concentration of zinc on the exchange site in the char sampled was below detection limit ( $BDL \leq 0.5$  mg/L).

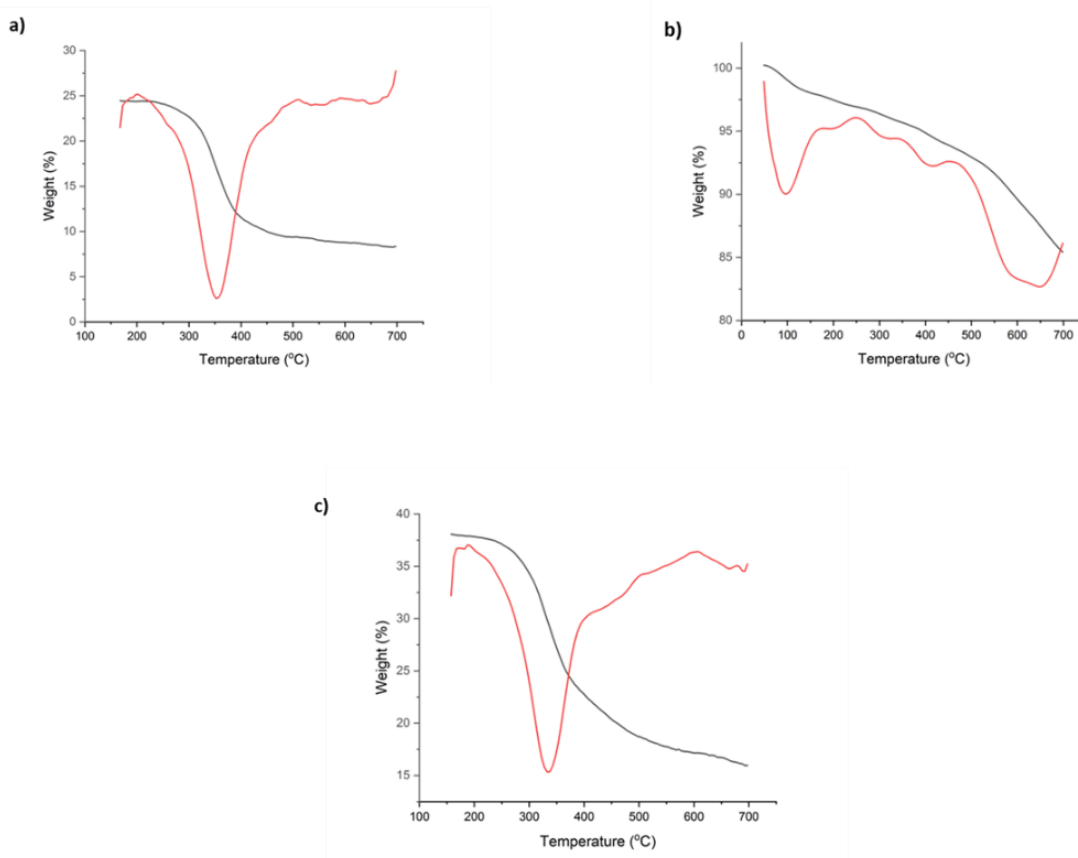
A hydrophobicity test was conducted using the Water Drop Penetration Time (WDPT) method on the MWB, CB and MWH char samples (Section 3.4c). The results indicated that the MWH sample was strongly hydrophobic in nature (Table 7-1), suggesting that when added to soil, it could repel water, leading to an increase in leachate. In contrast, the MWB and CB samples were hydrophilic, indicating their potential to improve water retention in the soil (Table 7-1).

SEM images of MWB, CB and MWH illustrate the surface morphology of the chars (Figure 7-1a, 7-1b and 7-1c). The surface of MWB showed a similar structure to that of palm waste biomass with the presence of few pores (Figure 7-1a). The surface of CB showed an irregular structure compared to the palm waste biomass with the formation of a well-defined tubular pore structure (Figure 7-1b). The SEM image of MWH depicts a rough granular structure, with fewer, irregular pores (Figure 7-1c).



**Figure 7- 1.** SEM images of a) microwave biochar (MWB) at x500 magnification, b) conventional biochar (CB) at x400 magnification and c) microwave hydrochar (MWH) at x120 magnification.

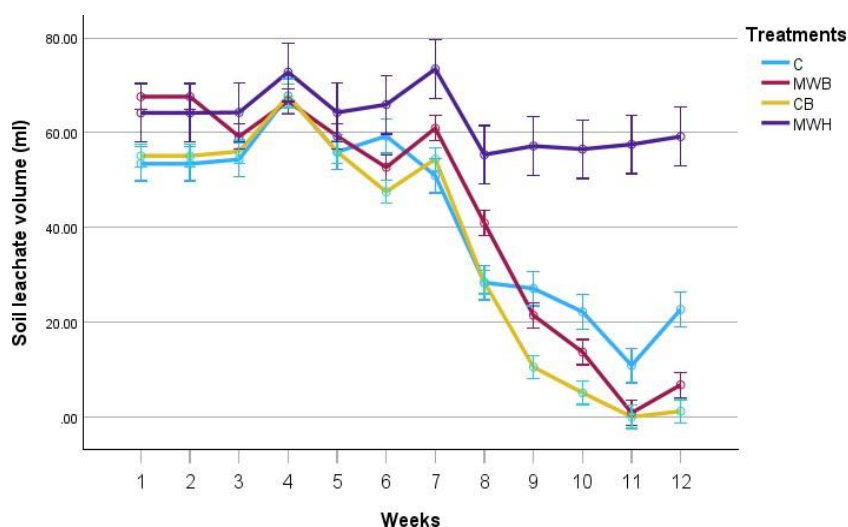
The thermal decomposition of MWB, CB and MWH chars was observed using TGA and DTG graphs presented in Figure 7-2a, 7-2b and 7-2c. The initial mass loss observed at 30 – 150 °C in all the three chars corresponds to moisture loss. The DTG curve for both MWB and MWH exhibits a sharp peak between 300-400 °C (Figure 7-2a and 7-2c). This mass loss occurs due to the combustion of the residual volatiles present within the char pores (Yi et al., 2012). The major weight loss observed was found to be lower in MWB (14.91 %) compared to MWH (22.03 %). The DTG curve in CB sample shows minimal weight loss (15 %), indicating that no rapid weight loss occurred over that temperature range (Figure 7-2b). This indicates that CB decomposes slowly and steadily, making it more thermally stable (Das et al., 2021).



**Figure 7- 2.** TG and DTG graph of a) microwave biochar (MWB), b) conventional biochar (CB) and c) microwave hydrochar (MWH). The thermogravimetric (TG) curves (black line, left y- axis) represent the weight loss (%) of the biomass samples as a function of temperature °C), while the derivative thermogravimetric (DTG) curves (red line, right y-axis) indicate the rate of weight loss (%) during thermal decomposition.

### 7.3 Effect of char treatments on soil leachate

This section reports how different char treatments influenced soil leachate over time. After a detailed description of the trends, there is a summary of the key points at the end of this section. A significant influence of biochar treatments, incubation periods and their interaction on soil leachate volume was observed ( $p \leq 0.001$ , Repeated measures two-way ANOVA) (Figure 7-3).



**Figure 7- 3.** Average weekly volume (ml) of water leached from control (C), microwave biochar (MWB), conventional biochar (CB) and microwave hydrochar (MWH) treated soils. Values are averages  $\pm$  standard errors,  $n=4$ . The bars in the graph represent stand errors.

In weeks 1 and 2, the soil leachate volume was significantly lower in the CB treatment compared to MWB treatment ( $p \leq 0.05$ , Bonferroni test), while no other significant differences observed between other treatments ( $p > 0.05$ , Bonferroni test). Although, C is lower than that of the CB leachate, the uncertainty means that the former is not significantly different from MWB. Between weeks 3 and 5, no further significant differences were observed between treatments ( $p > 0.05$ , Bonferroni test). In weeks 6 and 7, the soil leachate was significantly higher in MWH compared to CB ( $p \leq 0.05$ , Bonferroni test), and in week 7, MWH was also significantly higher than C ( $p \leq 0.05$ , Bonferroni test). No significant differences were observed between the rest of the treatments during this period ( $p > 0.05$ , Bonferroni test). In week 8, soil leachate was significantly higher in MWH compared to the rest of the treatments ( $p < 0.05$ , Bonferroni test) and MWH was significantly higher than CB and C ( $p < 0.05$ , Bonferroni test). No significant difference was observed between C and CB ( $p > 0.05$ , Bonferroni test). During weeks 9 and 10, the soil leachate was significantly lower in CB compared to C, MWB and MWH ( $p \leq 0.05$ , Bonferroni test). Additionally, MWH showed significantly higher leachate compared to C and MWB ( $p > 0.05$ , Bonferroni test), though no significant differences were observed between C and MWB ( $p > 0.05$ , Bonferroni test). In

week 11 and 12, soil leachate was significantly greater in MWH compared to C, MWB and CB ( $p \leq 0.05$ , Bonferroni test), while no significant differences were found between the other treatments ( $p > 0.05$ , Bonferroni test) and in week 12, soil leachate was significantly greater in C compared to MWB and CB ( $p \leq 0.05$ , Bonferroni test) (Figure 7-3)

Along with the treatments the incubation period also significantly influenced the soil leachate ( $p \leq 0.001$ , Repeated measures two-way ANOVA). In C, the leachate volume remained constant from weeks 1 to 7 ( $p > 0.05$ , Repeated measures two-way ANOVA), decreased significantly in week 8 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and remained constant until week 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 7-3).

In the MWB treatment, no significant difference was observed in soil leachate between weeks 1 to 7 ( $p > 0.05$ , Repeated measures two-way ANOVA), except for week 6, when leachate was significantly lower compared to weeks 1, 2 and 4 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). Leachate was significantly higher in weeks 1 to 7 compared to weeks 8 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). Week 8 showed significantly higher leachate than weeks 9 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), and in week 9, leachate was significantly greater than weeks 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant differences were observed between weeks 9 and 10 and weeks 10 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 7-3).

In the CB treatment, no significant difference in soil leachate was observed between weeks 1 to 3 ( $p > 0.05$ , Repeated measures two-way ANOVA), though leachate was significantly lower than in week 4 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and significantly greater than in weeks 8 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant differences were observed between weeks 1 to 3 compared to week 5 to 7 ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 4, leachate was significantly greater than in weeks 6 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant difference compared to week 5 ( $p > 0.05$ , Repeated measures two-way ANOVA). Leachate in weeks 5 to 7 was significantly greater than in weeks 8 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). And, in week 8, leachate was significantly greater than in weeks 9 to 12 ( $p \leq 0.05$ , Repeated measures two-

way ANOVA), with no significant differences observed between weeks 9 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 7-3).

In the MWH treatment, no significant difference in soil leachate was observed throughout the entire incubation period from weeks 1 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 7-3).

Summarising the general trends observed in soil leachate volume from weeks 1 to 12:

- a) The concentration of soil leachate was significantly higher in the MWH treatment, and lower in the CB treatment ( $p \leq 0.05$ , Bonferroni test). No significant difference was observed between MWB and C treatments ( $p > 0.05$ , Bonferroni test).
- b) In C, MWB and CB treatments, soil leachate volume were relatively stable from weeks 1 to 7, followed by a decrease from weeks 8 to 12.
- c) In the MWH treatment, soil leachate volume remained constant throughout the incubation period from weeks 1 to 12.

## **7.4 Effect of char treatments on nutrient concentration in soil leachate**

In this section the concentrations of primary macro-nutrients (nitrate phosphate potassium), secondary plant macro-nutrients (calcium and magnesium) and micronutrients (iron and zinc) in the soil leachates over time are presented.

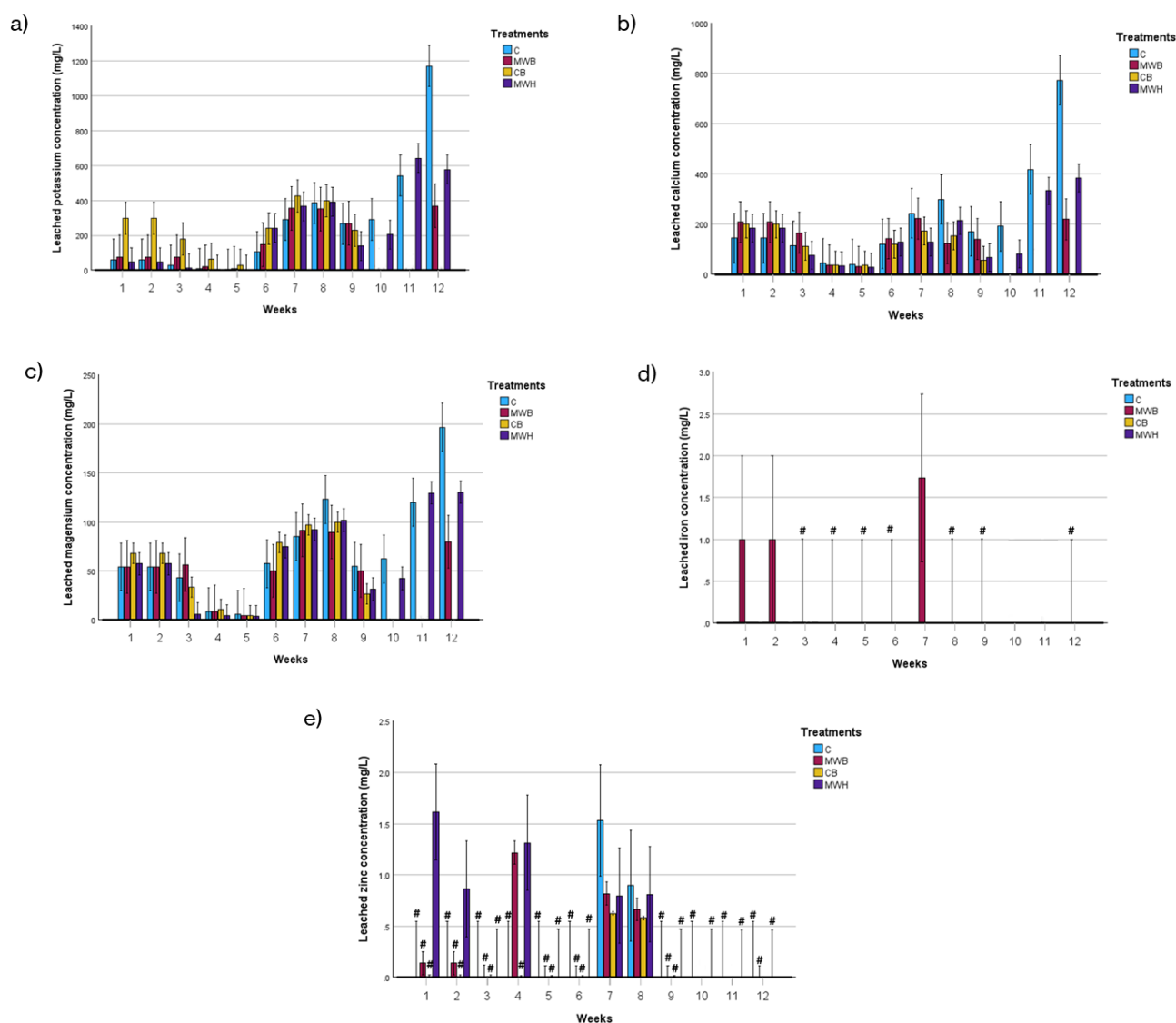
In the MWB treatment, insufficient leachate was collected for nutrient analysis in weeks 10 and 11, with a similar issue observed in CB treatment from weeks 10 to 12. Consequently, elemental concentrations during these weeks could not be measured (Figure 7-4 and 7-5). When comparing nutrient concentration across all treatments, nearly all nutrients exhibited similar trends, except for iron and zinc. The concentration of iron and zinc remained below detection limit ( $BDL < 0.5\text{mg/L}$ ) during certain weeks (Figure 7-4d and 7-4e). For potassium, calcium, magnesium, nitrate, and phosphate a concentration in the leachates decreased in week 4 compared to weeks 1 to 3. The addition of the Hoagland solution in week 5 led to an

increase in nutrient concentrations in the leachates from week 6 onwards (Figure 7-4 and 7-5). Variations observed over the incubation period are further described in the following sections.

After a detailed description of the trends, there is a summary of the key points at the end of each section.

#### **7.4.1 Leached potassium concentration**

A significant difference in leached potassium concentration was observed due to char treatment, incubation time, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA) (Figure 7-4a).



**Figure 7- 4.** Average weekly concentrations of leached cations a) potassium, b) calcium, c) magnesium, d) iron and e) zinc in control (C), microwave biochar (MWB), conventional biochar (CB) and microwave hydrochar (MWH) treated soils. Values are averages  $\pm$  standard errors,  $n=4$ . The bars in the graph represent standard errors. The # along the error bars for d) iron and e) zinc are below detection level (BDL < 0.5 mg/L) are expressed as the detection limit divided by the square root of two (Croghan & Egeghy, 2003).



In weeks 1 and 2, leached potassium was significantly greater in the CB treatment compared to C and MWH ( $p \leq 0.05$ , Bonferroni test), with no significant differences observed between other treatments ( $p > 0.05$ , Bonferroni test). Between weeks 3 to 11, no significant differences were observed between any of the treatments ( $p > 0.05$ , Bonferroni test). In week 12, leached potassium was significantly greater in C compared to MWB and MWH ( $p \leq 0.05$ , Bonferroni test) and no significant difference was observed between MWB and MWH (Figure 7-4a).

Along with the treatments, the incubation period also significantly influenced the leached potassium concentration ( $p \leq 0.001$ , Repeated measures two-way ANOVA). In C, no significant difference was observed in leached potassium from weeks 1 to 5 ( $p > 0.05$ , Repeated measures two-way ANOVA), and was significantly lower than in weeks 7, 8, 10 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant differences were observed between weeks 1 to 5 compared to weeks 6 and 9 ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 6, leached potassium was significantly lower than in weeks 8, 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant differences observed compared to weeks 7, 9 and 10 ( $p > 0.05$ , Repeated measures two-way ANOVA). There was no significant difference in leached potassium between weeks 7 to 11 ( $p > 0.05$ , Repeated measures two-way ANOVA) and the leached potassium was significantly lower than in week 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) (Figure 7-4a).

In the MWB treatment, no significant difference was observed in leached potassium between weeks 1 to 3 ( $p > 0.05$ , Repeated measures two-way ANOVA), and was significantly lower than weeks 7, 8 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant differences were observed in weeks 4 and 5 ( $p > 0.05$ , Repeated measures two-way ANOVA), and was significantly lower than in weeks 7, 8 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in leached potassium between weeks 6 to 9 and week 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 7-4a).

In the CB treatment, no significant difference was observed in leached potassium between weeks 1 and 2 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly higher than in week 4 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant differences observed in weeks 1 and 2 compared to weeks 5 to 9 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant difference in leached potassium was observed between weeks 3 and 5 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower than in weeks

6 to 9 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no further significant differences observed between weeks 6 to 9 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 7-4a).

In the MWH treatment, no significant difference was observed in leached potassium between weeks 1 and 2 ( $p > 0.05$ , Repeated measures two-way ANOVA), with significantly lower concentration than in weeks 6 to 8, 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and significantly higher than in weeks 4 and 5 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). There were no significant differences between weeks 3 and 5 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower than in weeks 6 to 8 and weeks 10 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 6, leached potassium was significantly lower than in weeks 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). Leached potassium in weeks 7 and 8 were significantly higher than in week 9 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), but lower than in weeks 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant differences were observed between weeks 9 and 10 ( $p > 0.05$ , Repeated measures two-way ANOVA) and were significantly lower compared to weeks 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no further significant difference between weeks 11 and 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 7-4a).

Summarising the general trends observed in leached potassium concentration from weeks 1 to 12:

- a) Insufficient leachate was collected for MWB during weeks 10 and 11, and for CB from weeks 10 to 12 hence, the concentration of potassium wasn't measured.
- b) The treatments in order of the highest concentration of potassium in the leachate are as follows  $C = MWH > MWB = CB$  ( $p \leq 0.05$ , Bonferroni test)
- c) During the initial watering period of wheat crop (weeks 1 to 4) using de-ionised water:
  - In all the treatments, leached potassium decreased in week 4 compared to weeks 1 to 3.
- d) From week 5 -12 with the introduction of the Hoagland solution:
  - In all the treatments, leached potassium remained lower between weeks 4 and 5.

- In the C and MWH treatment, leached potassium increased in week 6 and remained relatively constant till week 10 with a further increase in weeks 11 and 12.
- In MWB and CB treatments, leached potassium increased in week 6, followed by a decrease in week 9. In the MWB treatment, leached potassium remained constant in week 12 compared to week 9.

#### 7.4.2 Leached calcium concentration

A significant difference in leached calcium concentration was observed due to char treatment, incubation time, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA) (Figure 7-4b). Between weeks 1 to 9, no significant difference was observed in leached calcium between the treatments ( $p > 0.05$ , Bonferroni test). In weeks 10 and 11, no significant difference in leached calcium was observed between C and MWH treatment ( $p > 0.05$ , Bonferroni test). In week 12, the treatments in order of the most leached calcium are as follows: C > MWH > MWB, with significant differences between these groups ( $p \leq 0.05$ , Bonferroni test) (Figure 7-4b).

Along with the treatments, the incubation period also significantly influenced the concentration of leached calcium ( $p \leq 0.001$ , Repeated measures two-way ANOVA). In C, no significant difference in leached calcium was observed between weeks 1 to 6 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower compared to weeks 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). Additionally, leached calcium was significantly lower in weeks 3, 4 and 5 compared to week 8 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in leached calcium between weeks 7 to 11 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower than in week 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). Additionally, leached calcium was significantly lower in weeks 9 and 10 compared to week 11 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) (Figure 7-4b).

In the MWB treatment, leached calcium was significantly higher in weeks 1, 2 and 7 compared to weeks 4 and 5 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No other significant

differences were observed in leached calcium between the remaining weeks ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 7-4b).

In the CB treatment, leached calcium was significantly higher in weeks 1 and 2 compared to weeks 4, 5 and 9 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). Additionally, leached calcium was significantly higher in weeks 7 and 8 compared to weeks 4 and 5 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No other significant differences were observed in leached calcium between the remaining weeks ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 7-4b).

In the MWH treatment, no significant difference in leached calcium was observed between weeks 1 and 2 ( $p > 0.05$ , Repeated measures two-way ANOVA), however, leached calcium was significantly higher compared to weeks 4, 5 and 9 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and significantly lower than in weeks 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in leached calcium between weeks 3 to 7 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower than in weeks 8, 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), except for weeks 6, 7 and 8, where no significant differences were observed ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 8, leached calcium was significantly higher compared to weeks 9 and 10 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and significantly lower compared to weeks 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in leached calcium between weeks 9 and 10 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower than in weeks 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no further significant difference observed between weeks 11 and 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 7-4b).

Summarising the general trends observed in leached calcium concentration from weeks 1 to 12:

- a) Insufficient leachate was collected for MWB during weeks 10 and 11, and for CB from weeks 10 to 12 hence, the concentration of calcium was not measured.
- b) No significant difference was observed from weeks 1 to 9 in leached calcium ( $p > 0.05$ , Bonferroni test). The concentration of leached calcium was significantly greater in C

compared to the rest of the treatments from week 10 onwards ( $p \leq 0.05$ , Bonferroni test).

- c) During the initial watering period of wheat crop (weeks 1 to 4) using de-ionised water:
  - In all treatments, leached calcium was high in weeks 1 and 2, followed by a decrease from week 3 to week 4.
- d) From week 5 to 12 with the introduction of the Hoagland solution:
  - In the C and MWH treatment, leached calcium increased from weeks 6 to 8, decreased in week 9, with a further increase in weeks 11 to 12.
  - In MWB and CB treatments, leached calcium increased from weeks 6 to 7, followed by a decrease from week 8 to 9. In the MWB treatment, leached calcium increased in week 12 compared to week 9.

### **7.4.3 Leached magnesium concentration**

A significant difference in leached magnesium concentration was observed due to char treatment, incubation time, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA) (Figure 7-4c). During weeks 1, 2 and 4 to 9, no significant difference was observed in leached magnesium between the treatments ( $p > 0.05$ , Bonferroni test). In week 3, leached magnesium was significantly lower in MWH compared to C, MWB and CB ( $p \leq 0.05$ , Bonferroni test). In weeks 10 and 11, leached magnesium did not significantly differ between C and MWH ( $p > 0.05$ , Bonferroni test). In week 12, the leached magnesium was significantly greater in C compared to MWB and MWH ( $p \leq 0.05$ , Bonferroni test) and was significantly greater in MWH compared to MWB ( $p \leq 0.05$ , Bonferroni test) (Figure 7-4c)

Along with the treatments the incubation period also significantly influenced the concentration of leached magnesium ( $p \leq 0.001$ , Repeated measures two-way ANOVA). In C, no significant difference in leached magnesium was observed between weeks 1 to 5 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower compared to weeks 8, 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). Additionally, leached magnesium in weeks 4 and 5 was significantly lower than in weeks 7 and 10 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 6, leached magnesium was significantly lower than in weeks 8, 11

and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), and significantly higher than in week 9 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed between weeks 7 and 8 ( $p > 0.05$ , Repeated measures two-way ANOVA), however the leached magnesium was significantly higher than in weeks 9 and 10 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and was significantly lower compared to week 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). There were no significant differences between weeks 9 and 10 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower compared to weeks 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with a further increase in leached magnesium from week 11 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) (Figure 7-4c).

In the MWB treatment, no significant difference was observed in leached magnesium between weeks 4 and 5 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower compared to weeks 7, 8 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No other significant difference was observed in leached magnesium for the remaining weeks ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 7-4c)

In the CB treatment, no significant difference was observed in leached magnesium between weeks 1 and 2 ( $p > 0.05$ , Repeated measures two-way ANOVA) with significantly higher concentration than in weeks 3 to 5 and 9 and significantly lower concentration compared to weeks 7 and 8 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant differences were observed between weeks 3 and 4 ( $p > 0.05$ , Repeated measures two-way ANOVA), with significantly higher concentration than in week 5 ( $p > 0.05$ , Repeated measures two-way ANOVA) and significantly lower concentration compared to weeks 6 to 8 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 5, leached magnesium was also significantly lower compared to weeks 6 to 8 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and leached magnesium in week 6 to 8 was significantly higher than in week 9 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) (Figure 7-4c).

In the MWH treatment, no significant difference in leached magnesium was observed between weeks 1 and 2 ( $p > 0.05$ , Repeated measures two-way ANOVA), with significantly higher concentration compared to weeks 3 to 5 and 9 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and significantly lower than in weeks 7, 8, 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed between weeks 1 and 2 compared

to weeks 6 and 10 ( $p > 0.05$ , Repeated measures two-way ANOVA). There was no significant difference between leached magnesium in weeks 3 to 5 ( $p > 0.05$ , Repeated measures two-way ANOVA), but concentrations were significantly lower compared to weeks 6 to 8 and 10 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed between weeks 6 to 8 ( $p > 0.05$ , Repeated measures two-way ANOVA), with significantly higher concentration than in weeks 9 and 10 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and significantly lower concentration compared to weeks 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed between weeks 9 and 10 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower compared to weeks 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no further significant difference between weeks 11 and 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 74c)

Summarising the general trends observed in leached magnesium concentration from weeks 1 to 12:

- a) Insufficient leachate was collected for MWB during weeks 10 and 11, and for CB from weeks 10 to 12 hence, the concentration of magnesium wasn't measured.
- b) The treatments in order of the highest concentration of magnesium in the leachate are as follows: C > MWH > MWB = CB ( $p \leq 0.05$ , Bonferroni test).
- c) During the initial watering period of wheat crop (weeks 1 to 4) using de-ionised water:
  - In C, CB and MWH treatments, leached magnesium remained constant in weeks 1 and 2, followed by a decrease from week 3 to week 4.
  - In the MWB treatment, leached magnesium remained constant from weeks 1 to 3, with a decrease observed in week 4.
- d) From week 5 -12 with the introduction of the Hoagland solution:
  - In the C and MWH treatment, leached magnesium increased from weeks 6 to 8, decreased in week 9, and then increased again from weeks 10 to 12.
  - In the MWB and CB treatments, leached magnesium increased in weeks 6 and 7, followed by a decrease in week 9. In MWB treatment, leached magnesium in week 12 increased compared to week 9.

#### 7.4.4 Leached iron concentration

In weeks 1, 2 and 7, leached iron was significantly greater in MWB compared to the rest of the treatments ( $p \leq 0.05$ , Bonferroni test). In C and MWH treatments, the concentration of leached iron was below detection limits ( $BDL \leq 0.5\text{mg/L}$ ) throughout the incubation period between weeks 1 to 12 and in CB treatment from weeks 1 to 9, leached iron was BDL. Hence, no significant difference was observed between C, CB and MWH throughout the incubation period ( $p > 0.05$ , Bonferroni test) (Figure 7-4d).

The incubation period also significantly influenced the concentration of leached iron for MWH treatment ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In MWB treatment, leached iron was significantly higher in weeks 1, 2 and 7 compared to the rest of the weeks ( $p \leq 0.05$ , Repeated measures two-way ANOVA), and the concentration of leached iron was BDL for the remaining weeks. (Figure 7-4d). The concentration of leached iron in C, CB and MWH was BDL throughout the incubation period from week 1 to week 12, hence no significant difference was observed from weeks 1 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 7-4d).

#### 7.4.5 Leached zinc concentration

A significant difference in leached zinc concentration was observed due to char treatment, incubation time, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA, Repeated measures two-way ANOVA) (Figure 7-4e). In weeks 1 and 2, leached zinc was significantly greater in MWH treatment compared to the other treatments ( $p \leq 0.05$ , Bonferroni test), and no other significant differences were observed between the other treatments ( $p > 0.05$ , Bonferroni test). In week 4, no significant difference was observed in leached zinc between MWB and MWH treatments ( $p > 0.05$ , Bonferroni test) and were significantly greater than C and CB ( $p \leq 0.05$ , Bonferroni test). No significant difference was observed between C and CB treatments in week 4 because they were BDL ( $p > 0.05$ , Bonferroni test). In week 7, leached magnesium was significantly greater in C compared to



MWB and CB ( $p \leq 0.05$ , Bonferroni test), and no other significant differences were observed between other treatments ( $p > 0.05$ , Bonferroni test). No other significant differences were observed between the treatments for the remaining weeks ( $p > 0.05$ , Bonferroni test) (Figure 7-4e).

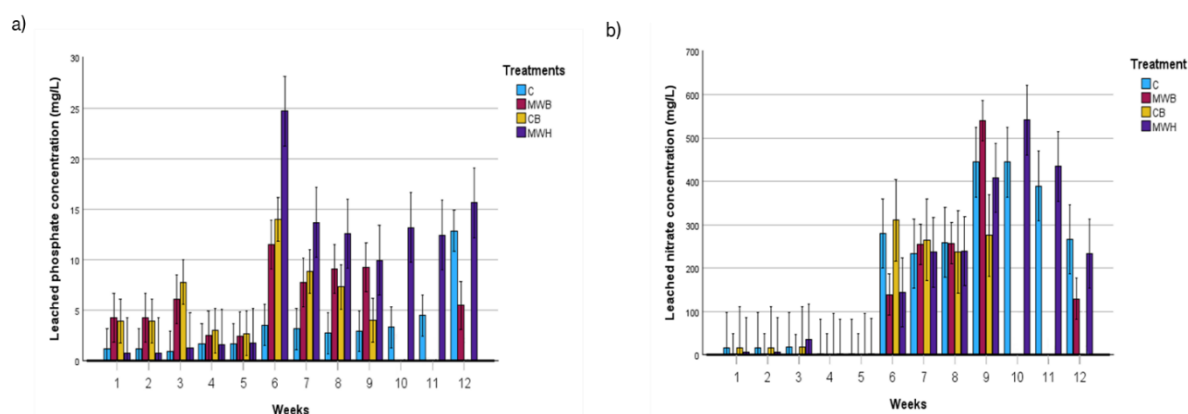
Along with the treatments, the incubation period also significantly influenced the concentration of leached zinc ( $p \leq 0.001$ , Repeated measures two-way ANOVA). For C, no significant difference was observed between weeks 7 and 8 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly higher than rest of the weeks ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In weeks 1 to 6 and 9 to 12, the concentration of leached zinc was below detection limit ( $BDL \leq 0.5\text{mg/L}$ ) (Figure 7-4e).

For MWB and CB treatment, no significant difference was observed in leached zinc between weeks 7 and 8 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly higher than rest of the weeks ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In weeks 1 to 6, 9 and 12, the concentration of leached zinc was BDL (Figure 7-4e).

For MWH treatment, no significant difference was observed in leached zinc between weeks 1, 2, 7 and 8 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly higher than rest of the weeks ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In weeks 3 to 6 and 9 to 12, leached zinc was BDL (Figure 7-4e).

#### **7.4.6 Leached phosphate concentration**

A significant difference in leached phosphate concentration was observed due to char treatment, incubation time, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA) (Figure 7-5a).



**Figure 7- 5.** Average weekly concentrations of leached anions a) phosphate and b) nitrate in control (C), microwave biochar (MWB), conventional biochar (CB) and microwave hydrochar (MWH) treated soils. Values are averages  $\pm$  standard errors,  $n=4$ . The bars in the graph represent standard errors.

In weeks 1, 2, 4 and 5, no significant difference in leached phosphate was observed between the treatments ( $p > 0.05$ , Bonferroni test). In weeks 3 and 8, the leached phosphate was significantly lower in C compared to MWB and CB ( $p \leq 0.05$ , Bonferroni test) and additionally, in week 3, the concentration was significantly greater in CB compared to MWH ( $p \leq 0.05$ , Bonferroni test). No other significant differences were observed between other treatments ( $p > 0.05$ , Bonferroni test). In weeks 6 and 7, leached phosphate was significantly lower in C compared to MWB, CB and MWH ( $p \leq 0.05$ , Bonferroni test) and additionally, in week 6, leached phosphate was significantly greater in MWH compared to MWB and CB ( $p \leq 0.05$ , Bonferroni test). No other significant differences were observed between C and MWB ( $p > 0.05$ , Bonferroni test). In week 9, leached phosphate was significantly lower in C compared to MWB and MWH ( $p \leq 0.05$ , Bonferroni test) and significantly lower in CB compared to MWB and MWH ( $p \leq 0.05$ , Bonferroni test). No significant differences were observed between C and CB, MWH and MWB ( $p > 0.05$ , Bonferroni test). In weeks 10 and 11, leached phosphate was significantly greater in MWH compared to C ( $p \leq 0.05$ , Bonferroni test). In week 12, leached

phosphate was significantly lower in MWB compared to C and MWH ( $p \leq 0.05$ , Bonferroni test) and no significant difference was observed between C and MWH ( $p > 0.05$ , Bonferroni test) (Figure 7-5a).

Along with the treatments, the incubation period also significantly influenced the concentration of leached phosphate ( $p \leq 0.001$ , Repeated measures two-way ANOVA). In C, no significant difference was observed in leached phosphate between weeks 1 to 11 ( $p > 0.05$ , Repeated measures two-way ANOVA), and was significantly lower compared to week 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) (Figure 7-5a).

In the MWB treatment, no significant difference was observed in leached phosphate in weeks 1 and 2 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower than in week 6 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 3, leached phosphate was significantly lower compared to week 6 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no further significant differences observed between the rest of the weeks ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in leached phosphate between weeks 4 and 5 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower compared to weeks 6 to 9 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed between weeks 6 to 9 ( $p > 0.05$ , Repeated measures two-way ANOVA), and the concentration of leached phosphate was significantly lower in week 12 compared to week 6 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No further differences were observed between weeks 7 to 9 compared to week 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 7-5a).

In the CB treatment, no significant difference was observed in leached phosphate in weeks 1 and 2 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower than in week 6 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no other significant difference observed with the remaining weeks ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 3, leached phosphate was significantly higher than in weeks 4 and 5 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and significantly lower compared to week 6 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant differences were observed between weeks 4 and 5 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower compared to weeks 6 and 7 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 6, leached phosphate was significantly higher than in weeks 7 to 9 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in leached phosphate between weeks 7 to 9 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 7-5a).

In the MWH treatment, no significant difference in leached phosphate was observed between weeks 1 to 5 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower compared to weeks 6 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). In week 6, leached phosphate was significantly higher compared to weeks 7 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no further significant differences between weeks 7 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 7-5a).

Summarising the general trends observed in leached phosphate concentration from weeks 1 to 12:

- a) Insufficient leachate was collected for MWB during weeks 10 and 11, and for CB from weeks 10 to 12 hence, the concentration of phosphate was not measured.
- b) The treatments in order of the highest concentration of phosphate in the leachate are as follows:  $MWH > CB = MWB > C$  ( $p \leq 0.05$ , Bonferroni test).
- c) During the initial watering period of wheat crop (weeks 1 to 4) using de-ionised water:
  - In the C and MWH treatment, leached phosphate remained stable from weeks 1 to 4.
  - In MWB and CB, leached phosphate was constant during weeks 1 and 2, increased in week 3, and then further decreased in week 4.
- d) From week 5 -12 with the introduction of the Hoagland solution:
  - Leached phosphate remained constant across all treatments during weeks 4 and 5.
  - In C, leached phosphate increased in week 6, remained constant until week 11, and then increase further in week 12.
  - In the MWB treatment, leached phosphate increased in week 6, decreased in week 7, and remained constant until week 9. Leached phosphate in week 12 was lower than in weeks 6 to 9.
  - In the CB treatment, leached phosphate increased in week 6 and decreased from week 7 onwards to week 9.
  - In the MWH treatment, leached phosphate increased in week 6, decreased in week 7, and remained constant till week 12.

#### 7.4.7 Leached nitrate concentration

A significant difference in leached nitrate concentration was observed due to char treatment, incubation time, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA, Repeated measures two-way ANOVA) (Figure 7-5b). In weeks 1 to 5, 7 and 8, no significant difference was observed in leached nitrate between any of the treatments ( $p > 0.05$ , Bonferroni test). In week 6, leached nitrate was significantly lower in MWB compared to C and CB ( $p \leq 0.05$ , Bonferroni test) and no other significant differences were observed between the treatments ( $p > 0.05$ , Bonferroni test). In week 9, leached nitrate was significantly higher in MWB compared to CB and MWH treatments ( $p \leq 0.05$ , Bonferroni test) and no other significant differences were observed between the treatments ( $p > 0.05$ , Bonferroni test). In weeks 10 and 11, no significant difference in leached nitrate was observed between MWH and C ( $p > 0.05$ , Bonferroni test). In week 12, leached nitrate was significantly greater in C compared to MWB ( $p \leq 0.05$ , Bonferroni test) (Figure 7-5b).

Along with the treatments, the incubation period also significantly influenced the concentration of leached nitrate ( $p \leq 0.001$ , Repeated measures two-way ANOVA). In C, leached nitrate between weeks 1 to 5 did not differ significantly ( $p > 0.05$ , Repeated measures two-way ANOVA), but they were significantly lower than in weeks 6 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). Leached nitrate in weeks 6 to 8 showed no significant difference ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower compared to weeks 9 and 10 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant differences compared to weeks 11 and 12 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in leached nitrate between weeks 9 to 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) and leached nitrate was significantly lower in week 12 compared to weeks 9 and 10 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed between weeks 11 and 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 7-5b).

In the MWB treatment, no significant difference in leached nitrate was observed between weeks 1 to 5 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower than in weeks 6 to 9 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant

differences compared to week 10 ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 6, leached nitrate was significantly lower than in weeks 7 to 9 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and no significant difference was observed compared to week 12 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed between weeks 7 and 8 ( $p > 0.05$ , Repeated measures two-way ANOVA) with significantly lower concentration than in week 9 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and significantly higher concentration compared to week 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). And in week 9, leached nitrate was also significantly higher compared to week 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) (Figure 7-5b).

In the CB treatment, no significant difference in leached nitrate was observed between weeks 1 to 5 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower than in weeks 6 to 9 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in leached nitrate between weeks 6 to 9 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 7-5b).

In the MWH treatment, no significant difference in leached nitrate was observed between weeks 1 to 5 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower compared to weeks 7 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) with no significant difference compared to week 6 ( $p > 0.05$ , Repeated measures two-way ANOVA). Similarly, no significant difference was observed in leached nitrate between weeks 6 to 8 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower than in weeks 9 to 11 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed between weeks 9 to 11 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly higher than in week 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) (Figure 7-5b).

Summarising the general trends observed in leached nitrate concentration from weeks 1 to 12:

- a) Insufficient leachate was collected for MWB during weeks 10 and 11, and for CB from weeks 10 to 12 hence, the concentration of nitrate wasn't measured.
- b) Leached nitrate concentration were significantly lower in CB treatment compared to C, MWB and MWH treatment ( $p \leq 0.05$ , Bonferroni test) and no other significant difference was observed between other treatments ( $p > 0.05$ , Bonferroni test)

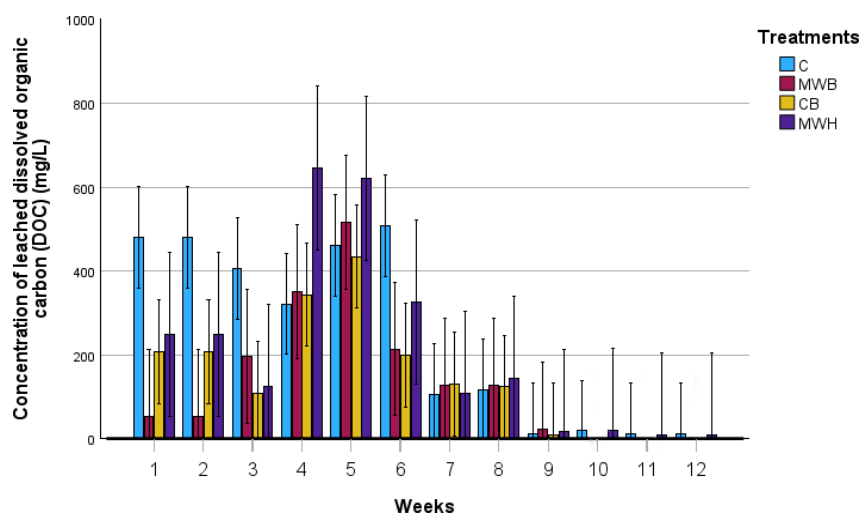
- c) During the initial watering period of wheat crop (weeks 1 to 4) using de-ionised water:
  - In all treatments, leached nitrate concentrations were low and remained constant from weeks 1 to 4.
- d) From week 5 -12 with the introduction of the Hoagland solution:
  - Leached nitrate remained constant in all treatments during weeks 4 and 5.
  - In the C and MWH treatment, leached nitrate increased in week 6, with a further increase in week 9, and decreased from week 11 to 12.
  - In the MWB treatment, leached nitrate increased in week 6, with a further increase in weeks 7 and 9. The leached nitrate in week 12 was lower compared to weeks 7 to 9.
  - In the CB treatment, leached nitrate increased in week 6 and remained constant through week 9.

## **7.5 Effect of char treatments on the concentration of dissolved organic carbon (DOC) in soil leachate**

In the MWB and CB treatments, the volume of leachate collected in weeks 10 and 12 was insufficient for the DOC analysis. Consequently, elemental concentrations during these weeks could not be measured. After a detailed description of the trends, there is a summary of the key points at the end of this section.

A significant difference in the concentration of leached DOC was observed due to char treatment, incubation time, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA) (Figure 7-6).





**Figure 7- 6.** Average weekly concentration of leached dissolved organic carbon (DOC) in control (C), microwave biochar (MWB), conventional biochar (CB) and microwave hydrochar (MWH) treated soils. Values are averages  $\pm$  standard errors,  $n=4$ . The bars in the graph represents standard errors.

In weeks 1 and 2, leached DOC was significantly greater in C compared to MWB and CB ( $p \leq 0.05$ , Bonferroni test), with no other significant differences observed between the rest of the treatments ( $p > 0.05$ , Bonferroni test). In week 3, leached DOC was significantly greater in C compared to CB ( $p \leq 0.05$ , Bonferroni test), with no other differences observed between other treatments ( $p > 0.05$ , Bonferroni test). In week 6, leached DOC was significantly greater in C compared to CB and MWB ( $p \leq 0.05$ , Bonferroni test), with no other significant differences were observed between other treatments ( $p > 0.05$ , Bonferroni test). In weeks 4, 5 and 7 to 12, no significant differences were observed between the treatments ( $p > 0.05$ , Bonferroni test) (Figure 7-6)

Along with the treatments, the incubation period also significantly influences the concentration of leached DOC ( $p \leq 0.001$ , Repeated measures two-way ANOVA). In C, no significant difference in leached DOC was observed between weeks 1 to 6 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly compared to weeks 7 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) (Figure 7-6).

In the MWB treatment, leached DOC in week 5 was significantly higher compared to weeks 1, 2 and 7 to 9 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No other significant differences were observed in DOC concentration between the remaining weeks ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 7-6).

In the CB treatment, leached DOC was significantly higher in week 4 compared to week 9 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), and in week 5, leached DOC was significantly higher compared to weeks 3 and 7 to 9 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No other significant differences were observed between the remaining weeks ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 7-6).

In the MWH treatment, leached DOC was significantly higher in weeks 4 and 5 compared to weeks 3 and 7 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No other significant differences were observed between the remaining weeks ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 7-6).

Summarising the general trends observed in leached DOC concentration from weeks 1 to 12:

- a) In MWB and CB, no leachates were collected between weeks 10 to 12, hence the concentration of DOC was not measured.
- b) Concentration of leached DOC was significantly higher in C compared to CB and MWB treatments in weeks 1 and 2 ( $p \leq 0.05$ , Bonferroni test) and no significant difference was observed between treatments from week 3 onwards ( $p > 0.05$ , Bonferroni test),
- c) In C, leached DOC remained stable from weeks 1 to 6 and decreased from weeks 7 to 12.
- d) In the MWB treatment, DOC concentration increased from week 3 to 5, followed by a decrease from weeks 6 to 9.
- e) In the CB and MWH treatments, leached DOC observed an increase in weeks 4 and 5, then decreased from weeks 6 to 9. The MWH treatment saw a further decrease from weeks 9 to 12.

## **7.6 Effect of char treatments on soil physical structure and chemistry at the end of 12-week experiment period**

Addition of different char to the soil can affect soil porosity, soil CEC, pH, and nutrient concentration on the soil exchange sites. This section examines how different char treatments impact physical structure and chemical properties of soil.

### **7.6.1 Effect of char treatment on soil porosity**

The soil porosity was significantly lower in C compared to MWB, CB and MWH treated soils ( $p \leq 0.05$ , one-way ANOVA) and the soil porosity was significantly higher in MWB compared to MWH and CB ( $p \leq 0.05$ , one-way ANOVA). No significant difference was observed in soil porosity between CB and MWH ( $p > 0.05$ , one-way ANOVA) (Table 7-2).

**Table 7- 2.** Physical and chemical soil properties for control (C), microwave biochar (MWB), conventionally biochar (CB) and microwave hydrochar (MWH) treatments. Data are presented as mean  $\pm$  standard error (n=4). Values across each column denoted by different superscripts <sup>a, b and c</sup> were found to be significantly different from one another according to Tukey's test ( $p < 0.05$ ).

Properties	C soil	MWB treated soil	CB treated soil	MWH treated soil
Soil porosity ( $\text{cm}^3 \text{cm}^{-3}$ )	$0.8577 \pm 5 \times 10^{-6} \text{ }^a$	$0.8723 \pm 10 \times 10^{-5} \text{ }^b$	$0.8692 \pm 5 \times 10^{-6} \text{ }^c$	$0.8678 \pm 6 \times 10^{-6} \text{ }^c$
Soil nitrate (mol/kg)	$6.3 \times 10^{-6} \pm 1.2 \times 10^{-6} \text{ }^a$	$1.9 \times 10^{-6} \pm 4.9 \times 10^{-6} \text{ }^a$	$9.1 \times 10^{-6} \pm 4.1 \times 10^{-6} \text{ }^b$	$2.8 \times 10^{-7} \pm 1.1 \times 10^{-8} \text{ }^c$
Soil ammonium (mol/kg)	$4.1 \times 10^{-6} \pm 4.1 \times 10^{-7} \text{ }^a$	$4.8 \times 10^{-6} \pm 8.6 \times 10^{-7} \text{ }^a$	$4.7 \times 10^{-6} \pm 7.2 \times 10^{-7} \text{ }^a$	$1.8 \times 10^{-6} \pm 1.5 \times 10^{-6} \text{ }^b$
Soil CEC (cmol/kg)	$4.78 \pm 0.44 \text{ }^a$	$4.45 \pm 0.33 \text{ }^a$	$11.08 \pm 1.14 \text{ }^b$	$5.85 \pm 0.37 \text{ }^a$
Soil pH	$7.63 \pm 0.07 \text{ }^a$	$7.81 \pm 0.02 \text{ }^a$	$8.00 \pm 0.02 \text{ }^a$	$7.88 \pm 0.01 \text{ }^a$
Concentration of potassium on soil exchange sites (mol/kg)	$0.0011 \pm 0.0004 \text{ }^a$	$0.0011 \pm 0.0005 \text{ }^a$	$0.013 \pm 0.0077 \text{ }^b$	$0.0023 \pm 0.0012 \text{ }^a$
Concentration of calcium on soil exchange sites (mol/kg)	$0.401 \pm 0.05 \text{ }^a$	$0.217 \pm 0.02 \text{ }^a$	$0.311 \pm 0.09 \text{ }^a$	$0.209 \pm 0.03 \text{ }^a$
Concentration of magnesium on soil exchange sites (mol/kg)	$0.005 \pm 0.0004 \text{ }^a$	$0.004 \pm 0.0003 \text{ }^a$	$0.004 \pm 0.0011 \text{ }^a$	$0.003 \pm 0.0002 \text{ }^a$
Concentration of iron on soil exchange sites (mol/kg)	$0.002 \pm 0.0002 \text{ }^a$	$0.002 \pm 0.0001 \text{ }^a$	$0.002 \pm 0.0005 \text{ }^a$	$0.001 \pm 0.0008 \text{ }^a$
Concentration of phosphate on soil exchange sites (mol/kg)	$0.0002 \pm 0.00004 \text{ }^a$	$0.0002 \pm 0.00001 \text{ }^a$	$0.0003 \pm 0.00004 \text{ }^a$	$0.0001 \pm 0.000005 \text{ }^b$

### **7.6.2 Effect of char treatments on nitrate and ammonium concentration on soil exchange sites**

The soil nitrate was significantly higher in CB compared to rest of the treatments ( $p \leq 0.05$ , Kruskal-Wallis test) and significantly lower in MWH treatment ( $p < 0.05$ , Kruskal-Wallis test). No significant difference was observed on soil nitrate between C and MWB treatments ( $p > 0.05$ , Kruskal-Wallis test) (Table 7-2). Soil ammonium concentration was significantly lower in MWH treatment compared to the rest of the treatments ( $p < 0.05$ , one-way ANOVA) and no difference was observed between C, MWB and CB treatments ( $p > 0.05$ , one-way ANOVA) (Table 7-2). The concentration of the ammonium and nitrate in soil were positively correlated ( $n = 16$ ,  $r = 0.235$ ,  $p < 0.05$ , Pearson correlation analysis) implying when the concentration of ammonium goes up, nitrate levels also go up and vice versa.

### **7.6.3 Effect of biochar treatments on soil cationic exchange capacity (CEC) and pH**

The CB treatment significantly increased the soil CEC compared to the other treatments ( $p \leq 0.05$ , one-way ANOVA). No significant difference was observed in the soil pH between the treatments ( $p > 0.05$ , one-way ANOVA) (Table 7-2). Furthermore, a significant positive correlation ( $n = 16$ ,  $r = 0.574$ ,  $p \leq 0.05$ , Pearson correlation analysis) was observed between soil pH and CEC.

### **7.6.4 Effect of char treatments on soil exchangeable cations**

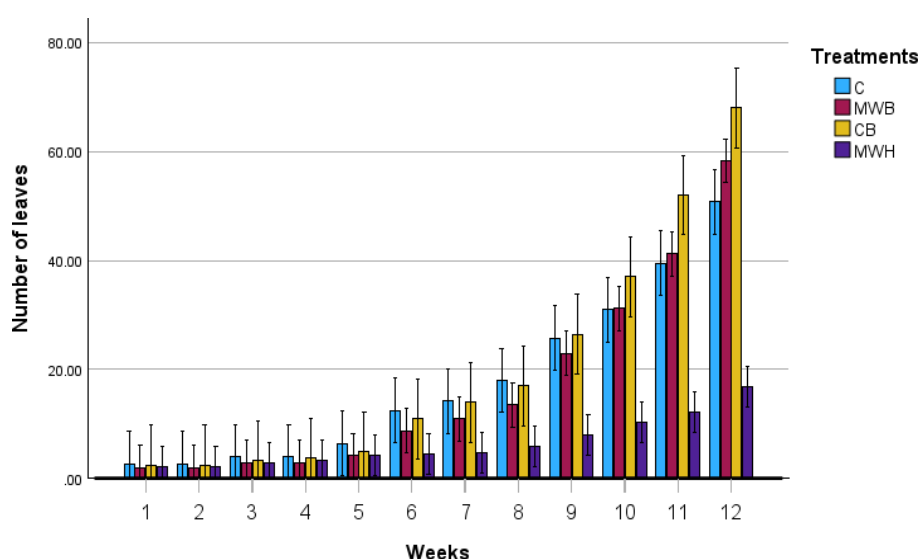
The concentration of potassium on the exchange sites was significantly higher in CB treated soil compared to C, MWB and MWH treated soils ( $p \leq 0.05$ , Kruskal-Wallis test) and the concentration of phosphorus on the exchange site was significantly lower in MWH treated soil compared to C, MWB and CB treated soils ( $p \leq 0.05$ , Kruskal-Wallis test). No other significant differences were observed in the concentration of calcium, magnesium, iron on

the exchange sites in the soil between the treatments ( $p > 0.05$ , one-way ANOVA). The concentration of zinc on the exchange sites in the soil was BDL ( $\text{BDL} \leq 0.5\text{mg/L}$ ) for all the treatments (Table 7-2).

## 7.7 Effect of char treatments on leaf number and biomass yield

This section investigates the impact of different char treatments on plant growth, specifically focusing on the number of leaves and biomass. After a detailed description of the trends, there is a summary of the key points at the end of this section.

A significant difference in the number of leaves was observed across biochar treatments, incubation times, and their interaction ( $p \leq 0.001$ , Repeated measures two-way ANOVA, Repeated measures two-way ANOVA) (Figure 7-7).



**Figure 7- 7.** Average number of leaves on wheat plants in the control (C), microwave biochar (MWB), conventional biochar (CB) and microwave hydrochar (MWH) treated soils. Values are averages  $\pm$  standard errors,  $n=4$ . The bars in the graph represents standard errors.

From weeks 1 to 7, no significant difference was observed in the number of leaves between the treatments ( $p > 0.05$ , Bonferroni test). In week 8, the number of leaves were significantly lower in MWH treatment compared to C ( $p \leq 0.05$ , Bonferroni test), with no other significant differences between the rest of the treatments ( $p > 0.05$ , Bonferroni test). In weeks 9 to 11, the number of leaves were significantly lower in MWH compared to C, MWB and CB ( $p \leq 0.05$ , Bonferroni test), with no other significant differences between the rest of the treatments ( $p > 0.05$ , Bonferroni test). In week 12, the number of leaves were significantly lower in MWH compared to C, MWB and CB ( $p \leq 0.05$ , Bonferroni test) and significantly greater in CB compared to C ( $p \leq 0.05$ , Bonferroni test). No other significant differences were observed between the rest of the treatments in week 12 ( $p > 0.05$ , Bonferroni test) (Figure 7-7).

Along with the treatments, the incubation period also significantly influenced the number of leaves ( $p \leq 0.001$ , Repeated measures two-way ANOVA). In C, no significant difference was observed in the number of leaves between weeks 1 to 5 ( $p > 0.05$ , Repeated measures two-way ANOVA), with significantly lower number of leaves than in weeks 8 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in the number of leaves in weeks 6 and 7 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower than in weeks 9 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant difference compared to week 8 ( $p > 0.05$ , Repeated measures two-way ANOVA). In week 8, the number of leaves was significantly lower than in weeks 10 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and no significant difference was observed compared to week 9 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in the number of leaves between weeks 9 and 10 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower than week 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA), with no significant difference observed compared to week 11 ( $p > 0.05$ , Repeated measures two-way ANOVA). No further difference was observed in weeks 11 and 12 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 7-7).

In the MWB treatment, there was no significant difference in the number of leaves between weeks 1 to 4 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower than in weeks 8 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant differences were observed in the number of leaves between weeks 5 to 7 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower compared to weeks 8 to 12 ( $p \leq 0.05$ , Repeated

measures two-way ANOVA). From week 8 onwards the number of leaves increased significantly till week 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) (Figure 7-7).

In the CB treatment, no significant difference in the number of leaves was observed between weeks 1 to 6 ( $p > 0.05$ , Repeated measures two-way ANOVA) and was significantly lower than in weeks 9 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in the number of leaves between the weeks 7 and 8 ( $p > 0.05$ , Repeated measures two-way ANOVA) but was significantly lower compared to weeks 10 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in the number of leaves in the weeks 9 and 10 ( $p > 0.05$ , Repeated measures two-way ANOVA), with significantly lower number of leaves compared to weeks 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and the number of leaves significantly increased from weeks 11 to 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) (Figure 7-7).

In the MWH treatment, no significant difference was observed in the number of leaves between weeks 1 to 7 ( $p > 0.05$ , Repeated measures two-way ANOVA) with significantly lower number of leaves than in weeks 11 and 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and no significant difference was observed in weeks 1 to 7 compared to weeks 8 to 10 ( $p > 0.05$ , Repeated measures two-way ANOVA). No significant difference was observed in the number of leaves between weeks 8 to 9 ( $p > 0.05$ , Repeated measures two-way ANOVA) with significantly lower number of leaves than in week 12 ( $p \leq 0.05$ , Repeated measures two-way ANOVA) and no difference was observed compared to weeks 10 and 11 ( $p > 0.05$ , Repeated measures two-way ANOVA) (Figure 7-7).

Summarising the general trends observed in the number of leaves from weeks 1 to 12:

- a) No significant difference in the number of leaves was observed between treatments from weeks 1 to 5. From week 6 to 12, the number of leaves in the MWH treatment was lower compared to the other treatments.
- b) During the initial watering period of wheat crop (weeks 1 to 4) using de-ionised water:
  - The number of leaves remained constant across all treatments.
- c) From week 5 -12 with the introduction of the Hoagland solution:
  - In C, MWB and CB treatments, the number of leaves increased from weeks 6 to 12.



- In MWH treatment, the number of leaves remained constant until week 8, with an increase observed from week 9 to 12.

In comparison between the treatments, the above ground biomass was significantly greater in CB treatment and the lowest in MWH treatment ( $p \leq 0.05$ , One-way ANOVA). No significant difference was found between C and MWB treatments for the above ground biomass ( $p > 0.05$ , One-way ANOVA) (Table 7-3). No significant difference was found between the treatments for below ground biomass ( $p > 0.05$ , One-way ANOVA) (Table 7-3).

**Table 7- 3.** Average aboveground and belowground biomass after the plant-pot experiment for control (C), microwave biochar (MWB), conventional biochar (CB) and microwave hydrochar (MWH). Data are the mean  $\pm$  standard deviation (n=4). Values within each column denoted by different superscripts <sup>a</sup>, <sup>b</sup> and <sup>c</sup> were found to be significantly different from one another according to Tukey's test ( $p \leq 0.05$ ).

Treatments	Above ground biomass (g)	Below ground biomass (g)
<b>C</b>	5.25 $\pm$ 0.47 <sup>a</sup>	4.75 $\pm$ 1.03 <sup>a</sup>
<b>MWB</b>	5.95 $\pm$ 0.15 <sup>a</sup>	6.25 $\pm$ 1.47 <sup>a</sup>
<b>MWH</b>	3.09 $\pm$ 0.39 <sup>b</sup>	4.50 $\pm$ 1.5 <sup>a</sup>
<b>CB</b>	7.12 $\pm$ 0.39 <sup>c</sup>	8.00 $\pm$ 0.63 <sup>a</sup>

## 7.8. Effect of char treatments on nutrient concentration at exchange sites in wheat biomass

Chars are known to influence soil nutrient availability, which in turn can impact the nutrient concentration in the crops. This section reports how different char treatments affects the nutrient concentration on the exchange sites in wheat biomass. Concentration of Potassium, calcium and magnesium in wheat biomass were significantly lower in MWH treatment compared to the C, MWB and CB treatments ( $p \leq 0.05$ , one-way ANOVA) (Table 7-4). No other significant differences were observed between the rest of the treatments ( $p > 0.05$ , one-way ANOVA). Iron concentration on the exchange sites in the wheat biomass was significantly

lower in CB treatment compared to the C, MWB and MWH treatments ( $p \leq 0.05$ , one-way ANOVA), with no significant differences observed with the rest of the treatments ( $p > 0.05$ , one-way ANOVA). No significant differences were found between treatments for zinc concentration ( $p > 0.05$ , one-way ANOVA) and phosphorus concentration ( $p > 0.05$ , Kruskal-Wallis test) on the exchange sites in the wheat biomass (Table 7-4).

**Table 7- 4.** Average nutrient concentration in wheat biomass after the plant-pot experiment for control (C), microwave biochar (MWB), conventional biochar (CB) and microwave hydrochar (MWH). Data are the mean values of 4 replicates. Values across each column denoted by different superscripts a, b and c were found to be significantly different from one another according to Tukey's test ( $p < 0.05$ , one-way ANOVA).

Properties	C soil	MWB treated soil	CB treated soil	MWH treated soil
Potassium (mol/kg)	$7.2 \times 10^{-5} \text{ a}$	$7.1 \times 10^{-5} \text{ a}$	$7.5 \times 10^{-5} \text{ a}$	$1.7 \times 10^{-5} \text{ b}$
Calcium (mol/kg)	$4.3 \times 10^{-5} \text{ a}$	$4.5 \times 10^{-5} \text{ a}$	$5.3 \times 10^{-5} \text{ a}$	$1.9 \times 10^{-5} \text{ b}$
Magnesium (mol/kg)	$5.6 \times 10^{-5} \text{ a}$	$5.2 \times 10^{-5} \text{ a}$	$5.4 \times 10^{-5} \text{ a}$	$1.7 \times 10^{-5} \text{ b}$
Iron (mol/kg)	$7.6 \times 10^{-7} \text{ a}$	$7.8 \times 10^{-7} \text{ a}$	$4.2 \times 10^{-7} \text{ b}$	$8.2 \times 10^{-7} \text{ a}$
Zinc (mol/kg)	$2.2 \times 10^{-6} \text{ a}$	$2.3 \times 10^{-6} \text{ a}$	$3.3 \times 10^{-6} \text{ a}$	$2.1 \times 10^{-6} \text{ a}$
Phosphorus (mol/kg)	$3.2 \times 10^{-6} \text{ a}$	$1.2 \times 10^{-7} \text{ a}$	$3.2 \times 10^{-6} \text{ a}$	$5.1 \times 10^{-7} \text{ a}$

## **Chapter 8: Discussion**

### **8.1 Introduction**

The application of char as a soil amendment has gained significant attention due to its potential to enhance soil fertility, retain nutrients and improve plant growth. Chars have the potential to enhance soil fertility, but their efficacy depends on factors like production method and soil characteristics (Beusch, 2021; Tan, 2023; Kandel et al., 2021; Irfan, 2017). This chapter examines the effects of potato peel derived chars produced via microwave pyrolysis pure biochar (PP-PB), microwave partially charred biochar (PP-PCB), conventional biochar (PP-CB) and microwave hydrochar (PP-MWH), alongside palm waste derived chars via microwave biochar (PW-MWB), conventional biochar (PW-CB) and microwave hydrochar (PW-MWH).

The initial section (Section 8.2) focuses on how these chars from different production techniques impact soil leachate volume, nutrient leaching, soil properties and plant growth. The later section (Section 8.3) explores the influence of different feedstocks on char properties, soil leachate, nutrient leaching, soil properties and plant growth. The study highlights the importance of production techniques and feedstock types in determining char effectiveness. Comparisons between potato peel and palm waste chars provided valuable insights into their application for sustainable agriculture.

### **8.2 Char characterisation**

#### **a) Carbon content**

In the potato peel study, PP-PB and PP-CB chars exhibited higher carbon content compared to PP-PCB and PP-MWH chars (Table 5-1 and 6-1). This increase in carbon content is attributed

to the decomposition and volatilisation of oxygen and hydrogen rich compounds during pyrolysis, resulting in the formation of stable aromatic carbons (Li et al., 2023). This process is evident in the TGA graphs for PP-PCB and PP-MWH chars (Figure 5-2 and 6-2), which show significant weight loss between 300 – 400 °C, corresponding to the release of volatiles. In contrast, PP-PB and PP-CB chars observed minimal weight loss within the same temperature range, reflecting their higher stability (Yi et al., 2012). PP-PB and PP-CB chars would have been subjected to higher pyrolysis temperature or longer durations compared to PP-MWH char. Higher temperature enhanced thermal decomposition and aromatisation, leaving behind a more carbon rich structure (Nguyen et al., 2010). Although PP-PB and PP-CB chars showed reduced weight loss compared to PP-PCB and PP-MWH, the overall thermal stability of these chars remains relatively low when compared to typical commercial biochars. Commercial biochars, especially those produced at higher temperatures (>600°C), often exhibit weight losses below 20% during TGA analysis, reflecting higher aromatic carbon and lower proportion of labile compounds (Almutairi et al., 2023 and Nan et al., 2021)

Similarly, in the palm waste study, PW-CB char had the highest carbon content, while PW-MWH char showed the lowest (Table 7-1). This trend is consistent with the TGA analysis (Figure 7-2) where PW-MWH char showed greater weight loss, indicating the release of a lower proportion of volatile compounds, along with low levels of aromatisation in PW-MWH during hydrolysis further contributed to its reduced carbon percentage compared to PW-CB char. Similarly, the carbon content was found to be lower in PW-MWB char compared to PW-CB char but higher than PW-MWH char, reflecting intermediate levels of volatile release and aromatisation during its production process.

### **c) pH**

In the potato peel study, PP-PB and PP-CB char samples exhibited significantly higher pH compared to PP-PCB and PP-MWH chars (Table 5-1 and 6-1). The increase alkalinity of these chars can be attributed to the presence of high concentrations of potassium and calcium on the exchange sites, which are more concentrated in PP-PB and PP-CB chars. The higher concentration of these minerals in PP-PB and PP-CB compared to PP-PCB and PP-MWH chars (Table 5-1 and 6-1) supports the observed higher pH, as these alkaline compounds contribute

to the overall alkalinity of the chars (Tomczyk et al., 2020; Pariyar et al., 2020). In contrast, the acidic nature of PP-MWH char could also have resulted from the formation of acid group on the hydrochar, a process that occurs at temperatures above 180 °C (Saha et al., 2019).

Similarly, in the palm waste study, PW-CB char also exhibited a higher pH than PW-MWB and PW-MWH chars (Table 7-1), which can be contributed by the significantly higher concentrations of potassium and calcium present on the exchange sites of PW-CB char. These elements contributed to the alkaline nature of CB. However, the lower pH observed in PW-MWH compared to PW-CB could be attributed to the formation of acidic functional groups during the production process (Saha et al., 2019).

#### **d) Cationic exchange capacity (CEC)**

PP-PB and PP-CB char samples exhibited significantly higher CEC than PP-PCB and PP-MWH chars (Table 5-1 and 6-1). This high exchange capacity of chars is largely attributed to the deprotonation of -COOH or -OH functional groups, creating negatively charged sites. The formation of these functional groups is influenced by the degree of volatilisation of cellulose, hemicellulose, and lignin present in the feedstock (Questell-Santiago et al., 2020; Deng et al., 2016; Tomczyk et al., 2020). TGA graphs of PP-PB and PP-CB (Figure 5-2 and 6-2) showed minimal weight loss, suggesting nearly complete depolymerisation of lignocellulose, resulting in the formation of carboxyl groups. In contrast, PP-PCB and PP-MWH chars exhibited incomplete decomposition, likely leading to fewer carboxyl groups on the surface (Chen et al., 2022).

In the palm waste study, similar results were observed, with PW-CB char exhibiting a higher CEC compared to PW-MWB and PW-MWH char samples (Table 7-1).

#### **e) Surface area**

Both PP-PB and PP-CB chars have a higher surface area compared to PP-PCB and PP-MWH chars (Table 5-1 and 6-1). The increase in the surface area of the char (Table 5-1 and 6-1) is

likely caused by the degradation of the organic material (hemicellulose, cellulose and lignin) which creates pores, thus exposing more surface (Lee et al., 2013; Kim et al., 2013). Chen et al. (2012) reported that the increase in the surface area is mostly attributed to the degradation of cellulose, which increases the release of the volatile matter and created more pores and surface area. The TGA graph of PP-PB and PP-CB chars shows less weight loss compared to PP-PCB and PP-MWH chars, suggesting that PP-PB and PP-CB char during pyrolysis underwent further degradation of organic materials, releasing volatile matter in the form of oil and gas compared to PP-PCB and PP-MWH char samples. This process resulted in an increase in the surface area and pore volume of PP-PB and PP-CB chars (Chatterjee et al., 2020).

In the palm waste study, equivalent results were observed where PW-CB char had a higher surface area compared to PW-MWB and PW-MWH (Table 7-1). As mentioned in Section 8.2.1 d), PW-MWB showed similar degree of volatilization to that of PP-PCB chars, which resulted in a similar trend in surface area between PW-MWB and PP-PCB.

#### **f) Hydrophobicity**

Chars with high levels of oxygen containing groups, such as hydroxyl (-OH), carboxyl (-COOH), and carbonyl (C=O) groups, are more hydrophilic due to their ability to form hydrogen bonds with water molecules. As mentioned above, PP-PB and PP-CB chars resulted in the formation of more oxygen containing groups making them hydrophilic in nature. In contrast, PP-PCB and PP-MWH chars, resulted with fewer oxygenated functional groups, making them more hydrophobic. The hydrophobicity in PP-PCB and PP-MWH chars could also be attributed to the presence of aliphatic functional groups and aromatic carbon structures on the char surface, which contributes to their hydrophobic nature. Non-oxygenated aliphatic compounds, being non-polar, do not readily interact with water molecules (Gray et al., 2014; Mao et al., 2019).

In the palm waste study, PW-MWH chars were also found to be hydrophobic compared to PW-MWB and PW-CB chars. This suggests that the production parameters used for hydrothermal carbonization need further assessment and optimization to improve the

properties of the hydrochars, especially in terms of their hydrophilicity/hydrophobicity.

### **8.3 Effect of potato peel and palm waste chars on leachate volume, nutrient concentration in the leachates and soil properties and plant growth**

#### **8.3.1 Effect of char treatments on soil leachate volume**

Two key properties of char that influence soil leachate volume when added to soil are surface area and hydrophobicity (Tanure et al., 2019; Mau et al., 2020; Mau et al., 2018). Generally, adding char to soil is expected to reduce water leaching, as the porous structure and large surface area can retain water and minimise water loss (Libutti et al., 2019). PP-PB and PP-CB samples demonstrated a high surface area and hydrophilic properties compared to the other chars (Table 5-1 and 6-1), which would explain the reduced leachate volume observed in these treatments (Figure 5-3 and 6-3). In contrast, the PP-PCB and PP-MWH treatments, with their lower BET surface area and hydrophobic nature (Section 8.2), led to increased soil leachate volume compared to other treatments and the control (Figure 5-3 and 6-3). The addition of hydrophobic PP-PCB and PP-MWH chars to the control soil likely inhibited water from entering the char's intrapore structure due to water repellence. This water-repellent characteristic could reduce the ability of the chars to retain water, resulting in increasing soil leachate volume when these chars were applied (Edeh and Mašek, 2021).

Soil leachate volume varied throughout the incubation period from weeks 1 to 12 across all treatments. An increase in leachate volume occurred from weeks 2 to 4, which can be attributed to frequent irrigation leading to higher leachate volumes. In PP-PCB and PP-PB treated soils, a subsequent increase in leachate volume was observed during weeks 6 and 7 (Figure 5-3). This increase coincided with an adjustment in irrigation volume from 150ml to 200 ml during these weeks. The likely explanation is that the soil had reached its maximum water-holding capacity, and therefore could no longer retain additional water, resulting in a balance between water input and output due to insufficient time for evaporation or plant uptake. From week 8 onwards, leachate volume began to decline, possibly due to increased plant growth in PP-MWH, PP-PCB and PP-PB treatments (Figure 5-7 and 6-7), which could result in greater water loss via plant transpiration (Makarieva et al., 2023). A similar trend was

observed in PP-CB treated soil, as the plant grew from week 11 onwards, resulting in the reduction in leachate volume (Figure 6-7).

In the palm waste study, similar results were observed, where PW-MWH treated soil exhibited significantly higher soil leachate volumes compared to the other char treatments and the control (Figure 7-3), likely due to the hydrophobic nature of PW-MWH. In contrast, the high surface area of PW-CB char likely enhanced water retention, reducing soil leachate volume. The PW-MWB char, with its lower surface area compared to PW-CB char, likely had minimal impact on the overall surface area, resulting in no significant effect on soil leachate volume compared to the control.

### **8.3.2 Effect of char treatments on nutrient leaching, soil properties and plant growth**

Plant nutrients can be categorised into three groups (Maathuis and Diatloff, 2012; Alabama Cooperative Extension System, 2018):

- Primary macro-nutrients: nitrogen, phosphorus and potassium (NPK) are needed in relatively larger quantities. They are critical for promoting green, leafy growth, energy transfer, root development and water movement within plants.
- Secondary plant macro-nutrients: calcium and magnesium, although required in smaller quantities, are vital for root and plant development, strengthening cell walls and supporting photosynthesis.
- Micronutrients: trace elements such as iron and zinc, required in tiny quantities are crucial for maintaining chlorophyll content and enzyme activity in plants.

In this study, nutrient concentrations in leachates from different treatments were influenced by various factors, including char additions, the Hoagland solution, soil cationic exchange capacity (CEC), pH and plant growth.

In the potato peel study, in all the treatments with respect to the control, an increase in the concentration of the nutrients was observed in the leachates from weeks 1 to 4. In the initial weeks, when plants were watered with de-ionised water, the leached nutrients were



primarily derived from the available nutrients already present in the soil. Since de-ionised water does not introduce additional nutrients, it would allow for the release of nutrients that were already available in the soil, including those from the char amendments. By week 4, the concentration of the leached nutrients diminished at a consistent rate, suggesting an initial flush of the nutrients from the soil. This decline likely limited nutrient availability for plant uptake, resulting in leaf withering, yellowing at the tips of the leaves, and slower leaf growth across all treatments (The Royal Horticultural Society, 2022). In addition to the effects of the chars, the application of Hoagland solution also significantly influenced nutrient concentration in the leachates. From week 5 onwards, there was an increase in leached potassium, calcium and magnesium concentrations (Figure 5-4 and 6-4), as well as phosphate and nitrate concentrations (Figure 5-5 and 6-5) across all treatments, including the controls. The addition of this nutrient solution corresponded with a significant increase in number of leaves and the length of the plant across treatments, suggesting that nutrient supplementation was essential for promoting plant growth. Similar findings were reported by Alotaibi and Schoenau (2016) and Ye et al. (2019), who observed that char alone did not significantly impact crop growth, but the crop yields increased with fertilizer addition.

In the palm waste study, a similar trend was observed in nutrient concentrations in the leachate over the incubation period (Figure 7-4 and 7-5). Along with the incubation period, the different char treatments also significantly influenced the concentration of the nutrients in the leachates through various factors.

In the potato peel study, higher concentrations of leached potassium were observed in PB and PP-CB treated soils compared to PP-PCB, PP-MWH treated soils and the controls. This aligns with previous findings that char with high potassium content increases potassium leaching when added to soil (Hardie et al., 2015; Buecker et al., 2016; Kuo et al., 2020; Dissanayake et al., 2023; Jahan et al., 2023). The concentration of potassium on the exchange sites in the char samples was found to be significantly higher in PP-PB and PP-CB compared to PP-PCB and PP-MWH samples (Table 5-1 and 6-1). At the beginning of the plant pot experiment, the addition of PP-PB and PP-CB chars to the soil increased the soil potassium concentration on the soil exchange sites by 31.8 % and 21.5 %, respectively, compared to the control. The addition of the Hoagland solution (potassium = 235 ppm) further contributed to the soil potassium concentration. By the end of the experiment, potassium concentrations on

the soil exchange sites remained significantly higher in PP-PB and PP-CB treated soils compared to PP-PCB, PP-MWH treated soils and the controls (Table 5-2 and 6-2). This suggests that the potassium was retained in the exchange sites for PP-PB and PP-CB treated soils due to their high CEC. However, the excess potassium in these soils likely contributed to increased potassium leaching, as some of the available potassium may have been leached out over time, reflecting a balance between retention on exchange sites and leaching from the soil.

This suggests that potassium on the exchange sites in PP-PB treated soil was sufficiently available for plant uptake, which likely supported essential functions like water and nutrient movement within plant tissue, photosynthesis, and overall crop quality (University of Minnesota, 2014; Prajapati, 2012). In contrast, PP-CB treated soil exhibited higher potassium concentrations on the exchange sites but lower levels in plant biomass compared to PP-MWH treated soil and the control, implying the potassium availability for plant uptake was limited in PP-CB treated soil. This limited availability likely contributed to slower leaf growth and reduced biomass, as insufficient potassium uptake can hinder plant development (University of Minnesota, 2014; Prajapati, 2012). At the beginning of the experiment, potassium concentration on the exchange sites was lower in the PP-PCB and PP-MWH char samples. The addition of these chars to the soil did not lead to any difference in soil potassium concentration (PP-PCB and PP-MWH = 0.04 mol/kg) relative to the control (0.04 mol/kg), resulting in a similar trend of potassium leaching compared to control.

In the palm waste study, potassium concentrations in the leachate were significantly lower in PW-CB treated soil compared to PW-MWB and PW-MWH treated soils, and the control (Figure 7-4). This reduction in leached potassium can be attributed to the high CEC of PW-CB char (Section 8.2), which increased the soil's overall CEC upon addition, facilitating the retention of potassium. This also explains the higher potassium concentrations observed at the exchange sites of PW-CB treated soil compared to the other treatments at the end of the experiment (Table 7-2).

In the potato peel study, the addition of PP-PB and PP-CB chars resulted in increased potassium leaching, but this trend was not observed for calcium and magnesium. One reason for this is that the addition of these chars did not increase the concentration of calcium and magnesium on exchange sites in PP-PB and PP-CB treated soils compared to the control. Although the application of the Hoagland solution likely increased nutrient concentrations on

the exchange sites in both treated soils and control, leached calcium and magnesium levels were significantly lower in char treated soils than in the control. This difference could be attributed to factors such as soil CEC. Addition of PP-PB and PP-CB chars with high CEC resulted in an increase in soil CEC. Soil CEC plays a crucial role in retaining cations like potassium, magnesium, iron, and zinc on soil exchange sites, thereby reducing nutrient leaching (Pariyar et al., 2020 and Clough and Condron, 2010).

This is reflected in the lower concentrations of calcium and magnesium in the leachates of PP-PB and PP-CB treatments throughout the incubation period (Figure 5-4 and 6-4), suggesting that these nutrients were retained in the soil exchange sites and made available for plant and root uptake (Hossain et al., 2020; Yu et al., 2018). Despite the PP-PCB and C having similar CEC, calcium leaching was significantly lower in PP-PCB treated soil than in control, possibly due to the increased root biomass observed in PP-PCB, which enhances calcium uptake. Calcium and magnesium are essential nutrients that strengthen the root cell wall and promote root growth (Zhang et al., 2020; Koch et al., 2020; Qu et al., 2023). Thus, higher calcium absorption in PP-PCB treated soil would explain greater root growth compared to control. The high percentage of calcium carbonate in soils, combined with the low CEC observed in the control soils, limited their ability to retain calcium, resulting in higher calcium leaching.

In PP-MWH treated soil, nutrient retention was not significantly different from the control, possibly due to PP-MWH char's hydrophobic nature, which reduced the interaction of dissolved ions with its surface, causing the nutrients to wash away with the water and not increasing the retention.

In the palm waste study, a similar trend was observed for leached calcium and magnesium concentrations, which were significantly higher in the control and lower in the char treated soil (Figure 7-4). This suggests that the char treatments improved the retention of these nutrients within the soil, reducing their leaching.

Changes in soil pH from char treatments were non-significant, indicating no substantial effect on soil alkalinity. As observed by Wang et al. (2024) and Gao et al. (2021), the effectiveness of char in increasing the soil pH is typically observed when there is a significant difference between the initial soil pH and the pH of the char, particularly when the soil is more acidic. In

the potato peel study, the initial pH of the soil was already alkaline in nature ( $8.17 \pm 0.08$ ) thus, explaining the negligible effect of the char treatments in the soil pH. Although PP-PCB and PP-PB exhibited an acidic pH, the high calcium carbonate content (46 %) in the soils likely neutralised the acidity of the chars, thus not affecting the soil's overall alkalinity (Akanji et al., 2022).

In the palm waste study, a similar trend was observed with soil pH, as no significant changes were detected following the addition of char treatments. Although PW-MWH exhibited an acidic nature, the presence of calcium carbonate in the soil likely neutralized the acidity of the char, maintaining the soil's alkalinity (Akanji et al., 2022).

The functional groups present on the char surface promote soil CEC, which is directly influenced by soil pH. In the potato peel study, the addition of PP-PB and PP-CB chars to the soil increased soil CEC, due to the presence of  $-\text{COOH}$  and  $-\text{OH}$  functional groups (Section 8.2). These groups deprotonate under alkaline conditions to form negatively charged sites, thereby enhancing cations retention (Gholami and Rahimi, 2022; Vilakazi et al., 2023; Joao et al., 2024 and Mia et al., 2017).

Similarly, in palm waste study, the addition of PP-CB char to the soil increased the CEC of the PP-CB treated soil. This enhanced CEC played a crucial role in retaining cations within the soil, thereby reducing nutrient concentrations in the leachate.

Adding char to alkaline soil would have further increased negatively charged functional groups, potentially causing electrostatic repulsion of anions like phosphate and nitrate on soil surfaces (Chen et al., 2018). This effect could explain the higher phosphate concentrations observed in leachates from char treated soils compared to the controls in the potato peel study (Figure 5-5 and 6-5). Plant growth increased significantly over time in all treatments except the PP-CB treatment, suggesting that some of the available phosphate on the exchange sites in the soil was being absorbed by the plants to support their growth. Despite PP-MWH treated soil exhibited greater plant growth than PP-CB treated soil, PP-MWH resulted in higher phosphate leaching compared to PP-CB. This could be due to the strong hydrophobicity nature of PP-MWH char, which may facilitate phosphate release into the soil solution, leading to higher leaching. Additionally, by the end of the plant-pot experiment, a lower concentration of phosphate was observed on the exchange sites in PP-MWH treated

soil compared to PP-CB treated soil and the control. This could be due to PP-MWH char's strong hydrophobicity, which causes the water to repel and may cause nutrients to leach out without significant interaction with the soil surfaces, thereby reducing the retention capabilities.

In the palm waste study, a similar trend was observed, where leached phosphate concentrations were significantly lower in the control compared to char treated soils (Figure 7-5). This can be attributed to the presence of negatively charged functional groups in the soil, which likely caused electrostatic repulsion of phosphate anions, reducing their retention in the soil. Among the char treatments, PW-MWH treated soil exhibited significantly higher concentrations of leached phosphate compared to PW-MWB and PW-CB treated soils (Figure 7-5). This is likely due to the hydrophobic nature of PW-MWH treated soil, which may have limited phosphate interactions with soil surfaces, thus increasing its leaching potential.

In the potato peel study, while phosphate concentration in leachates increased, nitrate leaching decreased with char treatments (Figure 5-5 and 6-5). The increased CEC in char treatments likely promoted retention of ammonium ions on the exchange sites in the soil, contributing to higher ammonium concentrations in the soils at the end of the plant growth experiment. This retained ammonium on the exchange sites was then converted to nitrate through nitrification, which explains the higher soil nitrate levels observed in char treated soils compared to controls at the end of the experiment (Table 4-2 and 5-2) (Ulyett et al., 2013; Cao et al., 2017; Major et al., 2012). The lower nitrate concentration in PP-PB and PP-MWH leachates (Figure 5-5 and 6-5) suggests that the available nitrate on exchange sites in the soil was efficiently absorbed by the plants, thereby reducing the nitrate leaching. Nitrogen is an essential nutrient for tillering, rapid growth and producing higher yields of biomass (AHDB, 2023). PP-PB and PP-MWH treatments displayed higher biomass yield (Table 5-3 and 6-3) and an increase in tillering of new leaves over time (Figure 5-7 and 6-7), indicating the presence of sufficient amount of nitrate in soil to promote plant growth (AHDB, 2023). The nitrate leaching was significantly lower in PP-PCB treated soil than in control and the soil nitrate concentrations on the exchange sites at the end of the plant-pot experiment wasn't significantly different compared to PP-PCB treated soil and control, suggesting that most of the available nitrate was absorbed by the plants improving the plant growth.

At the end of the experiment, soil nitrate concentration on the exchange sites in the PP-CB treatment were significantly higher than in the PP-MWH treated soil and control (Table 6-2), accompanied by increased nitrate leaching in both the PP-CB treatments and control (Figure 6-5). However, in comparison between the control and PP-CB treatment, the plant yield at the end of the experiment was significantly lower in PP-CB treatment compared to control (Table 6-3). This pattern in PP-CB treated soil suggests that, while nitrate accumulated in the soil, it was not effectively absorbed by the plants to support new leaf growth (Grzebisz et al., 2023 and Xu et al., 2020). Effective nitrate uptake typically requires accompanying cations such as potassium, calcium, and magnesium, however, these nutrients were largely retained in the soil of the PP-CB treatment rather than absorbed by the plants. This resulted in high concentration of these nutrients on the exchange sites in the PP-CB treated soil (Table 6-2) but low levels in plant biomass compared to PP-MWH treated soil and control by the end of the experiment (Table 6-3). This imbalance likely inhibited nitrate uptake, slowing plant growth and reducing biomass yield.

Root development plays an important role in nutrient uptake by serving as the primary interface between plants and the soil environment, enhancing nutrient absorption (Cochavi et al., 2020 and Goss et al., 1993). In the PP-MWH and PP-CB study, PP-CB treated soil showed lower root biomass compared to both PP-MWH treated soil and control (Table 6-3), potentially explaining the poor nutrient absorption in the PP-CB treatment. The limited root growth observed in PP-CB treated soil may be due to a phytotoxic effect of the PP-CB char, potentially caused by high potassium concentration on the exchange sites in the PP-CB treated soil. Increased potassium levels can hinder root development and reduce nutrient uptake from the soil (Růžicková et al., 2021). High potassium concentration on the exchange sites in the soil can interfere with the uptake of essential cations such as calcium and magnesium, potentially leading to deficiencies that adversely affect root growth (Xu et al., 2020).

In the palm waste study, the concentration of leached nitrate was significantly lower in PW-CB treated soil compared to PW-MWB, PW-MWH char treated soils, and the control (Figure 7-5). The high nitrate level observed on exchange sites in PW-CB treated soil at the end of the experiment suggests that nitrification derived nitrate was retained in the soil. Combined with increased plant growth, this could indicate that the available soil nitrate was effectively

absorbed by plants, contributing to new leaf formation and higher biomass yield. This nitrate retention and plant uptake likely explain the reduced leached nitrate concentration in CB treated soil. A similar trend was observed in the PP-PB and PP-MWH treated soil in the potato peel study, where the higher soil nitrate levels coincide with increased plant growth and reduced nitrate leaching, indicating effective nutrient retention and uptake.

In PW-MWB treated soil, leached nitrate concentration was significantly lower than in PW-MWH treated soil and the control (Figure 7-5). However, the soil nitrate levels on the exchange sites in PW-MWB treated soil were not significantly different from the control (Table 7-2), suggesting that most of the available nitrate on the exchange sites in PW-MWB treated soil was absorbed by plants, reducing nitrate leaching.

At the end of the experiment, both soil nitrate levels (Table 7-2) and biomass yield (Table 7-3) were significantly lower in PW-MWH treated soil compared to other char treatments and the control. This could be attributed to the hydrophobic nature of PW-MWH char, which repels water and potentially promotes nutrient leaching without substantial interaction with soil surfaces, thereby diminishing nutrient retention. While plant roots are essential for nutrient uptake and growth, no significant differences in root biomass were observed between treatments in the palm waste study (Table 7-3). This suggests that the limited availability of nutrients on the exchange sites in PW-MWH treated soil was the primary factor contributing to slower tillering of leaves (Figure 7-7) and reduced biomass yield (Table 7-3).

### **Micronutrients (Iron and Zinc)**

In the PP-PCB, PP-PB and, PP-MWH treated soil along with the controls, iron and zinc concentrations in the leachates remained below the detection limit (BDL < 0.5 mg/L) for most of the incubation period. This is likely due to the initially low concentrations of these micronutrients on the exchange sites in the soil. However, PP-CB treated soils exhibited higher iron and zinc concentrations in the leachates during the initial weeks, suggesting an initial release of these micronutrients from the char amended soil. These concentrations declined below detection level (BDL) after week 7. Under alkaline conditions, iron and zinc tend to form insoluble hydroxides, reducing their mobility. However, in the initial weeks, the buffering effects of the char and the soil likely maintained these elements in their soluble forms,

increasing their leaching potential (Hale et al., 2012; (Kicińska et al., 2021 and Wang et al., 2020).

In the palm waste study, a similar trend was observed in the concentration of iron and zinc in the leachates, where the concentrations were  $BDL < 0.5$  mg/L during the incubation period.

### **8.3.3 Effect of char treatments on the concentration of dissolved organic carbon (DOC) in leachate**

Differences in DOC leaching patterns emerged between the PP-PCB and PP-PB study and the PP-MWH and PP-CB study. In the PP-PCB and PP-PB study, DOC concentrations in leachates were lower from weeks 1 to 4 across all treatments, including the control, before increasing in week 5 and then gradually decreasing from week 7 onwards. Conversely, in the PP-MWH and PP-CB study, DOC leaching began with higher levels in the initial weeks for both char treatments and the control, which then gradually decreased over time. The differing trends between the two studies may be influenced by additional parameters affecting DOC release during plant growth experiments. One notable difference is the temperature shift from 20 °C from weeks 1 to 4 but lowered to 18 °C from weeks 5 onward, while the PP-MWH and PP-CB study maintained a constant 18°C. Typically, an increase in temperature is associated with enhanced microbial activity, accelerating organic carbon decomposition and DOC release (Clark et al., 2009; Pearson et al., 2024; Williams et al., 2015). Interestingly, in this case, DOC concentrations were lower at 20 °C and increased after the shift to 18 °C, suggesting that the temperature could also influence the DOC leaching patterns.

The concentration of DOC in the leachate was significantly higher in PP-PB treated soil from week 4 onwards and in PP-CB treatment in weeks 1 to 3. Dong et al. (2018) found that soil microbial activity is positively linked to the increase in DOC concentration in the leachate. The increase in the microbial activity in the soil could be directly influenced by the potassium concentration in the soil (Gabriel et al., 2022; Xia et al., 2024). As mentioned in Section 8.2.3, addition of PP-PB and PP-CB chars to the soil resulted in an increase in the potassium concentration on the exchange sites in the treated soils compared to PP-PCB, PP-MWH treated soil and controls. When potassium on the exchange sites is available in sufficient



quantities, it enhances microbial activity, allowing soil microorganisms to preferentially utilise labile organic carbon sources. This leads to the degradation of more easily decomposable carbon compounds, such as starch and hemicellulose thus increasing the concentration of DOC in the leachates (Song et al., 2020; Liu et al., 2022). In contrast, the addition of PP-PCB and PP-MWH chars did not result in any difference in potassium concentration with respect to control. As a result, minimum microbial activity was likely observed, leading to a reduction in DOC leaching. Over time, the decrease in DOC leaching was observed which could be due to the soil microbes would have degraded the labile carbon present in the char thus reducing the amount of DOC available for leaching.

In the Palm waste study, there were no significant differences in DOC among the treatments and the control. This can be attributed to the absence of significant differences in potassium concentrations between the char treated soils and the control, which likely results in similar microbial activity levels across all treatments. Consequently, microbial activity did not substantially impact DOC leaching. The addition of the Hoagland solution likely stimulated microbial activity by introducing additional nutrients, accelerating the decomposition of easily degradable organic components in the soil. This initially led to an increase in DOC leaching. However, as these readily leachable organic compounds were gradually depleted over time and the DOC concentrations in the leachates decreased.

## **8.4 Comparison between potato peel and palm waste feedstocks on leachate volume, nutrient concentration in the leachates and soil properties and plant growth**

### **8.4.1 Char characterization**

Although similar parameters were used for the microwave pyrolysis of PP and PW, the level of volatilisation observed is differed between the PP and PW chars. This variation is very likely due to the formation of hot spots in the microwave pyrolysis of potato peel, which led to the creation of two distinct types of chars: PB and PCB chars (Section 3.3). In contrast, during the microwave pyrolysis of palm waste, no hot spot formation was observed. However, the degree of volatilisation in palm waste chars were found to be similar to that of PP-PCB char,

as compared to the PP-PB char. This indicates that although both feedstocks underwent similar pyrolysis conditions, the characteristics of the resulting chars were influenced by the formation of hot spots in the potato peel pyrolysis process.

#### **a) Carbon content**

The carbon content in the potato peel study (microwave pyrolysis: PP-PCB = 46.56 %, PP-PB = 56.22 %, Hydrothermal carbonisation: PP-MWH = 47.83 % and conventional pyrolysis: PP-CB = 64.02 %) was found to be higher in the char samples compared to palm waste study (microwave pyrolysis: PW-MWB = 60.08 %, Hydrothermal carbonisation: PW-MWH = 53.51 % and conventional pyrolysis: PW-CB = 65.66 %). The difference in carbon content between feedstocks is attributed to their lignin content (Tomczyk et al., 2020). Palm waste feedstock (3.44 %) exhibited a higher lignin content compared to potato peel feedstock (0.15 %) (Table 4-1). Lignin, being a thermally stable component of biomass, contributes more to the residual fixed carbon during pyrolysis than and hemicellulose, which degrade and release volatiles. This process results in overall increase in the carbon content of char (Ippolito et al., 2020 and Almutairi et al., 2023). In addition to the thermal stability, lignin has higher C:O ratio than cellulose and hemicellulose, which further enhances its contribution to the carbon rich structure of biochar (Wang et al., 2018, Deng et al., 2016)

#### **b) Cationic exchange capacity (CEC) and alkali metals**

Biomass feedstock with high ash content produces chars with higher cation exchange capacity (CEC) due to the presence of alkali metals such as potassium, calcium, and magnesium, which promote the formation of oxygen-containing surface functional groups during pyrolysis (Tomczyk et al., 2020). In this study, potato peel feedstock exhibited a higher ash content (8.45 %), resulting in a higher CEC (Table 5-1 and 6-1), whereas palm waste feedstock had a lower ash content (5.48 %), leading to significantly lower CEC (Table 7-1). The main components of ash in biomass feedstock are soluble alkali metal salts (e.g., calcium, potassium, magnesium) and inorganic ions such as phosphate and carbonate ions (Ji et al., 2022; Shi et al., 2023). These alkali metal salts are thermally stable at pyrolysis temperatures

(300–700 °C) and do not volatilize like organic compounds, resulting in their concentration in the ash (Shi et al., 2023). Consequently, the potato peel feedstock's higher ash content increased the concentrations of potassium, calcium, and magnesium in potato peel chars (Table 5-1 and 6-1) compared to palm waste chars (Table 7-1). Additionally, the negative charged ions such as phosphate and carbonate in the ash promotes strong electrostatic attraction to cations, enhancing cation retention when the char is applied to soil (Ji et al., 2022).

### **c) Surface area**

Surface area is an important property of char that influences CEC, water holding capacity and adsorption capacity (Ji et al., 2022). A comparison of these two feedstocks in this study revealed that the surface area of potato peel derived chars was lower (Table 5-1, 6-1) than that of palm waste derived chars (Table 7-1).

This difference can be attributed to the high ash content of potato peel feedstock, as previously discussed. A high ash content leads to significant micropore blockage, reducing the overall surface area of the resulting chars (Leng et al., 2021; Ji et al., 2022). The ash particles, primarily composed of minerals and alkali salts, can fill or block pores during pyrolysis, particularly micropores, thereby limiting the surface area available for adsorption and other functions.

## **8.4.2 Effect of chars from different feedstocks on soil leachate volume**

In both the potato peel and palm waste studies, the addition of chars produced through microwave pyrolysis and conventional pyrolysis significantly decreased the soil leachate volume compared to the control. This reduction is attributed to the high surface area and hydrophilic nature of these chars, which enhanced water retention in the soil (Section 8.2.2).

The high surface area likely improved soil structure, enabling better water absorption and retention.

Conversely, the addition of chars produced via microwave hydrothermal carbonisation (MWH) from both feedstocks resulted in an increase in soil leachate volume. This increase is explained by the hydrophobic nature of MWH chars, which repelled water and reduced its interaction with the soil. The hydrophobicity of the MWH chars appears to be influenced by the production parameters rather than the type of feedstock used. This suggests that the conditions of microwave hydrothermal carbonisation, such as temperature, pressure, and residence time, played a critical role in determining the hydrophobic or hydrophilic characteristics of the resulting char.

#### **8.4.3 Effect of chars from different feedstocks on nutrient leaching, soil properties and plant growth**

In the potato peel study, the potassium concentration on exchange sites in PP-PB and PP-CB treated soil significantly increased by 31.8 % and 21.5 %, respectively, compared to the control (Section 8.2.3). This increase was attributed to the high potassium content in PB and CB chars. Although PP-PB and PP-CB treated soils exhibited high CEC, which facilitated potassium retention on exchange sites, a large portion of the excess potassium was still leached out by the end of the experiment.

In contrast, in the palm waste study, PW-CB treated soils exhibited significantly lower potassium leachate concentrations compared to PW-MWB, PW-MWH, and the control. Although PW-CB char contained a higher potassium concentration, its addition to the soil did not significantly increase the potassium concentration on the exchange sites in the PW-CB treated soil. The high CEC in PW-CB treated soil retained potassium ions, making them available for plant uptake, as reflected in the higher potassium concentrations in soil at the experiment's conclusion. This retention explains the reduced potassium leaching in PW-CB treated soils.

In both studies, a similar trend was observed with calcium and magnesium, where leachate concentrations were lower in char-treated soils compared to the control. The increased soil

CEC due to char additions improved the retention of these ions on soil exchange sites, making them available for plant uptake.

In the potato peel study, PP-MWH treated soil had significantly lower nitrate concentrations in the leachate compared to PP-CB treated soil. Conversely, in the palm waste study, PW-CB treated soil had significantly lower nitrate leachate concentrations compared to PW-MWH treated soil. However, in both studies, the final soil nitrate concentrations were significantly higher in CB-treated soils compared to MWH-treated soils. This divergence in nitrate leaching patterns is likely linked to differences in plant growth. Nitrogen is essential for tillering, rapid growth, and higher biomass yields (AHDB, 2023). In the potato peel study, tillering and biomass yield were significantly lower in PP-CB treated soils compared to PP-MWH treated soils, indicating reduced nitrate uptake by plants and higher nitrate leaching.

Conversely, in the palm waste study, PW-CB treated soils supported significantly higher tillering and biomass yields than PW-MWH treated soils, suggesting more efficient nitrate utilization by plants.

Despite the consistent improvements in soil properties by CB chars, the final wheat biomass yield was significantly lower in the potato peel study's PP-CB treated soils compared to palm waste study's PW-CB treated soils. This reduction in the potato peel study can be attributed to the high concentration of potassium observed in the PP-CB treated soils, which impaired root growth and nutrient uptake. High potassium concentration on the soil exchange sites can disrupt the uptake of calcium and magnesium, thereby affecting the plant health. Although CB chars from both the studies were produced using the same pyrolysis technique and parameters, the concentration of the potassium was significantly higher in the potato peel PP-CB char compared to palm waste PW-CB char, that resulted in increase in the concentration potassium on exchange sites in PP-CB treated soils.

#### **8.4.4 Effect of chars from different feedstocks on the concentration of leached DOC**

In the potato peel study, PP-PB and PP-CB treated soils significantly increased the concentration of leached DOC compared to the other char treatments and the control (Section 8.2.4). Conversely, in the palm waste study, no significant difference in leached DOC concentration was observed between the char treatments and the control.

The variation in DOC leaching can be attributed to microbial activity, which influences the breakdown of organic compounds in the soil and increased DOC concentrations in leachates (Dong et al. 2018). Microbial activity in soil is directly influenced by potassium concentration, as potassium is essential for enzymatic processes that support microbial growth (Gabriel et al., 2022; Xia et al., 2024). In the potato peel study, PP-PB and PP-CB char treatments increased the potassium concentrations in the soil, likely stimulating microbial activity and consequently increasing DOC leaching. However, in the palm waste study, char additions did not significantly increase soil potassium concentrations compared to the control. As a result, microbial activity and DOC leaching were unaffected.

The addition of the Hoagland solution from week 4 onwards likely stimulated microbial activity in the palm waste study by providing essential nutrients. This stimulation explains the observed increase in DOC leachate over time in the palm waste study.

However, a similar trend was not observed in the PP study. In this case, the microbial activity stimulated by the high potassium concentration in PP char treated soil resulted in the leaching of labile organic compounds during initial weeks. This early leaching significantly reduced the presence of labile compounds available for leaching in later stages, despite the addition of Hoagland solution. This difference highlights the distinct interactions between char characteristics, microbial activity, and nutrient dynamics in the two studies.

## **Chapter 9: Conclusion**

### **9.1 Introduction**

The chapter begins by summarising the key findings of the study, highlighting the influence of production methods (microwave pyrolysis, conventional pyrolysis, and microwave hydrothermal carbonisation) and the characteristics of feedstocks, specifically potato peel and palm waste, on the resulting char properties, soil properties and plant growth.

Next, the chapter identifies the environmental implications of the study, emphasising char's potential in waste valorisation, water conservation, nutrient retention for sustainable agriculture. Finally, it identifies directions for future research, including the optimisation of production techniques, mitigation of phosphate leaching, long term field trials, and investigating the energy requirement and economic viability of char production methods to support their implementation on a large and sustainable scale.

### **9.2 Main findings**

#### **9.2.1 Initial laboratory experiment**

In the initial laboratory-based experiment (Chapter 4), pyrolysis was conducted using a laboratory scale microwave reactor (CEM Discover). The objective of this chapter was to characterize chars produced at various temperatures and power settings for both potato peel and palm waste feedstocks to identify suitable parameters, such as temperature and power, for potential upscaling char production. However, the study encountered limitations in the pyrolysis process. While temperatures ranging from 180 to 250 °C were evaluated, the reactor

was only operable at a power setting of 100 W. Increasing the power resulted in equipment failure due to rapid pressure buildup, causing vial breakage within the microwave reactor (Section 4.3.2). Consequently, chars were produced and characterized at 100 W across the mentioned temperature range for both feedstocks to identify the optimal conditions for future large-scale production.

The biochar yield was highest at lower temperatures for potato peel at 150 °C (PP-150) and palm waste at 150 °C (PW-150) and decreased as the temperature increased. This is because, at lower pyrolysis temperatures, there is less decomposition, retaining more mass. At higher temperatures, increased volatilisation and carbonisation releases gases, reducing the biochar yield (Alperay et al., 2024; Mašek et al., 2013). An increase in pyrolysis temperature led to a corresponding increase in surface area of PP and PW biochar's. This temperature increase led to the collapse of biomass cell walls, creating micropores and mesopores that increased the surface area (Tong et al., 2020). The presence of these properties could reduce water and nutrient retention in char treated soils, significantly impacting plant growth.

Although biochar yield decreased with increasing production temperature, the properties of biochar produced at higher temperatures are reported to be more beneficial as soil amendments (Wystalska and Kwarciak-Kozłowska, 2021; Li et al., 2022; Liang et al., 2022; Khater et al., 2024; Kumar et al., 2024 and Liu et al., 2023). A drawback that was observed regarding the use of the laboratory scale microwave reactor, was in regards with the small feedstock input (1g per vial) that limited the quantity of char produced. Therefore, subsequent chapters (5,6 and 7) employed a larger scale microwave reactor for the char production. This large-scale reactor allows for increased char production quantities and operation at higher power outputs and enhanced process efficiency.

The plant pot experiments using PP chars (Chapter 5 and 6) and PW chars (Chapter 7) analyse their effects on soil properties and nutrient availability, considering chars produce through microwave pyrolysis, conventional pyrolysis and hydrothermal carbonization. The study (Chapter 8) reveals critical insights into how char's properties influence their performance as soil amendments.

In the potato peel study, during the microwave pyrolysis of the feedstock we observed hot spot formation, as a result two distinct chars were classified as partially charred biochar (PCB)



and pure biochar (PB) (Section 3.3). Potato peel chars produced through microwave pyrolysis biochar (PCB and PB), conventional pyrolysis biochar (CB) and microwave hydrothermal carbonization hydrochar (MWH) and palm waste produced through microwave pyrolysis biochar (MWB), conventional pyrolysis biochar (CB) and microwave hydrothermal carbonization hydrochar (MWH) exhibited distinct properties.

### 9.2.2 Soil leachate volume

Chars with greater surface area and hydrophilic properties (PP-PB, PP-CB and PW-CB) significantly reduced soil leachate volume. This aligns with the expectation that chars with porous structures promote water storage in soil and reduce soil leachate volume (Libutti et al., 2019). Conversely, hydrophobic char (PP-MWH and PW-MWH) increased the leachate volume, reflecting their inability to interact effectively with water molecules, which hindered water retention. This finding highlights the need to optimise hydrothermal carbonisation conditions to mitigate water repellence that is discussed further in Section 9.5.1 (Edeh and Mašek, 2021).

### 9.2.3 Nutrient retention and leaching

- **Potassium:** Potato peel derived PP-PB and PP-CB chars increased potassium leaching due to their high potassium concentration on the exchange sites of the treated soils. Despite this, the high CEC of these PP-PB and PP-CB chars did ensure adequate concentration of potassium on the exchange sites in the treated soils, making the nutrients available for plant uptake. Palm waste derived PW-CB chars demonstrated a significant decrease in the potassium leaching due to the high CEC of PW-CB chars, while PW-MWH char exhibited higher potassium leaching. The low CEC and the hydrophobic nature observed in PW-MWH treated soil, limited the potassium interaction with soil surfaces, thus increasing its leaching potential.

- **Calcium and magnesium:** Char with high CEC, such as PP-PB, retained these nutrients on the exchange sites in the soil effectively, resulting in lower leaching concentration and high availability for plant uptake. Hydrothermal chars (PP-MWH and PW-MWH), due to their lower CEC and hydrophobicity, demonstrated reduced nutrient retention.
- **Phosphate:** Phosphate leaching was higher in char treated soils compared to the controls, especially with hydrothermal chars PP-MWH and PW-MWH. This is due to the electrostatic repulsion of negatively charged phosphate ions on soil surfaces and the hydrophobicity of hydrothermal chars, which limited interactions between phosphate and soil properties.
- **Nitrate:** Nitrate leaching was reduced in PP-PB and PW-CB char treated soils, by retaining ammonium ions and subsequent conversion to nitrate through nitrification (Ulyett et al., 2013; Cao et al., 2017; Major et al., 2012). Although both nitrate and phosphate are negatively charged, nitrate retention in PP-PB and PW-CB treated soils influenced by the ability of these char treatments to retain ammonium ions on exchange sites, which subsequently undergo nitrification. The difference in nitrate and phosphate leaching may be attributed to soil pH. Under alkaline conditions, higher pH increases phosphate solubility, leading to greater leaching as phosphate remain in its soluble forms (Scholz and Brahney, 2022). In contrast, nitrate retention tends to improve with increasing pH due to cationic interactions (Bu et al., 2017). Additionally, plants generally have a higher demand for nitrogen, particularly in the form of nitrate, which is essential for leaf and shoot growth (Andrews et al., 2013). This increased uptake reduces nitrate leaching. However, the available phosphate in the soil is taken up at a slower rate, resulting in greater phosphate losses through leaching (M.A et al., 2020).

In contrast, PP-CB treated soil exhibited higher nitrate accumulation and reduced plant uptake, leading to increased nitrate leaching. PW-MWH treated soil showed increased nitrate leaching, likely due to the hydrophobic nature of PP-MWH char, which reduced its ability to retain nitrate effectively.

#### **9.2.4 Plant growth and biomass production**

Enhanced nutrient retention by PP-PB and PW-CB char treated soil promotes plant growth and increases biomass yield, highlighting the importance of char properties in influencing plant-nutrient dynamics. However, excessive potassium levels on the exchange sites in PP-CB treated soil inhibited root development, hindering nutrient uptake from the soil and highlighting the potential for nutrient imbalances (Růžicková et al., 2021; Xu et al., 2020).

#### **9.2.5 Dissolved organic carbon (DOC)**

The results were consistent with potato peel chars, particularly PP-PB and PP-CB, stimulating microbial activity due to their higher potassium concentration, which could lead to increased DOC leaching during the initial weeks (Gabriel et al., 2022; Xia et al., 2024). Over time, DOC levels decrease due to microbial degradation of labile organic compounds. No significant differences were found in DOC leaching between palm waste chars. This is due to the lack of significant changes in potassium concentration and microbial activity across treatments. However, the addition of the Hoagland solution is thought to have stimulated microbial activity in the soil, leading to an initial increase in DOC leaching, which then gradually decreased over time.

#### **9.2.6 Comparative analysis between feedstocks**

Potato peel chars had lower carbon content and surface area than palm waste chars, likely due to the higher lignin and ash content in palm waste. The elevated ash content in PP feedstock, rich in potassium, calcium, and magnesium, promotes the formation of oxygen-containing surface functional groups during pyrolysis (Tomczyk et al., 2020). However, this high ash content also reduced surface area by blocking pores, limiting adsorption capacity. Therefore, feedstock selection significantly influences the physical and chemical properties of char, affecting its effectiveness in soil improvement and nutrient management.

Palm waste chars had low ash content and CEC, making them less effective in retaining nutrients in the soil, which could lead to nutrient losses through leaching. However, the higher surface area observed in the PW-CB chars exhibited a reduction in the soil leachate volume, which help retain more nutrients in the soil. Thus, in turn contributed to improve nutrient availability for plant uptake and resulted in biomass yield.

### **9.3 Research limitations**

While chapters 5,6 and 7 each addressed some aspects of the knowledge gaps mentioned at the end of the literature review, there were limitations to each experiment.

In chapter 4, the laboratory scale microwave reactor exhibited pressure related issues which restricted pyrolysis to a maximum power setting of 100 W for both potato peel and palm waste. This limitation prevented the investigation of higher power levels and their effects on biochar characteristics. A potential solution to mitigate pressure buildup could involve conducting pyrolysis in an open vessel rather than a closed vessel, allowing gaseous byproducts to escape and preventing rapid pressure accumulation.

In Chapter 5 and 7, the microwave pyrolysis experiments were constrained by time, amount of biomass feedstock available and financial resources limiting the range of pyrolysis parameters such as power setting and residence time that could be investigated. Only a specific set of conditions were tested (Section 3.3), which may not fully capture the variability and potential optimizations in microwave pyrolysis. Future research should explore different power settings and residence time to better understand their effects on biochar properties.

Additionally, in Chapter 5, hot spot formation was observed during microwave pyrolysis of potato peel waste, potentially leading to variations in char properties. To mitigate this issue, grinding the biomass into finer particles would have improve heating uniformity. However, due to the limited amount of available feedstock, additional trials to produce a new batch of char using finer particles biomass feedstock was not feasible.

In chapter 6, the limited availability of potato peel waste restricted the plant pot experiments to only three replications. This reduced application may have influenced the statistical

analysis of the results, potentially limiting the detection of subtle treatment effects. This limitation highlights the challenges in maintaining consistency across experiments and emphasizes the need for adequate application in future studies to enhance statistical robustness.

Beyond these experimental specific limitations, certain key soil properties were not measured. Specifically, water holding capacity and anion's exchange capacity, both which could have provided further insight and deeper understanding on the biochar's role in retaining water and as well its interaction with negatively charged nutrients such as nitrate and phosphate. Another limitation was that the energy consumption was not measured for each production process. This information would have provided valuable insight into the efficiency and sustainability of different char production methods. Additionally, while this study focused on nutrient leaching, water retention, and plant growth, it did not include microbial analyses, which could provide further insight into nutrient cycling and long-term biochar stability. Another constraint was the pot size used in plant-pot experiments, which restricted root expansion and may have influenced plant growth responses compared to field conditions. Furthermore, this study primarily focused on short term effects, a longer-term investigation would be valuable to determine whether char benefits persist across multiple growing seasons.

## **9.4 Environmental implications**

This research has broader implications for sustainable agriculture and management practices by using agricultural wastes, that is, potato peels, date palm residues for sustainable production of char from them.

### **9.4.1 Waste Valorisation**

Char production supports the circular economy by transforming agricultural residues into valuable by products. This study demonstrates the potential of char derived from agricultural

residues, such as potato peel and palm waste, as effective soil amendments. By converting these byproducts into char through techniques like microwave pyrolysis, conventional pyrolysis, and microwave hydrothermal carbonisation, the research highlights a sustainable pathway for managing agricultural and organic waste. Most of the agricultural waste and the organic waste either end in landfills, incinerated or converted into a compost (Singh et al., 2021; Awogbemi and Kallon, 2022). Valorisation of waste into char not only reduces the environmental pollution associated with landfills and waste incineration but also enhances soil fertility and plant growth, connecting waste management and sustainable agriculture (Faiad et al., 2022; Pandey et al., 2019; Damilola, 2023)

Experimental results reveal that char properties, such as carbon content, cationic exchange capacity and hydrophilicity, are influenced by the type of feedstock and production methods. Sandy soils used in these experiments usually display poor soil properties such as increase in water leachate volume, nutrient leaching and, low soil CEC as well poor plant growth compared to the char treated soils. However, potato peel and palm waste chars, particularly from microwave pyrolysis and conventional pyrolysis, exhibited high CEC, reduction in water leachate volume and reducing nutrient leaching while enhancing their availability for plant uptake, demonstrating their utility in nutrient management.

The integration of waste valorization into soil amendment practices provides dual benefits: it addresses the challenges of organic waste accumulation and simultaneously improves agricultural productivity. The use of chars not only retains essential nutrients like potassium, calcium, magnesium, and nitrate but also reduces water and nutrient leaching, enhancing soil health and crop yield. This aligns with the principles of a circular economy, transforming agriculture residue into valuable resources.

Valorising agricultural waste through char production offers significant economic benefits by transforming low value waste residues into high value products, which can be utilised in agriculture. For instance, producing biochar from one tonne of agricultural waste, can yield approximately 300 kg of biochar (Climate SAN, 2021). In the United Arab Emirates in 2025, 5kg of biochar is valued at around £16.92 (85.18 AED), with potential revenue reaching approximately £3,384 (17,036 AED) per tonne of biochar (Persist, 2024). The global biochar market is projected to grow to \$3 billion by 2025 (Guo et al., 2019), offering an additional income stream for farmers and industries while encouraging responsible waste disposal

practices (Wang, et al., 2022). These initiatives promote sustainability while enhancing the economic viability of agricultural and industrial operations.

#### **9.4.2 Water conservation**

Chars are highly effective in enhancing soil water retention, reducing water loss through leaching. This improvement is crucial not only for water conservation but also ensuring that water remains accessible to plant roots during critical growth stages. These properties are particularly beneficial in sandy soils, which naturally have low water holding capacity, leading to a higher water requirement for irrigation. The use of chars offers a practical and cost-effective approach to conserve water in farming (Tsai, et al., 2021). In countries like the UAE, they observe high evaporation rates, low and irregular precipitations, with average rainfall reaching in maximum 160mm annually. Since the precipitations are low, the agricultural sector consumes a major part of the total water demand in the country estimated to be around 83% (Shahin and Salem, 2014). According to the statistics provided, in year 2020 around 6,2071 Mm<sup>3</sup>/y of water is consumed in UAE (Fanack Water, 2022). When compared to the statistics of the water consumption in UK that is 82-110 Mm<sup>3</sup>/y water was used for agriculture purpose in 2020, much higher water consumption is observed in UAE (Knox et al., 2020). As the results derived from the experimental studies did prove that with the addition of some chars specially those produced using conventional and microwave pyrolysis resulted in an increase in the retention of water thus reducing the volume of the leachate compared to the control soil. Soils treated with PP-PB, PP-CB, PW-MWB and PW-CB chars retained approximately 12 %, 8 %, 4 % and 10 % more water than the control, highlighting their potential for improving soil water conservation. This implies that treating these arid regions with biochar could improve water retention withing the soil structure, conserving water resources and making char an effective strategy for addressing water scarcity in agriculture.

In this study, it was observed that certain chars are more effective in water retention than others. The chars generated from hydrothermal carbonisation, due to their hydrophobic nature, resulted in an increase in both soil leachate volume and nutrient leaching. Higher

temperatures generally increase hydrophobicity and decrease surface free energy, water holding capacity and cationic exchange capacity of the resulting hydrochar (Mau et al., 2018; Röhrdanz et al., 2016). These changes are attributed to the degradation of hemicellulose and cellulose, leaving mostly lignin in the hydrochar (Mau et al., 2018). Based on the results derived from this study could highlight a broader implication on optimising the production techniques to produce hydrochar with suitable properties for soil amendment. Although, hydrochar can also be used for other applications like carbon sequestration, where hydrophobicity factor of the char is less critical.

### **9.4.3 Mitigation of Nutrient runoff**

Some of the chars has a high CEC, enabling them to retain essential nutrient cations such as potassium, calcium, magnesium, and ammonium. This retention helps prevents the loss of nutrients through leaching during irrigation or heavy rainfall, ensuring their availability for plant uptake. Using chars can lower the amount of nutrients like phosphorus and nitrogen that runoff into water bodies and help prevent problems like algal blooms in water bodies (Panday et al., 2020). This study found that the addition of chars produced through conventional and microwave pyrolysis increased soil CEC and reduced leaching, except for phosphate, compared to control soils. This nutrient retention capability enables farmers to reduce reliance on chemical fertilisers, minimising the risk of over fertilisation and its associated environmental impacts, thereby supporting sustainable farming practices. For instance, agricultural land in the UK typically requires approximately 180 kg of NPK fertiliser per hectare, whereas in the UAE, around 327 kg per hectare is used for crop cultivation (Globalen LLC, 2022). Fertilisers, often composed of nitrogenous, potash, and phosphate components, can generate revenues of approximately £3,000 (13,000 AED) per tonne (CEICdata.com, 2018). A back of the envelope calculations was performed to compare the costs of biochars and fertilisers in UAE, considering their application rates and potential benefits:



### 1. Biochar costs and application rate:

- Cost per tonne: Biochar prices vary widely based on the production methods and feedstocks. On average in 2025, a tonne of biochar costs around £3,384 (17,036 AED) (Persist, 2024).
- Application rate: Recommended application rate range is an average of 15 tonnes per hectare depending on the soil conditions (Hornaday, 2023). As a result, it would cost around £50,760 per hectare (255,540 AED per hectare).

### 2. Fertiliser costs and application rate:

- Cost per tonne: The average price for a tonne of NPK fertiliser in the UAE in 2025, is estimated to be around £3000 (13,000 AED) (CEICdata.com, 2018).
- Application rate: Recommended application rate range is an average of 0.327 tonnes per hectare depending on the soil conditions (Globalen LLC, 2022). As a result, it would cost around £981 per hectare (4,251 AED per hectare).

### 3. Potential reduction in fertiliser use with biochar application:

- Research suggested that biochar application can reduce fertiliser requirements by 30% over time due to improved nutrient retention (Gu et al., 2025).
- As a result, annual fertiliser cost with biochar would be approximately £687 per hectare (2,977 AED per hectare).

### 4. Time to recover biochar costs:

- Annual savings in fertiliser costs:  $£981 - £687 = £294$  per hectare (1,274 AED per hectare).
- To determine the payback period, we divide the initial biochar cost of £300,000 by the annual savings of £294. This results in a payback period of approximately 1,020 years.

Based on these estimates, the initial investment in biochar is substantial compared to the use of fertilisers. However, biochar offers additional long-term benefits, such as improved soil health, increased crop yield and increased water retention, which can provide significant economic and environmental benefits over time.

The nutrient retention properties of the char provide significant environmental benefits, particularly by reducing nutrient leaching and improving soil fertility. However, these advantages depend on several factors. For example, soil type plays a crucial role: sandy soils, which have low natural nutrient retention due to their low CEC and surface area, experience greater increase in nutrient retention upon the addition of the char. Similarly, the characteristics of the char itself, such as its porosity, surface area, and cationic exchange capacity, significantly influence its effectiveness. As observed in this current study, the chars with high CEC and surface area when added to the sandy soil, has resulted in an increase in the nutrient retention and reducing the leaching concentration. Additionally, the amount of char applied is a critical factor. For instance, as observed in this study, a 5% char amendment to sandy soil has been shown to increase soil pH, CEC, nutrient retention and increase the biomass yield.

## **9.5 Future research**

Future research should focus on optimising char production conditions to balance nutrient retention and leaching dynamics while investigating the long-term effects of potato peel and palm waste chars under field conditions. By addressing these factors, chars can play a crucial role in sustainable agricultural practices.

### **9.5.1 Optimization of Product Technique**

Future studies should primarily focus on enhancing pyrolysis and hydrothermal carbonization processes to produce chars that meet specific agricultural and environmental needs. This could include adjusting factors such as temperature and residence time for feedstocks to enhance carbon stability, nutrient retention and hydrophilic character and to remove unwanted properties such as hydrophobic character and phytotoxicity. Alternatively, developing economically sustainable methods for commercial scale char production, such as improving microwave pyrolysis could enhance efficiency. Incorporating techniques like stirring and rotation of the material during processing could help disrupt localised heating zones and promote uniform temperature distribution, thereby increasing energy efficiency in

production technologies (Topçam et al., 2020; Ye et al., 2017 and Potnuri et al., 2022).

However, during the microwave pyrolysis of chars in this study, the formation of hot spots was observed under the specific parameters used. Future research on char production via microwave pyrolysis should prioritise minimising hot spot formation by optimising parameters such as microwave power and residence time and also by amending the physical characteristics of feedstocks such as particle size and moisture content. Decreasing microwave power ensures more uniform energy distribution throughout the material, which minimises uneven heating and localised overheating. Additionally, increasing the residence time allows the heat to penetrate more evenly throughout the material, reducing the occurrence of hotspots. These adjustments are critical for achieving consistent thermal treatment and ensuring more uniform physiochemical properties in the treated material.

In this study, it was observed that hydrothermal carbonisation of potato peel and palm waste produced hydrophobic hydrochars. Further research should focus on optimising the temperature and residence time to further enhance the hydrophobicity of the chars. For instance, lower temperatures and shorter reaction times result in minimal decomposition of oxygenated functional groups, allowing them to retain on the surface and making them more hydrophilic, which would enhance its water and nutrient capacity (Fan et al., 2022; Wilsey et al., 2022).

Additionally, the study should examine the physical characteristics of the feedstocks, such as moisture content and particle size, which significantly influence the heating mechanisms during pyrolysis. Uneven moisture distribution in biomass causes inconsistent microwave energy absorption, with moister area heating faster and creating hotspots. Similarly, irregular particle sizes also impact energy absorption, with larger particles tending to form hotspots (Lin et al., 2022; Chen et al., 2024; Lei et al., 2009). Addressing these factors is essential for achieving uniform charring and enhancing the overall quality and characteristics of the char. Improved understanding and control of these variables can lead to better scalability and efficiency in microwave pyrolysis, making it more reliable method for producing high quality char.

### **9.5.2 Mitigating phosphate leaching**

Future research on nutrient retention in char amended soils should prioritise understanding and mitigating phosphate leaching. Although some of the char treatments in our experiments effectively reduced nitrate leaching, they also led to an increase in phosphate leaching. The increase in the phosphate leaching was due to the electrostatic repulsion of negatively charged phosphate ions on soil surfaces and the slow uptake of available phosphate by plants, leading to higher leaching. Phosphate leaching impacts the environment by contributing to eutrophication, which causes algal blooms, oxygen depletion, and aquatic ecosystem. It also degrades water quality, increases treatment costs and contaminates ground water (Akinawo, 2023; Arnob Dhar Pranta et al., 2023). During the plant-pot experiment, algae formation was observed in the vessel used to collect the leachates, indicating the presence of high concentration of phosphate levels in leachates. Possible approaches to mitigate phosphate leaching include combining biochar with materials or modifying the surface properties of biochar through specific coatings that enhance anion retention, such as zeolites, or metal oxides. These materials increase the soil's adsorption capacity by providing sites that can effectively bind negatively charged phosphate ions, thereby reducing their mobility (Keskinen, et al., 2021; Gerstein et al., 2019; Waller et al., 2023; Dey et al., 2023). These approaches not only improve nutrient retention but also minimise the environmental risks associated with phosphate runoff.

### **9.5.3 Long-term data and Field based studies**

Large-scale and long-term field trials are required for the actual implementation of using char in sustainable agriculture. Such studies need to assess soil fertility, nutrient recycling, water regimes, and crops production cycles on the subsequent year (Wang, et al., 2022). Also, studying the effects of char under prevailing climate conditions including drought, heavy rain, and temperature stress will also offer insights of the product's robustness.

#### 9.5.4 Energy consumption in biochar production techniques and economic viability

A comparative analysis of energy consumption across different production techniques, such as microwave pyrolysis, conventional pyrolysis, and hydrothermal carbonisation, could provide valuable insights into the efficiency and sustainability of these methods. The average energy consumption for microwave pyrolysis ranged from 2.001 to 6.139 kWh/kg, for conventional pyrolysis from 2.111 to 6.064 kWh/kg, and for microwave hydrothermal carbonisation from 1.344 to 6.001 kWh/kg (Allende et al., 2023; Shao et al., 2020). These findings highlight that microwave hydrothermal carbonisation generally requires less energy than the other techniques at its lower range, suggesting potential advantages in terms of energy efficiency. However, the broader range observed for hydrothermal carbonisation implies that under specific conditions, the energy demand may approach that of the others.

The implication of these energy costs becomes particularly significant when considering additional process steps. For instance:

1. Feedstock preparation: In this study, all feedstocks underwent the same preparation steps before char production. However, a potato peel waste had higher water content compared to palm waste, requiring a longer drying time. As a result, the drying process for potato peel waste consumed more energy than that for palm waste. This increased energy demand impacts the overall energy efficiency of using potato peel waste as a feedstock. Future research should focus on energy-efficient drying methods, to reduce energy demand for high moisture feedstocks.
2. Modification of the equipment: While energy consumption for microwave pyrolysis, conventional pyrolysis and hydrothermal carbonisation overlapped in the broader range, high temperature and pressure build up during microwave reactions posed challenges for equipment durability. This could necessitate additional safety and cooling mechanisms, further influencing energy requirements.

Further research should also focus on the economic feasibility of large-scale char production, identifying potential challenges, and finding solutions to address them. Such as adopting renewable energy sources, such as solar or wind, for these processes could offset energy costs and enhance sustainability. Large scale production could also benefit from economies of scale reducing per unit cost. Additionally, optimising reaction times and feedstocks

properties could improve energy efficiency and help integrate chars into sustainable farming practices more effectively.

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