Properties and Applications of Himalayan Nettle Fibre

by

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to Ada & Gino,

Francesca, Şenhan and Ayşe

'We should not impose artificial, external limits on ourselves. If you have dreams and ambitions, try to find the way to fulfill them. Sometimes an obstacle is just a message that life is sending you. You need to find another way, but that does not mean you can not reach your destination.'

ASI/ESA Astronaut – Captain Samantha Cristoforetti

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Ministry of Foreign Affairs
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Life, such as research, takes unpredictable roads, sometimes tortuous, sometimes, rarely, downhill. Each step should give us the awareness that, until we want it really, we can take the next step and a few more in the direction that we desire, the one that intrigues us the most and makes us passionate. Our will, our intellectual ability, allow us to continue our journey, without compromise, truthful to ourselves, respecting others and our moral integrity.
Thank you because now I know that every challenge can be accepted, if one sets the mind to it.

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Abstract

The persistent demand for sustainable and eco – friendly fibres able to achieve high performance materials drew the attention to bast fibres, which, in this regard, represent an attractive resource. They are characterised by their fineness, flexibility, and excellent tensile properties due to the cellulose volume fraction. In particular, the fibre of the Himalayan giant nettle plant (*Girardinia diversifolia*), which grows in Africa and Asia, simultaneously provides social and environmental benefits as well as attractive physical properties, making it an interesting fibre for high performance sustainable textiles.

This research provides an unprecedented investigation on giant Himalayan nettle fibres, currently processed and used in the handicraft, performed in parallel with common European nettle fibre (*Urtica dioica*) already used in the textile industry. The morphological, physical, and mechanical characterisation of *G. diversifolia* fibres provide encouraging results, indicating a substantial superiority (in terms of physical and mechanical properties) of Himalayan nettle fibres compared to the most common textile bast fibres. Himalayan nettle fibres displayed higher tensile strength, tensile modulus, and elongation at break with respect to European nettle fibres. Furthermore, the Himalayan nettle fibre is the longest bast fibre known so far. Its moisture content results indicate effective dielectric insulating capacity. The values of moisture regain, inferior to cotton, show that the fibre physical characteristics are not affect by humidity.

Treatment with increasing concentrations of sodium hydroxide (NaOH) was carried out in order to induce changes in the molecular structure, from Cellulose I to Cellulose II, of both Himalayan and European nettle fibres, and to assess how this variation could influence their physical and mechanical characteristics.

This research has identified as best result the fibre versatility: the ability to obtain, with alkaline treatment, characteristics of the fibre able to meet different purposes. To make the fibre attractive for the textile industry, Himalayan nettle fibre has to be competitive demonstrating that it can work in

technical textiles. The field of outdoor sportswear, where a high level of comfort is required, has been chosen to demonstrate the performance of the Himalayan nettle fibre hypothesising a multilayer fabric made of three Himalayan nettle nonwovens treated at three different concentrations of NaOH.

Based on its superior fibre length and mechanical strength, Himalayan giant nettle fibre could be a very promising material for advanced nonwovens where high – performance materials are required.

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Chapter 1

Introduction

> *The world will not evolve past its current state of crisis by using the same thinking that created the situation* [1].

This research concerns eco – sustainable fibres, defined as fibres with minimal environmental impact at every stage of their production. The subject of this study is the fibre commonly known as Himalayan nettle fibre extracted from the stem of nettle species *Girardinia diversifolia*. The fibre is used in Nepal, where the plant is most widespread, at a handcrafted level and in spite of its attractive characteristics it is not yet present in the textile industry and so far there is no in – depth literature regarding its investigation for industrial purposes. Therefore, as first objective, this research aims to provide a detailed description of *G. diversifolia* fibre to demonstrate its suitability for the industry. The achievement of this goal will hopefully encourage the economic, sustainable development of the poor areas where this nettle grows. This study also aspires to demonstrate the versatility of the fibre, which, subjected to specific treatments, could improve some of its several features and its potential to be used in the textile to meet different needs. For a natural and sustainable fibre, such as the *G. diversifolia* fibre, to firmly establish itself in a state of 'Pareto optimality' [2], it must demonstrate its competitiveness with respect to highly performing fibres used in areas such as the field of sportswear, where different features of

comfort and protection are required. The project concerns an examination of Eco – friendly fibres as possible candidates for the niche of High – Performance garments. The research includes:

Part I: *Girardinia diversifolia* fibre analysis

- An overview of issues related to the textile industry and in particular the high – performance fibres;
- A review of bast fibre features;
- An investigation and comparison of *G. diversifolia* (Himalayan nettle) fibre and *Urtica dioica* (European nettle fibre) fibre, both raw, scoured and treated with different concentrations of sodium hydroxide;

Part II: Versatility and application of treated *Girardinia diversifolia* fibre

- Study of the fibre versatility by nonwoven technology;
- Proposals for applications of treated *G. diversifolia* fibres;
- Conclusion and suggestions for further work.

1.1 Textile industry: global data and issues

Today the textile industry represents one of the major industrial sectors. In 2000, consumers spent around $ 1 trillion worldwide buying clothes: one third of sales were in Western Europe, one third in North America and only one quarter in Asia [3]. Total UK consumption of textile products until 2004 was approximately 2.15 million tonnes equivalent to approximately 35 kg per capita. With an increase in demand from 1990 to 2004 for natural fibres from 47 million to 67 million tonnes *p. a.* (Figure 1.1) [3].

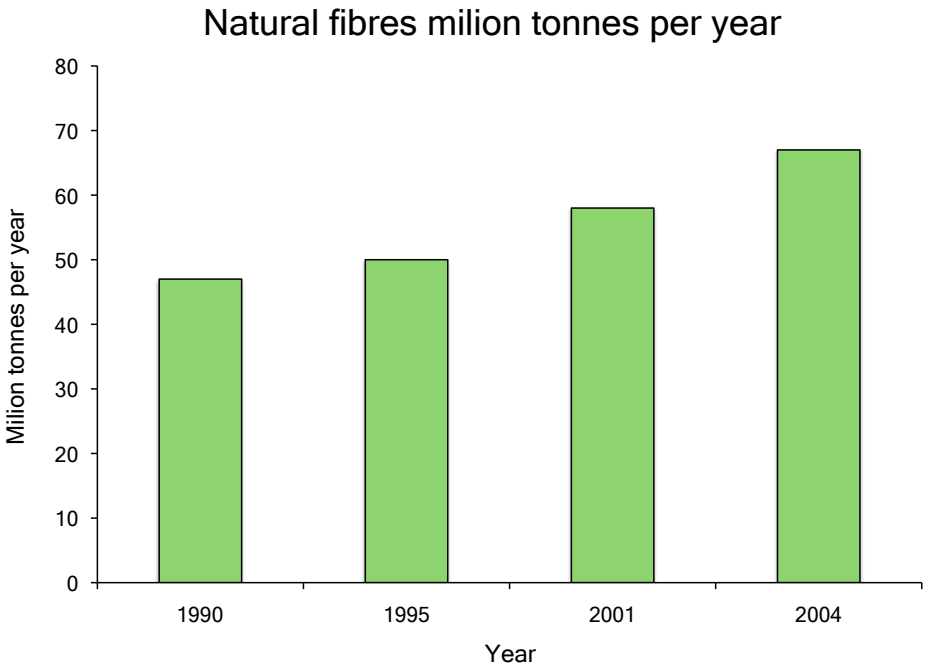

Figure 1.1: World demand for natural fibres from 1990 to 2004 [3].

After nearly a decade, the European Commission recorded an increase in textile, clothing and footwear production [4]. The consumption of textiles in the UK for 2010 was estimated at 2.7 million tonnes [5]. Due to the problems of destocking, turnover has higher growth with respect to production, even the export values increased slightly, thanks to the Middle Eastern and Asian countries attracted by the exclusivity and sophistication of European leather and clothing products. A phenomenon that hinders the economic growth of the European textile is represented by the strong competition South East Asian countries, where there is the highest concentration of textile factories, pushing for an increase in volume respect to the quality of the textile [4]. This is why European countries such as France and Italy, but also Spain and Portugal, maintain the headquarter company, the business and the flow of products in Europe, but they are relocating the manufacturing towards competitive countries, low – wage countries, for example in the Central / Eastern Europe (Turkey has an export of cotton of $ 777 m to EU and USA), in North Africa, in South

America and in the East Asian (India is the second largest producer of textile after China) (Figure 1.2) [3].

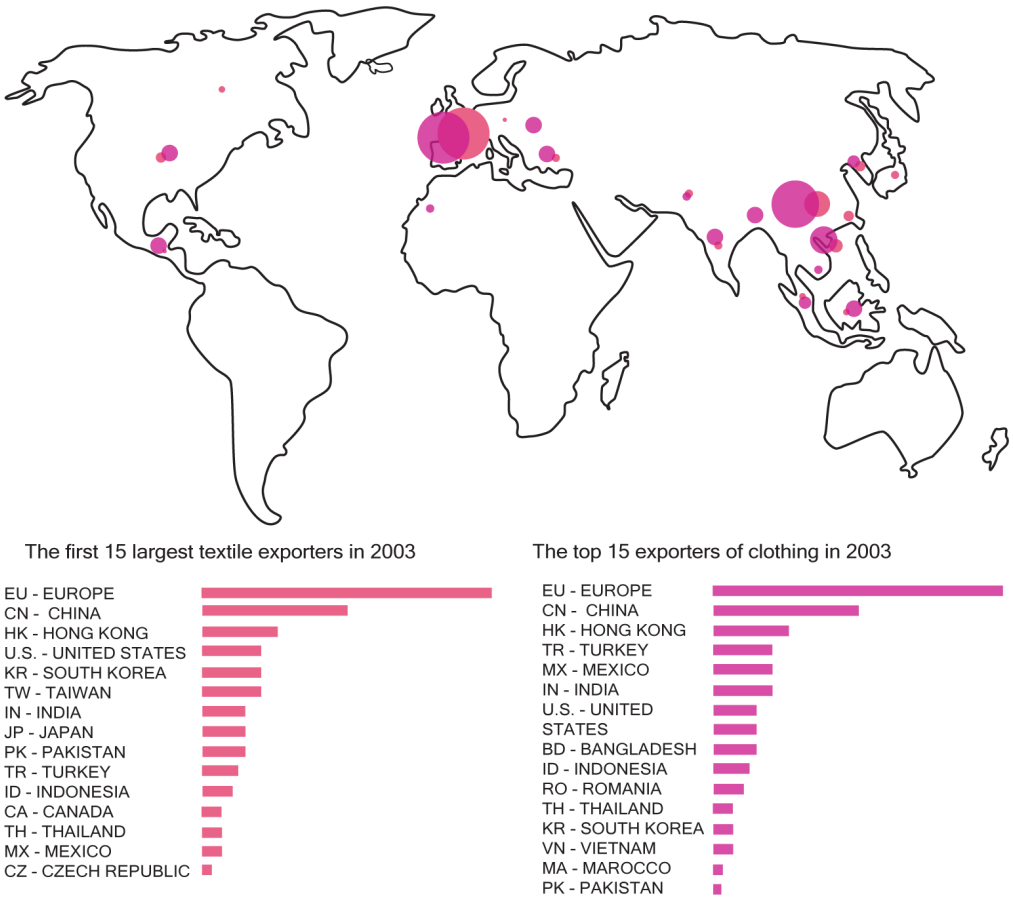

Figure 1.2: The largest 15 textile and clothing exporters in 2003 [6].

It is simple to imagine how an industrial sector of this magnitude can become a global phenomenon, not only in terms of economy, but also from a social and environmental point of view. This industry affects the environment with energy and toxic chemicals consumption, with biodiversity destruction, and finally for disposal of products / waste at the end of their life cycle. Ethical and social problems associated with production and labour exploitation also need to be considered [7]. A typical production cycle causes problems regarding energy consumption: it is consumed mainly by agricultural machinery, production machinery and by heating air and water in laundering. Chemicals are used in fibre crop production, like cotton, and at some stages of processing, such as

pre – treatment, dyeing and printing, and they can have a strong environmental impact. In particular, the consumption of water, required during dyeing and recycling, produces polluted water. Fibre crops grown in plantations, like cotton, require a large amount of water. Moreover, the economic advantages of extensive monocultures discourage the growing of other plantations, destroying the biodiversity. The problem of waste in this sector is growing also due to the advent of the so – called 'fast fashion'; in the UK, 30 kg per capita of clothing and textile are disposed of every year. The problems of the textile industry affect not only the environmental but also the society as very often the workers employed in this sector work in inhuman conditions and underpaid. There a many campaigns in the world to defend worker rights and to improve the conditions of the workplace. The UNICEF estimates that one quarter of the entire child population in Bangladesh is employed in the textile industry carrying three hundred different types of tasks [3], [5].

1.1.1 Need for a sustainable textile industry

One of the important solution for the environmental damages and the social situations caused by textile industry seem to be 'the green textile' (*i. e.* the textile industry which uses as raw materials vegetable fibres from organically crops) [8], [9]. These fibres have many positive aspects that can meet the demands of responsible consumers [10]:

- Low energy inputs;
- It can be processed locally;
- Wide range of products;
- Fibres for high quality textile [11].

Fibre plants are fully exploited in different fields. In fact, the plants not only provide raw material for textile, but also for food/feed, cosmetics, and phytomedicine [12]; areas where the use of natural products is growing in

step with the consumers' sense of responsibility towards the environment and body. In particular the consumers seek products that are healthy for the body, comfortable, with a low environmental impact, made from companies that have an ethical sense and traceability of their products is known. To cope with this demand, different solutions are being proposed such as the Sustainable Apparel Coalition (SAC), which considers a Quick Response (QR) code system that allows consumers to view product traceability using their smartphones [13], [14]. The data reported by Ethical Purchasing Index, launched in 1999, and updated annually by the Co – Operative Bank in its Ethical Consumerism Report, shows that the market respecting an ethical sense in the UK is valued at around 2%, but many indicators suggested a clear rising trend. In fact, the report of 2012 confirmed that, despite the onset of recession five years ago, the value of ethical markets in UK has risen from £ 35.5 bn to £ 47.2 bn. According to the most recent figures UK, spending on personal products, such as cosmetics and eco – fashion, (*e. g.* Fair – trade cotton) £ 1.8 bn, with a growth from 2000 to 2011 of 4.2% [15]. Even in countries traditionally more conservative in terms of habit changes, such as Italy and Spain, consumers' attention towards sustainable production is growing: from a market survey conducted in Italy by *Natural.Tex* [16] it emerges that for all the reasons listed above, consumers are willing to support a higher cost for organic textiles. The plant fibres may be a strong reality in the future of the textile industry; they can meet new market demands, tackle the insurmountable environmental problems, and help developing countries [16], [17].

1.1.2 Sustainable fibres – the green textile

As per the World Commission on Environment and Development (WCED), sustainable development meets the needs of the present without compromising the ability of future generations to meet their own needs [18]. The development of society operating in harmony with the environment should preserve the qual-

ity and quantity of heritage [19]. Vegetable fibres are considered a renewable resource when the production meets the following [3]:

- Use of renewable and clean sources of energy;
- Use of renewable materials;
- Use of materials and substances that are safe for humans and the environment;
- Minimal environmental impact at every stage of its production;
- It does not pollute the procurement of food and water;
- It replaces the existing product with one which provides the same feature improved;
- It creates a competitive price on the market.

Vegetable fibres are composed of cellulose, a natural polysaccharide which decomposes naturally through the chemical secretions and enzymes by reducing in a short time the cellulose fibre to simpler elements and compounds without release of toxic substances [20], [21]. In nature, the fibrous material is present in abundance, extracted from leaves, fruits, stems and shells seeds. It used for different purposes: as textile fibres; as composites for insulation materials in building; the components of the plant are also exploited in cosmetics, medicine, and for fine chemicals used as starting materials for specialty chemicals whose market includes primarily pharmaceutical, agrochemical and food feed industries [21], [22]. The products made by cellulosic fibres can be called sustainable when their life cycle has a minimal or no impact on the environment and no adverse influence on lives and health (Figure 1.3). This definition leads to the following prescriptions in every stage of production, consumption and disposal of the product [21]:

- Use raw materials from renewable resources;
- Do not use energy derived from non – renewable sources such as oil;

- Decrease the volume of waste;
- Increase the compostability and complete biological degradability in the natural cycle;
- Reduce carbon dioxide released to protect the climate;
- Eliminate environmentally harmful chemicals.

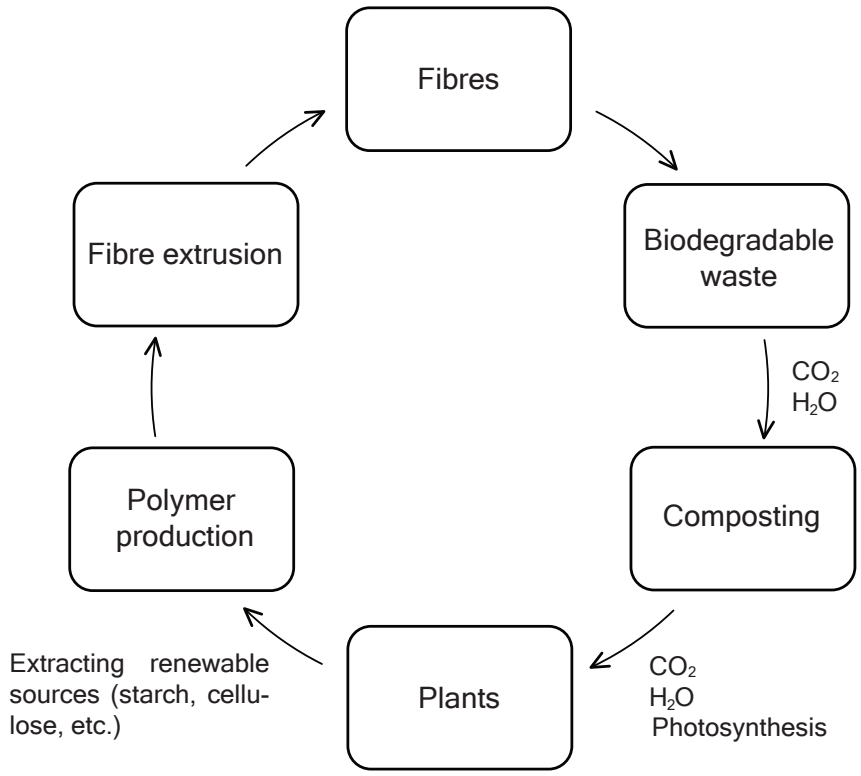

Figure 1.3: Life cycle of compostable, biodegradable fibres [21].

1.2 High Performance fibres

The textile market today is not asking only for eco – friendly fibres, but also and mostly, fibres able to meet high performance [4]; increasingly, highly technical fabrics are adapted to meet the comfort of everyday life. A good example to understand this trend is the line of towels Spaziale Splendy made

with TexWinner®, a fabric that was born from an idea by Emilio Mina, president of GIL, to take advantage of microfibre technology used by NASA, which dries 75 times faster than a regular towel (Figure 1.4) [23].

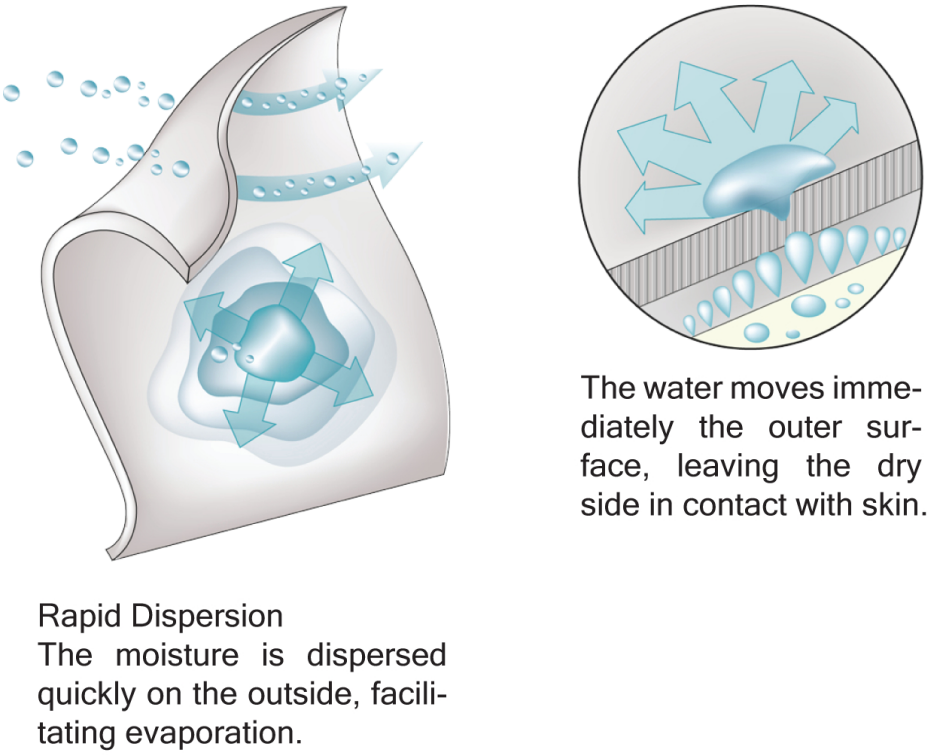

Figure 1.4: Splendy – TexWinner®technology [23].

In order to be competitive in the market, renewable fibres must prove strength and expectation in the more elitist sector, *i. e.* the field of high performance fibres. High Performance fibres have specific physical properties to meet specific technical requirements, including high tensile strength, temperature resistance, limiting oxygen index and chemical resistance. Each single fibre combines these properties in a unique way and fills a niche in the spectrum of High Performance fibres, namely:

- Protective properties;
- Well – being factors;
- Smart and interactive textiles.

Nowadays, engineering and high advanced technologies can create synthetic materials that combine high performance with aesthetic qualities [24]. High Performance fibres are sought in areas for protection and comfort for the body, when individuals are exposed to an adverse or difficult environment, or when in direct contact with harmful substances. Particular sectors of the market in which there is a continuous innovation are that of extreme sports (in which the aesthetic issue and branding are essential elements) and professional fields [25]. For example, sports that are practiced in the summer, with high temperatures, require fabrics that provide comfort and control of body temperature. Similarly, water sports clothes are designed to facilitate floating and to maintain the body warm for many hours. In professional fields, individuals who work with dangerous substances must be isolated from possible contact with them [26]. The textile industry must constantly change to satisfy consumer demands and it is driven by the development of new materials, innovative manufacturing processes and the requirements of a global market. Therefore High Performance fabrics are not only used to make us and our environment more attractive and comfortable, but they have to meet also further additional demands and supply new functions. As people become more and more concerned with health and general wellbeing, textiles can play an important role against harmful bacteria, for example through the release of substances as providing daily dose of vitamin C [27].

1.2.1 High Performance fibres are not sustainable, yet

At present, High Performance fibres, which dominate the market are all synthetic, therefore they can be neither disposed of nor easily recycled. Indeed, the problem to disposal of the fabrics used in high performance textile it is not only the synthetic fibres used, but the fact that often they are made from an assemblage of many synthetic fibres or films, often in complex arrangements or layers, which makes their recycling even more problematic. Among

the most widespread High Performance fibres are: Goretex®[28], Hyvent™[29], and H2No®[30] (Figures 1.5 and 1.6). Originally conceived for specific contexts, such as extreme environments, these are now in common use. Looking a little further ahead we will see garments containing interactive fabric elements. Smart textiles are those that respond to, or are activated automatically by external stimuli. These exciting developments will ensure that fabric innovation plays an important role in future technological advancement [21].

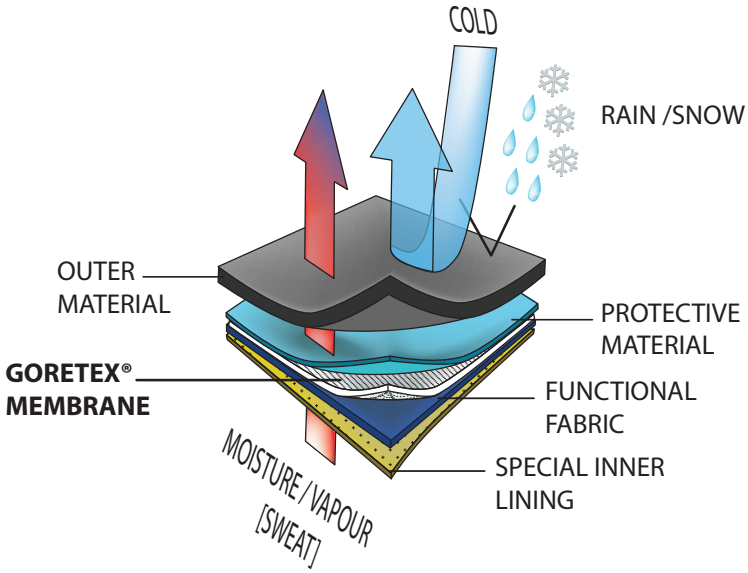

Figure 1.5: High performance fabric structure with the inner Goretex®membrane [28].

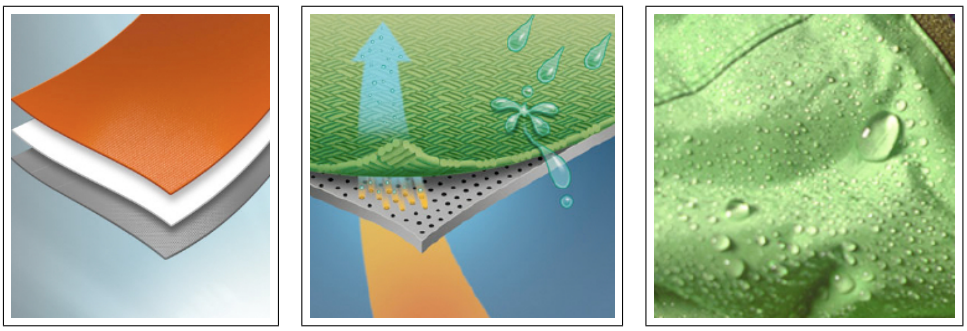

Figure 1.6: Examples of High Performance fibres: Goretex®(left), Hyvent™(center), H2No®(right) [28], [29], [30].

1.3 Field of investigation – Sustainable + High Performances fibres: a new product

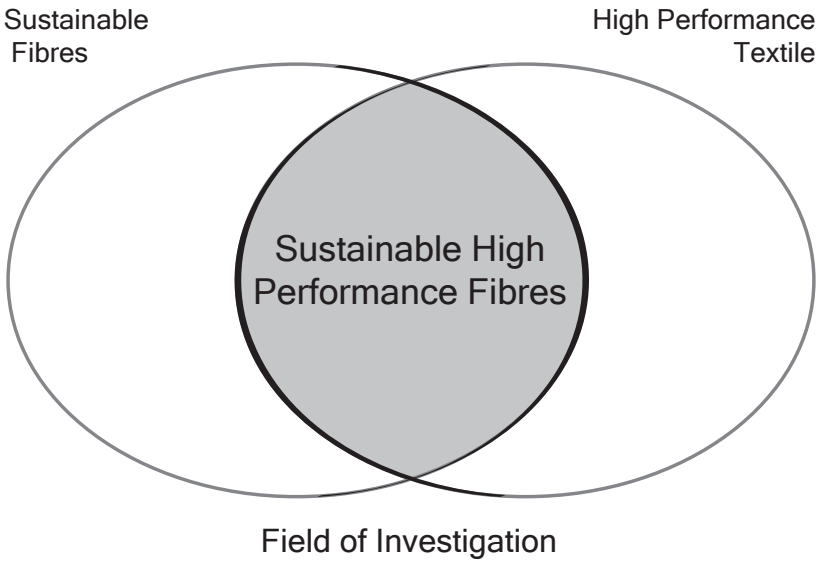

Figure 1.7: Field of investigation: High performance and sustainable fibres.

The field of investigation of this work is the intersection of the above sets (Figure 1.7), *i. e.* the study of natural fibres that have the potential to become High performance (eco) fibres. Eco – friendly fibres like flax, hemp, nettle, jute, to mention the most common, might meet demands for wear resistance, breathability, thermal insulation, resistance to traction, *etc*. Several companies already started experimental sustainable projects, for example *Nike with Nike Considered Design* [31], a new line of products that combines the principles of environmental sustainability with innovation for sport, Adidas with *Adidas Originals* continues its ecological campaign by presenting the new line of sneakers *Grün* (Green), strictly eco – friendly [32], and Timberland with the new ecological shoes *Earthkeepers*, made of recycled rubber and organic cotton, with finishes in recycled PET [33]. These examples are very interesting not only from an ethical viewpoint but also from the design one. However the problem that remains in these projects is that they seem to be more market oriented and

they do not provide performances able to replace existing High – performance products. Natural fibres must demonstrate that they are capable of competing with the materials now used in niche areas, replacing or combined with fibres that today's respond to the demands of high performance. The intersection of two of the current trends in textile industry, on one hand the Eco – Sustainable fibres and on the other the High Performance textiles represents one of the possible futures of the textile market: The **Eco + High Performance fibres**. The case study for this research is represented by a natural fibre: The Himalayan Giant Nettle (*Girardinia diversifolia*). This particular plant possesses both social and environmental aspects and physical characteristics, which make the fibre particularly interesting from the point of view of social / technological research.

1.4 Case study: Himalayan nettle fibre

Existing literature on the Himalayan Nettle fibre is not extensive; most investigations made so far were performed exclusively in Nepal and they are an account of the natural habitat of the *Girardinia diversifolia* [34], [35], of the craft activity currently related with this fibre and of the opportunity to open a way to the industrial development of Nepal and they do not allow a complete characterisation of the fibre [36], [37], [38], [39], [40]. The scarcity of specific information on this fibre is due to the fact that plant of Himalayan nettle, to date, has not been regarded as a raw material for textile and other industries, as a means for the development of the countries where it grows. For the purposes of this work the study has been structured as a framework in which the subject, the Himalayan fibre, is related to different fields of interest and to research undertaken for similar subjects, like other European nettle and bast fibres. In the remaining part of this dissertation when referring to Himalayan Nettle fibre, this specifically is Himalayan Giant Nettle (*Girardinia diversifolia*) and European nettle fibre is *Urtica dioica*.

1.4.1 Bast fibres

G. diversifolia fibre is a bast fibre. Bast fibres are: soft, woody fibre obtained from stems of dicotyledonous plants (flowering plants with two seed embryonic leaves) and used for textiles and cordage. Such fibres, usually characterized by fineness and flexibility, are also known as soft fibres, distinguishing them from the coarser, less flexible fibres of the leaf, or hard, fibre group [41], [42], [43]. Bast fibres are composed principally of cellulose which is the most important structural component of all green plants, hemicellulose (an amorphous branched or non – linear structures with little strength) and lignin that gives mechanical stability during the plant maturation [44]. The microstructure of a natural fibre is complex, having different lengths and different materials: the fibre consists of two cell walls arranged as concentric cylinders with a small central channel called the lumen, which contributes to the water uptake (Figure 1.8) [45]. The lumen produces additional porosity that reduces the capacity of the fibre to withstand load under tension.

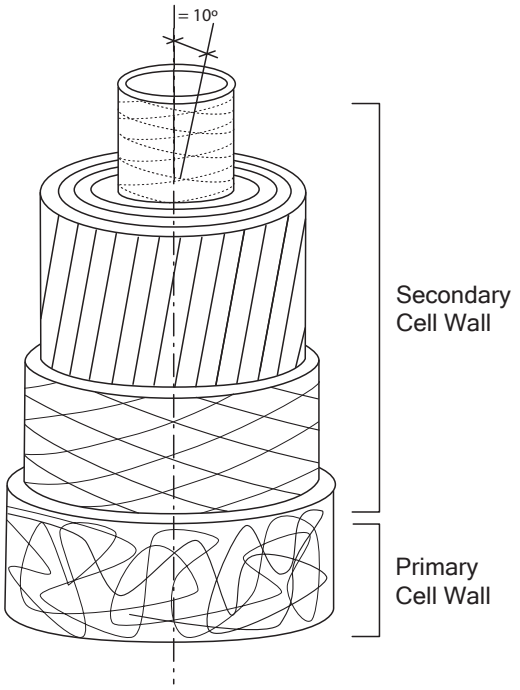

Figure 1.8: Structure of a flax fibre [45], [46].

Bast fibres have competitive mechanical properties; because of the volume fraction of cellulose (*e. g.* flax about 70 % [47]), they have good tensile property [48], which indicates how a material reacts to a force applied in tension [49], [50]. In particular, it is noted that flax and nettle, which are cellulosic hygroscopic fibres (*i. e.* they have the ability to absorb water molecules present in the environment [51]), have properties that vary depending on environmental conditions, and for this reason the physical tests on these plants are made in suitably air – conditioned environments, usually at 20 °C and 65 % relative humidity [52]. In natural fibres, strength typically increases with moisture content [44]. Another characteristic that influences the tensile behaviour of these fibres is the orientation of microfibril (a very fine fibril, or fibre like strand, consisting of glycoprotein and cellulose [53]), which comprises both crystalline and non – crystalline (amorphous) regions (Figure 1.9); the relative crystallinity of the fibre has significant effects on fibre properties. From an observation of fibrils under the microscope it is possible to discern the cellulose fibres: hemp and jute have a **Z** – twist, while flax and nettle an **S** – twist (Figure 1.10) [54].

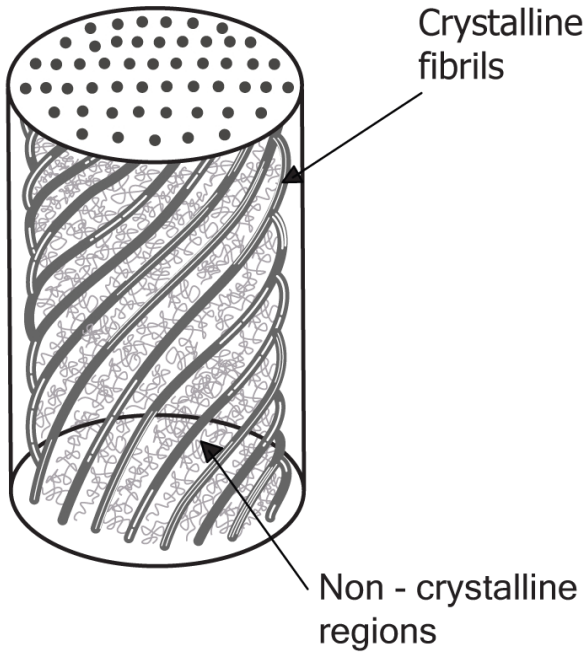

Figure 1.9: Principle of the flax fibre structure. Helical arrangement of fibrils in natural cellulose fibre [45], [46].

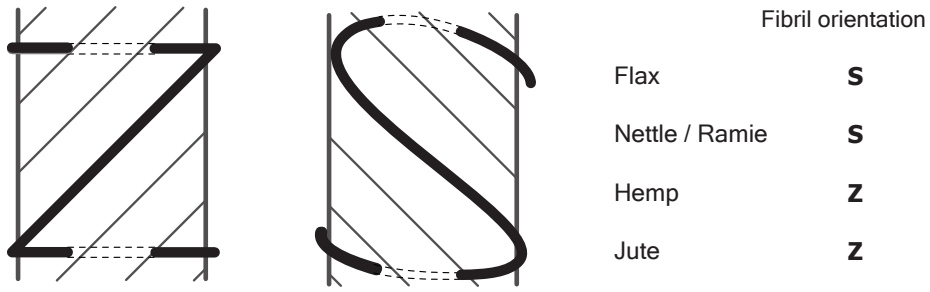

Figure 1.10: A diagram of **Z** – twist and **S** – twist [54].

Bodros & Baley define flax and nettle as high performance fibres [55]. Indeed this could be deduced by the tests on these fibres and from their physical characteristics, for example the stress – strain curves of stinging nettle and flax are linear (Figure 1.11) [49], [52].

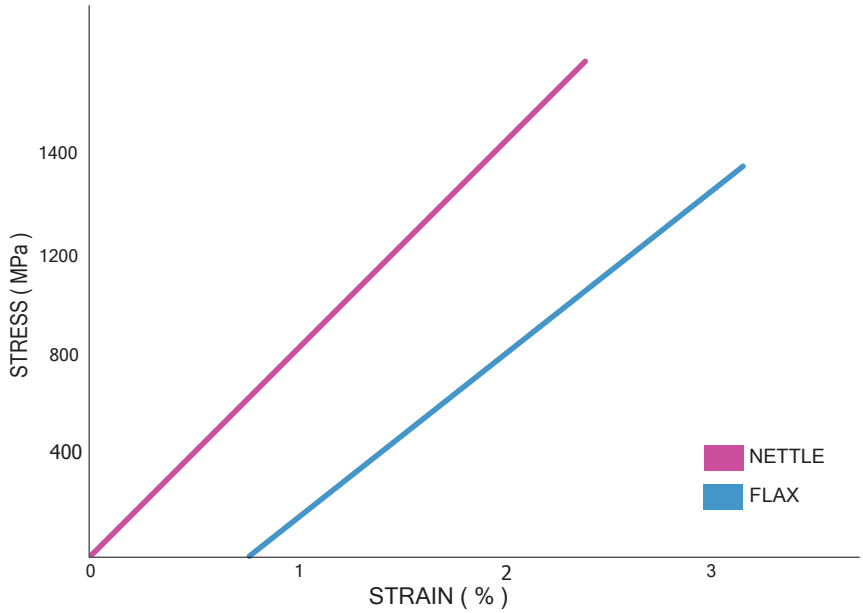

Figure 1.11: Typical Stinging nettle and flax fibre / strain curve [55].

Cellulosic fibres have the advantage of often being less expensive than synthetic fibres and have competitive mechanic features: cellulose fibres have a Young's Modulus [56] of ~ 140 GPa, comparable with values of manmade fibres such as aramid [44] (131 – 186 GPa [57]). Furthermore, they have low density,

which guarantees lightness of the material; they are found worldwide and they are recyclable [52]. From the literature [44], [54], [58] it is possible to infer that, stated the promising characteristics of cellulosic fibres, it is appropriate using these in composites materials as a substitute of mineral fibres, such as glass, carbon or asbestos fibres (Table 1.1) [54]. In addition to the use of bast fibres in composites they could be blended with other fibres, as viscose and silk, in order to obtain fabrics with most fitting and comfortable features [54].

Table 1.1: Typical properties of some bast fibres, with comparative values of the most common glass fibre, E – glass (composed of alumina – borosilicate glass with 1 % w / w alkali oxides [58]) [38], [44].

Properties	**E – Glass**	**Flax**	**Hemp**	**Jute**	**Kenaf**	**Nettle**	**Giant nettle**
Density kg / m^3	2250	2530	2520	1520	1193	–	–
E – Modulus (GPa)	71	58 ± 15	70	60	14 - 38	87 ± 28	5.8 - 22.5
Tensile Strength (MPa)	3400	1339 ± 486	920	860	240 ± 600	1594 ± 640	180 - 550
Specific Modulus	28	38	46	39	12-13	–	–
Elongation at failure (%)	3.4	3.27 ± 0.4	1.7	2	–	2.11 ± 0.81	–
Moisture absorption (%)	–	7	8	12	–	–	–

1.4.2 Economic aspects of bast fibre production

Natural fibres are generally produced by countries that are becoming textile – manufacturing giants such as India, China and Bangladesh as they offer the possibility of a low – cost manufacturing. European and American companies, connected with the textile industry, buy the production of their own goods, starting from the raw material, exclusively or for the most part from Asia, Eastern Europe and Central America companies enriching exponentially a bracket of private businessmen [41]. The major western commercial centres still have the leadership of marketing in terms of trend and sales / distribution [41]. The case of bast fibres is different from the natural ones used for the large scale distribution, such as cotton, a seed fibre. The bast fibres are generally produced in developing areas of countries that have become economic powerhouses in the textile industry and where a large gap in wealth between the regions exists. The activity related to bast fibres usually happens in villages, curated by medium, small and mostly family – run business [41].

The cut stems of bast fibre plants are left to macerate in water (retting) and further the fibres extracted manually or mechanically. Manufacturing is performed on old machinery, not entirely suitable for the type of fibre, which is one of the main reasons why the fibres obtained are of poor quality. The bast fibre business is financed by government funding, local or global, along with associations working for developing world poor areas. Bast fibre production is very expensive and limited for two main reasons [41], [59]:

1. The 'green decorticating' take a long time to obtain a satisfactory degree of clean fibres;
2. The fibres may be damaged by the biological extraction process, whereby to get a fibres amount satisfactory to the final sale, a much more large initial production is required.

One of the problems with bast fibres is to reduce the extraction cost through an eco – friendly process, which allows obtaining fibres of suitable

quality [59]. These fibres can be also used and processed in different ways, such as nonwoven and composite technologies. However, it is difficult to define and compare the price of all bast fibres since the costs vary between fibres depending on the quality considered, as in the case of flax, and depending on the exchange rate between the country that supply the fibres and the country that demands it. Examples of bast fibres production with an annual output of at least 50 thousand tonnes and distributed internationally, as in the case of jute with 2 million tonnes per year, are described below in order to understand how their business operates and in which situation it currently finds [41], [59].

1.4.2.1 *Jute: Withe jute (Corchorus capsularis) and Tossa jute (Corchorus olitorius)*

Compared to the 1980s, the market for jute declined from 1.3 million to 1 million tonnes of jute goods. Jute is a very expensive material due to the intensive labour that its production requires, therefore, the risk of labour exploitation would be much higher if this were not governed by the rules of production [41]. The production varies from country to country: in India the production is three times superior to the 0.5 million tonnes per annum achieved in Bangladesh. In India, the old machinery is used and a big percentage of the products are directed to the domestic market, while Bangladesh works in large part for the export then having to meet international standards [41], [60].

The largest producers of jute are India and Bangladesh (Table 1.2), and interest in this material in being revived by demonstrating its applicability in different sectors from the geo – textile, to textiles, to furnishing fabrics (whose demand is strong in Europe and America). The consumers of the richest countries, increasingly attentive to the eco – friendly perspective, appreciate the versatile qualities of jute fibre. Jute fibre offers advantages in terms of processing costs by requiring no special looms and blends with other fibres have interesting performances. Biological fibre extraction cannot be taken into consideration since too expensive [41], [60], [61].

Table 1.2: Developing countries world exports of jute products [41], [62].

Developing country	**Export in 1000 tonnes**
Bangladesh	408.5
India	176.5
Nepal	10.0
Thailand	6.9
China	6.5
Africa	1.4
Mexico	0.3
Syria	0.3
Turkey	0.3
Guatemala	0.2
Egypt	0.1
Total	**616.1**

1.4.2.2 *Flax (Linum usitatissimum)*

In the past, production of flax took place in Western Europe and it was of an excellent quality compared to those produced today. Following the collapse of the communism, companies have left the business centres in Western Europe and moved the flax – growing in Eastern Europe and African countries where the labour cost is so low as to cover also the costs of the re – import of the material. In the USA the plantations are made in Mexico for the same reason [41], [63]. Europe and the USA are the largest consumers of linen. Reducing the costs and the marketing of the big retail has encouraged a wide flax distribution. This policy of exploiting countries with low labour costs is also used for fibres such as cotton and polyester. The cost of linen in any case remains higher being a bast fibre must be subjected to a long and laborious

retting process that can damage a significant amount of fibre [41].

The subsidies that European countries receive serve to maintain low costs, otherwise Asian countries would increase the prices. Another factor is that there is not specific machinery to flax processing and this is reflected by the low qualities of the obtained fibre. There are only three companies that have used special machinery for flax, Bridge Mackie (China), Linimpianti (Italy) and Orioltexmash (Russia) but their business is not comparable to that of fibres such as cotton or synthetic fibres, whose industry is much more modern. Linen is a product intended for an exclusive upmarket. Its costs and quality varies depending on where it is produced. In 2003, the linen cost €1.95 to 3.2 per kg (depending on where in Asian countries it was produced) against the €15 per kg for flax produced in Europe. In Normandy, Belgium and the Netherlands are the most experienced flax growers and workers (Table 1.3) [41].

Since 2004, many Eastern European countries joined the European Union and they had to abide the rules and limit the growing areas to protect the bio – diversity and prevent environmental destruction. Having extensive plantations would be a solution in economic terms because the yield quantity and quality of fibre flax crops depends on weather conditions. Many manufacturers in Eastern Europe are not willing to deal with a business so risky. Employing an extraction process which avoids retting could reduce costs, while the quality of the fibres could be enhanced by cultivating flax in temperate areas. Unfortunately, nowadays European producers buy flax from the countries where it low cost and quality and mix it with their select linen; then selling it as fine linen produced entirely in Europe. Sadly, this type of policy is extended to many luxury products whose traceability is difficult [41], [63].

Table 1.3: European production of flax fibres in 2005 – 2006 [64].

Country	Tonnes
France	178,500
Belgium	26,547
Netherlands	8,412
Czech Rep	4,700
Lithuania	4,337
Poland	2,024
Austria	213
Finland	85
Germany	72
Italy	35
UK	13
Total	**226,762**

1.4.2.3 *Hemp (Cannabis sativa)*

Hemp is another bast fibre whose economy is not immediate. The spread of hemp has suffered a sharp decline, and the only demand in North America is not enough to raise its market. Hemp plantations require very long periods of seeding, retting processes and there is no specific machinery to processing its fibre. Furthermore, its characteristics are replaced by synthetic fibres. The successful use in composites seems to represent a hope for hemp fibre [41].

1.4.2.4 *Sisal (Agave sisalana)*

Sisal has been used mainly for making cordage, twine, which has now been largely replaced by polypropylene twine. Today, with the sisal fibre is employ in polishing cloths and composites for the automotive industry. But the

future of Sisal seems to be that of the paper. Its qualities in this way are better than wood pulp. Because of its high costs paper sisal is used for a narrow range of products [41], [65].

1.4.2.5 *Coir (Cocos nucifera)*

Although coir is not a bast fibre as it is extracted from the outer part of the coconut palm fruit it is however an important example to understand that natural fibres are an excellent resource [41], [66]. It includes advantageous features that cannot be replaced by man – made fibres neither by other natural fibres. Thus it promise a growing demand [41]. It is quite similar to other natural fibres such as jute and sisal, but its bigger volume makes the transportation costs higher. The production of Coir is certified and the sale takes place with agents: by orders the delivery time, usually a month, and level of quality are established. It is produced mainly in India and Sri Lanka by small company fibre [41].

Table 1.4: Price comparison of coir, sisal, jute and hemp fibres in 2003 [41].

Fibre	Coir	Sisal	Jute	Hemp
Price per tonne ($US^{\$}$)	600 – 650	1900 – 2000	550 – 600	550 – 600

Coir is extracted by natural retting. All this happens with low levels of toxicity and methodologies designed to reduce the level of pollution. Recently it was discovered that the pith from which the coir fibre is extracted are useful in agriculture as they release nutrients, retain moisture and have a reduced volume. This is an example of how some plants may be totally exploited [41]. The natural fibres, in order to penetrate the market, would need for a big raising awareness about environmental problems, cooperation between manufacturing and research, a very aggressive marketing policy that emphasises the benefits of this fibres and a cost analysis to understand how bring them down [41], [67].

Today an encouraging possibility for bast fibres is represented by composites that require greatly long fibres. Another possibility that is being proposed is to use them in the nonwoven [68], [69], [70]. Making predictions on bast fibres market based on past experience is difficult, but research must continue in order to find a solution in cost reductions as they have very good qualities and are, without a doubt, a chance for environmental protection [41].

1.4.3 Characteristics of common nettle fibre

In this research the analysis of *Girardinia diversifolia* fibre was performed in parallel with fibre from European nettle (*Urtica dioica*), which is already used in the textile industry as a potential replacement for glass fibre, as reinforcement and employed in luxury textiles (as Grado Zero™ [71] and G – Star [72]). Nettle fibre was employed as a textile plant before the advent of cotton although an actual production begun only in the nineteenth century [41]. This plant is very widespread and it is found in different parts of the world: the nettle family (*Urticaceae*) is divided in 5 sub – families and in 48 genera containing approximately 900 species. The 5 families are *Elastomeae*; *Boemerieae*; *Parietarieae*; *Forsskaoleae*; and *Urticaceae* (*Urereae*). The latter group includes some species commonly exploited to obtain fibre like *Boehmeria*, *U. dioica* (nettle) and *G. diversifolia*. Among these species: *Boehmeria nivea* (Ramie) fibre, native of East Asia, because of its brittleness and low elasticity, has limited applications in apparel (other than as a blend), although it is widespread as industrial thread, packing material, upholstery or canvas for home furnishings [41], [43].

Urtica is a species widely spread all over the world. Two British species are in this group: *Urtica dioica L.* and *Urtica urens L.* The best – known and employed in various textile sectors for a long time is *Urtica dioica L.*, called common nettle or stinging nettle. It grows in temperate areas of Europe and Asia and in the North up 2400 m. It is annual or perennial herb, sometimes shrubby at the base. *G. diversifolia* appears tall, short – living herbaceous plants. It

grows in Africa, tropical and East Asia. From this nettle the fibre extracted is long and strong, still handcrafted [73], [74]. The most investigated species, *U. dioica*, is a herbaceous plant with a stalk produced by rhizome, erect, quadrangular, generally from 30 to 150 cm long [54], [75]. Common nettle fibre is 100 % cellulose, like cotton, linen, lyocell and viscose [76]. The defining characteristics of a nettle fibre are [77]:

1. Hollow fibre;
2. High quality fibre;
3. Non – lignified cell wall;
4. Low specific weight;
5. Fibre – resistant, soft;
6. Good antistatic properties, breathable and temperature controlled.

In the last 20 years of research the agronomic aspects of the cultivation of stinging nettle and the technical methods of extraction of the fibre for textile industry have been investigated [73], [74]. The main reasons behind this interest are [74]:

- *Production of new materials of high quality for textile use;*
- *Local crops to eliminate both the costs and environmental damage caused by transport;*
- *Perennial crop that requires low energy inputs (herbicide and pesticides);*
- *Possibilities for the cultivation of areas occupy over – fertilized with nitrates and phosphates;*
- *Extensive cultivation for 10 to 15 years;*
- *Promotion of biodiversity.*

The fibre of *U. dioica L.* has been widely examined; various studies have been conducted in Germany, Finland, Austria, Italy, and UK [8], [78].

The Agricultural Research Centre of Finland, Fin Flax Ltd, Crafts and Design departments have developed cultivation methods and fibre processing in the project *From Nettle to Nettle I + II* (1997 – 2000; 2001 – 2003) [8]. *Nettle – reintroduction of Stinging Nettle Cultivation as a Suitable Raw Material for the Production of Fibers and Cellulose*, (1999 – 2001) is a project involving several European institutes: Institute of Agrobiotechnology (Austria), Institute of Applied Research Reutlingen (Germany), Zucchi S.p.A. (Italy), Heinrich Rimml and Textilpflenge Bruno Widmer (Switzerland). The areas studied are various, from developing fibre processing methods to testing spinning, weaving and manufacturing [8]. *Natural Textile Made of Nettle – Innovative Technology and Product Development for the Textile Industry*, a German project, has built an entire production process for nettle cloths, starting from cultivation through to processing. The cultivation methods are developed in organic farming. This project was conducted in the years 1999 – 2002 by the Institute of Plant Production and Breeding, University of Göttingen, Institute of Applied Botany, University of Hamburg, Langhein – Textile GbR (Germany) [8]. The Projects *LAMMATest*, developed during 2000 – 2006 and 2008 – 2009, have evaluated the production of Nettle fibre in Italy, in particular in the Tuscany region. It was conducted and funded by the Institute of Biometeorology (IBIMET), National Research Council (CNR), Regione Toscana (Italy) [79]. De Montfort University in Leicester, Defra, Sustainable Technologies Initiative (UK) from 2004 start – up *Sting project* to experiment sustainable technology in nettle growing. In these projects Clone – 13 was used. This clone, *i. e.* asexual reproduction, natural or artificial, of the variety of plant, stored at research institutes in Germany, was selected in 1959 by Bredemann who evaluated and selected agronomic and morphological characteristics of wild nettle over 30 years [80]. The clones Bredemann selected were characterized by [8]:

- *Frost tolerance;*
- *Optimum growth (long, straight, stable and unbranched stalk, abundance of leaves and strong tillering;*

– *High fibre content.*

Common data are emerged from the various studies conducted in Europe [8], [11], [12], [50], [55], [78]:

– The propagation of the plant is by cuttings or by grafting;

– In tests of three years, the best harvest occurs during the third year. During the first year the plant does not produce sufficient amounts of fibre, the stem has ramifications. Always in the first year can be problems of weed control solved without the use of herbicide;

– A planting of nettle can produce up to 4 years, then begins to decline, but the plant can continue to grow from 10 to 15 years without limits;

– The harvest may occur only once a year;

– The plant produces up to a maximum of 16 % fibre;

– During the extraction it has been noted that the fibre has different characteristics along the stem. So the stem is divided into three parts (bottom, middle, top) starting to cut 10 cm above the ground (Table 1.5);

– The diameter average of the nettle fibre is similar to other bast fibres. The diameter decreases from the bottom to the top of the stem (Figure 1.12);

– The fibre shows a flattened, irregular and ribbon – like shape, whose ends often ramify. Its walls are layered and section presents a medium – sized lumen containing a yellow substance;

– The fibre length is bigger than other bast fibres. The longer fibres are located at the top of the stem (Table 1.6).

– Tensile strength varies greatly along the stem. The lowest values are at the bottom where the fibre has a larger diameter and lower lignin content. While in other portions is approximately twice. The maximum value of tenacity is found to be 37 $cNTex^{-1}$. In general, the tensile strength remains

in the range of cotton; the elongation values do not vary along the fibre [11];

– The content of lignin determines the flexibility of the fibre and the potential uses in the textile. Nettle has 3 – 4 % of lignin content, comparable with Hemp 4 % and Flax 3.2 % [11];

– From the nettle stems, leaves phenolic compounds are extracted with antioxidants properties, making possible the applicability of these metabolites in various industries such as the phytomedicine and cosmetics, but also nutrition, food and feed, and finally in the textile industry, assuming biomedical textiles antimicrobial and antimycotic capacities [12].

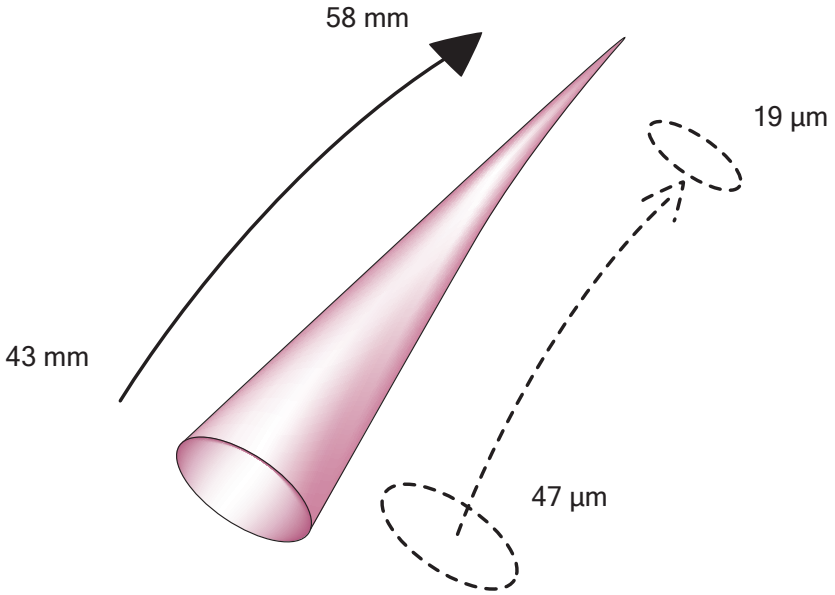

Figure 1.12: Nettle characteristics: diameter decreases and length increases from stalk bottom to top [11].

Table 1.5: Data of fibre extracted by different stalk portions of the second cultivation year nettle [11].

	Bottom	**Middle**	**Top**
Diameter mean (μm)	47	32	19
Length mean (mm)	43	50	58
Tensile Strength mean (cNTex^{-1})	24	62.1	58.7
Elongation mean (%)	2.6	2.3	2.5

Table 1.6: Nettle fibre diameter and length mean values comparable to those of Hemp and Flax fibre [11].

Fibre diameter mean values	(30 – 45 μm) **Nettle**
	(32 – 34 μm) **Hemp**
	(20 – 30 μm) **Flax**
Fibre length mean values	(48 – 52 mm) **Nettle**
	(20 – 28 mm) **Hemp**
	(20 – 30 mm) **Flax**

1.4.4 *G. diversifolia*: the Himalayan Giant nettle

This study focuses on the Himalayan Giant nettle (*G. diversifolia*) (Figure 1.13) commonly called *Allo*, Himalayan nettle, Nilgiri nettle or Nepalese nettle. This plant grows in tropical Africa, from Ethiopia to Madagascar, in Asia, Yemen, Nepal, India, Sri Lanka, southern China, Taiwan and Indonesia [35]. According to the report *Fibers exploitation in the Makalu – Barun con-*

servation area [39], Himalayan Nettle fibre (Figure 1.14) has several qualities of great interest to fabric and clothing manufacturers, including:

1. Length greater than any other plant fibre known to man, which increases spinning options and, hence, fabric possibilities;
2. Hollow core useful in creating fabrics with thermal properties, both warm and cool;
3. Great resistance to wrinkling;
4. Reputedly antimicrobial, antibacterial and fire retardant;
5. Socially responsible: it can provide income to millions through village – based work.



Figure 1.13: Plant and natural habitat of *G. diversifolia* [34].



Figure 1.14: The Giant Himalyan Nettle – *Girardinia diversifolia* – raw fibre.

Nowadays, the *Allo* plant is exploited to extract fibre, paper and dye (it is possible to obtain various colour like blue and green [81], [38]). At present

G. diversifolia fibre is used to make ropes, twine, fishing nets and sacking. In Nepal handicraft production is more focused on clothing often sold to tourists. Nettle fibre production, according to data of 2009, is still insignificant compared to other natural fibres [38]. In 2014, Himalayan nettle fabric sold at $ 10 – 14 per meter as reported by Bombay Hemp Company [36], [37]. The main ethnic groups that work with Allo in Nepal are the Magars, Gurungus, Tamangs, and Rais [81]. The plants grow at 1000 – 2500 m above sea level, in areas of partial shade. Currently, it is difficult for the plant to grow at low altitudes because the increasing population alters its habitat. The plant, which grows tall, strong and erect, needs an environment with the following characteristics [38]:

- Rank patch;
- Moisture content;
- High velocity winds;
- Lower temperatures than their neighbour common nettle (the plant is frost – resistant for 3 – 4 days);
- Fertile, deep, drained soil, for example where there is the presence of streams.

The nettle stem (Figure 1.15 to Figure 1.18) has a diameter of 1 – 2 cm slightly branched and hollow and it consists of concentric rings. The fibres, externally, are formed by fibrous cells separated from each other by parenchyma [82]. *G. diversifolia* can grow up to 1.5 – 3 m high. The cellulose microfibril are arranged close to each other, but they are not bound to each other, so a wall of pure cellulose would be weak (Figure 1.19). The hemicellulose and pectin are short branched molecules that interact to form bonds with several adjacent microfibrils, forming a solid three – dimensional network. The relative amounts of these components can vary, it will have, therefore, rigid or flexible walls, strong or weak [82].

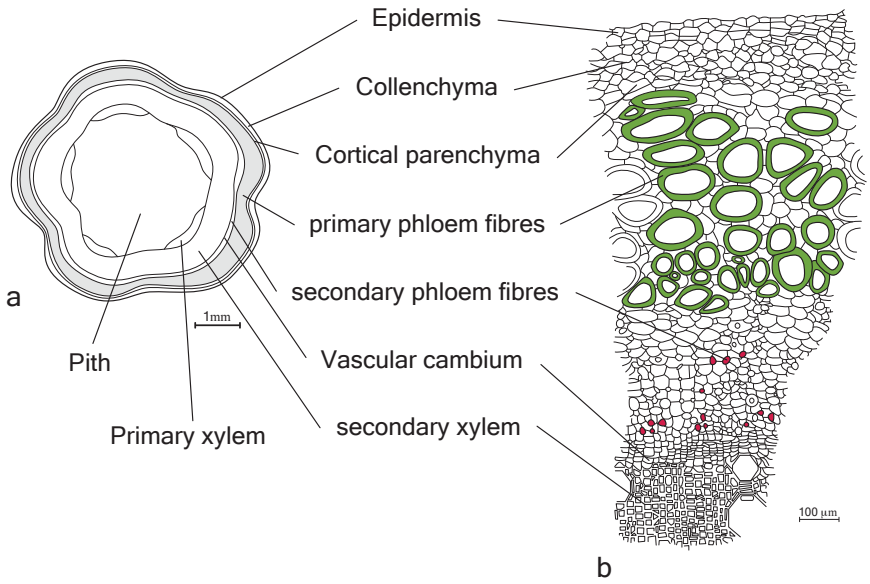

Figure 1.15: (a) Schematic stem section, (b) section of a *G. diversifolia* stem segment [83].

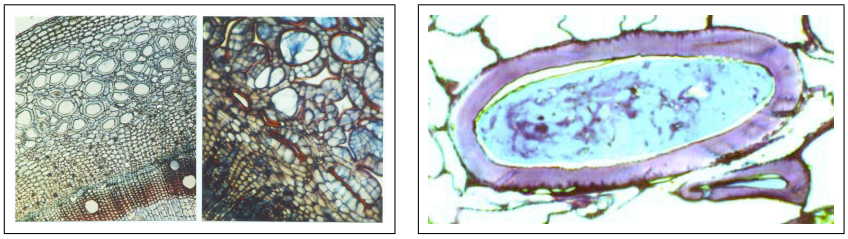

Figure 1.16: Section of *Girardinia diversifolia* stem extracts: the fibre cells with large lumens; collapsed fibre cells at the base of the stem; sample from Nepal [83].

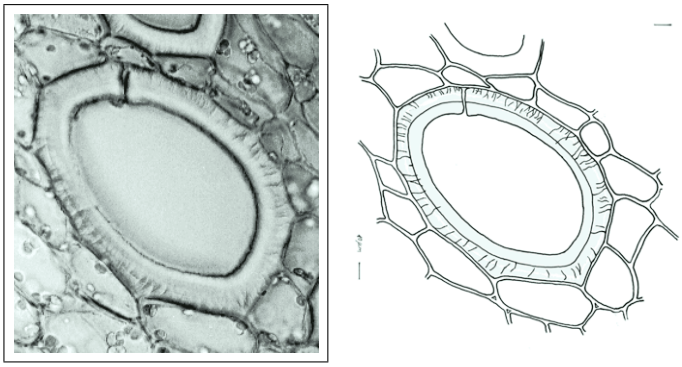

Figure 1.17: Micrograph and drawing of *G. diversifolia* fibre cell [83].

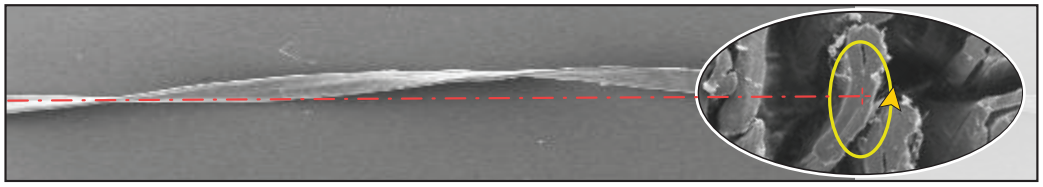

Figure 1.18: Longitudinal view and cross section of the *G. diversifolia* obtained by REM, highlighting the spiral rotation around the fibre axis [84].

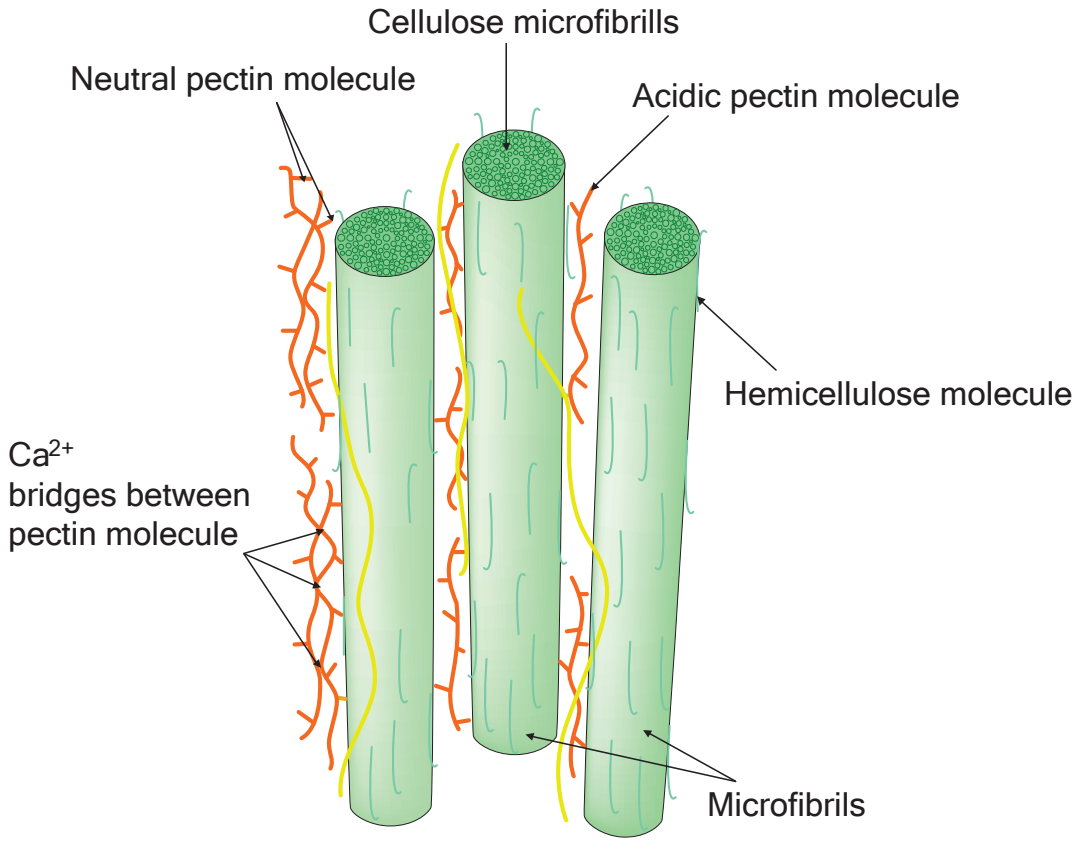

Figure 1.19: Structure of bast fibres in nettle stem [82].

Technical values differ along the stem whereby the stem is divided into portions of 10 – 20 cm [81]. *G. diversifolia* fibre has a good tensile strength (180 – 550 N mm^{-2}). The lowest values are recorded to the portion of the top

of the stem, where, although the fibre is very white, it is not resistant then not suitable for textile [38]. The modulus of elasticity is 5.8 – 22.5 GPa, with no differences along the stem. The fibre can be compared to ramie, but contains more gummy substance easily removable. The fibre is spun in long filaments [35]. The harvest of the *Allo* is between August and December, when the fibre is white and of a better quality. Local farmers usually hand – cut the plants during the cold seasons as the effect of stinging is considerably reduced by the lower temperature [39]. The processes of fibres extraction and spinning is still by hand (Figure 1.20). To extract the fibre it takes 3 – 5 nights. It can be extract up to 3 kg of fibres per day. During the extraction the fibre must be freed from pectins, hemicelluloses, lignin, substances that keep the fibres. This process could be a problem for the environment because the only way to get completely clean fibre is chemical. At the moment, however, the Nepalese workman refuse to use products such as caustic soda for this process and they only wash it with ash and water (in streams). Before processing, the fibres are separated by long and short, and softened with curcas oil for weaving [81]. A pedal device was introduced to speed up spinning and weaving, as before the work was carried out with the help of the teeth, creating, obviously problems to the workers' jaws. The processes of extraction and processing of this fibre are long and exhausting for the body. To try to reverse this situation several groups have shown interest in organising courses to start a modern production [38].

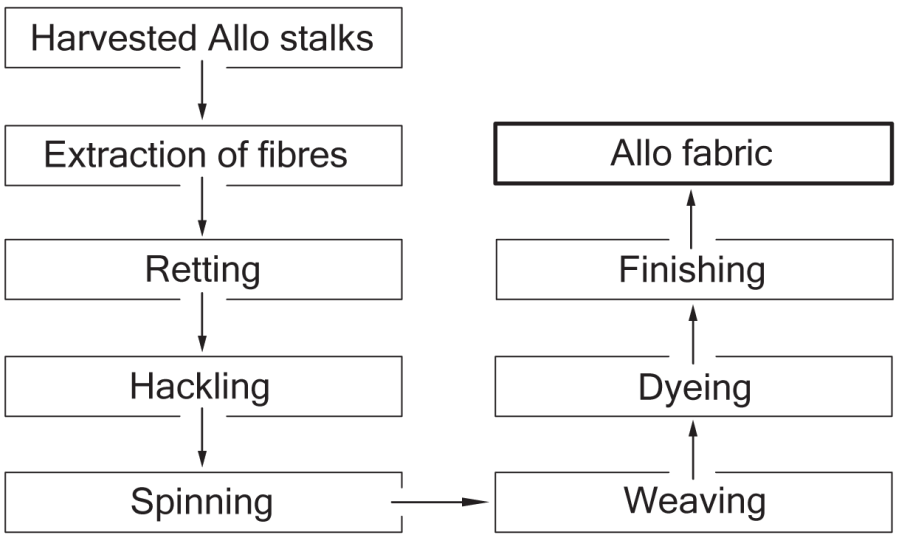

Figure 1.20: Flow chart of fibre processing [83].

The fibre obtained from *Allo* is withe and silky (Figure 1.21 and Figure 1.22). Weavers avoid the chemicals to cleaning the fibres using traditional methods: ash is used for bleaching and Kameromato, a clay that facilitates the boiling, is used to make the fibre soft for spinning [38].



Figure 1.21: Fibre extraction from the bark of the stem of the *G. diversifolia* plant [34].



Figure 1.22: *G. diversifolia* fibre [34].

It is reported [38] that when the plant of *G. diversifolia* grows in sunny areas, the fibres are dark and they are difficult to work, while, when the plant is growing in the shade, bleaching could be even superfluous. Singh & Shrestha have speculated that because the *G. diversifolia* needs shadow, crops could be arranged in subplanes [85]. In Nepal there are no real rules of the market. NGOs are trying to facilitate payments in local and foreign (UK, Germany, USA, *etc.*) market sales. If the *Allo* demand were to rise, a study of farming systems should be considered as in Europe (Italy, Austria, *etc.*), to understand the conditions of yield and performance. An attempt had been made by Sethmann in her PhD thesis on a plantation in the north – east of Hamburg [84]. Unfortunately, the plantation of *G. diversifolia* was abandoned and the work, of which there are some interesting data analysis of the fibre was not continued. This experiment suggests that the plant suffered moisture especially during the winter season. There are companies such as Himalayan Wild Fibers [34], an American Limited Liability Company (LLC), that have already embarked on the industrial development for Nepal. In addition, to increasing demand, marketing channels should be strengthened by using high – visibility sectors such as fashion [38].

1.5 Cellulose molecular structure

In order to develop nettle fibres for high – performance applications, a detailed understanding of cellulose chemistry is required, particularly in relation

to how the properties of cellulosic fibres may be changed through chemical processing. Vegetable fibres are composed mainly of cellulose, found in particular in the cell walls. Cellulose [$(C_6H_{10}O_5)_n$] is polysaccharide produced in living plants by biosynthesis [43], and the fibres are polymers consisting of repeat units of the monosaccharide β – D – glucopyranose [43], where the β – glycosidic bond is oriented equatorially (Figure 1.23). Each repeat unit of glucose has three hydroxyl groups (– OH) that bond to other polymer cellulose chains through hydrogen bonds; this produces a intermolecular force that, with a regular structure, is the cause of a high degree of crystallinity of the cellulose fibres [86].

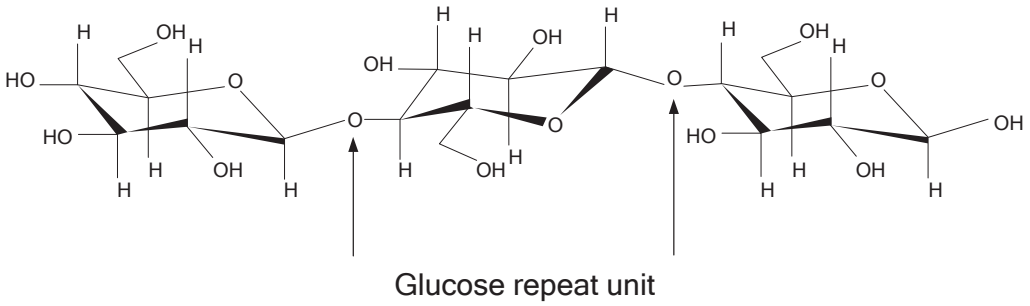

Figure 1.23: Cellulose polymer chain.

The fibre polymers present a fringed micellar structure, which is a structure that has both micelles and fringes; the former are crystalline regions (*i. e.* the internal arrangement of atoms, molecules or ions, is regular) and the latter are amorphous regions (*i. e.* the molecules are disorganised internally). In particular in the crystalline regions, the polymers possess an intermolecular hydrogen – bond and a regular form that extends in three dimensions (Figure 1.24) [43], [50].

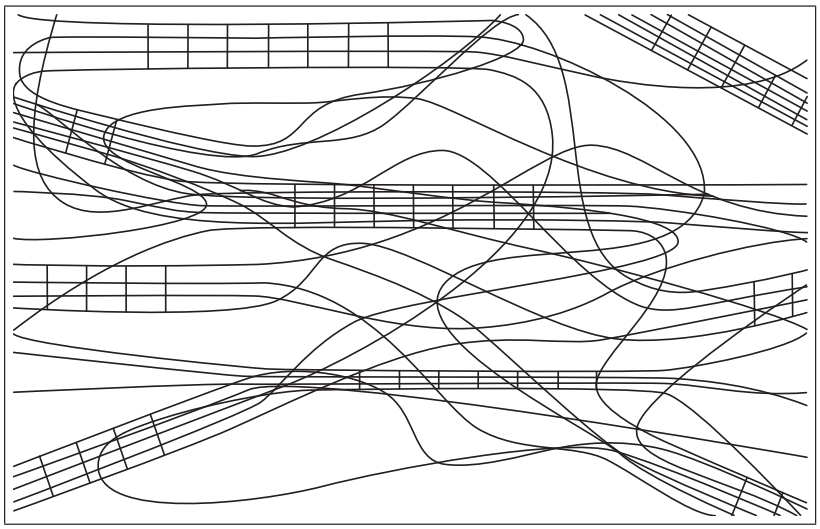

Figure 1.24: A fringed micelle structure of a long polymers. Crystalline and amorphous regions are present [43].

Crystalline regions prevent the penetration of water and other biological or chemical agents. In addition, the length of these regions and their alignment with the fibre axis (called high – orientation) affects the mechanical strength and stiffness of the fibres. Long polymer chains mean that the degree of polymerisation (DP) of the fibre is high; DP is the number of monomeric units in a polymer, and it gives to the fibre good mechanical properties like strength; this value, in this polysaccharide, is influenced by the degree of maturation of the cell wall, the aging of the fibre and the process it has been subjected; cellulose fibres useful for the textiles should have a DP above 2000 [87]. When such a fibre is formed (by a high percentage of crystalline regions with these features) it will be a stronger, stiffer, harder to dye and resistant to chemicals and ageing one [43]. The amorphous regions, in which parts of the polymer chain have freedom of movement, give to the fibre elasticity and flexibility. In these parts water or chemicals penetrate easily and this may cause chemical reactions or deterioration; these can occur even in the crystalline regions, but require more time and the breaking of secondary bonds between the chains.

The ratio of crystalline and amorphous regions plays an important role in fibre deterioration; polymer chains with few amorphous areas have a greater

resistance to factors that cause fibre degradation, although it should be noted that according to the fringed micelle theory, each polymer chain within the structure of the fibre has the opportunity to change between highly orientated crystalline areas and amorphous regions [43]. These polymer chains, about a hundred, form an elementary fibril with a diameter of 5 – 6 nm (Figure 1.25).

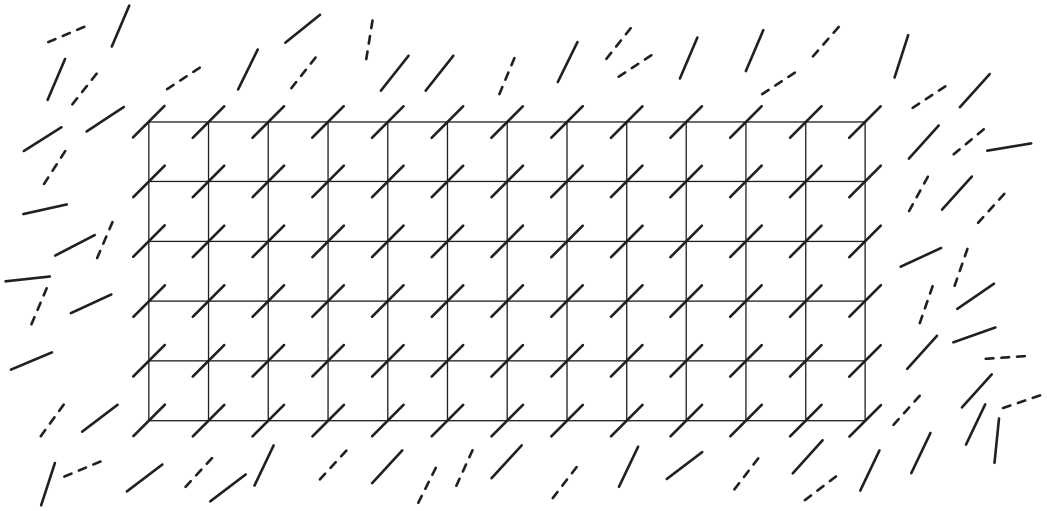

Figure 1.25: Diagrammatic representation of a cellulose microfibril cross – section. The central grid represents the crystalline core of which there are traces of cellulose (oblique lines). The lines that surround the grid represent the glucose (full lines) and traces of other sugars (broken lines) [88].

The elementary fibrils, aligned and grouped together in about 15 units, form the microfibril which has a diameter from 75 to 90 nm. The microfibrils form the macrofibril (or simply fibrils), with a diameter of 100 nm. The fibrils form the lamellae, divided into primary and secondary cell walls. In the primary wall, fibrils are disordered, but in the second are arranged spirally around the longitudinal axis of the cell (Figure 1.26) [43], [82].

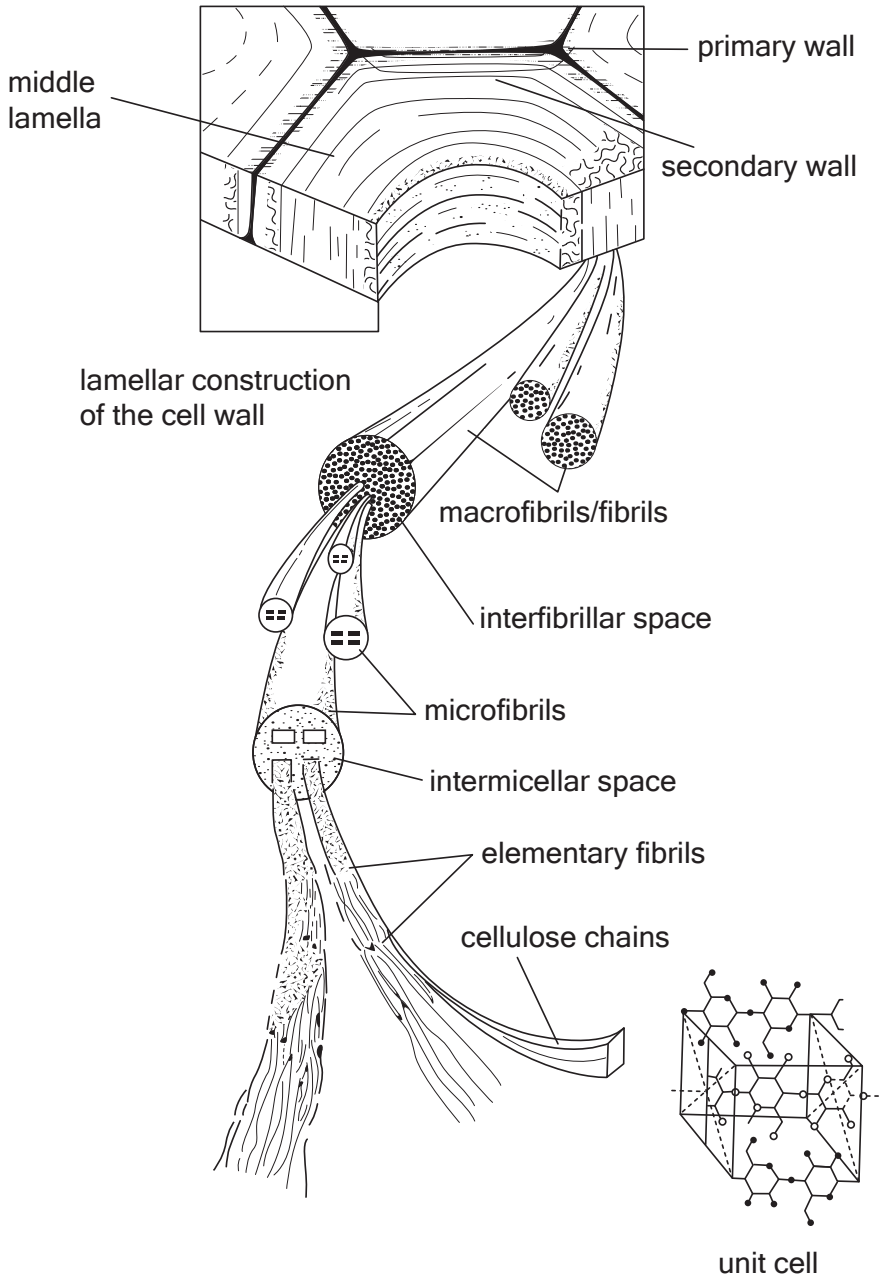

Figure 1.26: Organisation of cellulose chains into macrofibrils and in the cell wall of wood [43].

1.5.1 Extraction of textile fibres from bast fibres

Cell walls (primary and secondary) of cellulose fibres are composed of (in order of content) cellulose; hemicellulose; pectin (a non – cellulosic polysaccharide); protein; and lignin [44]. These components can also be found among the microfibrils and between cells of the middle lamella and form the matrix compound in which bast fibres are embedded within the plant stem. The removal of the fibres from cellular and woody tissues can be carried out naturally by bacterial decomposition retting or with acid chemicals treatments. This step largely determines the fibre fineness [41], [43], [89]. The fibres extracted from the stalk will not be composed only of pure cellulose, but of other components (listed above) the amount of which depends on the extraction method used. A further process is needed to remove these substances from the fibre, allowing for a pure cellulose, will improve the physical characteristics and the technical performance of the fibre [43].

Some micro-organisms and bacteria break glycosidic bonds in cellulose to form glucose for nourishment; the process can be accelerated by enzymes in humid conditions; this process is known as hydrolysis. Bacteria and microorganisms during cellulose decomposition cause oxidation and acids, excreted by certain microorganisms, cause acid hydrolysis of cellulose [43]. Some previous studies have demonstrated the efficacy of the enzymes treatments for textile fibres [8], [90], [91]. Currently there is a strong interest to obtaining alternative methods for the extraction and processing of cellulose fibres that avoid the use of chemicals such as sodium chlorite and sodium hydroxide, which present in waste maceration water, are a great cause of environmental pollution. New extraction methods trialled include the use of steam, high pressure water, ultrasound, effective to lignin removal, enzymes, and chelators [92]. Eco – friendly biological treatments do not give promising results as the process itself requires a long period of time in which the fibre is damaged. However, experimentation has not been abandoned as previous studies have shown the efficacy of the enzymes to treat textile fibres [8], [90], [91].

G. diversifolia fibre extraction by enzymes is currently being studied, as promising results have been obtained with industrial enzymes and selected microbes for *U. dioica* fibre. It requires a short period of retting, which is able not only to extract the fibres from the stem but also to separate them from each other. The success of enzymes decortication for *U. dioica* fibre is due to the low lignin content which in some cases makes the bleaching superfluous. The wastewater is biodegradable and does not represent a pollution problem [8], [41], [93].

1.5.2 Mercerisation

A process applied to many cellulosic fibres for textile applications is *mercerisation*, which changes the crystalline structure of cellulose [94], [95]. Molecules of native cellulose fibres are highly oriented, arranged parallel to one another, but their degree of orientation parallel to the fibre axis is reduced since they tend to form a spiral around the fibre [49]. Mercerisation disrupts the crystalline cellulose structure to a degree to be able to reform in a new cellulose structure [95]. Native cellulose is *Cellulose I* and in regenerated cellulose and mercerised fibres is *Cellulose II* (Figure 1.27). The crystalline forms of Cellulose I and II are the most important. It is possible to obtain *Cellulose III* and *Cellulose IV* with other chemical treatments [88].

With the mercerisation the following effects are obtained [96], [97], [98], [99]:

- Improvement of the lustre because of the change of the cross sectional morphology: from beam shape to round shape (Figure 1.28);
- Improvement of the strength:
  - Orientation (parallelisation) of molecular chains in amorphous region along the direction of fibre length;
  - Orientation of the crystallinity in the direction of the fibre length;
  - Increased crystallinity;

– Improvement the dye uptake and moisture regain.

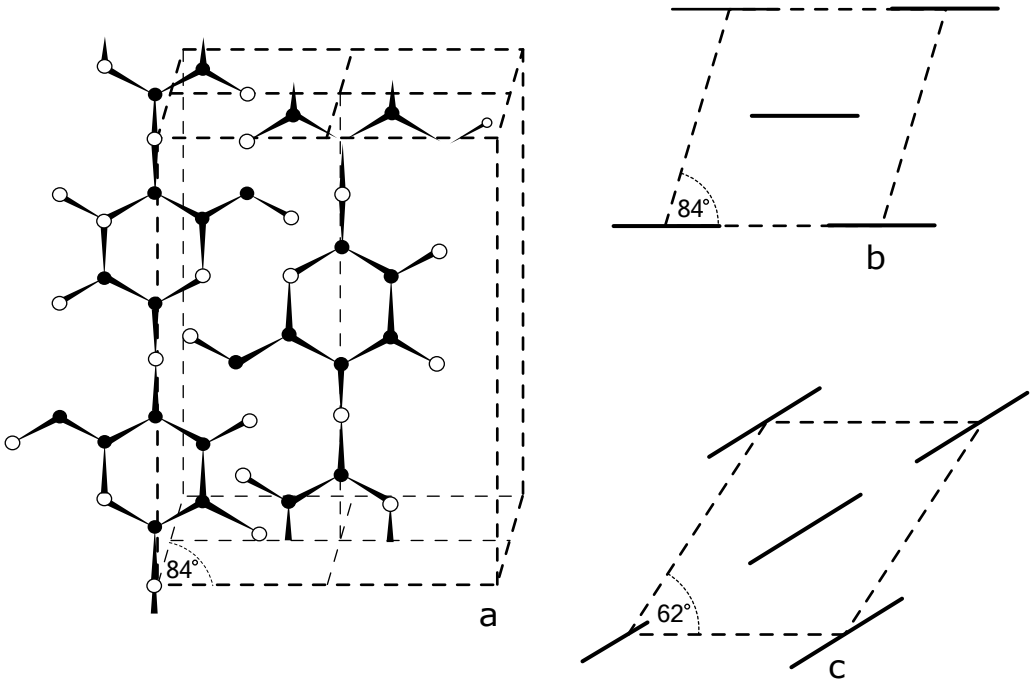

Figure 1.27: (a) Arrangement of molecules in crystal of cellulose; (b) Unit of cellulose I; (c) Unit of cellulose II [49].

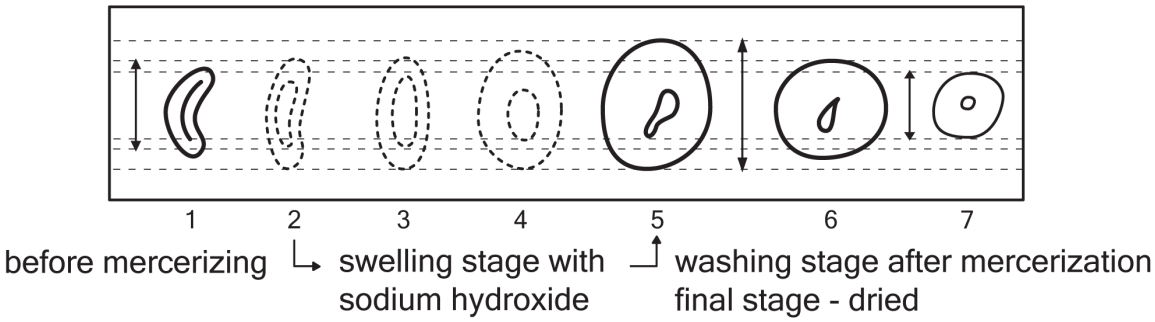

Figure 1.28: Changes of cotton fibre cross – section during the mercerisation process [43], [100], [101].

1.5.3 Alkali treatment of cellulose

Treatment of cellulose with alkali, notably sodium hydroxide, changes the crystal structure of cellulose from Cellulose I, *via* sodium Cellulose II, to hydrate Cellulose II (with water) on rinsing, to the final Cellulose II on drying (Figure 1.29) [43].

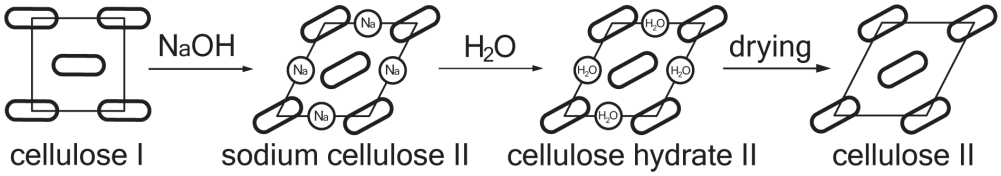

Figure 1.29: Cellulose crystal structure change on alkaline treatment (mercerisation or regeneration), and change of the cross – section cotton fibre during the mercerisation process [43], [102], [103].

At certain NaOH concentrations and temperatures, the morphological, molecular and supramolecular features of cellulose may be changed, as demonstrated by Goswami et al. [102]. This causes changes in stiffness, in the orientation of the fibril, in crystallinity and in particular the transformation from Cellulose I to Cellulose II which has a much more stable crystal structure compared to the former [102], [103]. Analysis on different types of cellulose fibres (regenerated cellulose, lyocell [102], bast fibres, jute and hemp, leaf fibres, sisal and seed fibre as the kapok [104], [105], [106]) show that morphological and molecular changes determined by NaOH treatment on the fibre influence their physical and mechanical characteristics improving their performance.

1.6 Purpose of this research

The fibre of the Himalayan giant nettle (*G. diversifolia*) appears to have potentials to encourage a deeper scientific investigation. The advantages of *G. diversifolia* fibres are not only social and environmental since the cultivation of

the plant and processing of fibres is entirely eco – sustainable and might deliver substantial industrial development in Nepal; but it is also believed that this fibre provides technical advantages for the textile industry. Other fibres are already extracted from the *Urticaceae* family, for example from the *Boehmeria nivea* (Ramie) and from the *Urtica dioica* (Common nettle) and they all have interesting features for the textile industry. Taking the above into account and also knowing from the literature [39] that *G. diversifolia* fibre is a very long strong fibre with an excellent potential for weaving, it is believed that it could also bring benefits in terms of performance in textiles. This is why the investigation of this fibre has been carried out.

The purpose of this research is to verify whether the *G. diversifolia* fibre can be used as a textile industrial material. It represents a resource both from a social and environmental point of view. To demonstrate its competitiveness it must find a place in a niche sector such as technical clothing currently dominated by synthetic fibres.

In work herein *G. diversifolia* fibre will be subjected to an alkaline process with sodium hydroxide. This method is considered the most suitable as it penetrates easily and eliminates encrusting materials allowing to obtain pure cellulose fibres of excellent quality changing its structure and consequently its physical and mechanical characteristics. Then as made by Hassan & Blendzki for jute fibre [104], *G. diversifolia* and *U. dioica* fibres will be treated at different concentrations of NaOH and compared morphologically, physically and mechanically analysing the influence of the NaOH treatment on structural and mechanical properties of the fibre. Although the intent of this research is to propose alternative performance materials fully sustainable, being eco – extraction method for bast fibre in testing phase, as preliminary purpose an investigation of the Nepalese nettle fibre must provided in order to understand its suitability for use in the textile industry since there are no data in this regard.

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Chapter 2

Materials and methods

2.1 Introduction

This chapter provides an account of the materials and experimental techniques employed in this project (Chapters 3 and 4); techniques specific to a particular area are outlined in the respective experimental chapter (Chapter 5). Details are also provided of the test methods that were used, and the principles which underlie them, to assess the various characteristics of the fibres.

2.2 Materials

The investigation of Himalayan nettle fibre (*Girardinia diversifolia*) (Figure 2.1, left) was carried out in comparison with European nettle fibre (*Urtica dioica*) (Figure 2.1, right). Tests were performed on two samples of fibres provided by:

– Himalayan Wild Fibers LLC (HWF) [1] provided 1.84 kg of raw *Girardinia diversifolia* fibres, with an average length of 471.6 mm, extracted by boiling the bark in water at pH of 11 – 12 obtained by addition of sodium carbonate;

– 1 kg *Urtica dioica* fibres (Clone 13), length on average 52.8 mm, were kindly provided by De Montfort University, UK [2].

In this study, for each type fibre, eleven samples were analysed: 1 raw, 1 scoured, and 9 treated from 0 – 8 mol dm^{-3} aqueous sodium hydroxide solution.



Figure 2.1: Raw Himalayan nettle fibre – *Girardinia diversifolia* (left) and raw European nettle fibre – *Urtica dioica* (right).

2.3 Physical and mechanical analysis

Physical characteristics and mechanical properties of the fibres are important to hypothesise their behaviour in terms of textile products. A yarn, a fabric or a composite will be largely characterised by the properties of fibres from which is made and from their arrangement. In fact, for some mechanical properties the value of the yarn will not be greater than the sum of the maximum values of the fibres that it is composed of. The knowledge of the characteristics of a fibre is imperative to identify its effective use [3]. The properties that best determine the 'character' of a fibre for industrial purposes are mechanical ones; studies investigating bast fibres are concerned with their mechanical properties and often the study concerns how the morphology of the fibre affects tensile properties and what factors may change these properties. Baley [4] examined flax fibre and collected data for the fibre length (33 mm) and diameter (19 μm), in which a very wide range was observed, and considered many factors

that would influence the stress – strain curve of the flax fibre, which are:

- The rate of Load;
- Sample Moisture Content;
- Specimen Length.

The last point seems to have a particular influence because the length could be affected by the imperfections arising from the growth of the plant and from the decortication to the fibres extraction from the stem. It is found that the significant change in the fibre diameter and the fibre flaws are a practical problem to determining the tensile properties. Kink bands in flax fibres (Figure 2.2), which are defect areas of fibre where it is assumed are the weak links in molecular fibre structure, represent problem as they will cause a loss in fibre strength [5]. It has been found that also a larger diameter results in a loss of strength.

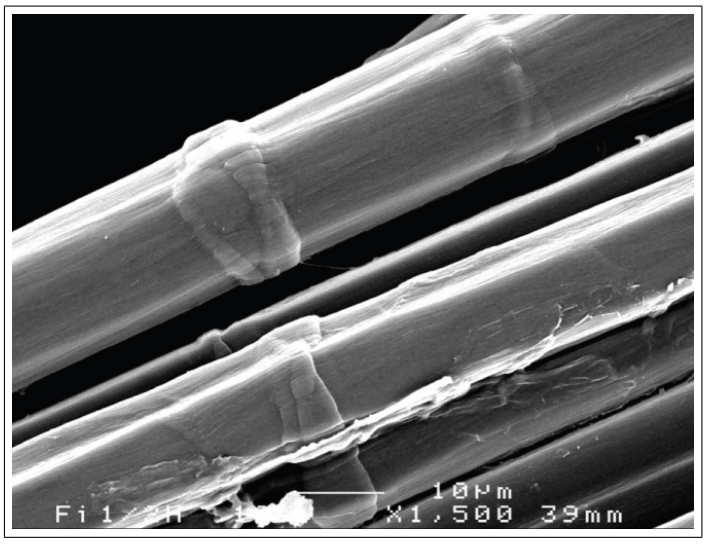

Figure 2.2: Kink bands of a bundle of flax fibres [5].

In examining the fibre diameter of the Hèrmes flax variety [6], fibre porosity is considered, *i.e.* the presence of a lumen (lumen area divided by the area of the cross – section of the fibre); the area considered is that of the entire section minus that of the lumen. It is noted that the lumen increases with the

diameter; the cause of the change in diameter is due to the fibre twist and its convolutions. The mechanical properties are then considered in relation to the effective cross – sectional area. It is found that the main cause of mechanical properties scattering is due to the change of fibre size along its axis.

A problem in the analysis of bast fibres is to determine their cross – sectional area both in practical method and in the calculation. Virk *et al.* studied jute [7], where fibres were embedded in epoxy resin to be perpendicular to the polishing plane. They cut with a microtome and observed under an optical microscope; the cross – section outlines are traced manually, and the area is calculated using an image analysis program. For each section, the perimeter, the diameter of the fibre and the lumen, and thus the porosity, were calculated, and they concluded that the lumen does not have great influence on Young's modulus. In further work on jute [8], they analysed fibre cross – section using LEXT OLS3000 Confocal Laser Scanning Microscopy (CLSM) and LEXT OLS 6.03 image analysis software. The specimen consists of the fibre with a cellulose acetate sheet attached to it using epoxy resin adhesive and washed with diluted detergent in an ultrasonic bath at each stage. The images are acquired with a CLSM and processed using Matlab R2008a. The outlines are drawn manually (Figure 2.3).

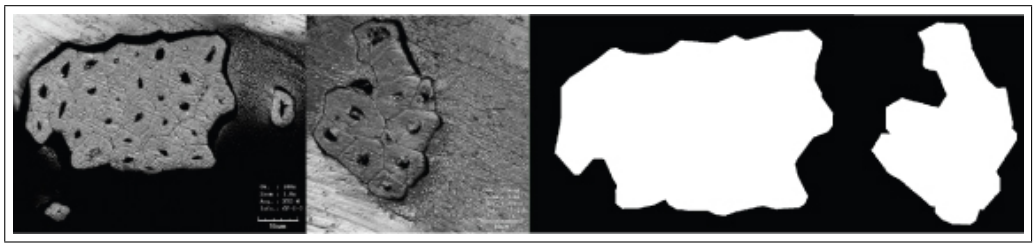

Figure 2.3: Typical Jute fibre cross – sections and mask to identify true fibre shape [8].

Analysis of the section is conducted using a circle diameter (minor and major), and referring it to the other three models: the ellipse, the super – ellipse and convex hull (Figure 2.4). In particular, projections on the axes are

made following the angular rotation that the section makes along the fibre twist (Figure 2.5). In her work on *Girardinia diversifolia*, Sethmann [9] measured cross – section by rotating on the coordinate plane (Figure 2.6); from this complex calculating method, it is possible to note that the areas obtained considering the minor ellipse were most reliable. This method underestimates the area of the cross – section, but enables more reliable Young's Modulus and strength data.

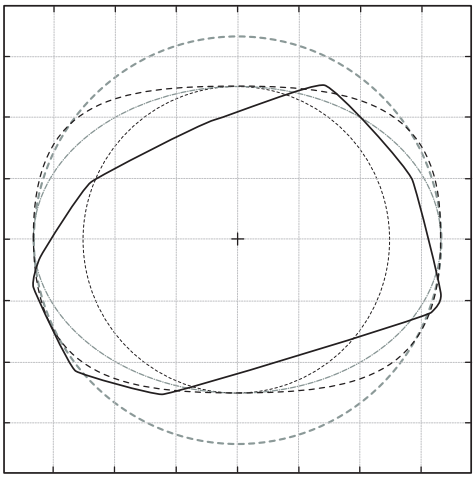

Figure 2.4: The five forms considered to determine the size of the cross – sectional area of Jute fibre: convex hull, super – ellipse, ellipse, circle (minor and major) [6].

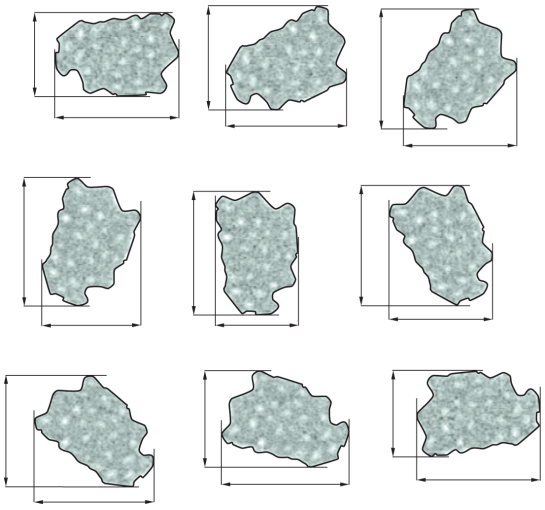

Figure 2.5: The measurement of cross – sectional area of Jute fibre based on the rotation angle of the assumed shape [6].

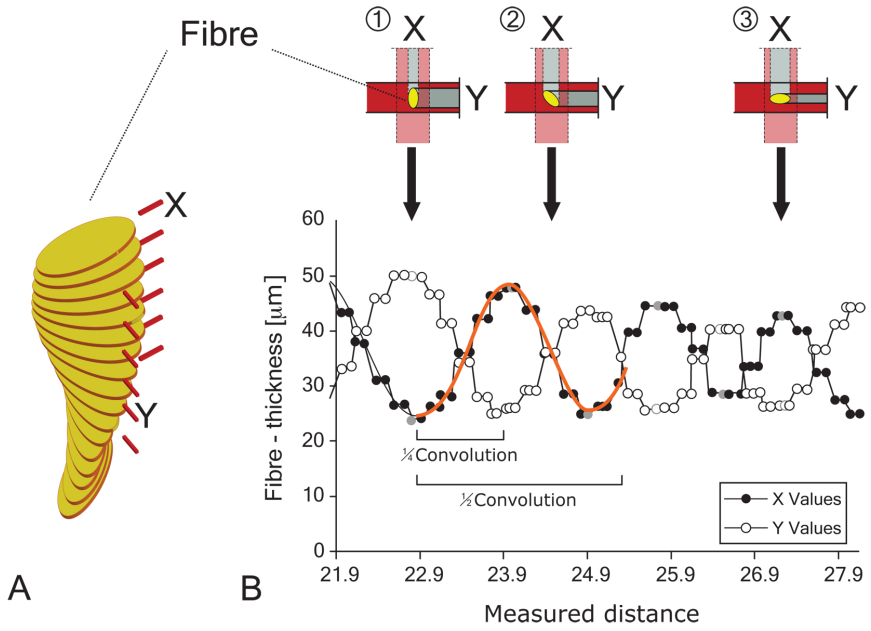

Figure 2.6: Measurement method of nettle fibre cross – section by Sethmann [9].

Other studies determine the fibre section, in the work by Charlet *et al.* [10] the fibre is initially encapsulated in epoxy resin and then polished with different grit papers. Then it is analysed using an Olympus Lext confocal microscope and by image analysis software. In this study the fibre section is considered circular, although the authors claim this is not correct in the calculation of the mechanical properties. The tensile test is carried out using samples of different lengths. For the prediction of tensile properties the Weibull statistic method was used: the probability theory of Weibull is a continuous probability distribution defined on positive real numbers used to describe the size distribution of particles [11]. Zhang Wang [12] used the conventional model weibull / weakest – link to determine the diameter variation of fibre with geometric irregularities, for both animal and vegetable fibres. Weakest – link is the theory under which the working probabilities of a system are equal to the working probability of the weakest component [13], which shows that the strength decreases with increasing length. Virk *et al.* [14] used the same prediction weakest – link model to define the tensile properties of jute fibre; in particular, the fibres in this study were measured with a steel ruler and the diameters determined at intervals of

1 mm but considering a circular section. The tensile tests were performed with an Instron machine on different groups of fibre lengths; it is shown that the dispersion of results increases with the increase in length and that the strength decreases with the increase in length because a long fibre has a higher probability of flaws in comparison with shorter fibres. In this work a method of analysis of the mechanical properties of the fibre in relation to the area of the section is proposed.

Bodros & Baley's work on nettle fibres collected in France [15] has been considered as an interesting starting point for understanding the mechanical properties of Himalayan nettle fibre; the study correlates Young's modulus of the fibre with its respective diameter calculating an average diameter and the elastic modulus for each fibre. This method [16] asserts that the vegetable fibres have a very irregular cross – section so for practical reasons it is convenient to consider them as perfectly cylindrical and the lumen, present in these fibres, is not considered. In this way the exact area involved in the tensile stress is not examined. Despite these faults, this work was considered very reliable for the results, which shows the stiffness of the European nettle fibres greater than of glass fibres and a tensile larger than that of ramie, hemp and sisal, and for the interesting applied methods in the determination of fibre cross – section images, the diameter calculation and its relation with the Young's modulus, which determines the elasticity of a fibre.

In the work herein, it was considered more accurate and reliable not to approximate the irregular shape of the fibre section area to other of simple calculation, *e. g.* a regular circumference. The cross – section has been studied relatively to the molecular fibre variations and compared with the mechanical properties changes of the fibres at different NaOH concentrations. The tests for the calculation of mechanical properties (strength, elongation, elastic modulus, moisture contain and regain) were performed by standard methods for the bast fibres [12], [17], [18], [19], [20]. The cross – section variation in size, the tensile properties and moisture content ability were followed as parameters to deter-

mine the properties of performing non – woven fabrics made with Himalayan nettle fibre.

Morphological studies and physical tests were carried out on raw, scoured and undergoing alkaline treatment fibres. The fibres are subjected to a scouring washing process using surfactants and detergents to emulsify the natural impurities that cover and protect the fibres from drying or from the microbial penetration to obtain high quality cellulose fibres for the industry [21]. As mentioned before the Himalayan and European nettle fibres are 100 % cellulose like other textile fibres as cotton, linen, lyocell and viscose [22] and they are highly crystalline [23], [24], [25], [26], [27]. Treatments with increasing concentrations of aqueous sodium hydroxide solution (0 to 8 mol dm^{-3} NaOH) were carried out in order to induce changes in the molecular structure of both fibres and to assess how this variation could influence their morphological and physical characteristics. Therefore a crystallinity analysis of the fibre treated at different concentrations of NaOH has been carried out and the changes compared with the results obtained from the morphological and physical tests performed on the treated samples. The aim is characterising the fibre and examining how changes in crystallinity, the structure and the molecular order, affecting the physical morphological fibre properties. A comparison of the fibre cross – section was carried out with the physical properties, ability to moisture regain and moisture content, and with the tensile strength, with which are closely related to the study of Young's modulus.

2.3.1 Length

The length is one of the important properties of a fibre. A longer fibrous material has the advantages in terms of processing. More yarns can be produced as there are fewer fibres ends along the yarn [16]. At the same time from long fibres it is possible to make stronger yarn using the same twist level or to generate a higher strength softer yarn with a low level of twist [16]. There

are some techniques to calculate the length of the fibres based on a numerical average, on the section or mass. Being natural fibres irregular in section, this work employed a calculation method frequently used for research purposes: the unbiased mean length, based on number of fibres L (see Equation 2.1) [24], [28].

$$L = \frac{l_1 + l_2 + l_3}{3} \tag{2.1}$$

The Length per each fibre was measured using a steel ruler and graph paper. The sample size was determined for 50 raw European nettle fibre specimens and for 50 raw Himalayan nettle fibres specimens.

2.3.2 Scouring

Some parts of the plant are covered with lipids that have the function to protect it from drying or from microbial penetration. These fatty, waxy and other gummy substances affect the processing of natural fibres. To obtain high – quality cellulose fibres for the industry, the substances that cover the fibres must be removed by a washing process that uses surfactants and detergents to emulsify the natural impurities [29]. This type of washing to clean the fibres is called scouring [12]. Himalayan giant and European nettle fibre samples, prior to washing, were conditioned for at least 48 hours at 65 % humidity (RH) and $20 \pm 2\,^{\circ}$C. Scoured samples were produced using sealed, 1 L capacity, stainless steel dye – pots housed in a Roaches International Pyrotec Rotodyer dyeing machine. Raw fibres were scoured using the procedure shown in Figure 2.7, employing an aqueous solution comprising $2\,\mathrm{g\,dm^{-3}}$ non – ionic detergent (*Sandozin NIN*) and $1\,\mathrm{g\,dm^{-3}}$ of Sodium carbonate (Na_2CO_3), a moderate alkalinizing reagent, with a liquor ratio (LR) 1 : 100. Temperature was raised to $60\,^{\circ}$C at $2\,^{\circ}\mathrm{C\,min^{-1}}$ and the samples held at $60\,^{\circ}$C for 30 minutes, then cooled. Baig & Carr [30] scoured fibre at different temperatures and demonstrated that

at 60 °C up to 5 g dm^{-3} Na_2CO_3 is effective for scouring and does not cause severe loss of weight or strength. At the end of scouring the sample was removed from the process bath, squeezed, to remove surplus liquor, and rinsed with warm water for 5 minutes and then with cold water for further 5 minutes. After rinsing, the fibre sample was opened manually and left to dry in the open air for 24 hours (Figure 2.8). The fibres were conditioned for at least 48 hours at 65 % humidity (RH) and 20 ± 2 °C prior to analysis.

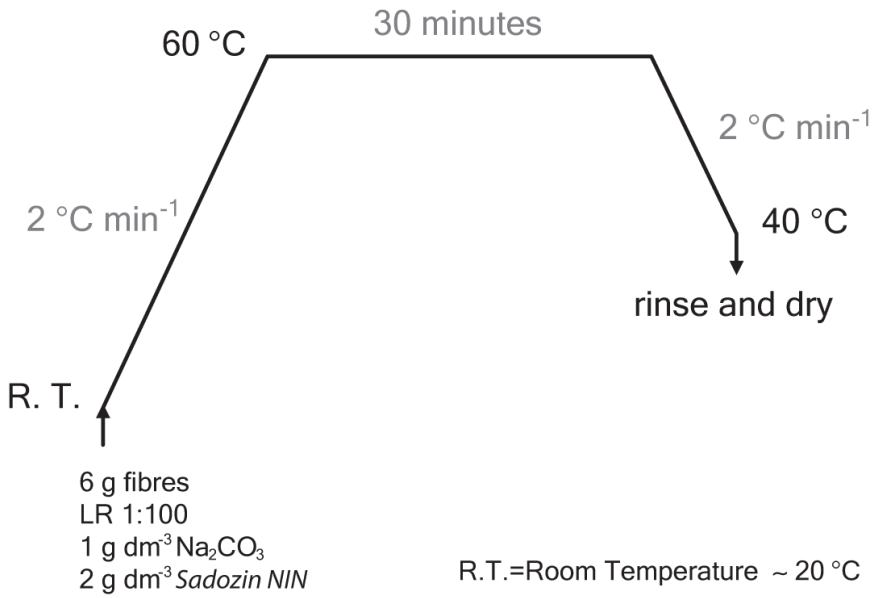

Figure 2.7: Scouring profile for raw Himalayan and European nettle fibres with *Sandozin NIN* and Na_2CO_3.



Figure 2.8: Dry scoured sample of *Girardinia diversifolia* (left) and *Urtica dioica* (right) nettle fibres.

2.3.3 Sodium hydroxide treatment

The alkaline treatment was performed on 9 samples of fibre and it consists of a series of aqueous sodium hydroxide solutions from 0 to 8 mol dm^{-3} NaOH, using a liquor ratio of 1 : 100 processed in the rotary drum dyeing machine at 40 °C at 2 °C min^{-1} for 2 hours (Figure 2.9). At the end of alkaline treatment the sample was removed from the process bath, squeezed to remove surplus liquor and rinsed as for scouring process and neutralised by immersing them in a 1 mol dm^{-3} of Hydrochloric acid (HCl) aqueous solution for 1 hour. After neutralisation the samples were following the rinsing procedure described previously. Then they were opened manually and left to dry in the open air for 24 hours and conditioned for at least 48 hours at 65 % humidity (RH) and 20 ± 2 °C prior to analysis.

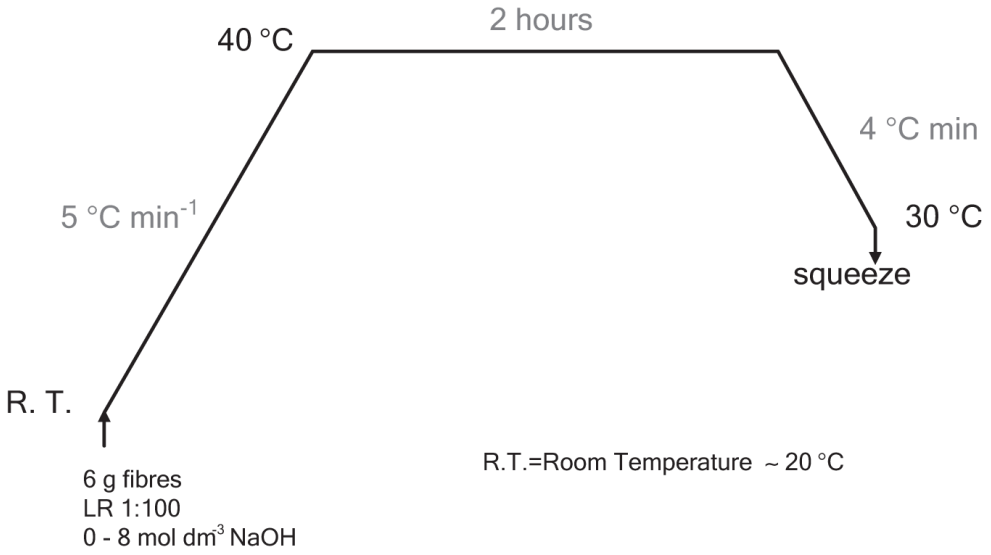

Figure 2.9: Alkaline treatment with NaOH profile of scoured Himalayan and European nettle fibres [24].

2.3.4 Cross – sectional analysis

When working with a cellulosic fibre some problems can arise because rarely the fibre has a regular shape. The cross – section study was known to be the most arduous obstacle [6], [7], [8], [9], [10], [11], [12]. With microscopic analysis it is possible to observe the longitudinal view of the fibres and to note that nettle fibres, in a manner similar to cotton fibre, presents convolutions, whereby the cross – section is varied along the length of the fibre [9]. The methods experimented aimed to relate the cross – section to the results of tensile strength in order to calculate the Young's modulus of the raw fibres and to understand the characteristics of the natural fibre not modified with chemical treatments. To achieve this, three trial were carried out. The first two have failed, while the third was successful and gave the desired results for the analysis. The first method consists to obtain the measurement of the section by an analysis of the longitudinal section. The images recorded have no satisfactory resolution, due to their very small dimensions, and a tensile test it is not reliable because for microscopic analysis the fibre is immersed in a silicone solution and this significantly changes the mechanical properties of the fibre. The second attempt was carried out using two sets of samples: the first set was used for fibre cross – sectional analysis and second set for tensile testing. The experiment consisted of immersing the whole fibre in a capsule with resin to obtain a number of sections describing the behaviour of fibre cross – section. The images obtained were considered not sufficiently clear as the resin used, BJ – 4 resin, was not appropriate. The tensile stress for a particular specimen was not related with the cross – section area of the same one. This method was considered not valid.

In the third attempt the procedures were different for the two types of nettle fibre. The Himalayan nettle fibre is long enough to be divided into two parts, one to be submitted to tensile tests and the other to be used for cross – section measurement. In this way the fibre is not physically affected and its mechanical properties remain unchanged. The same procedure is not

possible for the European nettle fibre because it is generally very short. For this reason a number of raw samples were selected for tensile test and an equal number for cross – section analysis. The sample size determined should be 100 European nettle fibres, 50 for each type of test, and 25 Himalayan nettle fibres. The Himalayan nettle fibres were cut into four parts at regular intervals. The outer parts were used in tensile tests, the interior retained for analysis under the microscope. Each fibre or part of the fibre that will be used for the cross – section will be cut in several sections (Figure 2.10). The reliable measurements were 50 (x 2) for Himalayan nettle fibres and 50 for European nettle fibre. For fibres scoured and treated with NaOH were not considered less than 5 measurements per sample.

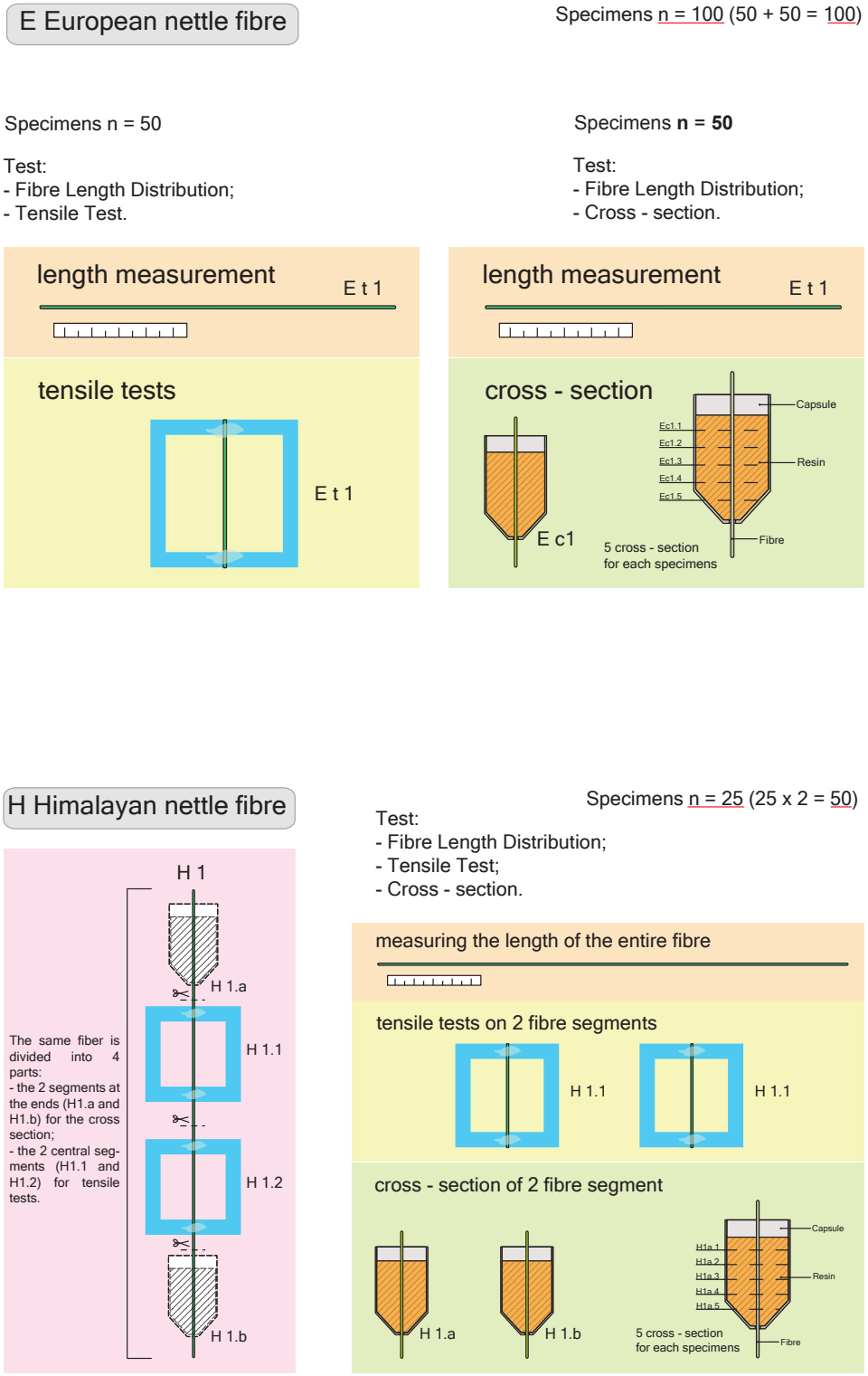

Figure 2.10: Sampling method of fibres for tensile tests and microscopic analysis of cross – section.

The procedure for this analysis follows the procedure made in the work by Bodros & Baley [15], fibre samples were embedded in a resin solution + ethanol series. LR White resin was used to prepare the specimens for its very low viscosity suitable in light and electron microscopy applications and because the reagents of the resin penetrate easily into the section showing show minimal staining [31]. To 50 ml of the LR White resin was added 0.99 g of catalyst. The fibre was immersed in the resin using *Gelatine Capsules Size 4*, recommended with this resin (Figure 2.11). The capsules were kept in the oven for more than 24 hours at temperature of $60 \pm 2\,^{\circ}C$ to ensure a good resin solidification.



Figure 2.11: *Gelatin Capsules Size 4* with specimen of European nettle fibre embedded in solidified LR White resin.

Once the resin hardened, the gelatine capsules were removed with the help of a blade, then the sample mounted on a microtome (Ultra cut Reicher – Jung) where it was sliced with a glass knife to make the surface as even as possible. When the result obtained was satisfactory, the glass knife was replaced with another one on which was attached a plastic boat filled with water. This was useful to collect the small pieces of resin and take them to bring on a numbered printed glass slide. In any sample location of the slide it was placed a drop of water to make it easier to release the sample. Before being observed

to the microscope, the sample was placed on a hot plate (2208 Multiplate) to evaporate the water (Figure 2.12).

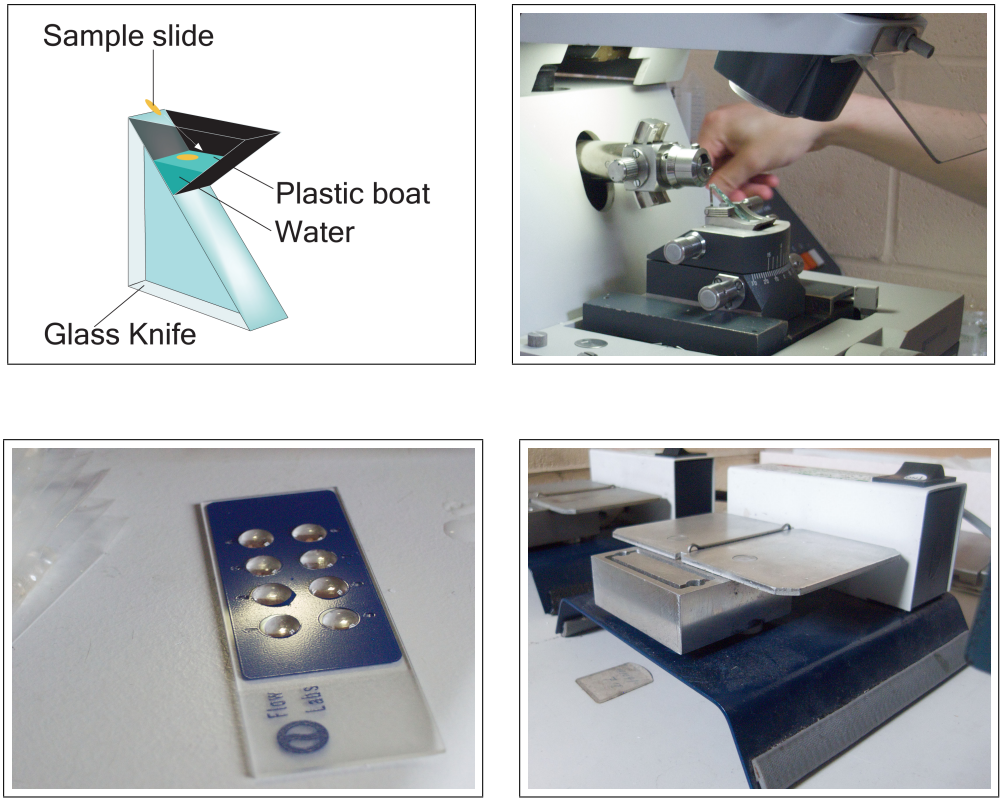

Figure 2.12: Instruments to obtain slides suitable for the cross – section analysis: glass knife with plastic boat; microtome *Ultra cut Reicher – Jung*; printed glass slide; hot plate *2208 Multiplate*.

The morphology of the section is examined by observing the resin slides and detecting the images with SEM (Scanning Electron Microscopy). The images have been taken by the Biology Department of the University of Leeds, UK. The size and the outline of the fibre cross – sections were obtained by processing the SEM photographs using the program of image processing and calculating *Image – Pro (6.2)*. For this analysis the program option segmentation was used, which uses a gray scale with the values of 1 = black and 0 = white. With a reference scale of the image magnification (entered by the program *Image J*) it was possible to calculate the area of the entire cross – section, the size of the lumen and then the effective surface of the fibres.

2.3.5 Tensile and elongation tests

The knowledge of the characteristics of a fibre is imperative to identify its effective use [3]. The properties that best determine the character of a fibre for industrial purposes are the mechanical ones; in particular, the tensile properties are the most studied, which indicate the fibre behaviour when it is subjected to forces and deformations along its axis. The tensile test detects the elongation of a fibre, up to its breaking point, when it is subjected to a gradual increasing load; this quantity is called elongation at break, and may be expressed as the percentage increase in length. The elongation of a fibre according to its definition in Equation 2.2:

$$Elongation = \frac{Extension}{Initial\ Length} \times 100 = \% \tag{2.2}$$

where extension is increased length of a test specimen, produced by a force on that specimen, expressed in units of length; initial length is the length of a test specimen under specified pretension at the beginning of a test expressed in units of length. The fibre behaviour during tensile test is shown in a load – elongation curve [19] (Figure 2.13):

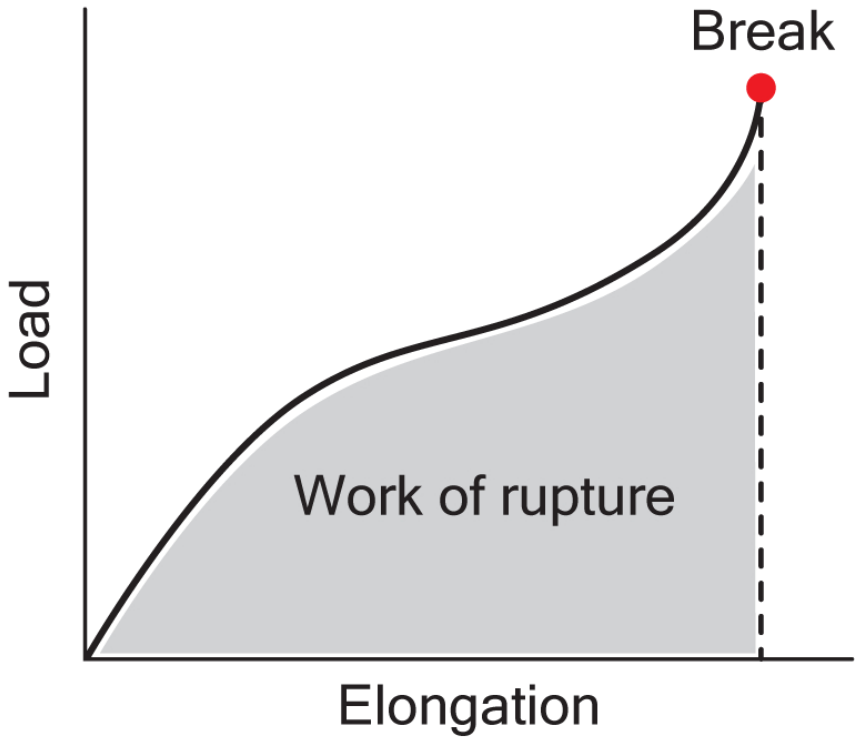

Figure 2.13: Force extension curve.

With a tensile test, it is possible to calculate the Young's modulus; this value indicates how a material resists to the Elongation, *i.e.* its stiffness. Young's modulus shows the tensile elasticity that is the longitudinal fibre deformation during the application of opposing forces along the axis. Young's modulus, or elastic modulus, (E) is given in Equation 2.3:

$$|E| = \frac{Stress}{Strain} \tag{2.3}$$

Young's modulus has the same unit of stress (N / m^2 or Pa) because Strain is dimensionless. The stress expresses the resistance of a fibre at break in relation to its cross – section [16] (Equation 2.4):

$$Stress = \frac{force\ applied}{cross - section\ area} \tag{2.4}$$

Strain indicates the elongation of a specimen during a tensile test in proportion to its original length Equation 2.5:

$$Strain = \frac{Elongation}{Initial\ Length} \tag{2.5}$$

In order to prepare the fibre for the tensile test, they are conditioned for 24 hours in a room at a standard humidity of 65 % and at a temperature of $20 \pm 2\,^{\circ}$C. The samples have been analysed using an *Instron 1026 UK* apparatus, a tensile tester for resistance of a fibre or of a yarn. It consists of two movable jaws, where it is possible to fix the sample, and of a force sensor. These parts are located in the centre of the machine and they move up and down at a set speed. As the yarn is moved with constant and known velocity, its displacement is proportional to the time elapsed. The fibre specimens were mounted on a square paper of 20 mm size (this is considered as the initial length of the sample) and subjected at a speed of 50 mm min^{-1} and a weight of 0.5 kg (Figure 2.14) [3], [19]. This load was ideal to test European nettle fibre, but after having observed, in the first attempt, the strength of Himalayan nettle fibre, a load of 5 kg was used instead. Tensile testing was performed, in accordance with BS 5079 [19], on 50 raw Himalayan nettle fibre samples and on 50 samples of raw European nettle fibre. For scoured fibres 100 samples were analysed for each type of nettle fibres and 20 samples were tested for each sample of both fibres treated from 0 to 8 mol dm^{-3} NaOH (Figure 2.15).

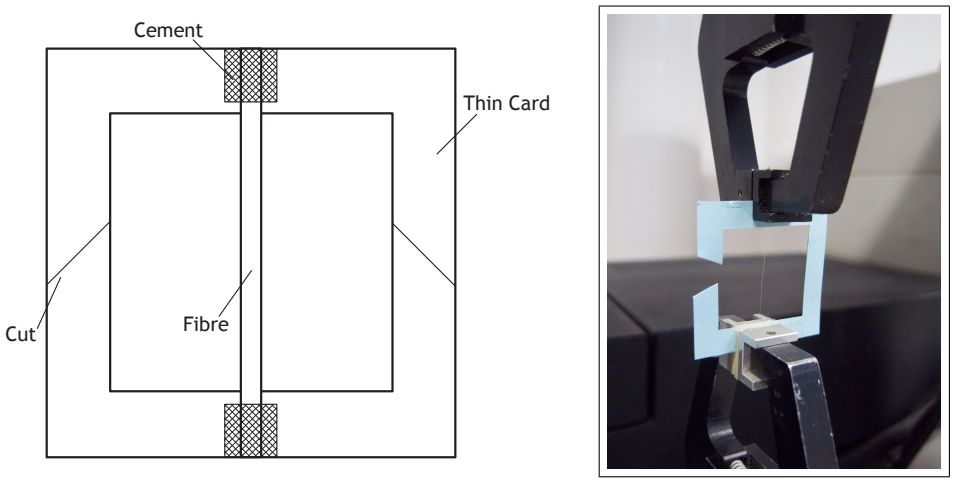

Figure 2.14: Specimen of Himalayan nettle fibre for Instron apparatus.

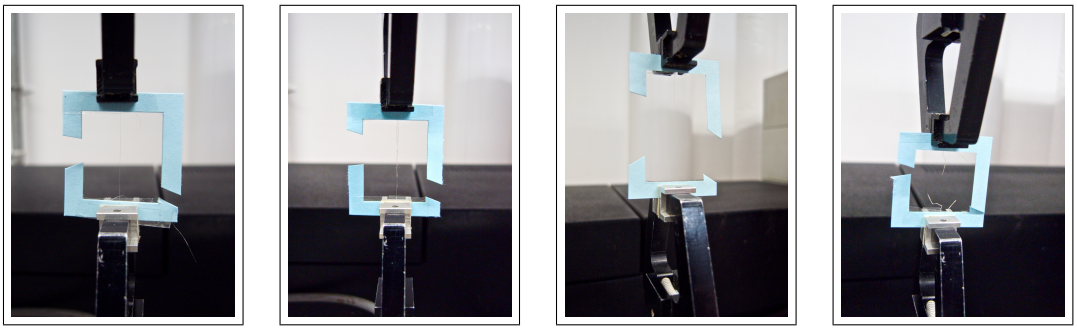

Figure 2.15: Instron test, elongation at break of Himalayan nettle fibre.

2.3.6 Moisture regain and moisture content

Moisture properties related to a textile fibre are determined by its reaction to external conditions such as the weather. For example, the water absorbed by a fibre can affect dimensions, tensile strength, elastic recovery, electrical resistance, rigidity, and so on. Some fibres have the capacity to absorb water vapour from the atmosphere and, vice versa, to lose water in a dry atmosphere. Fibres that have these characteristics are called hygroscopic. In particular, vegetable fibres have a considerable quantity, in comparison with other constituents, of hydrophobic lignin structure, which determines the amount of water that can penetrate the fibre itself. The water acts as a plasticiser which

gives the fibre softness and flexibility. Since the vegetable fibres are made of organic material, they tend to seek equilibrium with the moisture of air through the moisture absorption and desorption. The humidity is absorbed by fibres through pores and capillaries. The water penetrates in the amorphous region and is bound. The moisture content represents the weight of moisture present in a material expressed as a percentage of the dry fibre weight [32]. Moisture regain is the ability of a fibre to absorb moisture [20], [27], [33], and defined as: the amount of water a completely dry fibre will absorb from the air at a standard condition of $20 \pm 2\,^{\circ}$C and a relative humidity of 65 % [34].

Himalayan and European nettle fibres 8 samples of 5 g raw fibres and 8 samples of 5 g scoured fibre were tested. For the samples of fibres treated with different concentrations of NaOH were analysed 3 samples of 5 g. In order to undertake the Moisture Content and Moisture Regain test, the following preliminary actions must be performed [32]:

1. The first step consists of leaving the fibre in the room with standard humidity of 65 % and temperature $20 \pm 2\,^{\circ}$C for 48 hours;
2. 4 glass containers with their lid are washed with acetone and distilled water and carefully dried;
3. The glass containers and their lids are weighed separately, W_1 and W_2, with an electronic balance. Then they are put (lid open) in a conditioning oven for 24 hours at $105\,^{\circ}$C;
4. After 4 hours the glass containers (lid open) are put in the desiccators with silica gel for approximately 1 hour, to cool at room temperature. Then they are weighed separately, W_3 and W_4;
5. Samples (5 g each) of Himalayan and European nettle fibre are weighed accurately W_a and put in the respective numbered containers;
6. The samples in the containers (lid open) are put in a conditioning oven for 24 hours at $105\,^{\circ}$C;

7. After 24 hours the samples in the containers (lid open) are put in the desiccators with the desiccant Silica gel, for approximately 1 hour, to cool at room temperature;
8. Closed the lid, it is measured the combined weight W_b (glass container + lid + sample);
9. The weight of the sample W_d is calculated using equation 2.6:

$$W_d = W_b - (W_3 + W_4) \tag{2.6}$$

10. Moisture Content (MC) is calculated by equation 2.7:

$$MC = \frac{W_a - W_d}{W_d} \times 100 \tag{2.7}$$

11. The samples are weighed after being conditioned for 48 hours W_5, for 24 hours W_6 and after 48 hours W_7;
12. Moisture Regain (MR) is calculated by equation 2.8 in the three different times:

$$MR = \frac{W_i - W_d}{W_d} \times 100 \tag{2.8}$$

where $W_i = W_5$, W_6 and W_7.

2.4 Molecular structure analysis

In studies on cellulose – based fibres subjected to alkalisation an intersection of characterisation methods are used to observe the molecular changes during irreversible conversion from Cellulose I to Cellulose II which has a much more stable crystal structure. In the work of Mwaikambo & Ansell [35] on

chemically modified fibres of Hemp, Sisal, Jute and Kapok four characterisation techniques are performed: SEM, Scanning Electron Microscopy, FTIR, Fourier transform infrared spectroscopy, WAXS, Wide – angle X – ray and DSC, Differential Scanning Calorimetry. The methodology of X – ray is widespread [36], [37] but not exhaustive as it does not provide a quantitative analysis and the methodology of sample preparation according to [38], [39] may alter it.

2.4.1 Attenuated Total Reflectance – Fourier Transform Infrared (ATR – FTIR)

FTIR analysis provides a vision of qualitative and quantitative cellulose crystallinity changes studying three spectra LOI (Lateral Order Index), TCI (Total Crystallinity Index) and HBI (Hydrogen Bond Intensity). Nelson and O'Connor first developed an empirical crystallinity index for raw cotton not applicable to mercerized fibres, subsequently develop TCI which is an absorbance ratio, at specific wavenumbers ($1420/893\,cm^{-1}$) related with the quality of the measurements *i.e.* accessibility and density [40], [41], [42]. Previously Hurtubise & Krässing have demonstrated that the crystallinity degree depends as much on crystallinity as the degree of fibre mercerisation, and then it is appropriate to analyse the crystallinity index with the values of LOI, whose linear variation is due to amount of mercerized cellulose present [43]. LOI provides qualitative information about the type of cellulose crystallinity as Cellulose I, Cellulose II and amorphous cellulose have a different LOI spectrum [44], [40]. Also the spectrum of HBI, introduced by Nada [45], provides qualitative information about cellulose as shown by Kondo and Sawatari [46]. It describes three factors of the molecular structure, *i.e.* the system crystal, the degree of intermolecular order and the water presence in the molecular bonds. The HBI trend is indicative of the of hydrogen bonds amount that are forming among the hydroxyl groups in and between the cellulose chains; it generally increases during the conversion from Cellulose I to Cellulose II while the crystallinity de-

creases. TCI instead reflects exactly the crystallinity degree of the fibre being proportional to it [40]. The work by Kljun *et al.* on cotton fibre [44] shows that with increasing NaOH concentration, the values of LOI or TCI decrease, and HBI values increase, indicating a loss in crystallinity, and a change from Cellulose I to Cellulose II is observed. Research on lyocell by Goswami *et al.* [24] and by Široký *et al.* [47], [48] demonstrate similar trends in crystallinity changes, although this is with fibres that are already Cellulose II.

The analysis of FTIR fibres of hemp, sisal, jute and kapok made by Mwaikambo [35] it has verified the ability of these fibres to react to alkaline treatment. The kapok is very reactive because it contains amorphous materials such as lignin and hemicellulose and hydroxyl groups available to react with the chemicals. Fibres such as hemp instead have proven to be much more crystalline, because the less reactive to NaOH having few hydroxyl groups available and a little amorphous region.

Before performing an Attenuated Total Reflectance – Fourier Transform Infrared (ATR – FTIR) test, samples of raw, scoured and treated with 0 – 8 mol dm^{-3} NaOH of both European and Himalayan nettle fibres were conditioned at 65 % humidity (RH) and $20 \pm 2\,^{\circ}$C for 48 hours and then held in a desiccator over P_2O_5 to maintain the same atmosphere as the FTIR measurement equipment not located in the same place. In this test a Perkin – Elmer Spectrum BX spotlight spectrophotometer with diamond ATR attachment has been used to subject the nettle samples to FTIR spectroscopy (at four different points in the sample). Scanning was conducted from 4000 to 600 cm^{-1} with 64 repetitions scans averaged for each spectrum. Resolution was 4 cm^{-1}, and interval scanning was 2 cm^{-1}. Obtained spectra were normalised to the absorbance of the O – H in – plane deformation band at 1336 cm^{-1} due to any obtained changes in this band among all examined samples [44]. The trends of corrected height of Total Crystallinity Index TCI (α_{1372} / α_{2900}) [49], Lateral Order Index, LOI (α_{1429} /α_{893}) [40], and Hydrogen Bonding Intensity, HBI (α_{3336} / α_{1336}) [50], were calculated as described by Široký *et al.* [47].

2.4.2 Differential Scanning Calorimetry (DSC)

The DSC method is also used to estimate the drop in crystallinity degree by heat dehydration and the cellulose fibre decomposition. In the thermal analysis two peaks are showed: the exothermic peak corresponding to decomposition and the endothermic peak showing the crystallinity reduction. The former peak is approximately 350 °C for the plant fibres indicating the thermal degradation of the crystallites [51], [52]. Ciolacu *et al.* [53] explained that the physical behaviour and chemical reactivity of cellulose is strongly influenced by the proportion of amorphous or crystalline domains. The chemicals can penetrate easily in the amorphous areas. A non – crystalline zone is not necessarily amorphous: it can consist of liquid crystalline and nematic cellulose. DSC analysis is based on the fact that the thermal degradation is related with the supramolecular structure of cellulose and its organisation. A less crystalline structure will have dehydration peaks at higher temperatures as well as that of the thermal degradation that begins in the amorphous region with the splitting of the glycosidic bonds. Furthermore, Nada [45] has shown that the samples conditioned at greater humidity levels have highest endothermic peak of dehydration due to the water contained. It is known that the water is absorbed in the amorphous domain and that the endothermic peak area is determined by the amount of water absorbed that is lost and that therefore proportional with part of amorphous cellulose. Several researchers agree in affirming that increasing the alkali concentration the first exothermic peak, *i.e.* the decomposition temperature of the plants fibres, decreases sharply, indicating an increase in the amorphous domain of cellulose that has little thermal resistance [54], [55], [56], [57]. The Mwaikambo's study [57] on chemical modification of various cellulose fibres shows that as the kapok fibre has the first exothermic peak temperatures lower than the fibres such as hemp highly crystalline and stable to thermal degradation (Figure 2.16). The endothermic peak area is directly related to the amorphous fraction of cellulose. Native cellulose has its endothermic peaks between 50 – 150 °C in DSC curves. When the crystallinity degree decreases

the value of dehydration shifts on a higher value.

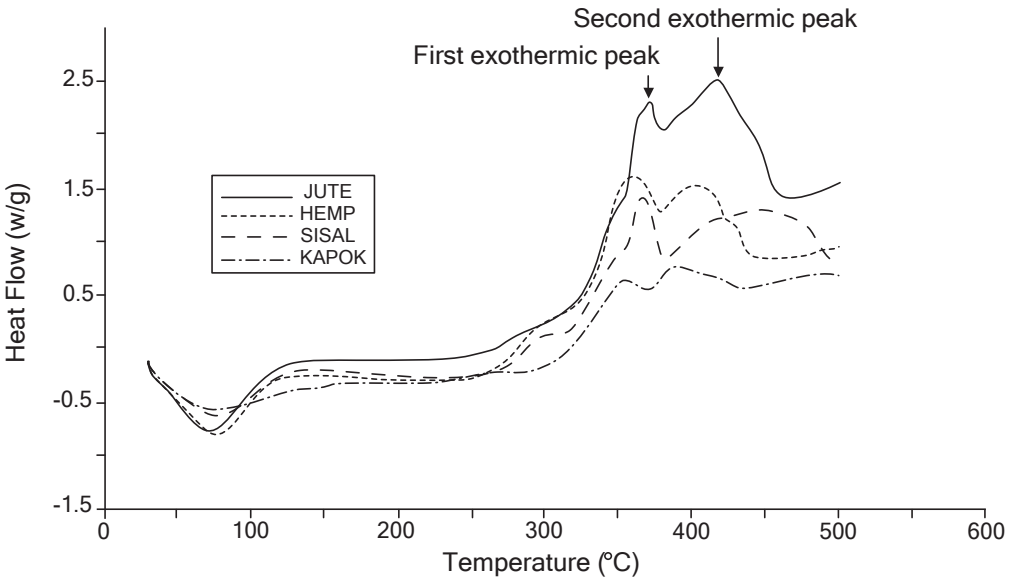

Figure 2.16: Thermograms realised by DSC of jute, hemp, sisal and kapok fibres not subjected to any treatment ([35] p.2226).

Differential Scanning Calorimetry is used to see the effect that alkaline treatments have on the involved nettle fibres and their crystallinity changes plotting the peak temperature *Vs* the NaOH concentration [48]. DSC test was performed only on the samples of Himalayan and European nettle fibres treated with NaOH from 0 to 8 mol dm^{-3}. Prior to the experiment, the samples were conditioned for 72 hours in a standard atmosphere of 65 % humidity (RH) and $20 \pm 2\,^{\circ}$C and maintained in the transport in these standard condition in a desiccator over P_2O_5 to maintain the same atmosphere as the DSC measurement equipment is not located in the same place. For each sample the measurement was repeated with three specimens weighing 5 mg, enclosed in an aluminium container and sealed [57]. A *Jade DSC* scanner calorimeter was used set to operate from 30 °C to 430 °C at 20 °C min^{-1} in a nitrogen environment purged at 30.1 ml min^{-1}.

2.4.3 Interferometry

The interferometry method was used for the first time by Goswami *et al.* [24] to study the structural changes and optical characteristics of fibres that change their section from circular to irregular. The method uses the refractive index ($n^{\parallel}$ and $n^{\perp}$) and birefringence (Δn) of fibres. These parameters show changes in the fibres orientation. The orientation angle indicates the cross – link bonds between hydrogen and cellulose chains that are parallel in completely oriented fibre while it is harder to obtain cross – link in fibre with amorphous chains. The increase of the orientation angle is coincided with a trend of hydrogen bonding to make cross – link [24]. The birefringence method is useful to understand the orientation degree of the fibre by calculating f which is the orientation factor obtained from the ratio of birefringence (Δn) with that of perfectly oriented cellulose (Δn_0).

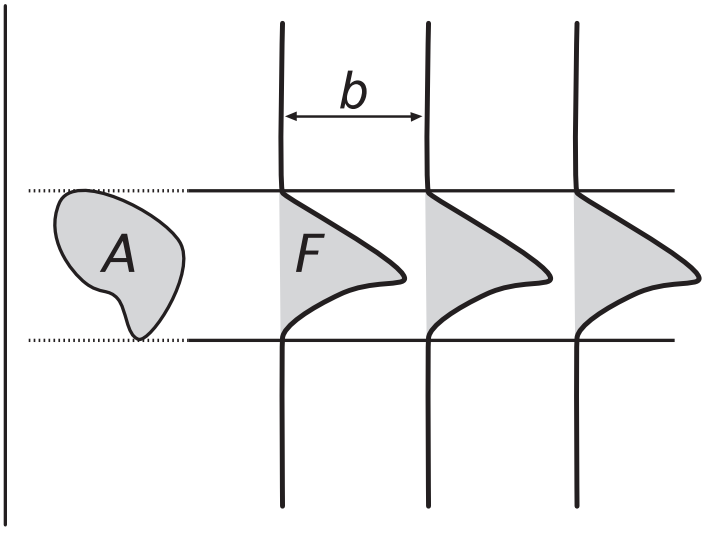

Figure 2.17: Interference fringes of fibre with irregular cross – section area (A) where F is the area between fringe shift ([24] p.458).

Interferometry is used to study the optical characteristics of fibres that treated with sodium hydroxide change the morphology of the cross – section to obtain information on the optical and structural properties of the fibres by

calculating the refractive indices ($n^{\parallel}$ and $n^{\perp}$) and birefringence (Δn) of fibres [24], [58]. In this work the interference (*Pluta*) microscope [59] equipped with a CCD camera and computer for image storage was used for a direct measurement of the birefringence by Equation 2.9:

$$\Delta n = \frac{\Delta F \cdot \lambda}{b \cdot A} \tag{2.9}$$

in which: A is the fibre cross – sectional area; F is the area enclosed under fringe shift; b is the inter – fringe spacing, λ is the wavelength (550 nm) of light used (Figure 2.17)

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Chapter 3

Characterisation of raw and scoured nettle fibres. *Girardinia diversifolia Vs Urtica dioica* and other bast fibres

3.1 Introduction

Himalayan nettle fibre has not been exhaustively investigated previously. In this section, a study of raw and scoured *G. diversifolia* fibres is presented. To support data results, the analysis was carried out in parallel with the fibre extracted from *U. dioica*, a plant of a related species, which is already used in the textile industry and in particular in luxury textiles. From the limited information available, there seemed to be advantages of *G. diversifolia* over *U. dioica* due to the size of the plant, so a comparative investigation was conducted. Raw fibre is as provided by the supplier, *i. e.* pulled from the bark; scoured fibre is where lipids, wax and other gummy impurities have been removed [1]. This analysis outlines the morphological and mechanical characteristics of the fibre providing an insight into which properties make it attractive for the textile industry.

3.2 Testing and analysis

A description of the materials and methods employed are provided in detail in Chapter 2. The data analysis in this section concerns:

- Average length (Section 2.3.1);
- Measurement of total and hollow cross – sectional area (Section 2.3.4);
- Tensile, elongation and Young's modulus (Section 2.3.5);
- Moisture regain and moisture content (Section 2.3.6);
- Fourier Transform Infrared (ATR – FTIR) to quantify the degree of the fibre crystallinity (Section 2.4.1);
- Birefringence to understand the molecular orientation of the fibre (Section 2.4.3).

3.3 Results and discussion

3.3.1 Length

The average length was measured in this study for 50 samples of raw *G. diversifolia* fibre and 50 samples of raw *U. dioica* fibre, and respective lengths were found to be 483.5 mm and 53.5 mm showing that the former fibre is substantially longer than the latter (Figure 3.1).

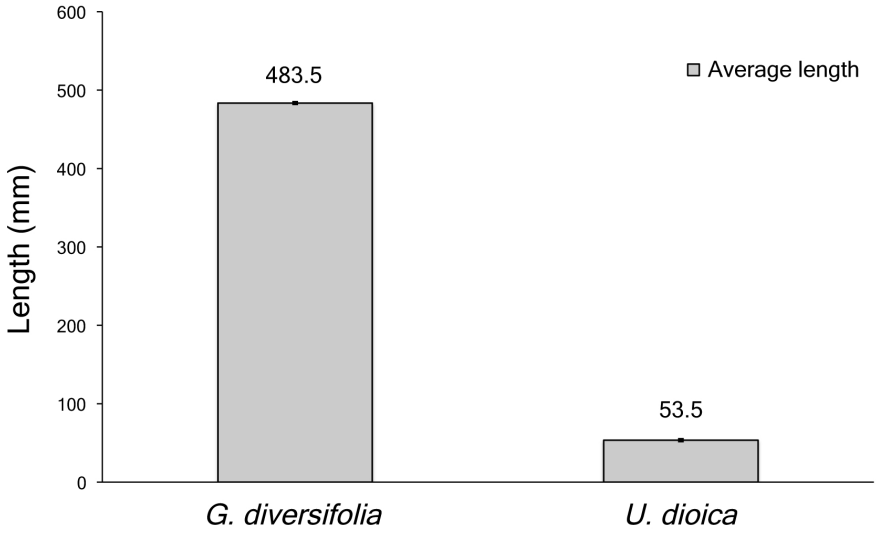

Figure 3.1: Average length of raw *G. diversifolia* and *U. dioica* fibres. Error bars show coefficient of variation (CV %).

Considering the lengths of other common bast fibres (see Table 3.1) [2], [3], [4] it should be noted that the fibre length values of *G. diversifolia* (483.6 mm) found in this study are close to, but higher than, those reported by Sethmann (211 – 400 mm) [4], where the *G. diversifolia* fibres analysed were extracted from a plantation developed to study the adaptation of Himalayan nettle in Germany. The greater length of the nettle cultivated in Nepal is to be attributed to the favourable climatic conditions that allow for good growth of the plant, resulting in longer fibres. In literature, the ideal climatic conditions for *G. diversifolia* plant are not properly described, but it is reported that the plants require frequent and consistent irrigation; it can be assumed that moist and shady Nepal forests are the ideal habitat for this plant [5], [6]. Climatic conditions and processing methods affects the structure and chemical composition of the fibre [7], [8]. Nettle fibres consist of a high percentage of cellulose, which confers a highly crystalline structure with few amorphous regions along the fibre [8], [9].

U. dioica fibre is shorter than the *G. diversifolia*; its length values (53.5 mm) are similar to those reported by Bacci *et al.* (48 – 52 mm) in their

work on *U. dioica* cultivated in Italy [2]; it was noted that these values are over the range of hemp and flax.

Evidence seems to confirm that the *G. diversifolia* fibre is the longest bast fibre known so far. As mentioned in Chapter 2, the length is an important quality of the fibre as it guarantees good performance during processing, but also enables resistant and soft yarns.

Table 3.1: Length values of the common bast fibres from the literature along with values reported from a study of *G. diversifolia* grown in Germany [2], [3], [4].

Fibre	**Flax**	**Hemp**	**Ramie**	***U. dioica***	***G. diversifolia*** grown in Germany
Fibre Length (mm)	25 – 30	15 – 25	120 – 150	48 – 52	211 – 400

The strength of the long *G. diversifolia* fibre is confirmed by the results presented in the following paragraphs about tensile tests (Section 3.3.3) and birefringence (Section 3.3.6), where the fibre shows a high fibre crystallinity and orientation degree [10]. Since the two nettle fibre types have lengths substantially different, it was considered appropriate to check whether there was a relation between length and tensile strength of the fibre. Results show that there is no significant relationship between length and strength as the linear regression is very low, with $R^2 = 0.07$ for *G. diversifolia* and $R^2 = 0.05$ for *U. dioica* (Figures 3.2 and 3.3).

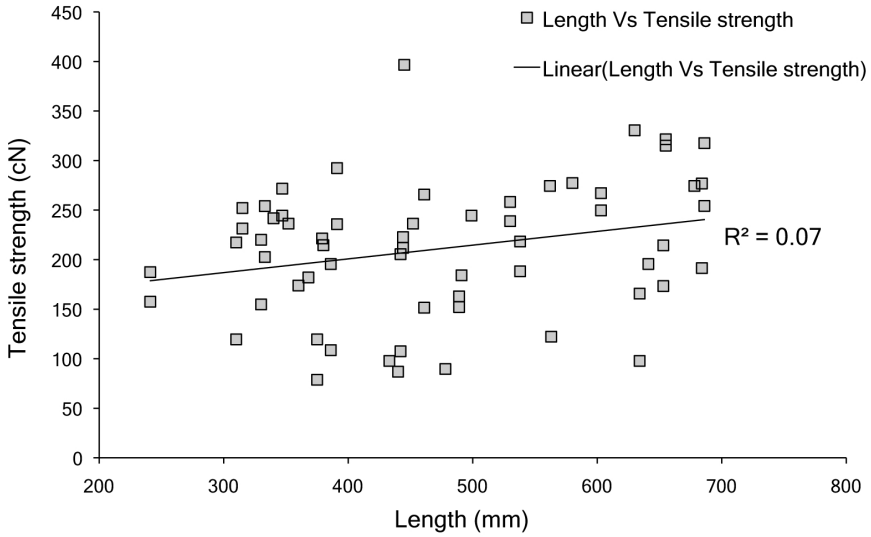

Figure 3.2: Trend line of plotted values of *G. diversifolia* length *Vs* strength (By conversion factor 1 kilogram – force (kgf) equal to 980.665 centinewtons (cN), tensile strength 0.21 kgf = 210.70 cN).

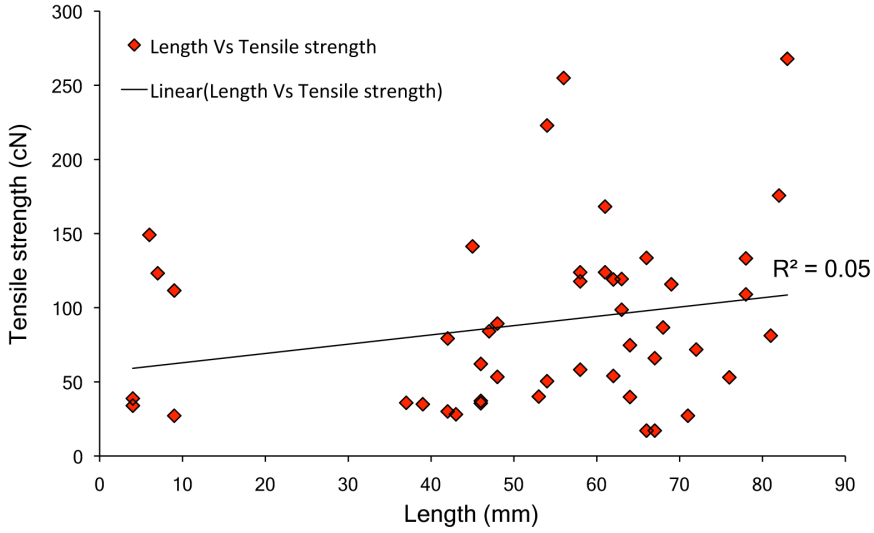

Figure 3.3: Trend line of plotted values of *U. dioica* length *Vs* strength (By conversion factor 1 kilogram – force (kgf) equal to 980.665 centinewtons (cN), tensile strength 0.09 kgf = 89.72 cN).

3.3.2 Cross – sectional area

Tables 3.2 and 3.3 show that raw *G. diversifolia* fibre has a smaller total cross – sectional area in comparison with *U. dioica*. However, when the nettle fibres are scoured an increase of the cross – sectional area is observed (Figures 3.4 and 3.5), with a narrowing cross – sectional area of the lumen observed. After scouring, the cross – sectional area of *G. diversifolia* fibre is more than twice that of scoured *U. dioica* fibre.

Table 3.2: Cross – sectional area with CV % of raw and scoured *G. diversifolia* fibres.

	Raw *G. diversifolia*	Scoured *G. diversifolia*
Total cross – sectional area (μm^2)	**488.28**	**2740.77**
CV %	1.11	0.69
Lumen area (μm^2)	**83.93**	**67.58**
CV %	3.12	3.97
Actual cross – section area (μm^2)	**344.35**	**2684.51**
CV %	0.32	0.33

Table 3.3: Cross – sectional area with CV % of raw and scoured *U. dioica* fibres.

	Raw *U. dioica*	Scoured U.dioica
Total cross – section area (μm^2)	**511.32**	**1146.21**
CV %	0.62	0.24
Lumen area (μm^2)	**13.52**	**4.48**
CV %	0.36	8.89
Actual cross – section area (μm^2)	**378.22**	**1006.38**
CV %	0.96	1.18

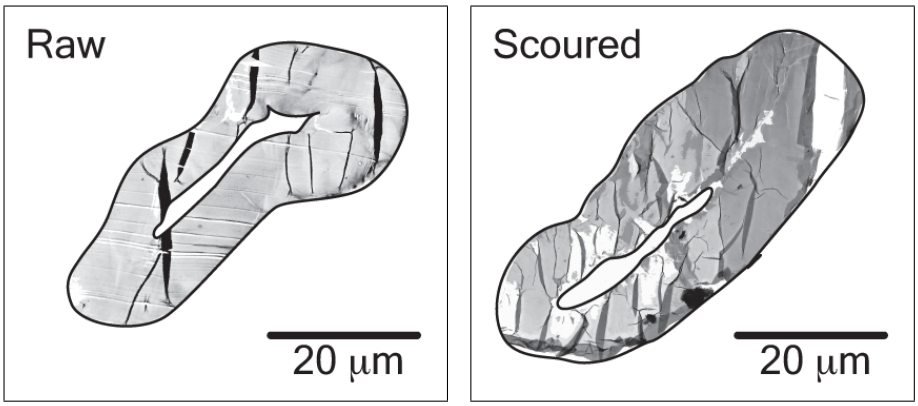

Figure 3.4: Examples of cross – sectional area of raw and scoured *G. diversifolia* nettle fibres. SEM photographs 700 x magnification.

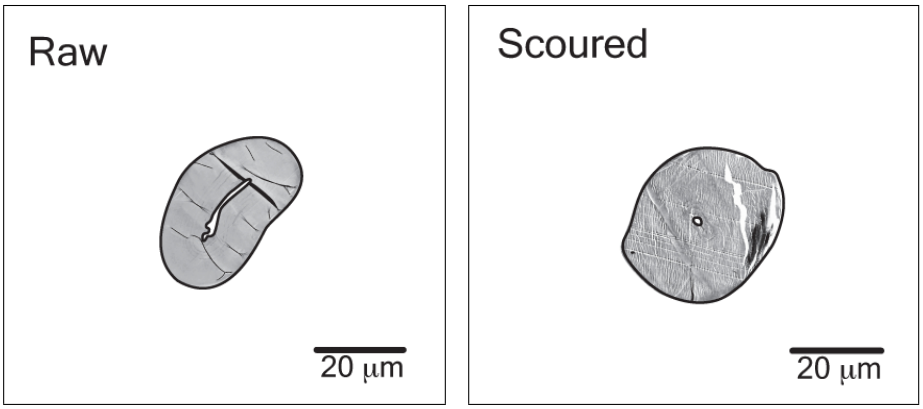

Figure 3.5: Examples of cross – sectional area of raw and scoured *U. dioica* nettle fibres. SEM photographs 700 x magnification.

Cellulose fibre in contact with aqueous solutions undergoes a phenomenon of swelling; water penetrates the amorphous regions allowing the cellulose molecules to move creating spaces into which water can penetrate; absorbed water creates swelling pushing the fibre molecules apart [11]. Cellulose molecules do not dissolve as they are maintained in the crystalline regions by intermolecular H – bonding. Swelling raises dimensional, increases accessibility and reduces the crystallinity of the fibre [12]. Swelling causes a dimensional increase of diameter, length, cross – sectional area, and volume (Figure 3.6).

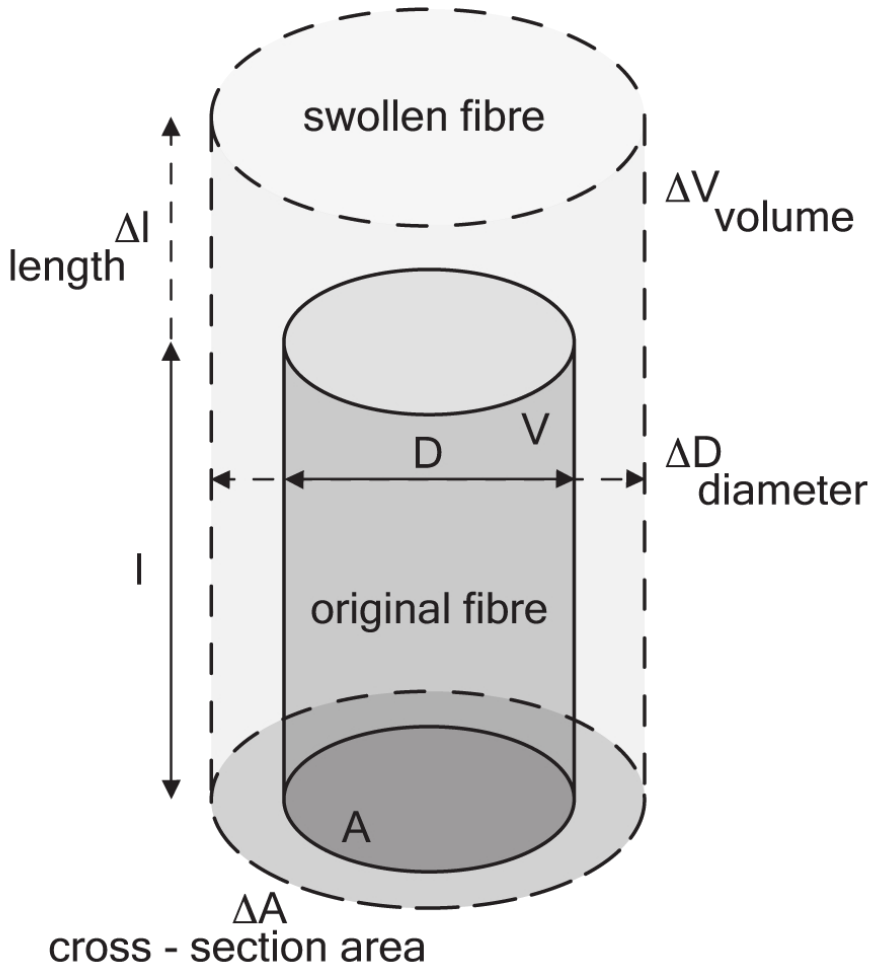

Figure 3.6: Dimensional changes of fibre caused by swelling [11].

Considering the cross – sectional area of the most common bast fibres and of cotton reported in literature [13], [14], [15], [16], [17], it is observed that nettle fibres analysed in this study have a wide cross – section, within the range of values recorded for jute and hemp (Table 3.4).

Table 3.4: Cross – sectional area of common bast fibres and cotton [13], [14], [15], [16], [17].

Fibre	**Cross – sectional area** (μm^2)
Cotton	110
Ramie	270
Flax	272
Jute	299 – 780
Hemp	441 – 911
G. diversifolia	488.28
U. dioica	511.32

3.3.3 Tensile and elongation tests

Tensile tests were performed on 50 specimens of raw *G. diversifolia* fibre (obtained from 25 fibres), 50 raw *U. dioica* fibres, 100 scoured *G. diversifolia* fibres, and 100 scoured *U. dioica* fibre. From the performed tensile tests some preliminary observations can be made about the results of elongation and tensile strength of the raw fibres. From results it is evident that the tensile strength of *G. diversifolia* fibre is substantially higher than that of *U. dioica* fibres (Table 3.5).

Table 3.5: Tensile Strength values of raw *G. diversifolia* and *U. dioica*.

	G. diversifolia	***U. dioica***
Tensile Strength (kgf)	**0.21**	**0.09**
STD	0.07	0.06
SE $\pm$	0.01	0.01

Stress average [force load (kgf) converted to $\mathrm{N\,m^{-2}}$ (MPa)], has been calculated to make a comparison with the strength values of the commonly used bast fibres referred from the most studies in MPa (Table 3.6) [8], [18], [19], [20]. It is observed that both nettle fibres have a higher tensile strength than other common bast fibres and cotton. Tensile strength of *U. dioica* is about three times higher than those of flax and eight times than that of cotton. The values of tensile strength calculated in this study for the *G. diversifolia* fibre (5099.8 MPa) are higher than those of some industrial fibres such as S – glass (4570 MPa) and Carbon (4000 MPa) [21].

Table 3.6: Tensile Strength comparison of raw *G. diversifolia*, *U. dioica*, common used bast fibres and the widely used cotton fibre [8], [22], [23], [24], [25].

Natural Plant	**Tensile Strength (MPa)**
G. diversifolia	5099.81
U. dioica	1777.50
Flax	500 – 1500
Jute	393 – 773
Hemp	690
Ramie	400 – 938
Cotton	287 – 597

With regards to elongation, the experimental results show that the average elongation % is 6.54 ± 0.02 % for *G. diversifolia* fibre and 2.80 ± 0.01 % for *U. dioica* fibre (Table 3.7); the value for *G. diversifolia* is higher than other bast fibres (flax 2 – 3 %; hemp 1 – 6 %; jute 1.5 %; ramie 4 %) [26]. Values of *G. diversifolia* fibre are greater than those of the most common bast fibres; elongation of *U. dioica* fibre is in the range of values determined for flax [27]. Furthermore, the value detected for the elongation of the *U. dioica* fibre, 2.80 ± 0.01 % is close to that found for the same fibre by Bodros & Baley (2.11 ± 0.01 %) [28].

Table 3.7: Elongation values of raw *G. diversifolia* and *U. dioica*.

	G. diversifolia	***U. dioica***
Elongation (%)	**6.57**	**2.80**
STD	0.02	0.01
SE ±	0.00	0.00

The tensile test shows that *G. diversifolia* fibre is more flexible and stronger than *U. dioica* fibre; *G. diversifolia* fibre extends more while the other under the applied force is quicker to break (Figure 3.7).

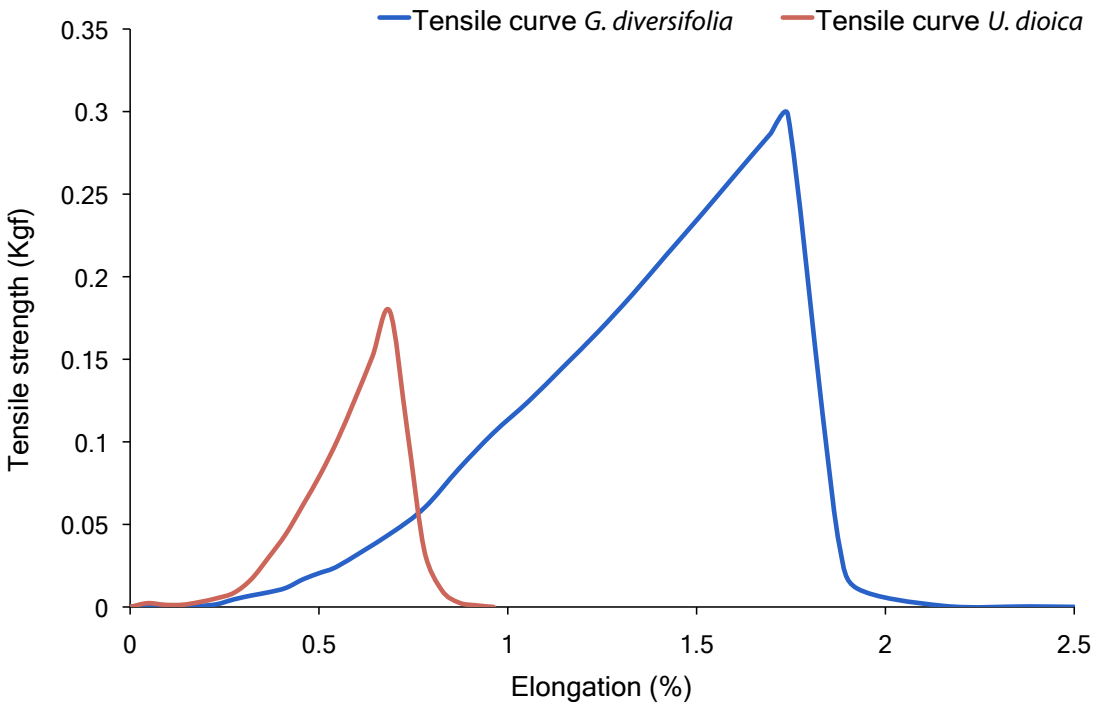

Figure 3.7: Tensile curve examples of *G. diversifolia* and *U. dioica* fibres.

When subjected to the scouring process *G. diversifolia* fibre showed a decrease in tensile strength and elongation while *U. dioica* fibre values remain unchanged for tensile strength and slight increase in elongation (Tables 3.8 and 3.9). The decrease in resistance is due to the scouring temperature as mild hydrolysis causes fragmentation of polymer chains into low molecular weights. Baig & Carr [29] demonstrated a strong correlation between weight loss and tensile properties of treated fibres. Natural cellulose fibres contain percentage of natural impurities, non – cellulose materials, as hemicellulose, representing 8 – 12 % of the total fibre weight and when removed cause the loss in tensile strength [30]. The strength and the elongation of natural fibres increases with moisture content and the presence hemicellulose favours water absorption [31], [32].

Table 3.8: Tensile strength values of scoured *G. diversifolia* and *U. dioica*fibres.

	G. diversifolia	***U. dioica***
Tensile Strength (kgf)	**0.15**	**0.09**
STD	0.06	0.09
SE $\pm$	0.01	0.01

Table 3.9: Elongation values of scoured *G. diversifolia* and *U. dioica*.

	G. diversifolia	***U. dioica***
Elongation (%)	**3.54**	**3.57**
STD	0.06	0.09
SE $\pm$	0.01	0.01

Young's modulus values found for the raw nettle fibre are higher for *G. diversifolia* fibre in comparison with *U. dioica* (Table 3.10). Once the fibres are scoured, values of E decrease and in particular those of *G. diversifolia* fibre which become considerably lower. This phenomenon is explained by the relationship between Young's Modulus and cross – sectional area of the fibre as demonstrated by Lamy *et al.* on flax fibre [28], [31], [33] and by Prasad *et al.* on hemp fibres [34], [35]; E decreases when cross – section increases [36]. Nettle fibres subject to scouring have a wider cross – section due to the swelling caused by washing. Bast fibres have a high Young's modulus due to the large cellulose content. Comparing the values of common bast fibres, cotton and E – glass fibre (Table 3.11), it is observed that *G. diversifolia* and *U. dioica* fibres have higher E and cellulose content. The values are lowered when the fibre is subjected to scouring, since water has penetrated the amorphous regions, disrupting fibre

hydrogen bonds and breaking those with the adjacent crystalline regions. This means that raw fibres have E values close to the crystalline cellulose while the treated fibres lose crystallinity and then stiffness becoming more elastic [37].

Table 3.10: Young's modulus with SE $\pm$ and total cross – sectional area of raw and scoured *G. diversifolia* and *U. dioica*.

	raw		**scoured**	
	G. diversifolia	***U. dioica***	***G. diversifolia***	***U. dioica***
Young's Modulus (GPa)	**102**	**79**	**20**	**29**
SE $\pm$	0.02	0.29	0.89	0.69
cross – sectional area (μm^2)	**488.28**	**511.32**	**2740.77**	**1146.21**

Table 3.11: Young's modulus of the most common bast fibres [3], [38], [39].

	Flax	**Hemp**	**Jute**	**Ramie**	**Cotton**	**E – glass**
Young's Modulus (GPa)	80	70	30	59	12	73

3.3.4 Moisture regain and moisture content

Tests to calculate moisture content and moisture regain were carried out as described in the Chapter 2 for 0.5 g x 8 samples of raw nettle fibres, 4 *G. diversifolia* and 4 *U. dioica*, and for the same amount of scoured nettle fibres. From Table 3.12 it is noted that the moisture content found for *G. diversifolia* (6.00 %) is about a third less than that of *U. dioica* (9.40 %). Moisture content values are important because they are related to mechanical performance of the fibres; as moisture content increases, tensile properties improve. When the nettle fibres are scoured moisture content decreases compared with the raw fibre and, consequently, tensile strength decreases.

Table 3.12: Moisture content with SE ± of raw and scoured fibres of *G. diversifolia* and *U. dioica* fibres.

	raw	scoured	raw	scoured
	G. diversifolia	***G. diversifolia***	***U. dioica***	***U. dioica***
Moisture content (%)	**6.00**	**5.92**	**9.40**	**9.39**
SE ±	0.002	0.003	0.001	0.001

The moisture content is also interesting because it is an internal factor that influences the dielectric properties of the fibres [40]. All materials, including fibres, possess their own ability to acquire a static charge, which can be transferred from one material to another. The moisture content of a fibre, and the humidity to which it is exposed, affect significantly the ability of the fibre to dissipate these charges. In particular the cellulose fibres do not accumulate high static electricity and is instead interesting to know their degree of electrical insulation. [41]. It was shown, with respect to dielectric properties of cotton, viscose, and many other fibres [13], that the permittivity (*i. e.* the susceptibility

of a material to transmit or allow an electric field [42]) increases with increasing moisture content; resistance (the electrical resistance of an object is a measure of its opposition to the passage of a steady electric current [42]) is inversely proportional to the moisture regain [42]. In particular, according to Saville [43], in the case of fibres that absorb water the approximate relation between the resistance and the moisture content is given by Equation 3.1:

$$RM^n = k \tag{3.1}$$

where R is the resistance, M is the moisture content (%) while n and k are constants.

The relation holds for relative humidity between 30 and 90 %. The changes in resistance are considerably large, indeed, the resistance decreases about ten times any increase in relative humidity of 13 % [43]. It follows then that to have a significant variation in the dielectric properties moisture content should be at least about 10 %, while values close or equal to zero do not cause any kind of variation [44]. Herein it is observed that *G. diversifolia* fibre moisture content is 6.0 %, therefore it is possible to hypothesise a good insulating capacity of the fibre in question while this cannot be asserted for the *U. dioica* fibre which has a moisture content value of 9.4 % to the limit for good insulation. The values of *U. dioica* fibre are closer to those found for the other most common bast fibres (Flax 8 – 12 %; Hemp 6.2 – 12 %; Jute 12.5 – 13.7 %; Ramie 7.5 – 17 %) and cotton (7.85 – 25 %) [45], [46], [47], [48].

Table 3.13: Moisture regain with SE ± of raw and scoured fibres of *G. diversifolia* and *U. dioica* fibres.

	raw	scoured	raw	scoured
	G. diversifolia	***G. diversifolia***	***U. dioica***	***U. dioica***
Moisture regain (%)	**6.79**	**6.68**	**8.80**	**8.62**
SE ±	0.003	0.003	0.010	0.010

Moisture regain of raw nettle fibres is 6.79 % for *G. diversifolia* fibre and 8.80 % for *U. dioica* (Table 3.13). Considering the values of moisture regain of the four main bast fibres (Flax 12 %; Hemp 12 %; Jute 13.8 %; Ramie 6 %) and that of the cotton (8 %) [49], [14], it is concluded that the moisture regain of raw *G. diversifolia* fibre (6.79 %) is close to ramie (6 %), the lowest, while the moisture regain of the *U. dioica* fibre (8.80 %) is same to that of cotton (8 %) and lower than other common bast fibres. The slight decrease from the two nettle fibres in absorbing water from the environment when they were scoured is most probably due to hemicellulose removal, as hemicellulose readily absorbs water [31], [32]. Furthermore, it is known [49] that moisture regain of the fibres is directly proportional to the amorphous region as the water penetrates more easily when the fibre presents a higher percentage of amorphous regions; this applies only to water and not for other liquids [49]. Values of total crystallinity index (TCI) [49] increase when moisture regain decreases (section 3.3.5), which is when the fibres are scoured. In most vegetable fibres the moisture is taken mostly from the middle lamella [49]; it can be observed how, for example, a bast fibre such as flax has a moisture regain higher (12 %) than a seed fibre like cotton (8 %) which is devoid of a middle lamella. Another explanation in the absorption of water due to the morphology is that fibres like flax are

straight, porous and with large capillaries facilitating the penetration of water while fibres such as cotton, *G. diversifolia* and *U. dioica* are twisted and present convolutions, hence accessibility is reduced [49]. Moisture regain is the ability of a fibre to adapt to environmental conditions [49]. This affects the properties of fibre. A moisture regain of 6 – 8 % is required to avoid harshness, loss in strength and to reduce wrinkles [14], [49]. A value in this range provides a good physical stability of the fibre. *G. diversifolia* at 6.79 % is an interesting fibre because the moisture has no significant effect on its mass and other physical properties. It remains appropriate in the event of warm weather since it is a natural vegetable and hygroscopic fibre.

3.3.5 Fourier Transform Infrared (ATR – FTIR) Spectroscopy

ATR – FTIR (Attenuated Total Reflectance – Fourier Transform Infrared) spectroscopy was performed on raw and scoured samples of both *G. diversifolia* and *U. dioica* fibres as described in Chapter 2. The analysis of Total Crystallinity Index (TCI $\alpha_{1372/2900}$) [50], Lateral Order Index (LOI $\alpha_{1429/893}$) [51], and Hydrogen Bonding Intensity (HBI $\alpha_{3336/1336}$) [52], trends were calculated to verify the morphological characteristics of the fibres and their qualitative behaviour when the fibre are scoured. In the studies of O'Connor *et al.* [53], Nelson & O'Connor [54], and Colom & Carrillo [55], it was found that through the study of IR spectra that fibre crystallinity can be understood. Infrared radiation absorbed from cellulose molecules creates a vibration that is affected by inter – and intra – molecular interactions and by hydrogen bonds. The molecules vibration in the cellulose polymer chain will be different depending on whether there is an ordered crystalline phase rather than an unordered amorphous phase [56]. Then the absorption bands can be associated to crystalline or amorphous regions as well, as introduced by Nada *et al.* [57] to a hydrogen bond intensity. The absorption band typical of a crystalline phase of

a polymer has been associated by O'Connor *et al.* [53] at a ratio of absorption obtained from the spectra changes at 1429 cm^{-1}, while the amorphous at 893 cm^{-1}. The ratio of these two bands provides a 'crystallinity index', which will later be referred to as 'lateral order index' (LOI) since a new ratio given by the ratio of the absorption bands at 1372 cm^{-1} and 2900 cm^{-1} will be named 'total crystallinity index' (TCI) [54]. From here it is possible to deduce that when the ratio of LOI or TCI is high, the fibre will have a large percentage of crystalline regions. Cellulose has three hydroxyl groups that can interact with other hydroxyl groups to form valence bonds. The network of hydrogen bonds is important for the crystallinity and the structure of the molecular chains, which in the transformation from Cellulose I at Cellulose II, which occurs with the alkaline treatment, are configured into a model (Cellulose II) antiparallel [58]. During the transformation intermolecular hydrogen bonds are formed. They bind the hydroxyl groups of a molecule of cellulose with the ring oxygen of an adjacent cellulose molecule. The second hydrogen bonds make intermolecular bonds between hydroxyl groups and carbon ring of neighbouring chains [58]. The absorption bands ratio at 3336 cm^{-1} and 1336 cm^{-1}, introduced by Nada *et al.* [57] is called 'hydrogen – bond intensity' (HBI) and shows the state of crystalline order and the degree of intermolecular regularity [59].

The analysis at this stage does not concern molecular modification from Cellulose I to Cellulose II. Here the fibre is investigated raw and subjected to scouring. The results of ATR – FTIR are listed in Table 3.14.

Table 3.14: ATR – FTIR analysis of raw and scoured fibres of *G. diversifolia* and *U. dioica* fibres.

	G. diversifolia		***U. dioica***	
	Raw	**Scoured**	**Raw**	**Scoured**
TCI ($\alpha_{1372/2900}$)	0.52	1.76	0.32	0.51
LOI ($\alpha_{1429/893}$)	0.68	1.09	2.22	1.77
HBI ($\alpha_{3336/1336}$)	12.45	15.39	3.30	18.00

Table 3.14 suggests that *G. diversifolia* fibre is more crystalline than the *U. dioica*, which, however, has a greater LOI, the degree of the ordered regions perpendicular to the chain direction [60]. The ratio of HBI in raw *U. dioica* fibre is much shorter than that of *G. diversifolia*. Scouring causes an increase in crystallinity in the fibres due to the removal of impurities and the tension created by high temperatures [61]. *G. diversifolia* fibre, which is more crystalline has an increase in LOI, while *U. dioica* decrease. This latter seems to be more affected by the phenomenon of swelling because the order of the molecular chains decreases and the ratio of HBI increases substantially. Also in the *G. diversifolia* fibre HBI increases but slightly, suggesting that this fibre has fewer available hydroxyl groups able to interact by inter and intramolecular hydrogen bonding [61].

3.3.6 Birefringence

The test was performed as described in Chapter 2 on 5 samples of raw fibre and 5 of scoured for *G. diversifolia* and *U. dioica* fibres. A minimum of 3 images were captured from each of the 5 samples. Birefringence (Δn) was determined using the mean value of the respective cross – sectional area of raw

and scoured fibres (*G. diversifolia* fibre raw: 488.28 μm^2; scoured: 2740.77 μm^2. *U. dioica* fibre raw: 511.32 μm^2; scoured: 1146.21 μm^2). Hermans [62] determined the orientation factor (f) related to the birefringence (Δn) by the ratio of the birefringence of the fibre with that of an ideal fibre in which the molecules are perfectly oriented parallel to the fibre axis denominated Δn_0 (Equation 3.2); Δn_0 for cellulose fibre is calculated to be 0.055 [63], [64], [65]. Birefringence (Δn) and orientation factor (f) calculated for the fibres of both nettle species are shown in Table 3.15.

$$f = \frac{\Delta n}{\Delta n_0} \tag{3.2}$$

Table 3.15: Birefringence (Δn) and orientation factor f of raw and scoured fibres of *G. diversifolia* and *U. dioica* fibres.

	G. diversifolia		***U. dioica***	
	Raw	**Scoured**	**Raw**	**Scoured**
Δn	**0.091**	**0.018**	**0.135**	**0.044**
SE $\pm$	0.01	0.00	0.00	0.00
f	**1.66**	**0.32**	**2.46**	**0.80**
SE $\pm$	0.23	0.02	0.06	0.08

Figure 3.8 shows examples of interferograms of samples of raw and scoured *G. diversifolia* fibres and raw and scoured *U. dioica* fibres. Birefringence for both fibres is higher than that of the intrinsic birefringence of perfectly oriented cellulose. When the fibres are scoured it decreases in particular that of the *G. diversifolia* fibre. Considering the orientation factor f and knowing that the values of a perfectly oriented fibre are equal to 1 and a non – oriented fibre are equal to 0 [11], it can be deduced that the *G. diversifolia* fibre is

very oriented when raw and little once scoured. While the raw *U. dioica* fibre has values considered very high for an orientation factor and that continue to be closer to 1 even when the fibre is scoured. Hermans' orientation factor *f* is the quantity most used for the molecular orientation of polymers [63]. The birefringence is inversely proportional to the fibre swelling, as when a fibre undergoes the phenomena its area increases. For this reason birefringence (Δn) and the related orientation factor *f* decrease. Looking at the *U. dioica* fibre values of the previous analysis (moisture regain and the huge rise from raw to scoured fibres in the trend of HBI), the fibre demonstrates a susceptibility to the penetration of water and the rupture of inter – molecular bonds. Even the *U. dioica* LOI trend is in agreement with the values of f for which the molecular orientation decreases when the fibres are treated. It can be assumed as reported by Kljun *et al.* [66] and Poletto *et al.* [67] that the *U. dioica* fibre consists of ordered cellulose chains that allow ease of inter – molecular hydrogen bonds between neighbouring chains giving also a high degree of crystallinity. An inconsistency between the values of *f* and the trend of LOI occurs for the *G. diversifolia* fibre showing an increase in LOI when the fibre is scoured and a decrease of *f*.

This variation is explained by the fact that the *G. diversifolia* fibre as the cotton is a twisted fibre which has several convolutions. Treatments such as scouring, bleaching or alkaline, lead this type to swell and untwist [49]. *G. diversifolia* fibre has shown so far to be difficult to penetrate by water, showing to be a fibre with a high crystallinity. After scouring, it increases its amorphous regions also decreasing the orientation factor of the molecular chains (f) and the birefringence (Δn) but increases LOI, the degree of the ordered regions perpendicular to the chain direction, because the fibre acquires a new morphology that makes it more regular and flat. To confirm this, that the twisted form influences the values of fibre birefringence, it is the fact that fibres as cotton and other cellulose fibres, in this case *G. diversifolia*, the birefringence is lower (for example than for *U. dioica* fibre) as the molecules are arranged

helically around the fibre axis [11].

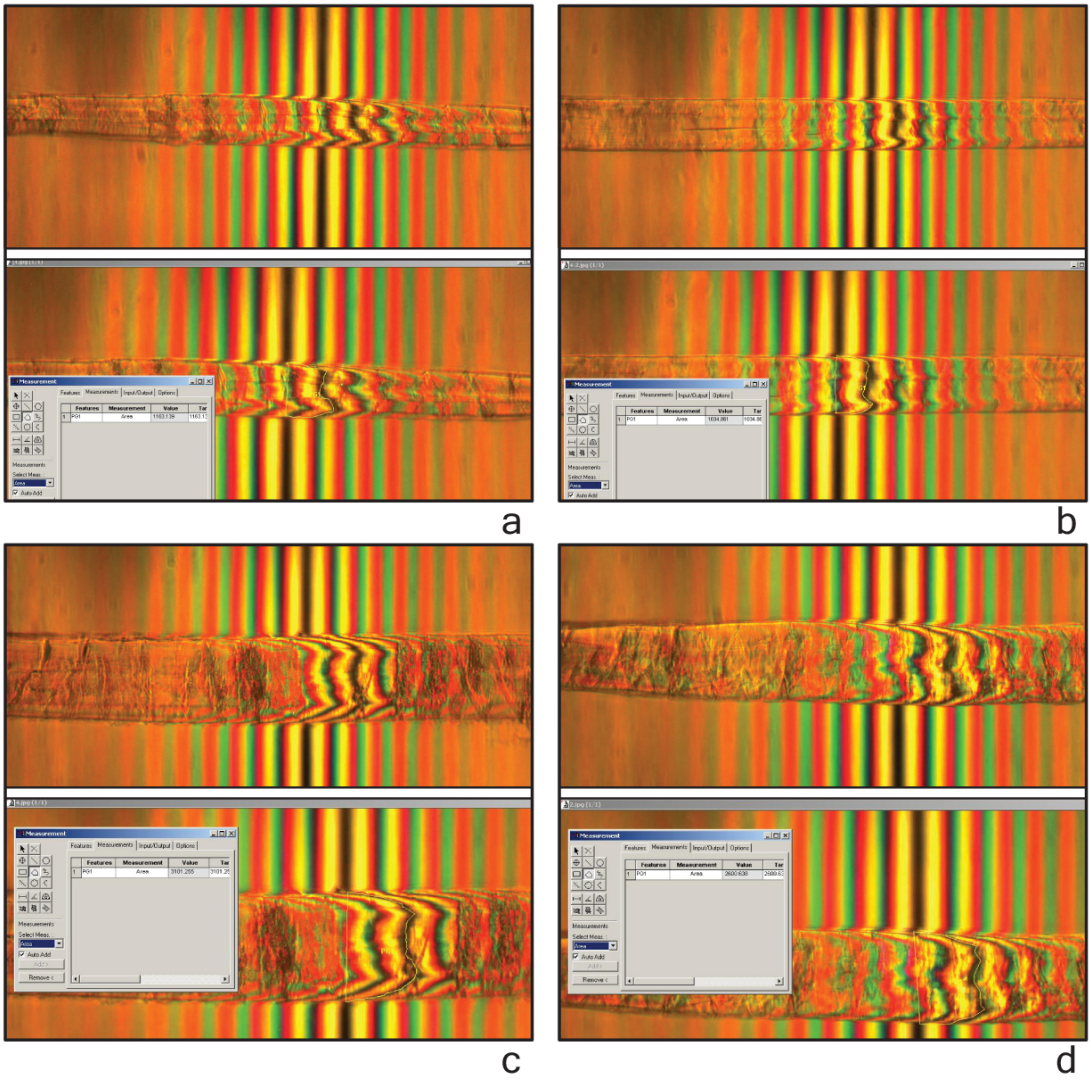

Figure 3.8: Examples of interferograms (Magnification 20 x 0.01 mm) of two samples of raw (a) and scoured (b) *G. diversifolia* fibres and of raw (c) and scoured (d) *U. dioica* fibres. For each specimen the analysis of the final contoured – line interferograms by Image – Pro Plus software used to calculate the inter – fringe spacing b and the area enclosed under the fringe shift ΔF.

3.4 Conclusion

This chapter investigates *G. diversifolia* fibre, in comparison with that of *U. dioica*, in order to obtain an understanding of the relative physical and mechanical characteristics and suitability for the textile industry. Experimental results obtained reveal that *G. diversifolia* is the longest bast fibre known so far (Table 3.1). The moist and shady habitat of Nepalese forests favours this characteristic. In fact a slight decrease in fibre length was reported for *G. diversifolia* fibres obtained from an experimental plantation in northern Germany (possibly due to this regions' Atlantic climate). Both nettle fibres have a larger cross – section than the other common bast fibres, close to the size of hemp and jute (Table 3.4). In particular, fibre *G. diversifolia* has a wider lumen (Tables 3.2 and 3.3) When the fibres are scoured, they are subject to swelling, increase considerably their cross – section (Table 3.2). From the tests of resistance, measuring tensile strength and elongation, *G. diversifolia* fibre shows a clear superiority compared to the *U. dioica* (Table 3.5) Both have a superior resistance to common bast fibres and in particular the values of *U. dioica* are very close to those of flax (Table 3.6) *G. diversifolia* fibre once scoured decreases its strength (Table 3.8) and elongation as the high temperatures of the cleaning process causing the polymer chains rupture. Raw *G. diversifolia* fibre has a Young's modulus higher than that of the *U. dioica*. Once scoured this value, in *G. diversifolia* fibre, decreases substantially being inversely proportional to the section, which, because of the swelling during the scoured process, increases (Table 3.10). *G. diversifolia* fibre, raw and scoured, has moisture content values lower than those of the *U. dioica* fibre and other bast fibres (Section 3.3.4). A low moisture content is an indication of a good dielectric insulating capacity of the material (discussed in Section 3.3.4). *G. diversifolia* and *U. dioica* fibres have a moisture regain in a range of values that does not influence their physical stability (Table 3.13). *G. diversifolia* fibre has values lower than *U. dioica* and thus higher stability to humidity. When the fibres are scoured moisture regain slightly decreases as with

scouring hygroscopic hemicellulose is removed (Table 3.13). The mechanical properties of the fibre, such as strength, elongation and the ability to absorb humidity from the environment, may be affected by the molecular chains orientation. The study, by ATR – FTIR, of the trend of TCI, LOI and HBI describe qualitatively the molecular arrangements, explaining the strength properties of the fibres. *G. diversifolia* fibre is, from its trend of TCI and LOI, highly crystalline conferring greater strength and lower moisture absorbency. *U. dioica* fibre is less crystalline than *G. diversifolia* but it has a higher molecular chain orientation. Having more amorphous regions, this fibre interacts more readily with water. Its trend of HBI, considering the raw fibres is much lower than that of the *G. diversifolia* fibre; but when the fibres are scoured, *U. dioica* fibre undergoes a sharp increase, while the *G. diversifolia* increases slightly (Table 3.14). The birefringence analysis confirms a high orientation of the molecular chains of the both fibre, which remains even when scoured (Table 3.15). The *G. diversifolia*, when scoured, has an improvement in terms of orientation of the molecular chains that are around the fibre axis, as it is regularised and flattens. The analysis shows that both fibres are very strong and resistant, particularly the *G. diversifolia* fibre, by their degree of crystalline when they are raw. Once scoured, the resistance of the fibres decreases and the elongation increases: decreases since the crystalline and increase the hydrogen bonds between the molecular chains within the fibre. In *G. diversifolia* fibre this phenomenon is influenced by hemicellulose percentage that favors water penetration. The fibre even after the scouring remains crystalline and does not undergo an increase in elongation.

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Chapter 4

Effect of NaOH treatment on *Girardinia diversifolia* and *Urtica dioica* fibres

4.1 Introduction

In this Chapter treatment with increasing concentrations of sodium hydroxide (NaOH) was carried out in order to induce changes in the molecular structure, from Cellulose I to Cellulose II, of both Himalayan and European nettle fibres, and to assess how this variation could influence their physical and mechanical characteristics. *G. diversifolia* and *U. dioica* fibres at various concentrations of NaOH were analysed morphologically, physically and mechanically.

Cellulose crystal structure changes subject to alkaline treatment (as described in detail in Section 1.5.3): at lower concentrations of NaOH, intermolecular hydrogen bonds between neighbouring cellulose chains are broken. As the concentration of NaOH is increased, intramolecular hydrogen bonds within individual cellulose chains are broken. The hydrogen bonds reform into a new order that re – orients cellulose chains in the fibre to form Cellulose II (Figure 4.1). This treatment causes changes in the molecular structure of cellulose, removal

of non – structural material, variations in mechanical performance, and shape regularisation.

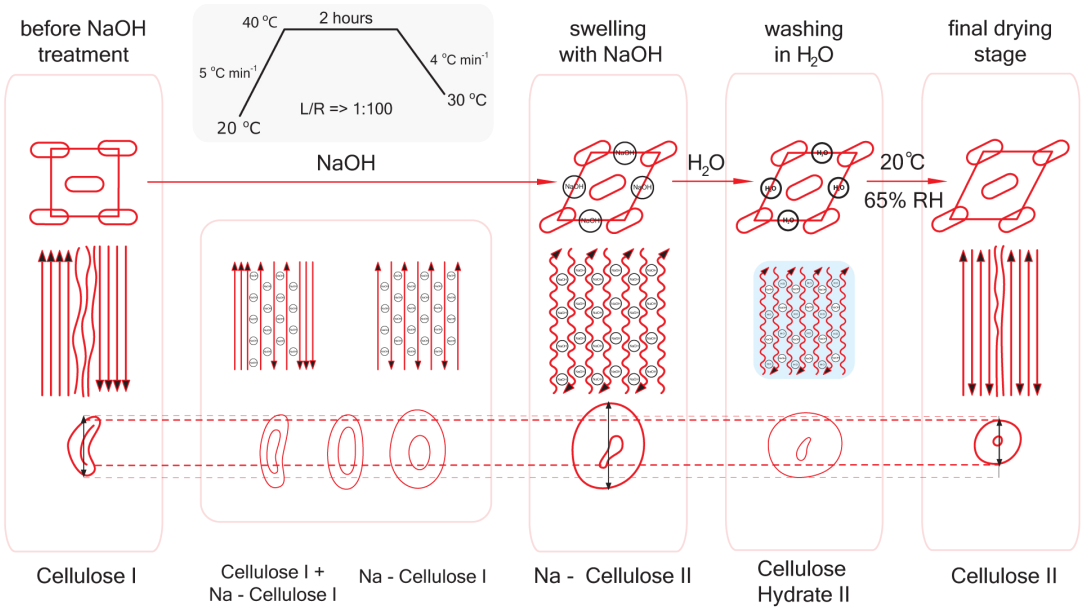

Figure 4.1: Cellulose crystal structure and cross – sectional changes on alkaline treatment [1], [2].

This work aims to characterise the *G. diversifolia* and *U. dioica* fibres and to understand how the alkaline treatment affects its morphological characteristics and its mechanical properties. The following data relate to *G. diversifolia* and *U. dioica* fibres treated with different NaOH concentrations, starting from the analysis of the changes in the molecular structure of the fibre and then mechanical tests and observation of the cross – section.

4.2 Testing and analysis

For both *G. diversifolia* and *U. dioica* fibres, 9 samples were subjected to alkaline treatment with increasing concentrations of sodium hydroxide (an incubation series in varied aqueous NaOH solutions from 0 – 8 mol dm^{-3}), as described in Chapter 2, Section 2.3.3, and then tested following the procedures described previously in Chapter 2. The data analysis in this section concerns:

- Measurement of total and hollow cross – sectional areas (Section 2.3.4);
- Tensile, elongation and Young's modulus (Section 2.3.5);
- Moisture regain and moisture content (Section 2.3.6);
- Fourier Transform Infrared (ATR – FTIR) to quantify the degree of the fibre crystallinity (Section 2.4.1);
- Differential Scanning Calorimetry (DSC) to estimate the drop in crystallinity degree by heat dehydration and the cellulose fibre decomposition;
- Birefringence to understand the molecular orientation of the fibre (Section 2.4.3).

4.3 Results and discussion

4.3.1 Changes in crystallinity

G. diversifolia and *U. dioica* fibres were treated at different concentrations of aqueous NaOH solution. Changes in crystallinity were analysed by ATR – FTIR spectroscopy, as done by Mwaikambo *et al.* [3] on alkalisation of hemp, sisal, jute, and kapok fibres, and by DSC and birefringence, following the work done by Goswami *et al.* [4] on lyocell. Differences in fibres were compared morphologically, physically and mechanically according to work done by Gassan & Blendzki on jute fibre [5]. Changes in crystallinity influenced structural and mechanical properties of the fibre [3], [4], [5], [6], [7].

4.3.1.1 Fourier Transform Infrared (ATR – FTIR) analysis of NaOH treated nettle fibres

The study of TCI (Total Crystallinity Index), LOI (Lateral Order Index) and HBI (Hydrogen Bond Intensity) are performed to understand the qualitative behaviour of the fibre crystallinity when subjected to treatments with aqueous

solutions of NaOH, as can be seen for *G. diversifolia* fibres (Table 4.1) and *U. dioica* fibres (Table 4.2).

Table 4.1: ATR – FTIR analysis of *G. diversifolia* fibres treated with varying NaOH concentrations.

NaOH mol dm^{-3}	**TCI** ($\alpha_{1372/2900}$)	**LOI** ($\alpha_{1429/893}$)	**HBI** ($\alpha_{3336/1336}$)
0	0.69	1.07	11.15
1	0.79	0.70	9.62
2	0.66	1.53	11.49
3	0.71	0.29	14.47
4	0.51	0.15	12.62
5	0.52	0.15	18.94
6	0.60	0.17	14.69
7	0.50	0.16	17.56
8	0.55	0.18	19.00

Table 4.2: ATR – FTIR analysis of *U. dioica* fibres treated with varying NaOH concentrations.

NaOH mol dm^{-3}	**TCI** ($\alpha_{1372/2900}$)	**LOI** ($\alpha_{1429/893}$)	**HBI** ($\alpha_{3336/1336}$)
0	0.16	2.19	20.21
1	0.36	1.79	15.30
2	0.48	1.83	15.57
3	0.33	0.88	18.13
4	0.51	0.70	27.86
5	0.56	0.47	18.72
6	0.57	0.48	20.78
7	0.39	0.45	21.56
8	0.60	0.39	22.33

In agreement with studies on other cellulosic fibres [8], [9], [10], for both species of nettle fibre increase in NaOH concentration leads to generally decreased LOI, while HBI values are higher in treated fibres (Figures 4.2 and 4.3). Gassan & Bledzki in their investigations of alkaline treatments for jute fibre [5], [6] report that during the reaction with NaOH hemicellulose, which separates some chains of cellulose, is removed allowing the formation of new hydrogen bonds. Examining the trends in more detail, NaOH treatment causes, in both fibres, an initial increase in LOI to 2 mol dm^{-3}, followed by a significant reduction between 2 and 4 mol dm^{-3} NaOH, which is associated with a decrease in crystallinity and a transformation of Cellulose I to Cellulose II. Conversely HBI heightens with increasing NaOH concentrations showing significant changes in HBI between 3 and 5 mol dm^{-3} NaOH associated with a decrease in crystallinity and a transformation of cellulose I to Cellulose II. Above 4 mol dm^{-3} NaOH, no further crystallinity change was observed therefore it is possible to deduce that from 5 mol dm^{-3} NaOH the fibres are constituted of Cellulose II.

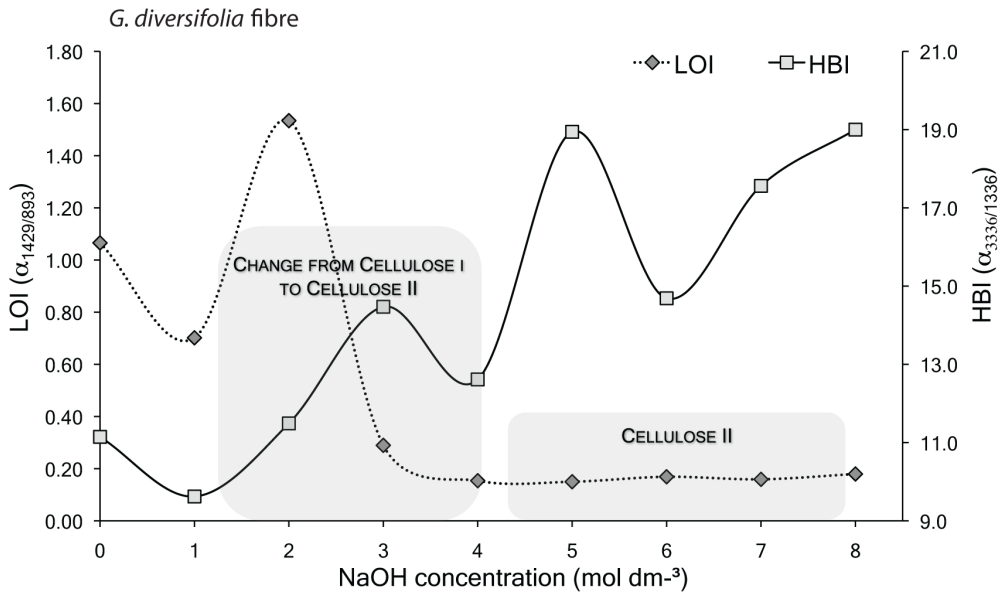

Figure 4.2: *G. diversifolia* LOI ($\alpha_{1429/893}$) *Vs* HBI ($\alpha_{3336/336}$) trends at increasing NaOH concentration. At 2 mol dm^{-3} NaOH the transformation from Cellulose I to Cellulose II starts and from 5 mol dm^{-3} NaOH the change in Cellulose II is completed.

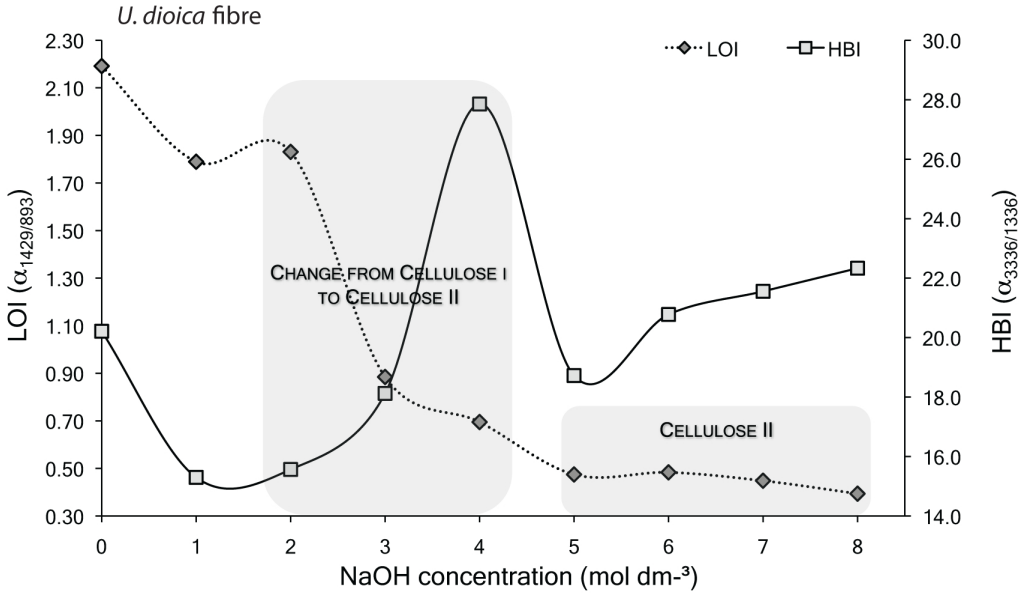

Figure 4.3: *U. dioica* LOI ($\alpha_{1429/893}$) *Vs* HBI ($\alpha_{3336/336}$) trends at increasing NaOH concentration. At 2 mol dm^{-3} NaOH the transformation from Cellulose I to cellulose II starts and from 5 mol dm^{-3} NaOH the change in cellulose II is completed.

The observations for TCI are not so straightforward (Figure 4.4). For *G. diversifolia* fibre a decrease in TCI values is observed with increasing concentration of NaOH; the opposite situation is observed for *U. dioica* fibre, which shows an increasing of TCI trend at higher concentration of NaOH, doubling its original values at end of treatment. According to Mwaikambo *et al.* [3], fibres with high content of amorphous material, hemicellulose, lignin and reactive hydroxyl groups on the fibre surface and in the amorphous region, are more reactive to alkaline treatment. Conversely, fibres with high crystallinity index, and consequently less amorphous regions, show a lower reactivity as they have less hydroxyl groups available to react. The values of TCI obtained by FTIR test show that the *U. dioica* fibre to 8 mol dm^{-3} NaOH has a value close to that recorded for untreated *G. diversifolia* fibre.

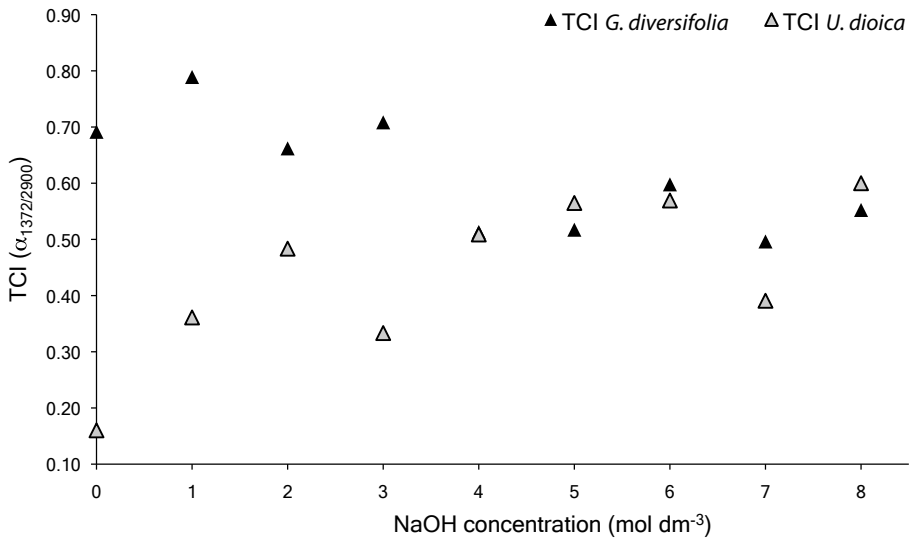

Figure 4.4: TCI ($\alpha_{1372/2900}$) trend of *G. diversifolia Vs U. dioica* fibres.

4.3.1.2 Differential Scanning Calorimetry (DSC) investigation of NaOH treated nettle fibres

DSC investigation was carried out as an alternative way to estimate the crystallinity of the fibre. Water absorption takes place mainly in the amorphous domains of cellulose; by measuring the area of the endothermic peak the heat

dehydration, loss of water absorbed, is determined, the higher value, the greater the fraction of amorphous cellulose [11]. From the performed DSC test, it is possible to note that *G. diversifolia* and *U. dioica* recorded decomposition peaks higher than the most common bast fibres [3]. *G. diversifolia* fibre has the decomposition peak in the range of 360 – 380 °C. Furthermore, it is noted that, although generally stable in the decomposition temperature, as well as the *U. dioica* fibre, *G. diversifolia* fibre records a sharp decline of the peak when treated with 2 and 6 mol dm^{-3} NaOH, indicating an increase of amorphous cellulose, which is known to have low thermal resistance and the decrease in cellulose crystallite length [3], [11], [12], [13], [14]. To confirm this, it can be observed as the trend of endothermic peak grows in accordance with the HBI values, at higher concentration of NaOH (Figure 4.5 and 4.6).

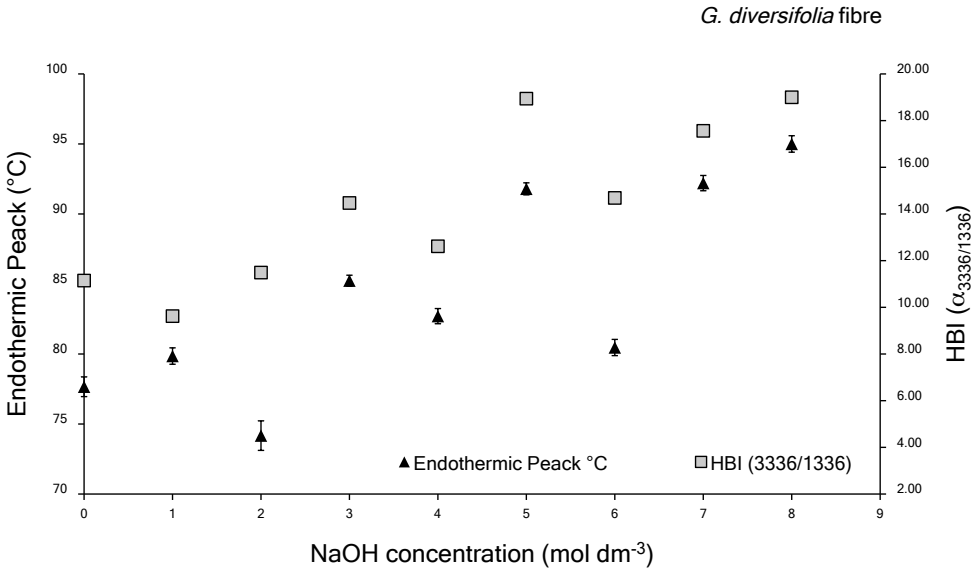

Figure 4.5: Endothermic peak (°C) with SE ± *Vs* HBI ($\alpha_{3336/336}$) of *G. diversifolia* fibre.

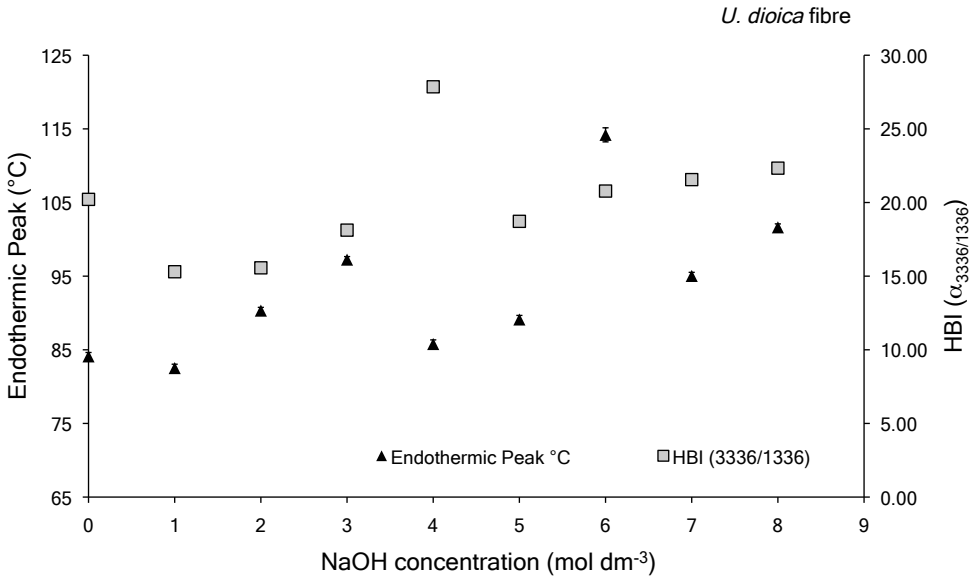

Figure 4.6: Endothermic peak (°C) with SE ± *Vs* HBI ($\alpha_{3336/336}$) of *U. dioica* fibre.

4.3.1.3 Birefringence analysis of NaOH treated nettle fibres

Changes in the molecular structure of the fibre are also shown by the values of ΔH J g^{-1}, peak area obtained with the melting of the polymer, which in *G. diversifolia* fibre has a decreasing trend. In agreement with Goswami *et al.* [15], birefringence (Δn) values and the proportional orientation factor f of *G. diversifolia* fibre decrease with the increase of NaOH concentration. Two major peaks are calculated at 2 and 4 mol dm^{-3} NaOH (Figure 4.7). *U. dioica* Δn values show an abrupt drop between 2 and 6 mol dm^{-3} NaOH (Figure 4.7) and the orientation factor f tends to increase with the concentration of NaOH, recording higher values compared to *G. diversifolia* fibre. Both fibres have Δn values higher than the known value $\Delta n_0 = 0.055$ of the perfectly oriented cellulose, this makes impossible to calculate the orientation factor f, normally used to obtain the molecular orientation of the polymers [16]. From this it is possible, however, to infer that the fibres are more oriented of the normal cellulose and lyocell [15].

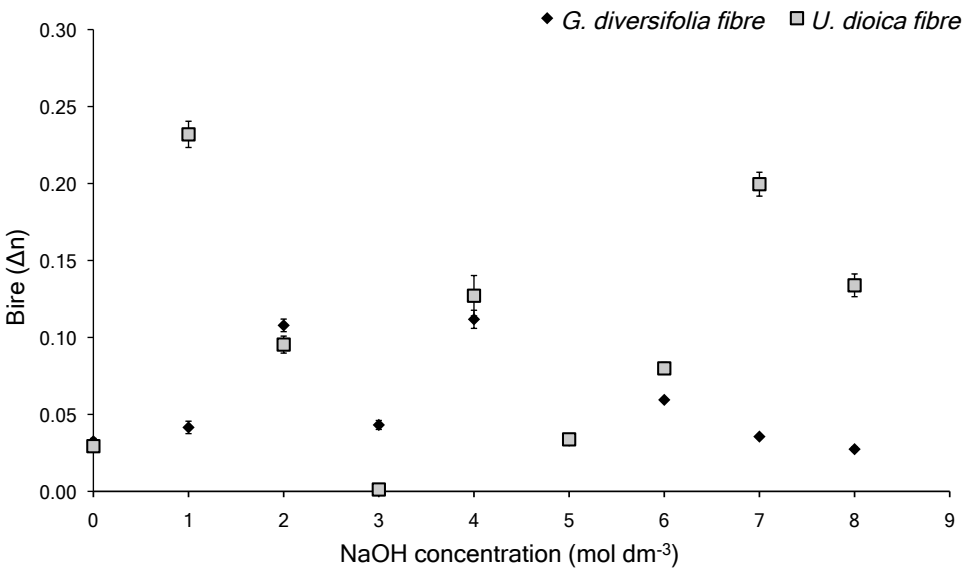

Figure 4.7: Birefringence (Δn) with SE $\pm$ of *G. diversifolia Vs U. dioica* fibres.

4.3.2 Mechanical properties of the fibres in relation to their crystallinity changes

4.3.2.1 Tensile strength, elongation and Young's modulus of *G. diversifolia* and *U. dioica* fibres treated with varying NaOH concentrations

Having analysed the changes in the molecular structure of both nettle fibres, *G. diversifolia* and *U. dioica*, at increased concentrations of NaOH, it is interesting to investigate how these variations relate with the mechanical properties and morphology of the fibres [17]. When the fibres are treated with increased concentrations of NaOH tensile strength values decrease (Table 4.3) compared to the values reported for the raw fibre.

Table 4.3: Tensile strength and elongation values of *G. diversifolia* and *U. dioica* nettle fibres treated with increasing NaOH concentration.

		NaOH mol dm^{-3} concentration								
Fibre	Property	**0**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**
G. diversifolia	Tensile strength (kgf)	**0.12**	**0.17**	**0.21**	**0.19**	**0.15**	**0.15**	**0.13**	**0.09**	**0.14**
	SE ±	0.01	0.01	0.01	0.01	0.00	0.01	0.01	0.01	0.01
	Elongation (%)	**4.39**	**6.21**	**6.47**	**10.64**	**11.86**	**10.61**	**10.09**	**8.05**	**6.95**
	SE ±	0.00	0.00	0.00	0.01	0.01	0.01	0.01	0.00	0.00
U. dioica	Tensile strength (kgf)	**0.05**	**0.06**	**0.04**	**0.04**	**0.04**	**0.04**	**0.05**	**0.04**	**0.04**
	SE ±	0.01	0.01	0.00	0.01	0.01	0.00	0.01	0.00	0.01
	Elongation (%)	**4.29**	**4.93**	**4.47**	**5.94**	**7.31**	**6.89**	**7.61**	**5.72**	**6.32**
	SE ±	0.00	0.00	0.00	0.01	0.01	0.01	0.01	0.01	0.00

The decrease of the strength trend happens for both types of fibres whose results may appear fluctuating due to the fact that the alkaline treatment is removing surface cementing elements as lignin, waxy substances and natural oil that confer fibre resistance [3]. The decrease in tensile is due to molecular changes caused by the conversion from Cellulose I to Cellulose II and the consequent decrease in crystallinity (Figures 4.2 and 4.3) [15]. The *G. diversifolia* tensile trend is quite linear, probably due to the fact that the fibre is more crystalline and chemical in aqueous solution penetrate with difficulty. The *G. diversifolia* fibre continues, as for raw fibres, to retain values higher than those of *U. dioica* fibre. Elongation increases with increasing the NaOH concentration (Table 4.3). The increase in elongation, for both fibres, is proportional to the increase of the HBI (Figure 4.8 and 4.9), explained by the breaking of hydrogen bonds with the penetration of NaOH in aqueous solution. In the change of Cellulose I to II the unit molecular angle changes giving greater flexibility to the fibre [18].

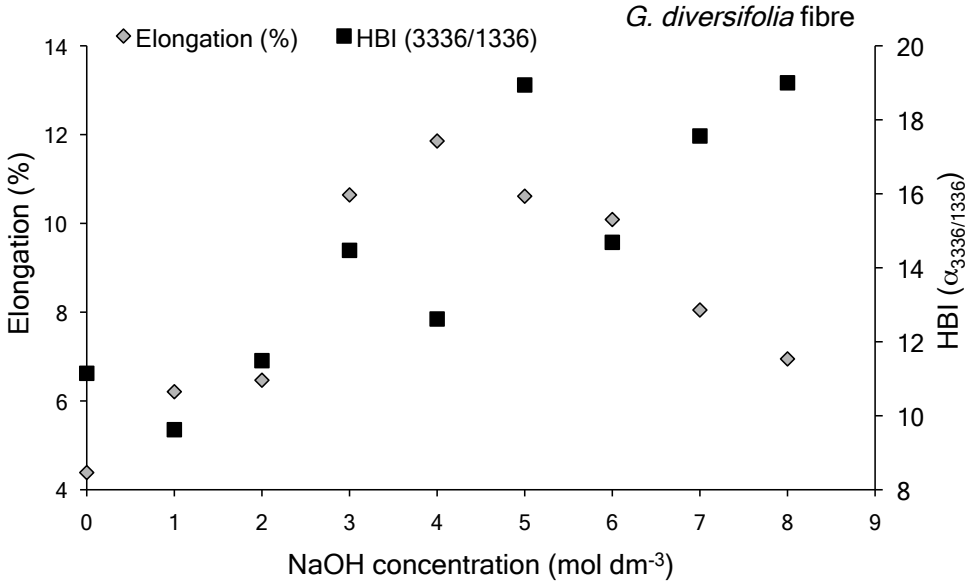

Figure 4.8: Elongation (%) *Vs* HBI ($\alpha_{3336/336}$) of *G. diversifolia* fibre.

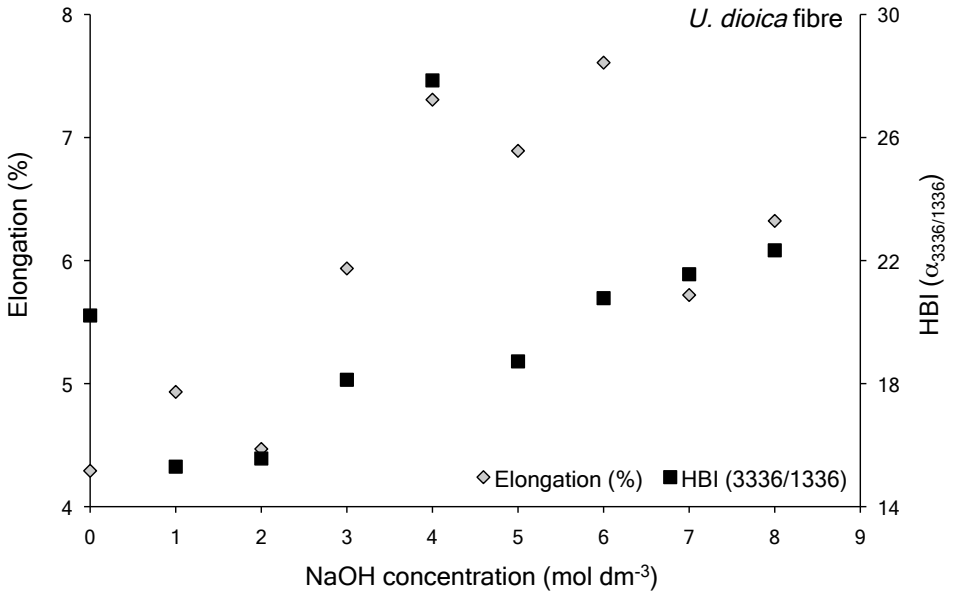

Figure 4.9: Elongation (%) *Vs* HBI ($\alpha_{3336/336}$) of *U. dioica* fibre.

To confirm that the formation of a new molecular structure gives more elasticity to the fibre, Young's modulus was calculated (Table 4.4), as a stiffer fibre will have a higher elastic modulus [19]. Both nettle fibre show a decreasing Young's modulus trend to high concentrations of NaOH. The modulus of elas-

ticity as expected following the trend of the LOI, which at high concentrations of NaOH decreases, indicating a loss of crystallinity (Figure 4.10 and 4.11) and is inverse to HBI which is indicative that a new orientation of the molecular chains is being formed in the conversion of Cellulose I to Cellulose II (Figure 4.12 and 4.13).

Table 4.4: Young's modulus with SE ± of *G. diversifolia* and *U. dioica* fibres.

		NaOH mol dm^{-3} concentration								
Fibre	Property	**0**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**
G. diversifolia	Young's modulus (GPa)	**6.20**	**8.61**	**16.96**	**5.06**	**9.07**	**3.67**	**8.15**	**2.43**	**5.96**
	SE ±	0.43	0.55	0.74	0.35	0.51	0.28	0.63	0.29	0.39
U. dioica	Young's modulus (GPa)	**11.10**	**44.27**	**16.17**	**5.28**	**7.64**	**5.81**	**7.52**	**26.75**	**13.56**
	SE ±	0.25	0.08	0.57	0.00	0.67	0.39	0.34	0.57	0.67

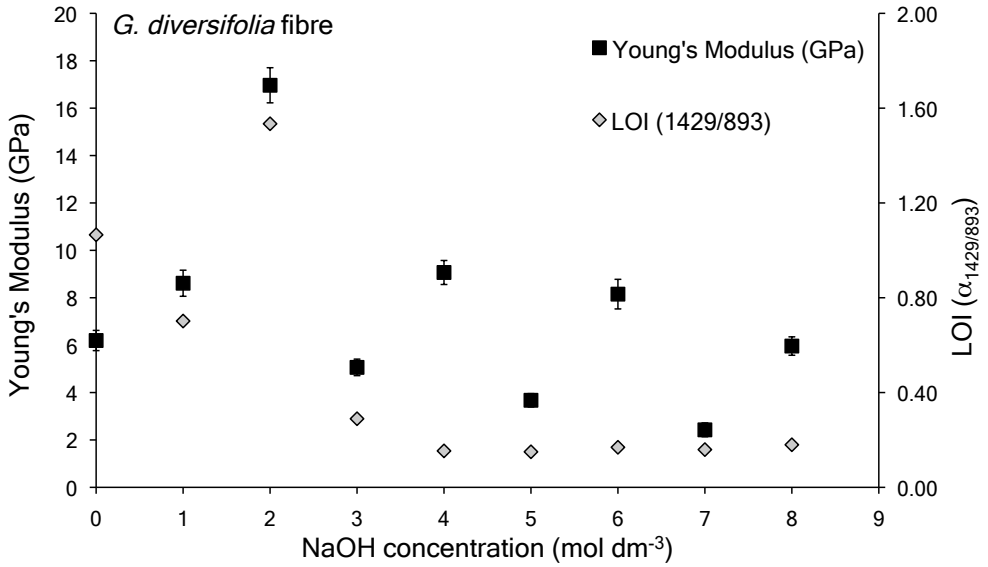

Figure 4.10: Young's modulus *Vs* LOI ($\alpha_{1429/893}$) of *G. diversifolia* fibre.

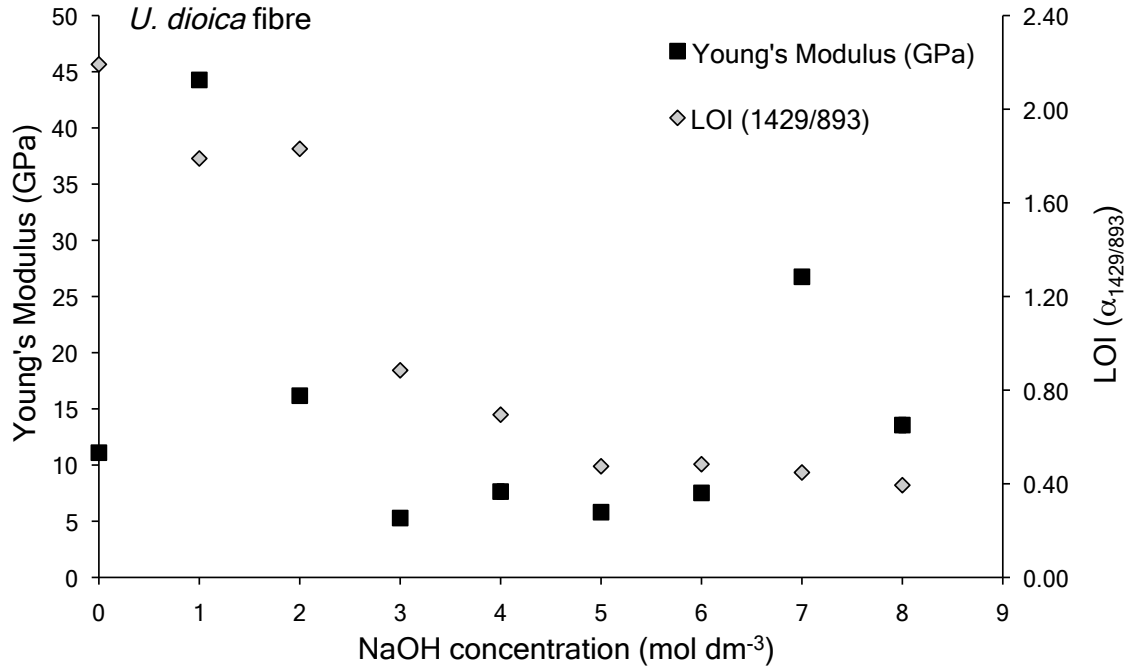

Figure 4.11: Young's modulus *Vs* LOI ($\alpha_{1429/893}$) of *U. dioica* fibre.

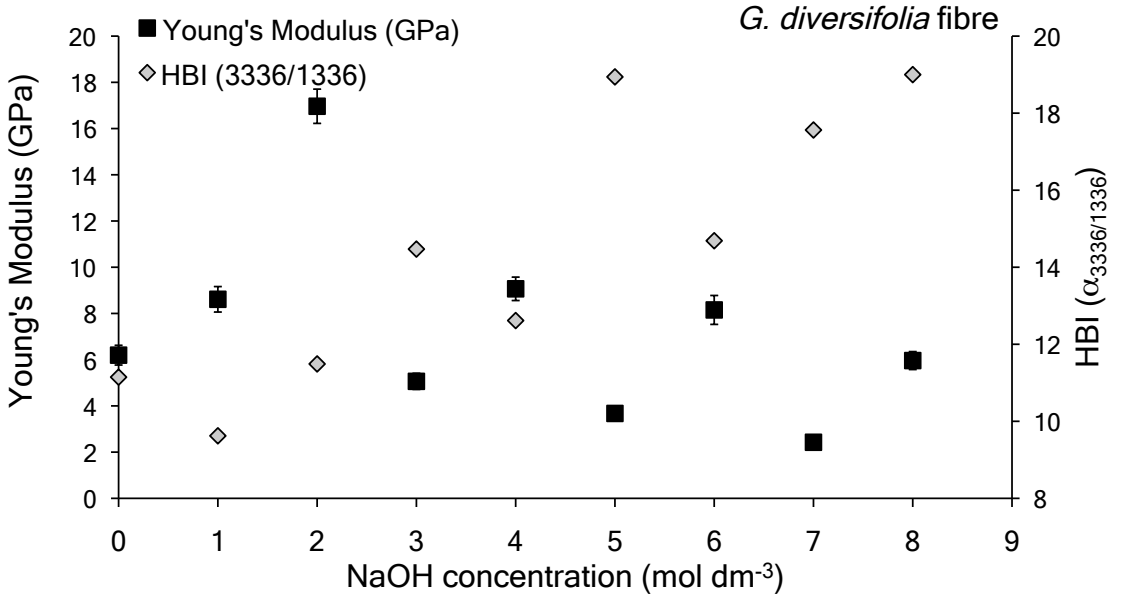

Figure 4.12: Young's modulus *Vs* HBI ($\alpha_{3336/336}$) of *G. diversifolia* fibre.

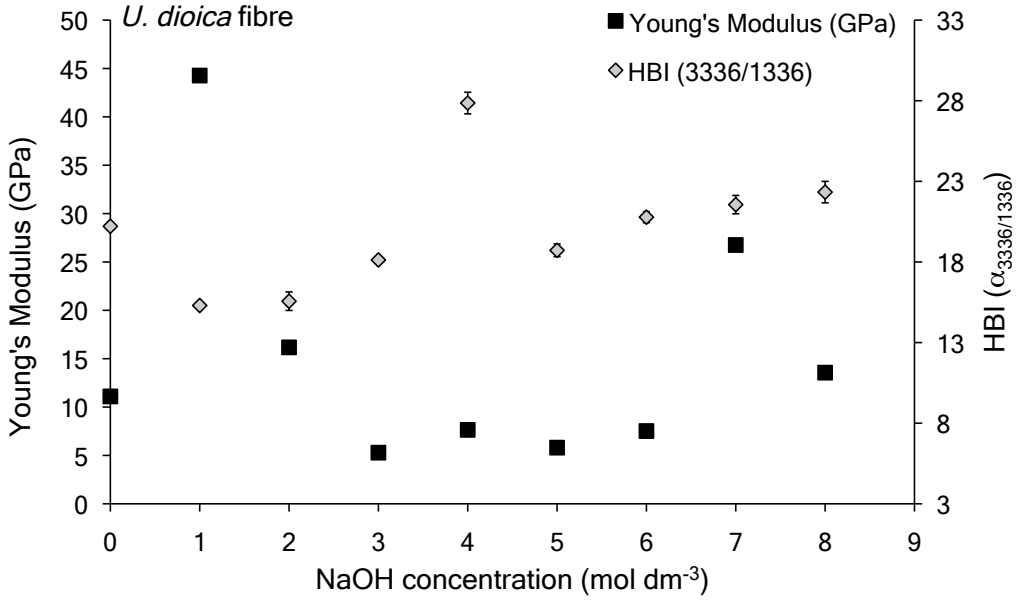

Figure 4.13: Young's modulus *Vs* HBI ($\alpha_{3336/336}$) of *U. dioica* fibre.

4.3.2.2 Moisture regain and moisture content of *G. diversifolia* and *U. dioica* fibres treated with varying concentrations of NaOH

Moisture regain was determined for *G. diversifolia* fibres to be 6.79 % for raw fibre and in a range from a minimum of 5.08 % to a maximum of 7.42 % for fibres treated with increasing NaOH concentration. *U. dioica* has moisture regain values for raw fibre of 8.80 % and in a range between 5.55 % – 7.52 % for NaOH treated fibres (Table 4.5). For both fibres the values are in a range, remaining within 8 %, that ensures their good physical stability [20].

Table 4.5: Moisture regain and moisture content with SE ± of *G. diversifolia* and *U. dioica* fibres.

		NaOH mol dm^{-3} concentration								
Fibre	Property	**0**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**
G. diversifolia	Moisture regain (%)	**5.08**	**4.66**	**5.86**	**5.96**	**7.42**	**5.96**	**5.77**	**5.90**	**6.70**
	SE ±	0.00	0.00	0.01	0.01	0.01	0.01	0.01	0.00	0.01
	Moisture content (%)	**6.30**	**6.44**	**5.97**	**7.40**	**8.95**	**10.19**	**11.06**	**9.77**	**9.80**
	SE ±	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.01	0.00
U. dioica	Moisture regain (%)	**6.17**	**5.55**	**6.00**	**6.93**	**7.17**	**7.27**	**7.52**	**6.55**	**7.26**
	SE ±	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00
	Moisture content (%)	**9.10**	**9.63**	**8.86**	**10.12**	**10.39**	**11.41**	**10.45**	**11.21**	**10.69**
	SE ±	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01

G. diversifolia and *U. dioica* fibres in agreement with their trend of LOI, showing an increase of moisture regain with increasing concentration of NaOH. Moisture regain of *G. diversifolia* fibre at higher NaOH concentration decreases slightly compared to the values calculated for the raw fibre. A peak in moisture regain is recorded at 4 mol dm^{-3} NaOH concentration, where there is evidently an increase of amorphous regions (Figure 4.14 and 4.15). Comparing the fibres, it is possible to deduce that *U. dioica* fibre has higher values of moisture regain, due to a large proportion of amorphous regions, and that the physical properties of the *G. diversifolia* fibre are less influenced by humidity than those of the *U. dioica* fibre (Figure 4.16).

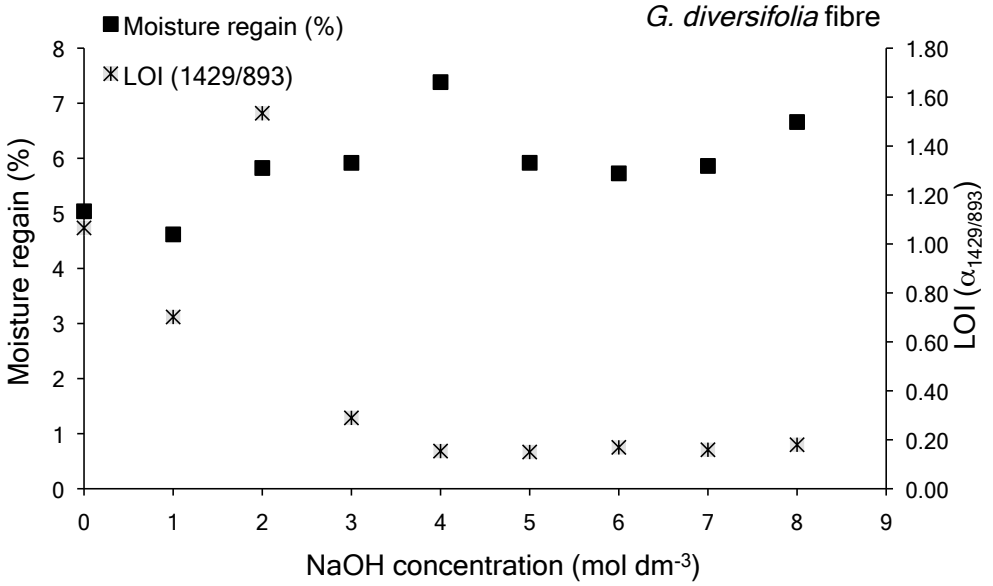

Figure 4.14: Moisture regain (%) *Vs* LOI ($\alpha_{1429/893}$) of *G. diversifolia* fibre.

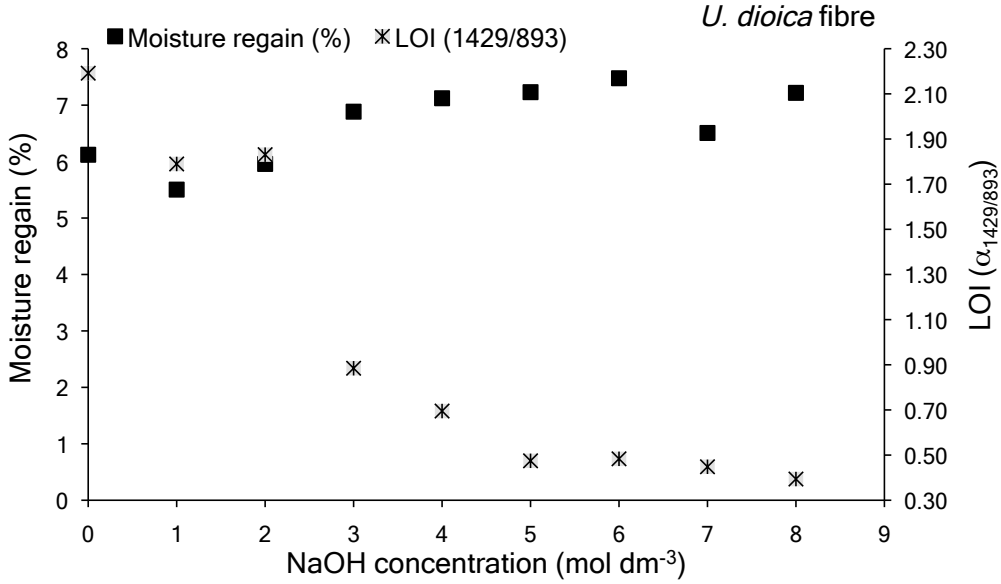

Figure 4.15: Moisture regain (%) *Vs* LOI ($\alpha_{1429/893}$) of *U. dioica* fibre.

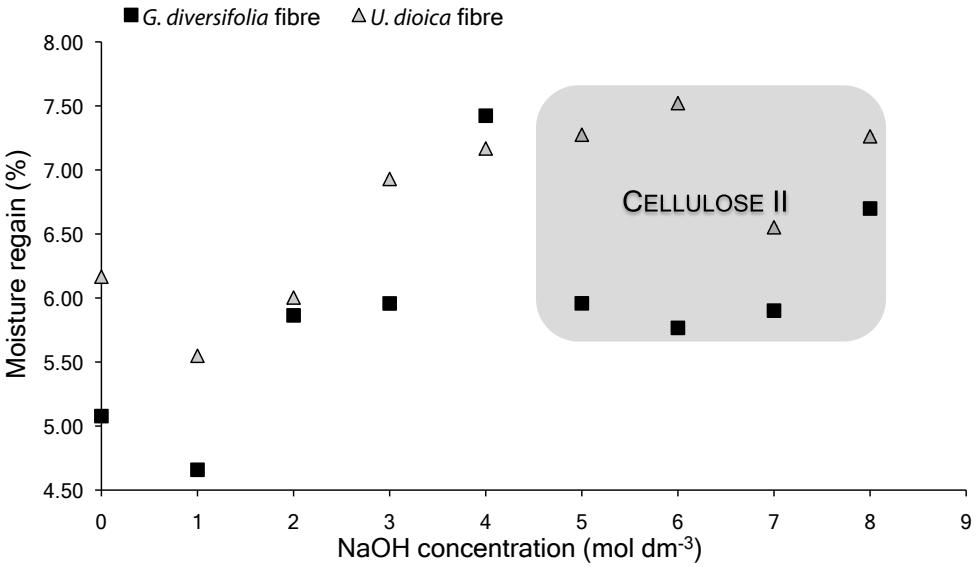

Figure 4.16: Moisture regain of *G. diversifolia Vs U. dioica* fibre.

Moisture content is an internal factor that influences the dielectric properties of the fibres; permittivity increases with increasing moisture content [21], and content lower than 10 % does not cause significant variation in the dielectric properties [22]. It is observed that the percentage of *G. diversifolia* fibre moisture content is 6.00 % for raw fibre and 9.80 % at 8 mol dm^{-3} NaOH. The maximum peak, 11.06 %, has been calculated at 6 mol dm^{-3} NaOH and the minimum, 5.97 %, at 2 mol dm^{-3} NaOH. It is possible to hypothesise a good electrical insulating capacity of the fibre in question (Table 4.5). The values of moisture content of the *U. dioica* fibre are slightly higher but still close to the value of 10 %: raw fibre 9.40 %; 10.69 % at 8 mol dm^{-3} NaOH concentration; the highest value, 11.41 %, at 5 mol dm^{-3} NaOH; the lowest value, 8.86 %, at 2 mol dm^{-3} NaOH (Table 4.5).

4.3.3 Morphological changes in the fibre cross – section of *G. diversifolia* and *U. dioica* fibres treated with varying NaOH concentration

Cellulose and alkaline solutions interact causing a variation in the fibre cross – section (Table 4.6) [23]. Factors such as the temperature and NaOH concentration cause the fibre swelling. *G. diversifolia* increases its cross – section and lumen at 5 mol dm^{-3} NaOH and *U. dioica* at 3 mol dm^{-3} NaOH. In both cases the fibres remain swollen when the cellulose is under conversion from Cellulose I to II. In this phase, the fibres do not have a regular structure being the cellulose chains not re – oriented in a new order as the concentration of NaOH is not enough to penetrate up to break intramolecular hydrogen bonds within individual cellulose chains. The morphology of the cross – sections for both *G. diversifolia* and *U. dioica* fibres changes to a more regular circular cross – section following alkaline treatment (Figures 4.17 and 4.18) [20], [24], [25]. In *U. dioica* fibres the change is clearly visible (Figures 4.19 and 4.20): the section and the lumen sizes at 8 mol dm^{-3} NaOH concentration (actual section area: 378 μm^2; lumen: 2 μm^2) is slightly narrower than at 0 mol dm^{-3} (actual section area: 1441 μm^2; lumen: 40 μm^2). *G. diversifolia* fibre at 8 mol dm^{-3} NaOH undergoes narrowing of the lumen (actual section area: 2338 μm^2; lumen: 85 μm^2) compared to the fibre at 0 mol dm^{-3} (actual section area: 4107 μm^2; lumen: 430 μm^2) (Figures 4.17 and 4.18).

Table 4.6: Cross – sectional area (A) (μm^2) of *G. diversifolia* and *U. dioica* fibres.

		NaOH mol dm^{-3} concentration								
Fibre	Property	**0**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**
G. diversifolia	A total (μm^2)	**4537**	**3035**	**1803**	**4003**	**1379**	**4315**	**2405**	**4585**	**2323**
	CV %	0.5	0.6	0.1	0.0	1.2	0.9	6.1	0.3	2.2
	A lumen (μm^2)	**430**	**208**	**186**	**34**	**25**	**103**	**17**	**60**	**85**
	CV %	0.2	11.1	0.0	0.0	0.8	1.0	49.1	49.7	32.1
	A actual (μm^2)	**4107**	**2797**	**1617**	**3969**	**1354**	**4212**	**2388**	**4525**	**2238**
	CV %	0.6	1.3	0.2	0.0	1.2	0.9	6.5	0.3	0.9
U. dioica	A total (μm^2)	**1481**	**363**	**631**	**1922**	**1097**	**1366**	**653**	**205**	**379**
	CV %	0.4	1.9	0.1	5.3	0.1	3.0	0.9	0.4	1.1
	A lumen (μm^2)	**40**	**5**	**15**	**42**	**10**	**54**	**8**	**5**	**2**
	CV %	5.2	71.1	56.4	1.2	9.0	6.4	41.0	100.0	1.7
	A actual (μm^2)	**1441**	**358**	**616**	**1880**	**1323**	**1323**	**645**	**200**	**378**
	CV %	0.2	0.9	1.3	5.5	0.2	2.8	1.4	2.8	1.1

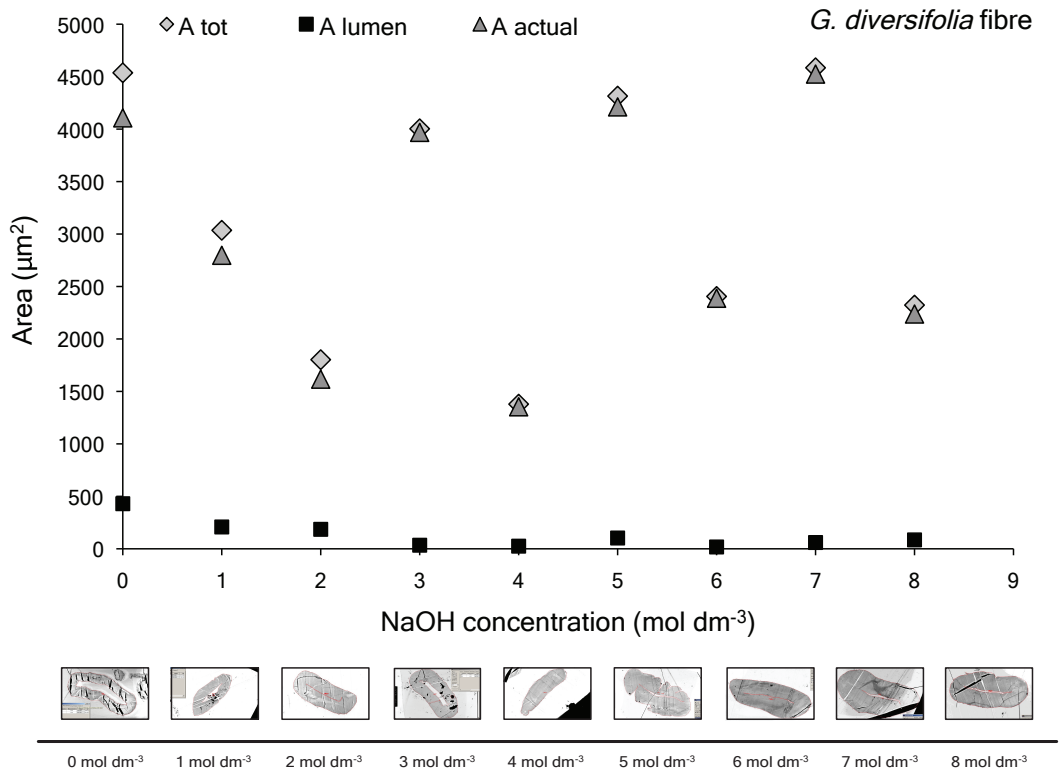

Figure 4.17: Cross – section area (μm^2) (total – lumen – actual) with CV % of *G. diversifolia* fibre treated with NaOH. SEM photographs 400 – 700 x magnification.

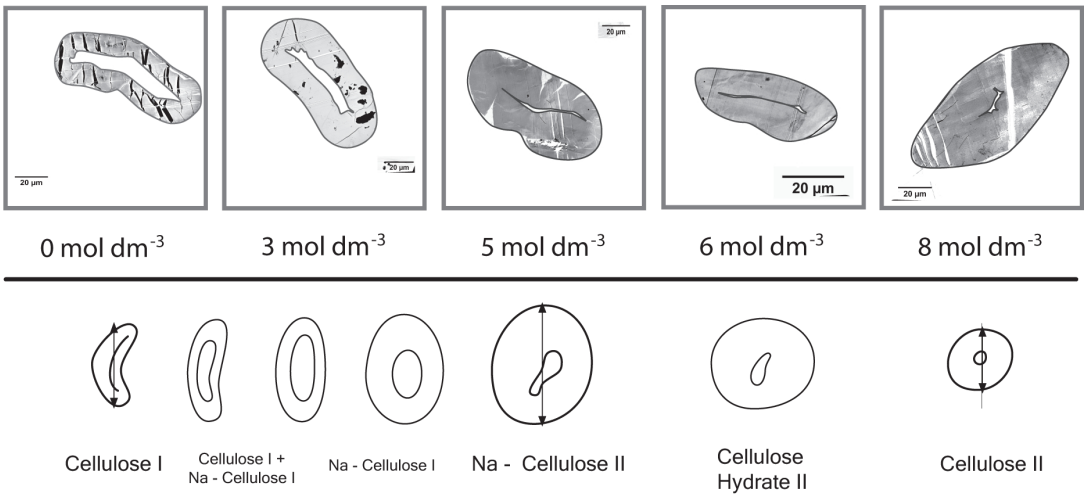

Figure 4.18: Variation of cross – section during the reaction of cellulose with different NaOH concentrations for G. diversifolia fibres.

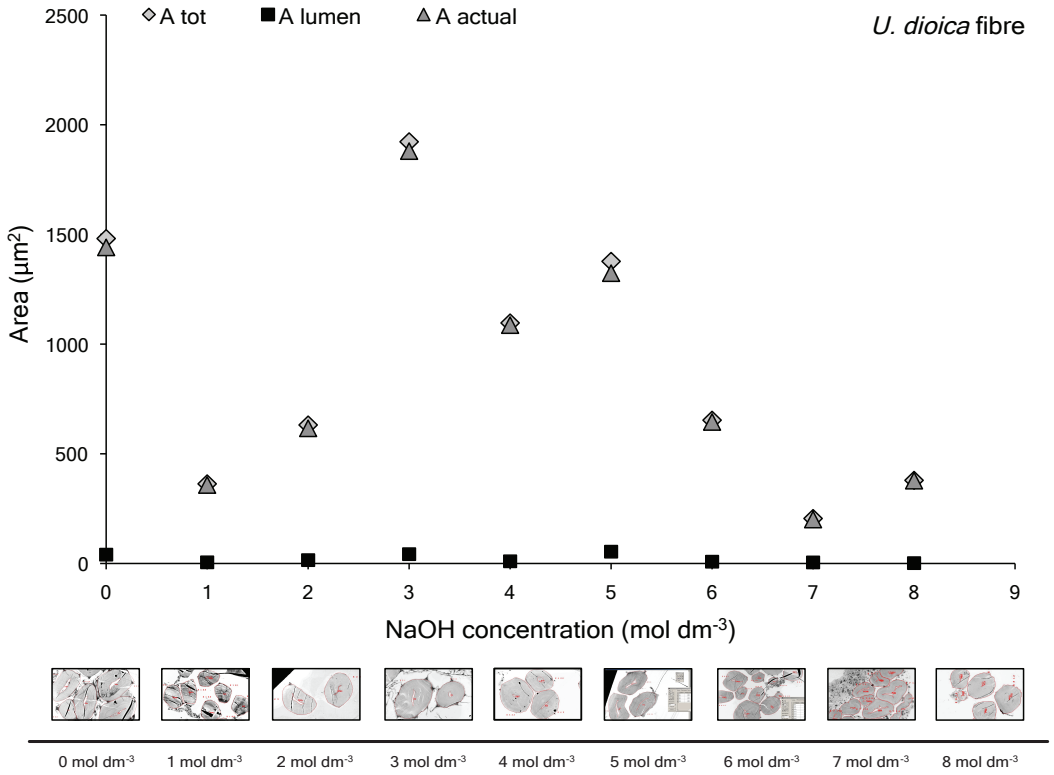

Figure 4.19: Cross – section area (μm^2) (total – lumen – actual) with CV % of *U. dioica* fibre treated with NaOH. SEM photographs 400 – 700 x magnification.

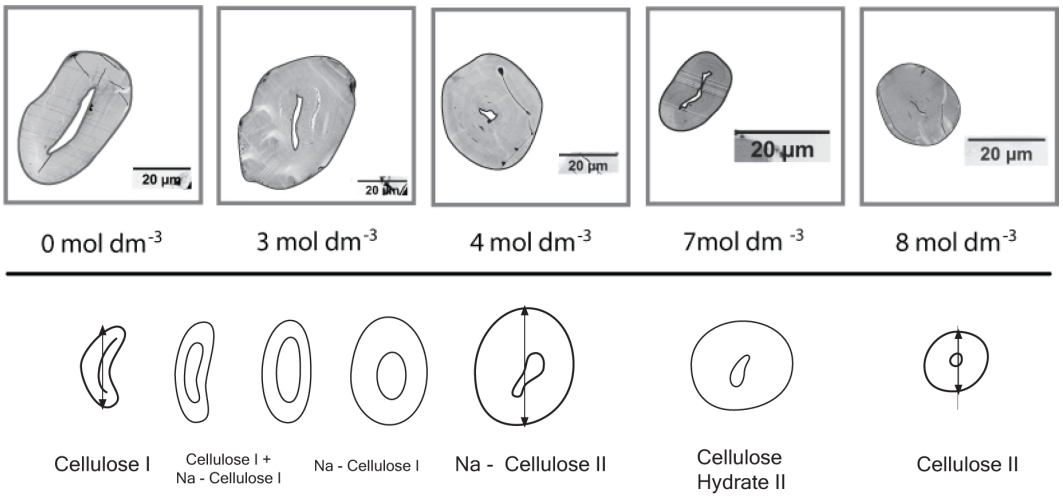

Figure 4.20: Variation of cross – section during the reaction of cellulose with different NaOH concentrations for U. dioica fibres.

4.4 Conclusion

The fibres treated with increasing concentration of NaOH reduce their degree of crystallinity. High NaOH concentrations cause the transformation from Cellulose I to Cellulose II. During this process the values of HBI increase and consequently elongation of the fibre, while LOI decreases (Figures 4.2 and 4.2). In the case of *U. dioica* fibre the data obtained show that it is more affected by alkaline treatments increasing its TCI at higher concentration of NaOH (Figure 4.4). The transformation of Cellulose I to Cellulose II occurs between 2 and 4 mol dm^{-3} NaOH when a significant LOI reduction and appreciable changes in HBI take place. The complete transformation into Cellulose II takes place above 5 mol dm^{-3} NaOH, where no further crystallinity change was observed.

The endothermic peak trend, detected by DSC analysis, grows in accordance with the HBI values at higher concentration of NaOH. This test confirms the loss in cellulose crystallite length as at 6 mol dm^{-3} and 4 mol dm^{-3} NaOH, respectively for *G. diversifolia* and *U. dioica* (Figures 4.5 and 4.6), low thermal resistance is recorded indicating an increase of amorphous cellulose. The results also demonstrate the good thermal resistance of the fibres, having decomposition peaks, in the range of 360 – 380 °C, higher than the most common bast fibres.

Both fibres are very oriented, generally more then the perfectly oriented cellulose. *U. dioica* has an orientation factor f greater than that of *G. diversifolia* fibre.

With regards to the mechanical properties of the nettle fibres and how the alkaline treatment influences them, it is possible to note that at increased concentrations of NaOH tensile strength values decrease compared to the values reported for the raw fibre (Tables 3.5 and 4.3), due to the decrease in crystallinity. *G. diversifolia* has values very close to raw fibre and a linear trend which indicates that the fibre is highly crystalline and consequently NaOH acts

with more difficulty. Contrary to tensile strength, the elongation increases at high NaOH concentration according to the HBI trend and to the unit molecular angle that changing in the transformation from Cellulose I to II, confers greater flexibility to the fibre (Figures 4.8 and 4.9). The Young's modulus analysis remarks the fact that with increasing concentrations of NaOH the fibres lose crystallinity, in fact, the trend of the elastic modulus follows the LOI trend (Figures 4.10 and 4.11) and is inverse to that of HBI (Figures 4.12 and 4.13) and therefore to elongation, reducing the fibre stiffness.

The values of moisture regain are proportional to the percentage of the amorphous region present in the fibre. In the case of *G. diversifolia* and *U. dioica* moisture regain increases with NaOH concentrations showing an opposite trend to that of LOI (Figures 4.14 and 4.15). The percentages of moisture regain of both nettle fibres are in a range that ensures a good physical stability to adapt to environment humidity. By the comparison of the fibres it is deducted that *G. diversifolia* fibre is less influenced by humidity than *U. dioica* fibre (Figure 4.16) The low moisture content of *G. diversifolia* fibre confers good electric insulating capacity.

With the alkaline treatments, it is possible to give the fibre a more regular shape of the section. *G. diversifolia* increases its cross – section and lumen at 5 mol dm^{-3} NaOH and *U. dioica* at 3 mol dm^{-3} NaOH when the Cellulose conversion from I to II is ongoing (Figures 4.18 and 4.20).

One of the most promising results of *G. diversifolia* fibre is its versatility: treating the fibre with different concentration of sodium hydroxide it reacts with characteristics potentially spendable in different fields of the high performance activities where a combination of waterproofness, breathability and resistant to abrasion is required. *G. diversifolia* fibre is presented as a very promising sustainable candidate for this niche of textile and deserves further investigation.

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Chapter 5

Application of *Girardinia diversifolia* fibre

5.1 Introduction and background

In the first part of this work, fundamental research was done to characterise the properties (*e. g.* the fibre morphology and mechanical properties) of Himalayan nettle fibre (*G. diversifolia*) after being treated with different concentrations of sodium hydroxide solution (n = 0 – 8 mol dm^{-3} NaOH). The aim of this work is to perform a preliminary analysis of the capabilities and possible applications of the Himalayan nettle fibre subjected to alkali pre – treatments.

5.1.1 Comfort in outdoor sports

The apparel market is highly competitive and evolved to anticipate and satisfy the needs of the consumers [1]. Synthetic fibres have evolved to impose their supremacy in the sportswear market in terms of their comfort performance and have demonstrated their convenience to the fashion industry [1]. However natural fibres such as Himalayan nettle fibre have several other advantages (*e. g.* hypoallergenic and sustainable) and one cornerstone of this research is to benchmark the properties of Himalayan nettle fibre against the requirements of

sportswear material.

The sports industry is in continuous search for high – performance fabrics as consumers' demand high level of comfort [1]. The mechanical and thermal sensations are amplified by dynamic interaction with the body during any physical activity [2] and therefore, for thermo – physiological comfort, a crucial factor in sports is to keep the body temperature and the moisture close to normal levels of the body [1], [3]. The fibres used in high – performance sports textiles must have different functionalities to meet various needs. This is why the sport garments and accessories are often a combination of several materials with different specifications. Basic essential characteristics are [2]:

- Mechanical: tensile strength, elongation at break, tensile modulus;
- Chemical: glass transition temperature, melting point, heat stability, specific electrical resistance, resistance to humidity;
- Surface characteristics: hydrophilic or hydrophobic, wettability, softness, *etc.*;
- Geometry: diameter, hollow fibre;
- Fibre length.

Since the 90's, the clothing sector has become very competitive. A crucial factor to ensure the success and survival of a business is the ability to meet, and even predict the demands and needs of consumers [1], [2]. Synthetic fibres are very successful in the sportswear sector due to their thermal and movement comfort [1]. Comfort is a concept that relates with wellbeing and living conditions. Indeed, a focus of textile research in recent years has been to improve the conditions around our body for better well – being, for example, research in physical and psychological comfort. Textile companies make clothing comfort innovation as this ensures the survival and competitiveness of a company with strong economic repercussions [1].

For physical comfort, clothing represents an interface between the environment and the body; clothing consists of a protective layer or multiple layers

that ensures a condition suitable for the survival of the body by creating a proper thermal state. Clothing also protects from external agents such as abrasions, radiation, wind, water, electricity, chemicals and microbiological toxic substances [1].

Keeping the human body warm and dry is one of the fundamental conditions that occur in a state of comfort. The importance of this function is especially visible in apparel for cold weather sports [4]. The main function of winter sportswear is to ensure a *comfort zone* where temperature, humidity and air circulation creates an optimal microclimate. This microclimate is not altered by changes in the condition of the body (at rest or under stress), and changes in weather and external temperature. Therefore the clothes must ensure a rapid adjustment of the microclimate and adaptation to climatic conditions through a good transport of heat, humidity and air flow (Figure 5.1) [4]. The performance of these functions can provide a sense of mental and physical comfort [5], [6]. For a long time, the natural fibres such as wool and cotton have performed functions of absorbency, breathability and thermal insulation while the synthetic fibres met the requirements of resistance. Natural fibres are generally hydrophilic and hygroscopic, *i. e.* able to absorb water and retain it; whereas the synthetic ones are generally not water absorbent and do not alter their physical characteristics in a wet state. Combination of natural and synthetic fibres has created a clothing system that ensures comfort [7], [8].

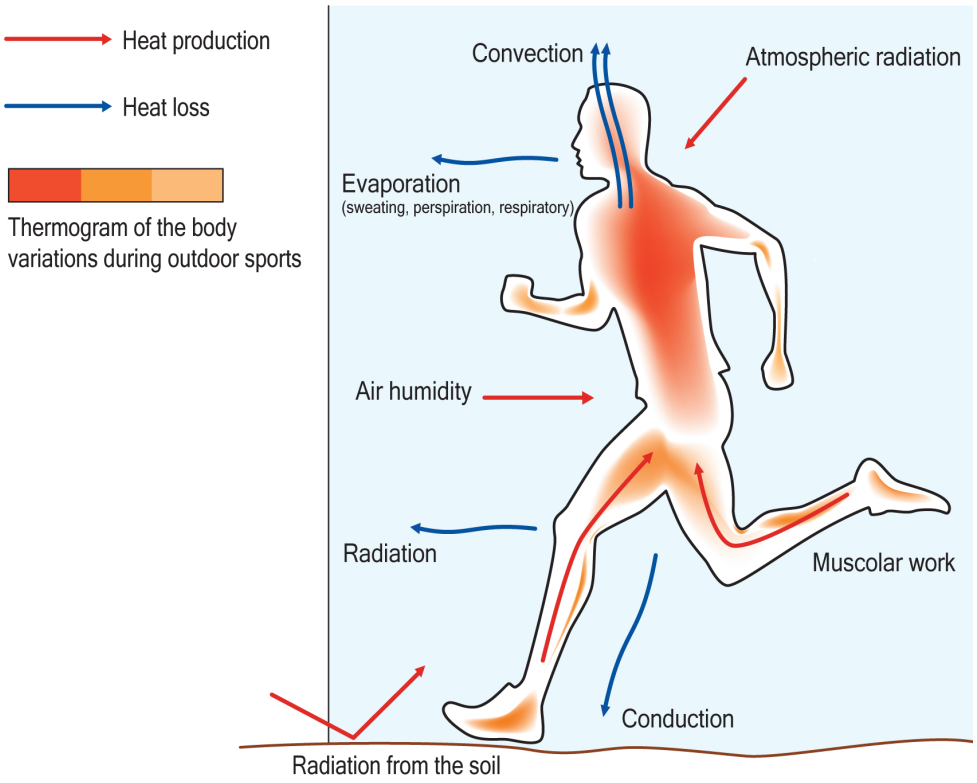

Figure 5.1: Thermogram of the body variations during outdoor sports showing the heat production and the heat loss [1], [2], [4], [9], [10].

5.1.2 Three layer outdoor clothing system

For standard *Protective clothing* against cold [11] the most important property that a garment must have is to protect the wearer from the cold. Cold could be defined as an environment which is at a lower temperature as characterised by the combination of humidity and wind [11]. The fabric property, including its performance as a component in a complex multi – layered clothing, should be considered. A garment that provides breathability and ventilation could be considered more effective than a 'closed garment' in which the humidity and heat caused by physical activity is trapped (Figure 5.2). Low water vapour resistance is a desirable property in a garment for comfort [1], [4], [11].

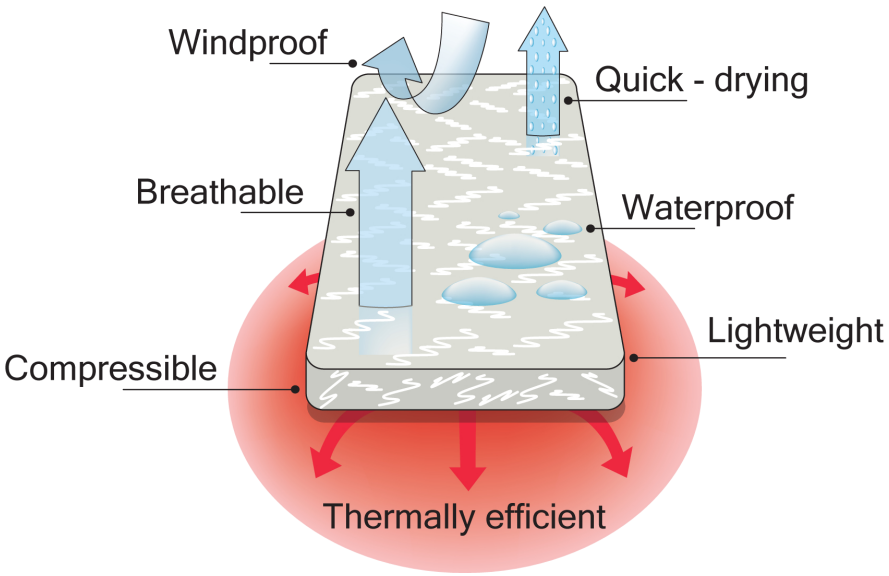

Figure 5.2: Thermal performance of the fibre technology in extreme conditions as in Primaloft®jacket [12].

A layered system, either by a single component or by multiple components, is proven to be the best way to ensure a state of comfort in outdoor sports. It allows a fast adjustment to weather conditions and to the physical changes. Generally, the outdoor sportswear is built on three layers [4]:

- **The base layer** next to the skin, breathable and moisture regulator [4], [13];
- **The middle layer**, insulating, it protects from the cold [4];
- **The shell layer**, the outer, resistant to abrasion and it protects from external environment, wind and rain [1], [4].

The base layer (in contact with the skin) must be able to regulate the body temperature by conducting away the moisture during perspiration [4]. It should be able to keep the body dry and cool in the summer and avoid hypothermia in the winter [2].

Vapour and heat are removed from the body through the convective heat or due to the clothing worn. The vapour passes through the system of tissues to reach the outermost surface of the garment and evaporates unless there

is an impermeable layer that creates condensation [14]. This situation could be avoided by ensuring that all the layers in the clothing have an adequate breathability [4]. The layer next to the skin has to remove the moisture and it must not be absorbent. The breathable part of the clothing must be composed of hydrophobic fibres (moisture repellent), like merino wool, polyethylene terephthalate, propylene etc. Materials such as cotton retain moisture and therefore create a feeling of cold body [13], [15], [16].

The base layer has to leave the body dry quickly [2], [4]. The base layer fabric should be breathable to perform its primary function of temperature control and freeze prevention [4], [5]. It should also be flexible and soft for a good fit, and strong and elastic to resist friction during physical activity [13], [15], [17], [18], [19], [20], [21].

The function of the middle layer is to maintain the body heat and isolate it from the outside temperature changes. Trapping air it acts as an insulator [4]. The best – known textile insulating materials are merino wool, which performs this function even if it is wet and goose down which only works if it is kept dry [15]. The goose down is an excellent insulator especially in a dry and cold climate [4]. It has excellent warmth to weight ratio and an ability to be compressed better than all the other textile materials. Other fibres that have good insulating performance are synthetic fibres such as polyethylene terephthalate (PET). PET fabrics are lightweight, breathable and efficient even when wet and have a warmth to weight ratio better than fibres such as wool [4]. In general, the insulating layer is composed of hydrophobic fibres and protected by an additional cover layer that should be water – repellent and breathable [4], [15]. There is a difference between 'water – repellent' and 'waterproof' fabrics. Waterproof fabrics are manufactured, for example, by coating the fabric with rubber [20]. Such a treated fabric will not be breathable and will be uncomfortable to wear. A water – repellent finish remains permeable to air and is achieved by the application of hydrophobic chemicals to the fabric [4], [11], [20], [22]. The best performing middle layer is a material called Gore –

Tex membrane patented by W. Gore & Associates [21], [22], [23]. The Gore – Tex fabric is simultaneously waterproof and highly breathable. It is made from polytetrafluoroethylene (PTFE) micro – porous material expanded thermo mechanically [21], [23].

The outer (shell) layer acts as a protective layer and its function is to shield the inner layers from rain and wind [4]. Ideally this layer should be water repellent and breathable. The breathable functionality prevents condensation in the inner layers. The outer layer is treated with a water repellent finish and this layer could be laminated with the inner layer to eliminate friction [4]. Fabrics made out of nylon is normally used as water – resistant and breathable shell fabric; nylon is lighter and stronger than PET. Polyurethane (PU) coating is often used to manufacture waterproof and windproof garments [4], [15], [22].

The breathability property of outdoor sportswear garments are considered more important than the waterproof property [1], [2]. The important design feature is to produce lightweight comfortable (breathable) garments and this is achieved by innovative middle layer technologies (*e. g.* Gore – Tex) [2], [23]. The shell, depending on the sport activity, must meet several functions; elasticity, protection from the wind, a required degree of thermal insulation, and water repellency [2]. A preferred material for the shell is fleece with good thermal insulation and with a water – repellent surface finish. The inner (base) layer should be soft and must have the ability to transfer the perspiration outwards [1], [4]. Tight clothing improves thermal insulation and has a reduced bulk to improve the ease of movement (Table 5.1) [2].

Research has shown that the most important element to ensure comfort for the sports clothing in cold weather, is the utilisation of trapped air; trapped air is an excellent thermal insulator [2], [24], [25]. The trapped air is essential to ensure thermal regulation and insulation, it could be considered as the main element to create lightweight garments [4], [26]. Novel sportswear designs utilises fabrics that can trap air and minimise the contact between the skin and the garment [2], [4], [6]. Fabrics such as the sports knits: piqué honey-

comb or raised ribbed textures, allows the body to take advantage of its natural thermoregulation [24], [25]. The morphology of the hollow natural fibres are of particular relevance in this regard [24], [25]; the air trapped in the hollow core of the fibres could provide thermal insulation [5], [23].

Table 5.1: The evolution of the three layers system: soft shell [2].

Extreme condition: three layers garment system	**Normal condition: soft shell**
Base Layer → wicking (knit)	**Base Layer** → wicking (knit)
Middle Layer → warmth (fleece)	**Middle Layer** → elasticity, wind protection, breathability, water resistance (soft shell)
Shell Layer → waterproofness, windproofness, breathability (membrane)	**Shell Layer** → waterproofness (hard shell)
Waterproofness + breathability = **comfort**	Wearability + breathability + Wind protection + water resistance = **Optimum comfort in action**

5.1.3 Technical analysis of the three layer properties

To design outdoor sportswear the following characteristics must be considered [2]:

- Breathability, to prevent the accumulation of moisture and chilling;
- The thermal insulation, essential to maintain a thermal balance;
- Wearability, crucial for sports performance.

The inner layer controls the humidity and is in direct contact with the skin [2]. Direct contact with perspiration during any physical activity could

make this layer wet [2], [13]. The humidity is absorbed into and from the fabric by diffusion, *i. e.* the water vapour molecules move through the fabric to the outer surface from which the vapour dissipates into the air by desorption [2], [16], [27], [28]. The structure of the fabric and the fibre type influences this phenomenon [2], [27]. The synthetic fibres, through capillary action, allow water to move away from the skin to the garments' outer surface. The voids in the fabric structure acts as tubes; narrower the tubes the more effective is the capillary action (Figure 5.3) [28]. Microfibres that are effective in absorbing moisture allows a faster drying rate [29].

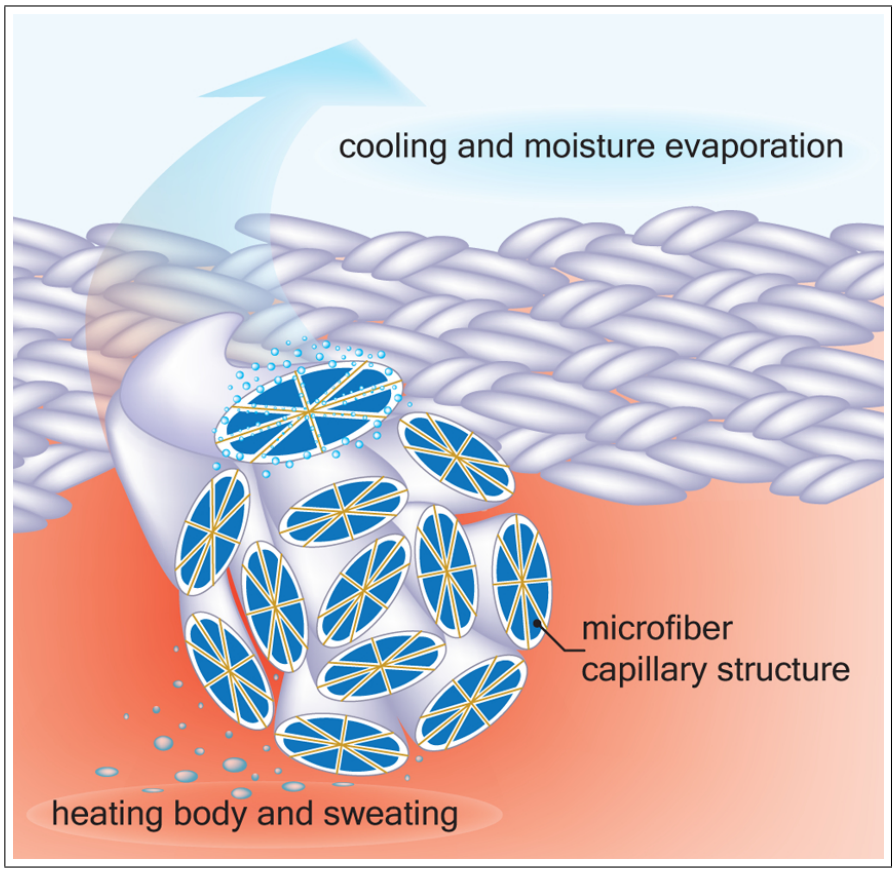

Figure 5.3: Capillary system of microfibre for absorbency and moisture evaporation [28].

Natural hygroscopic fibres absorb the perspiration and carry it into the fibre by swelling [15], [27], [30]. Fick's law (Equation 5.1) regulates this transfer of steam in stationary conditions [31].

$$F = -D\frac{\partial C}{\partial x} \tag{5.1}$$

where F is the rate of moisture flux (in mol / m^2·s), C is the concentration of diffusing substance (in mol / m^3), D is the diffusion coefficient or mass diffusivity of the component (in m^2/s), and x is the space coordinate measured in normal to the section of the medium (in m).

It states that the ratio of water vapour transfer from the inner to the outer side is proportional to the difference in vapour pressure between the two faces of the fabric [2], [30], [32], [33], [34]. The physical and environmental conditions where a breathable fabric works could be very different. Second Fick's law describes the concentration changes due to the diffusion over time [13]. In this equation (Equation 5.2), variables added are time (t) and the elements of space coordinate measured (x = thickness, y and z = unit area that are considered as one – dimensional diffusion; Figure 5.4).

$$\frac{\partial C}{\partial t} = D\left(\frac{\partial^2 C}{\partial x^2} + \frac{\partial^2 C}{\partial y^2} + \frac{\partial^2 C}{\partial z^2}\right) = D\frac{\partial^2 C}{\partial x^2} \tag{5.2}$$

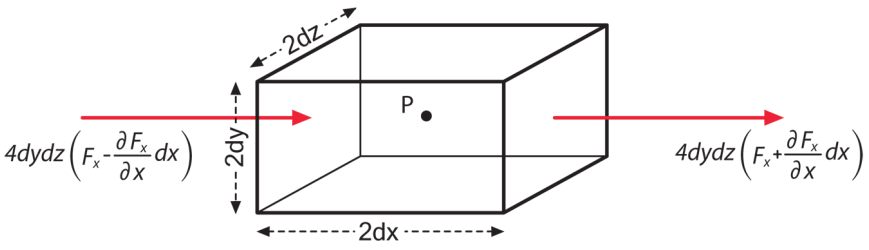

Figure 5.4: Change in diffusion volume as a factor of time in non – steady state diffusion. C is the concentration in the point P, which indicates the centre of the volume [2], [35].

The porosity of the material and the ability of the fibre to release water vapour define the ratio of water vapour diffusion through a fabric [31], [36].

The hydrophilic fibres have an initial rapid absorption of moisture governed by Fick's law. The absorption causes fibre swelling and thereby reduces the porosity of the fabric. This causes a decrease of humidity diffusion in the fabric by slowing down its absorption. This phenomenon of decreasing the absorption is referred to as non – Fick's law [13], [16], [29], [31], [37].

The diffusion of water vapour through a material determines its breathability and could be analysed as water vapour permeability [14], [16]. The methods for testing water vapour permeability are aimed to have a complete vision of the phenomenon of breathability. They differ in the conditions to which the samples are subjected to and the equipment used for testing [37], [38], [39].

The fabric permeability can be measured according to two methodologies [38], [39], [40]:

- Gravimetric methods;
- Sweating hot plate methods.

The gravimetric method includes 'cup method' and 'dish method', and can be performed using desiccant material or water, in normal or inverted position; the inverted position is recommended for fabrics with a water repellent finish [39]. This type of test is widely used by researchers as it does not require a complex procedure and is not expensive. It describes a comfort state that occurs in low physical activities where excessive sweating is not involved [39]. This methodology has the limitation of not providing information regarding the condensation that occurs on the fabric surface. Furthermore, the results obtained are influenced by the air that is trapped between the sample and the medium used for testing *e. g.* water [39]. Water vapour permeability (WVP) is calculated using Equation 5.3 [37], [38], [39]:

$$WVP = \frac{24M}{At} \tag{5.3}$$

The WVP is measured in g/m^2/day, as reported in BS 7209 [41]: M the mass loss (in g) of the assembly over time period t; t the time between successive weighing of the assembly (in h); A the area of the exposed test fabric (equal to the internal area of the test dish) (in m^2).

A is obtained by using Equation 5.4:

$$A = \frac{\pi d^2}{4} \times 10^{-6} \tag{5.4}$$

where d is the inner diameter of the test dish (in mm).

The method 'sweating hot plate' is the new generation of the gravimetric method and is widely used by researchers and manufacturers. It places the fabric in close contact with a wet plate and then the amount of water vapour eliminated from the garment is measured. The values of water – vapour resistance R_{et} (in m^2·Pa/W) could be obtained using Equation 5.5 [41].

$$R_{et} = \Delta p \cdot \frac{A}{H - \Delta H_e} \tag{5.5}$$

where Δp is the difference of partial pressure between two sides of specimen (in Pa); A is the area of the measuring unit (plate) (in m^2); H is the heating power supplied to the measuring unit (plate) (in W); ΔH_e is the correction term (in W).

It is possible to calculate water – vapour permeability index, i_{mt}, by using Equation 5.6, which is dimensionless with values between *0*, when a material is totally impermeable to water – vapour, and *1*, when it has the same thermal resistance and water – vapour resistance of an air layer with equal thickness [40], [41], [42].

$$i_{mt} = S \cdot \frac{R_{ct}}{R_{et}} \tag{5.6}$$

where $S = 60$ Pa/K; R_{ct} is the thermal resistance of the sample, which will be defined below.

The sweating hot plate method has practical disadvantages due to the high cost, large sample size (300 X 300 mm), and time required to test. Equipment was therefore developed to simulate the phenomenon of body perspiration: PERMETEST [39], [43].

This tool can work on various fabrics simulating the human skin and detecting the heat flow. Relative Water Vapour Permeability, $RWVP$ is obtained in the steady state isothermal condition. $RWVP$ is calculated as a percentage

> 'and is the ratio of heat lost when the textile is placed on the measuring head to heat lost from the bare measuring head [43].'

The results of this methodology are very close to those obtained for sweating hot plate [39], [40].

Previous work [44], [45] has shown that for permeable textile materials made from hydrophobic fibres, the 'sweating skin' and 'upright cup' test methods give very similar results; for semi – permeable materials, the water vapour permeability is similar for hydrophobic membranes and less in agreement for hydrophilic ones.

A new method was proposed by Huang [38], constituted by a cylinder and flow cell [45], offers the possibility to measure the ability of a fabric to carry the water vapour using small specimens measured at small intervals of time. The data provided is reliable, but the disadvantage is represented by the temperature changes that occur during the test [38], [46].

In this research, the Upright Cup with distilled water was used to characterise the water vapour permeability of the fabrics. This method was used by other researchers to characterise the comfort of the inner layer [5], [7], [45].

The technical characteristics for the thermal protection is explained below. As described by Farnworth Equation (Equation 5.7) [46], the volume of air present in a textile system can determine the effective thermal conductivity of

a textile system (K_{sys}) since air is eight times more thermally insulating than the fibres [4].

$$K_{sys} = (1 - P_{\varphi})K_A + P_{\varphi}K_F \tag{5.7}$$

P_{φ} is packing factor; K_A is the thermal conductivity of air volume fraction; K_F is the thermal conductivity of the fibrous component.

From Equation 5.7 it can be deduced that increase in the volume of air increases the thermal insulation of textiles [7], [44]. The function of the fibres is therefore to trap as much air as possible inside the structure of the fabric [4]. The physical characteristics of the fibres such as fineness, shape, crimp, length are of importance for the thermal insulation performance of the fabric [44]. Thin fibres have a higher surface area – to – volume ratio compared to thicker fibres and therefore thin fibres could potentially have more free spaces (bigger pores) in the fabric that could trap air [44]. Crimp in the fibre improves the loft of the fabric and therefore its thermal insulation property [4], [44], [47]. Hollow fibres trap air and therefore act as a superior thermal insulator [48]. Several manufacturers have approached this theory with innovative products. Du Pont and Teijin [49] have created Aerocapsule®material composed of fibres with spiral structure and very wide hollow cross – sections [4], [50]. These features allow both to trap air for insulation as well as to obtain very light fabrics with a high capillary action which ease the passage of moisture [2], [4], [29]. Microfibre with these characteristics is used successfully in swimsuits while the hollow fibres continue to be ideal for wearing in cold weather [4].

A layered clothing provides adequate and efficient insulation at low temperatures. The main function of the mid layer is to provide the thermal insulation and as such, this layer must be made of materials that trap a large amount of air. However, a study by Wilson *et al.* [51] has not found a linear relationship between the thickness and the thermal resistance of the mid layer fabric.

For single layer garment, the thickness of a fabric is proportional to the

thermal resistance and inversely proportional to the heat loss (Equation 5.8) [52]. Equation 5.8 is not always true if the fabric has special finishes or features in its structure that allow a large amount of air to be trapped. Frydrych *et al.* [52] deduced from the tests performed on fabrics made of Tencel®[49] and cotton that the yield of fabric comfort depends largely on the properties of the fibres used.

$$R = \frac{\sigma}{\lambda} \tag{5.8}$$

Where R is the thermal resistance (in $m^2 \cdot K/W^{-1}$), σ is fabric thickness (in m) and λ thermal conductivity (in m·K/W).

Other studies on natural fibres (*e. g.* Bandyopadhyay *et al.* [53] on the jute and jute – blend fabrics) showed a linear relationship between the thermal resistance and thickness as well as the bulk density of the fabric.

Gericke *et al.* [54] in their study on regenerated bamboo, cotton and viscose rayon fabrics assert that the thermal resistance, the resistance of a garment against the loss of body heat, is proportional to the thickness of the fabric that compose it. The thermal resistance of a garment could be obtained by adding the thermal resistance of all the fabric layers and the thermal resistance of the air layer between the skin and the inner face of the garment [54]. Moreover, since water is a good heat conductor, the presence of humidity in a fabric can influence the thermal resistance; more absorbent the textile is the higher will be the frequency of conduction of heat [54].

One of the most effective methods to test the comfort properties of a fabric is the sweating thermal manikin that simulates the body perspiration [29], [52]. There are heat flow sensors positioned on specific areas of the manikin that detect the temperature and are connected to a computer that monitors the loss of heat and sweating [29]. This type of test is among the best to simulate the physical conditions but is not suitable for this research, as a garment is required to do this testing. European standard for testing the thermal resistance

of textiles (EN 31092) [42] describes a procedure to test the insulation of a textile material against cold. This feature is called Thermal resistance (R_{ct}) and refers to a table (Table 5.2) that indicates the class of an insulating material; the first class gives little insulation [4].

Thermal resistance (R_{ct}), expressed in m^2·K/W, is the temperature difference among the two faces of a material divided by the heat flow per unit area gradient. The resultant heat flux is obtained by a constant temperature gradient which acts on a given area [42].

Table 5.2: Classification of textiles based on their thermal insulation (R_{ct}) property [4].

R_{ct} (m^2K/W)	**Class**
0.06 – 0.12	1
0.12 – 0.18	2
0.18 – 0.25	3

Thermal resistance is the sum of the heat transferred by radiation, conductivity, and convection. It is an intrinsic property of the material but the values are affected by the interaction with the surrounding environment [2], [4], [10]. An effective method, among the several used to measure this property for clothing textile, is the sweating guarded hot plate because it simulates the thermal processes occurring close of the skin [42].

The Thermal resistance unit of measure (m^2·K/W) can be converted to 1 tog, defined as the temperature difference of 0.1 °C caused by a heat flow between two surface and equivalent to 1 Watt/m^2 (1 tog corresponds to 0.1 m^2·K/W).

The degree of stiffness of a fabric is relevant to the wearability of a garment and could be measured using *FAST – 2* [55], [56], [57]. The stiffness

of a material is directly proportional to the bending length [58]. The *FAST – 2* meter is a cantilever bending stiffness measurement equipment developed by Shirley Institute used to measure the bending length of 50 X 15 mm strips of nonwoven according to the standard ISO 9073 [56], [57], [58].

The sample is mounted on a horizontal support on top of the apparatus, then moved slowly until the edge of the fabric reaches the plane inclined at an angle of 41.5 ° (Figure 5.5). The stiffer the fabric, the greater is the length necessary to ensure sufficient bending [56].

Bending length indicates the draping quality of the fabric defined as the length of a given fabric strip sample that will bend under its own weight to a particular extent [56]. Bending length could be calculated using Equation 5.9.

$$c = l f_1(\theta) \tag{5.9}$$

Where

$$f_1(\theta) = \left(\frac{\cos \frac{\theta}{2}}{8 \tan \theta} \right)^{\frac{1}{3}} \tag{5.10}$$

c is the bending length (in mm), l is the length of the fabric projecting (in mm), θ is the angle fabrics bends. For $\theta = 41.5\,^{\circ}$, the value obtained from the function is $f_1(\theta) = 0.5$.

From the bending length, flexural rigidity of a fabric could be calculated using Equation 5.11 (unit in μNm).

$$G = 9.81 \times M \times C^3 \times 10^{-3} \tag{5.11}$$

Where M is the mass of the sample per unit area (in gm^2), C is the mean bending length (in cm, g = 9.81 m/s^2). The flexural rigidity values are

strongly related to the fabric thickness; doubling the thickness of the fabric sample could increase the flexural rigidity eightfold [58], [59], [60].

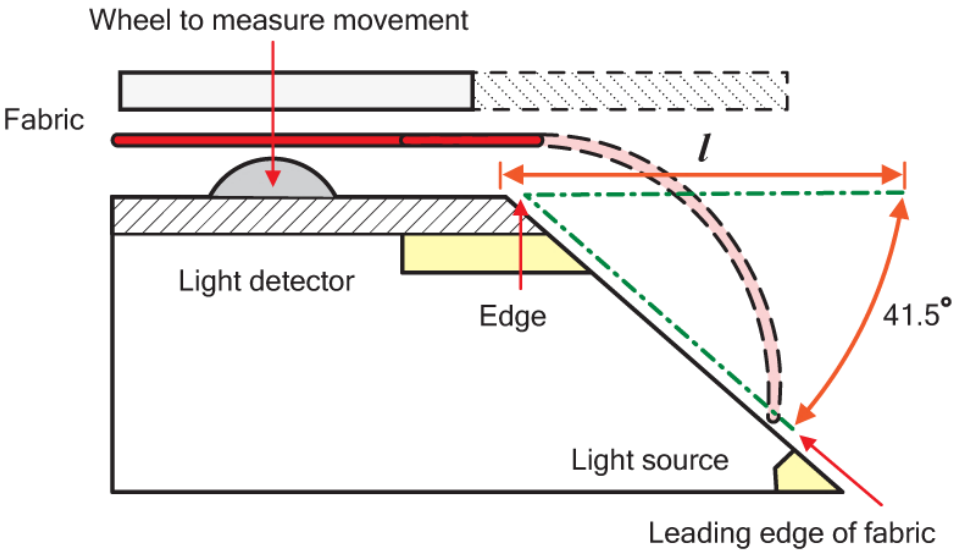

Figure 5.5: Schematic diagram of bending length measurement using a *Siro – Fast* stiffness meter. l is the bending length and the angle $\theta = 41.5\,^{\circ}$ [55], [57], [58], [59].

5.1.4 A three – layer assembly using Himalayan nettle fibre

A layered system, with each layer performing a given role, is proven to be the best way to ensure a state of comfort in outdoor clothing [1], [2], [3], [4]. The treatment concentration of NaOH required to achieve *G. diversifolia* fabrics with different functionality suited to perform the functions required from each layer have been studied in this section.

It is proposed that *G. diversifolia* fibres treated with 4 mol dm^{-3} NaOH will be ideal for the base layer; the function of the base layer is to provide breathability and wearability. Compared to other treatment concentrations, at 4 mol dm^{-3} the fibres show high elongation (Chapter 4, Section 4.3.2.1), implying that fabrics made out of these fibres will be flexible and comfortable to wear [60]. These fibres also show low moisture content and high moisture regain (Chapter 4, Section 4.3.2.2). This should facilitate the absorption of

water vapour and its flow outwards [13], [14], [38], [39]. Fibres treated with 4 mol dm^{-3} NaOH also show the lowest cross – sectional area (Chapter 4, Section 4.3.3) and therefore will show higher pore volume in the fabric; this will help the water to move to the next layer by capilliary action [29].

G. diversifolia fibres samples treated with 0 mol dm^{-3} NaOH will be ideal for the middle layer; as discussed previously, the middle layer should be a thermal insulator, breathable and wearable. The fibres, when treated with 0 mol dm^{-3} NaOH, show a very wide cross – section and especially the cross – sectional area of lumen are the greatest amongst all treatment concentrations (Chapter 4, Section 4.3.3). This means that it is possible to make a bulky layer using these fibres to trap air [11]. The values of moisture content and moisture regain are low (Chapter 4, Section 4.3.2.2) which is ideal for the middle layer [19], [38], [39].

The outer layer of the garment should be resistant to abrasion and external factors such as a drastic change in weather. It is proposed that *G. diversifolia* fibres treated with 8 mol dm^{-3} NaOH will be ideal for the outer layer. When treated with 8 mol dm^{-3} NaOH, the fibres show good strength and elongation properties (Chapter 4, Section 4.3.2.1). Higher strength and elongation implies better processes ability of the fibre and a better fit of the fabric [37]. It is proposed that a multilayered fabric where fibres treated with 4 mol dm^{-3} for the base layer, 0 mol dm^{-3} for the middle layer, and 8 mol dm^{-3} for the outer layer will be ideal for outdoor clothing (Figure 5.6 and 5.7).

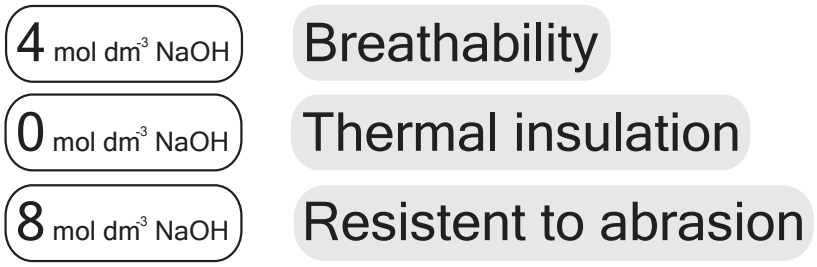

Figure 5.6: *G. diversifolia* fibre treatment concentrations with NaOH to perform the functions required of each layer.

To check the proposed hypothesis and to verify if the high performance requirements of outdoor clothing could be met by Himalayan nettle fibres, fabrics were made and tested using a nonwoven manufacturing process.

5.1.5 Nonwoven technology

The application of nonwovens in technical textile applications are increasing and the nonwoven manufacturing process allows large volume production at high speed and low cost, compared to traditional processes. According to the INDA nonwovens could be defined as

> 'A fabric made directly from a web of fiber, without the yarn preparation necessary for weaving and knitting [61], [62]'.

The Nonwovens industry is a growing sector globally, where academic and industrial efforts are directed to design biodegradable nonwoven materials in order to provide an answer to sustainable issues [63], [64].

Natural fibres are renewable resources and in addition, they are biodegradable, soft, durable, and absorbent [65], [66]. Market research predicts that the use of natural fibres will increase with increasing environmental awareness of the consumers [65], [66], [67]; cotton, hemp, jute, flax, kenaf, coir, sisal, wool are at present used in the nonwovens sector [63], [65].

In reference to this work, the thickness of a nonwoven fabric is proportional to the thermal resistance (see Equation 5.8) [4], and clothing worn in extreme situations usually have a fabric area density of $200\,g\,m^{-2}$ or more. A $100 - 150\,g\,m^{-2}$ synthetic nonwoven padding normally offers effective protection against the cold [4], [12]. While for the breathable clothing used in physical activities such as running, the area density of the fabric is normally between $25 - 70\,g\,m^{-2}$ [2]. The UK brand Mountain Equipment®[68], which uses the material Primaloft®for fill outdoor sport jackets, uses an area density of $70\,g\,m^{-2}$ in applications where the garment have to protect the body from the cold and only $40\,g\,m^{-2}$ in areas where greater breathability is required [12], [69].

In this work a sample of Primaloft®Sport, provided by Albany International Corp. [70], was used as a reference to measuring the thermal resistance. Primaloft®Sport $100\,g\,m^{-2}$ fabric is made from 100 % PET fibres and is used to keep the body warm and dry in extreme conditions; [12] this fabric is claimed to be heat resistant and breathable [4], [12].

The mid layer should normally be thick for better protection from the cold, while the base layer should normally be lightweight to allow the ease of movement [1], [2], [4]. The outer layer should be resistant to external agents and it is hypothesised in this study that for adequate strength, this layer should be of the same area density as the second layer; higher area density gives a stronger and more durable material [63].

The density used in this work are:

- base layer $= 40\,g\,m^{-2}$;
- middle and outer layer $= 120\,g\,m^{-2}$.

5.2 Experimental

5.2.1 Samples preparation: Himalayan nettle nonwoven

To check the proposed hypothesis and to verify if Himalayan nettle fibres could meet the high performance requirements of outdoor clothing, fabrics were made and tested using a nonwoven manufacturing process (Figure 5.7).

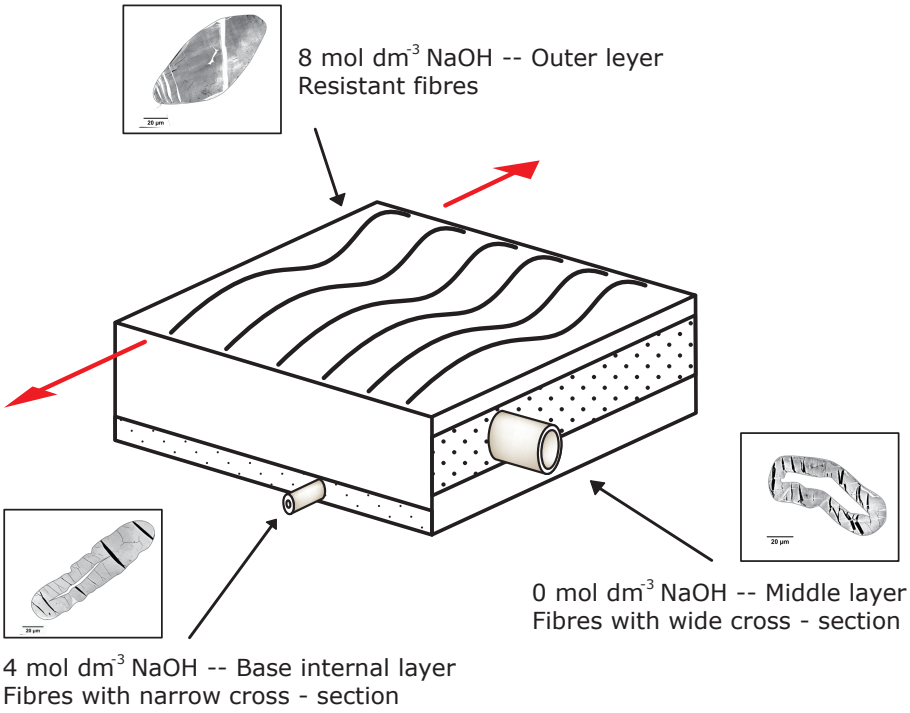

Figure 5.7: Scheme of a multilayer fabric made with *G. diversifolia* fibres treated at three different concentrations of NaOH.

Three groups of *G. diversifolia* fibres treated at three different concentrations of sodium hydroxide, 0 mol dm^{-3}, 4 mol dm^{-3}, 8 mol dm^{-3}, were used to make nonwoven fabric samples. The samples were carded followed by thermal bonding step (Through – air oven). As *G. diversifolia* fibres are non – thermoplastic fibres, a bi – component fibre was used to bond the webs into fabrics [71].

Since the nineties, the bi – component fibres are widely used to bond the webs in the thermal bonding stage [71], [72]. Usually they are classified according to their cross – sectional orientation *e. g.* side – by – side, sheath – core *etc.* [72]. The side – by – side consists of one or more different regions that run along the fibre. The sheath – core structure are constituted by a sheath covering a core, which can be positioned according to requirements. The sheet acts as a binder while the core is the structural part of the fibre. The two parts have different melting temperatures; the core remains stable even at high temperatures, which allow nonwoven fabrics of high quality [71], [72], [73], [74]. In this work a polyethylene therephthale (PET) bi – component sheat – core fibre was used as binder (4.8 dtex; staple length 52 mm; sheat melting point 110 °C; the core melting point 250 °C).

The *G. diversifolia* fibres were cut to a staple length of 6 cm before carding. The fibres were then carded manually with a pair of rectangular hand carders fitted with thin steel teeth, which work against one another to disentangle the mass of fibres until they are aligned. Then the NaOH treated *G. diversifolia* fibres were blended manually with given percentage of bi – component fibres and carded in a single cylinder worker – stripper card manufactured by Tatham (Figure 5.8) [72]. This card gives webs with an area of 0.24 m^2 (0.6 X 0.4 m) in the machine direction. After the first pass, the webs are rotated 90 ° and passed through the machine again. The webs are passed twice for greater uniformity of the web.

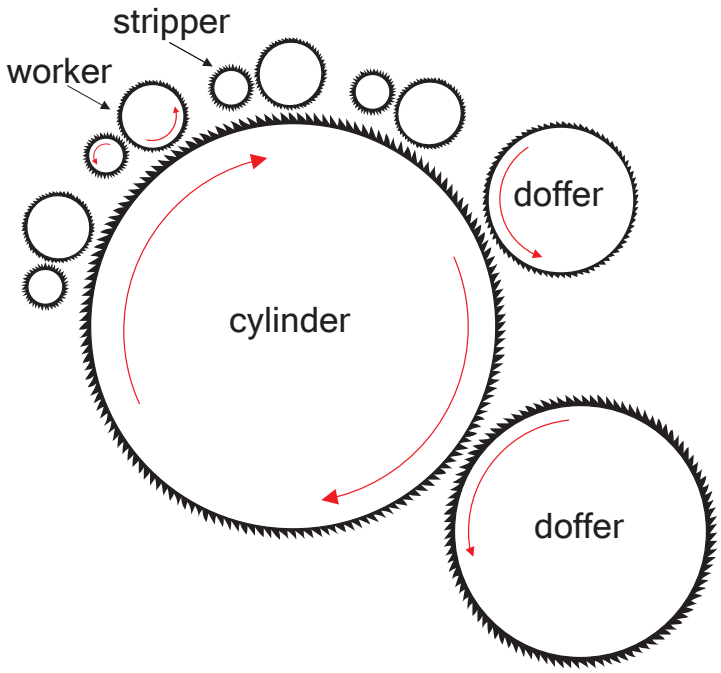

Figure 5.8: Diagram of the rollers positioning in a carding machine [71].

Once carded, the webs were thermally bonded using a through – air oven manufactured by Spooner (conveyor speed of 1.2 m min^{-1}). The machine consists of an oven that heats the web using a jet of hot air at a temperature which allows the bonding of the web. At elevated temperature the sheath of the bi – component fibre becomes viscous or melts and bonds the web [71], [74].

A first range of samples were made (40 and 120 g m^{-2}) by varying three process parameters as shown in Figure 5.9 using *G. diversifolia* fibres. This is to identify the best process parameters for manufacturing 40 and 120 g m^{-2} fabrics.

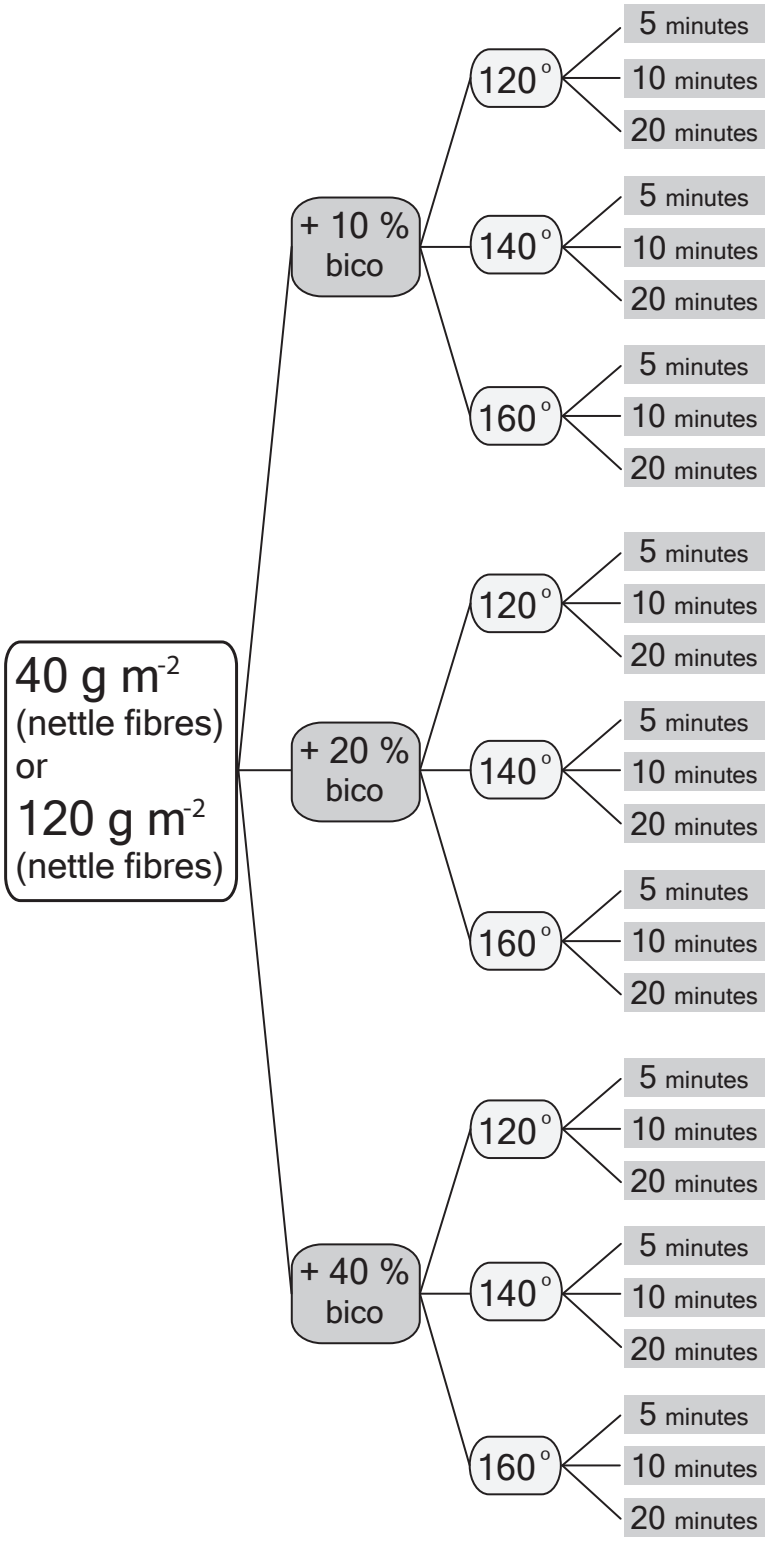

Figure 5.9: Combinations of the processing parameters in order to identify the best process parameter for manufacturing 40 and 120 g m^{-2} nonwovens made by *G. diversifolia* fibre.

5.2.2 Determination of breaking strength and elongation of fabrics

BS EN ISO 9073 was used to test the tensile strength properties of the nonwoven fabrics [75]. It determines the breaking force (F_{max} in newton (N)) and the elongation (%) of the nonwoven fabric.

The specimens (50 X 200 mm) were conditioned for at least 48 hours (65 ± 2 % relative humidity; $20 \pm 2\,^{\circ}$C) before testing.

The *Zwick* tensile testing machine was used for testing. The load cell was 200 N, the gauge length was 150 mm, and the rate of extension was 200 ± 10 mm min^{-1}. The processor *TestXper II* of *Zwick* machine automatically calculates the values of mean, standard deviation and variance of braking strength and elongation of the samples. Samples were tested in the machine direction, cross direction and in a 45 ° bias (Figure 5.10).

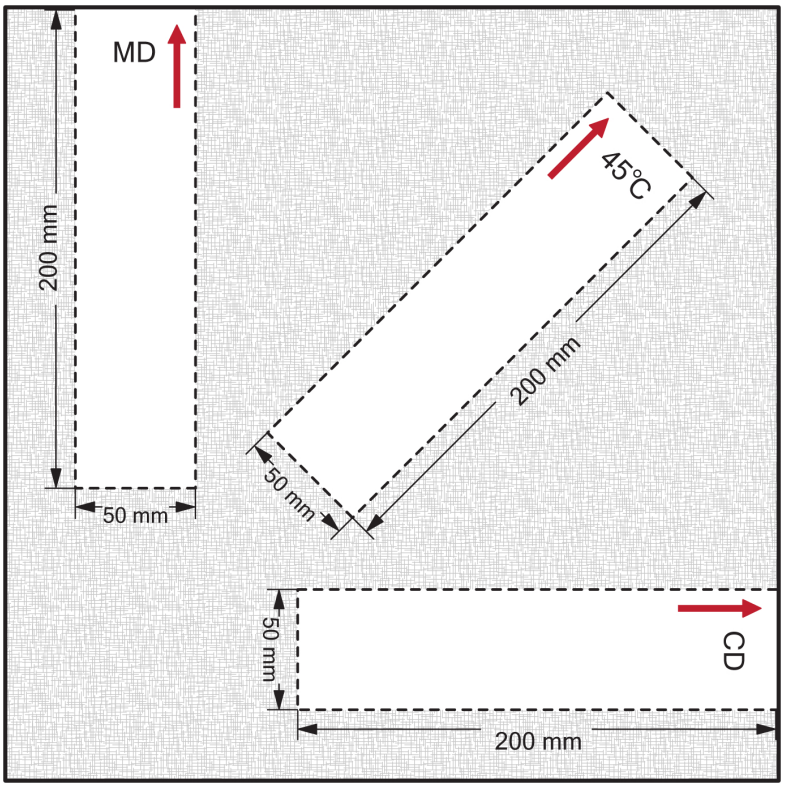

Figure 5.10: Method of cutting nonwoven fabric strips for tensile and bending length test [57].

5.2.3 Gravimetric upright cup with distilled water

Gravimetric upright cup determines the level of water vapour that is able to permeate through a fabric over an extended period of time [41], [76]. This can then be compared with a reference sample to calculate a permeability index value. The method follows BS 7209 [41].

The size of the specimen is 96 mm in diameter and obtained by a circular cutting device. The specimens were conditioned for 24 hours before the experiment and the tests were performed in the conditioned atmosphere ($20 \pm 2\,^{\circ}C$ and relative humidity of $65 \pm 2\,\%$) [77]. The cups were filled with $46\,cm^3$ of distilled water in order to obtain an internal still air layer of 10 ± 1 mm. Then the specimens were placed over the rim of the cup and sealed with an elastic ring (Figure 5.11).

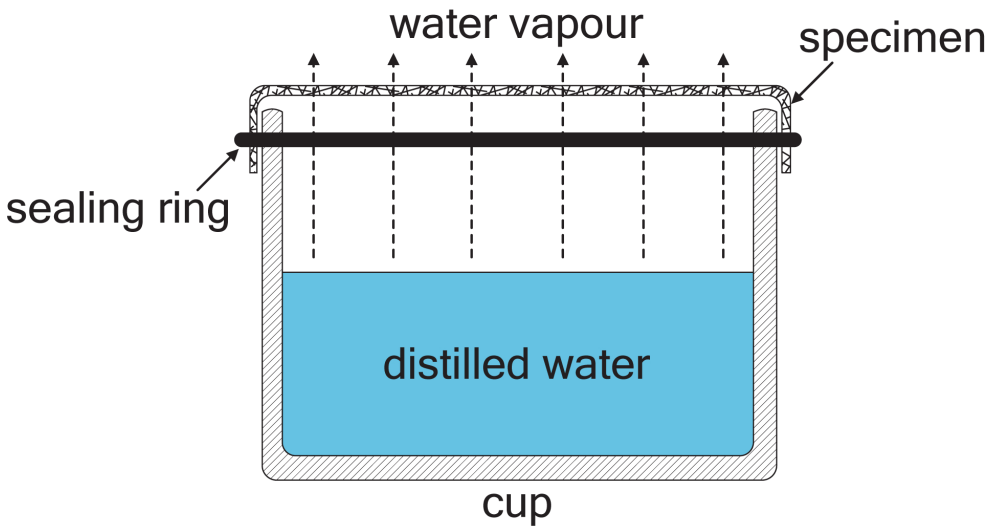

Figure 5.11: Assembly: container, water, sample and seal ring, for the water vapour permeability test [76].

This assembly (container, water, sample and seal ring) is weighed and then placed on a turntable that supports eight samples. The turntable rotates at a speed below 6 m/min. The dishes are rotated for a minimum of 1 hour to allow the equilibrium to form. The dishes are then weighed again. The dishes

are then returned to the turntable and allowed to rotate further for an extended period. The dishes are then reweighed. In this test, the samples were reweighed after 24 hours [41], [76].

The samples should be compared to a reference fabric having the following specifications [41]:

Polyester woven fabric	
Yarn diameter (μm)	32
Mesh aperture (μm)	18
Yarn density (per cm)	196.1
Open area (%)	12.5

The water vapour permeability index I can be calculated as the percentage of the rate between water vapour permeability of the fabric under test $(WVP)_f$ and that of the reference fabric $(WVP)_r$ (Equation 5.12) [41].

$$I = \left\{ \frac{(WVP)_f}{(WVP)_r} \right\} \times 100 \tag{5.12}$$

5.2.4 Sweating guarded hot plate

The sweating guarded hot plate is a method that simulates the process of heat and mass transfer between the skin and the clothing in different environmental situation. BS EN 3109240 [42] test standard was used in this research (steady – states condition) to measure the thermal resistance of non-woven fabrics made with *G. diversifolia* fibres.

The specimens were cut into squares of 310 X 310 mm and conditioned for 24 hours in a conditioned atmosphere ($20 \pm 2\,^{\circ}$C; 65 ± 2 % relative humidity) [42].

This apparatus consists of a porous plate and a guard ring both heated at a steady temperature of $\sim$35 $^{\circ}$C to simulate the human body temperature.

The ring is kept at the same temperature of the central plate, to prevent heat loss. The schematic diagram of the test assembly is shown in Figure 5.12 [4], [29], [42], [76], [78].

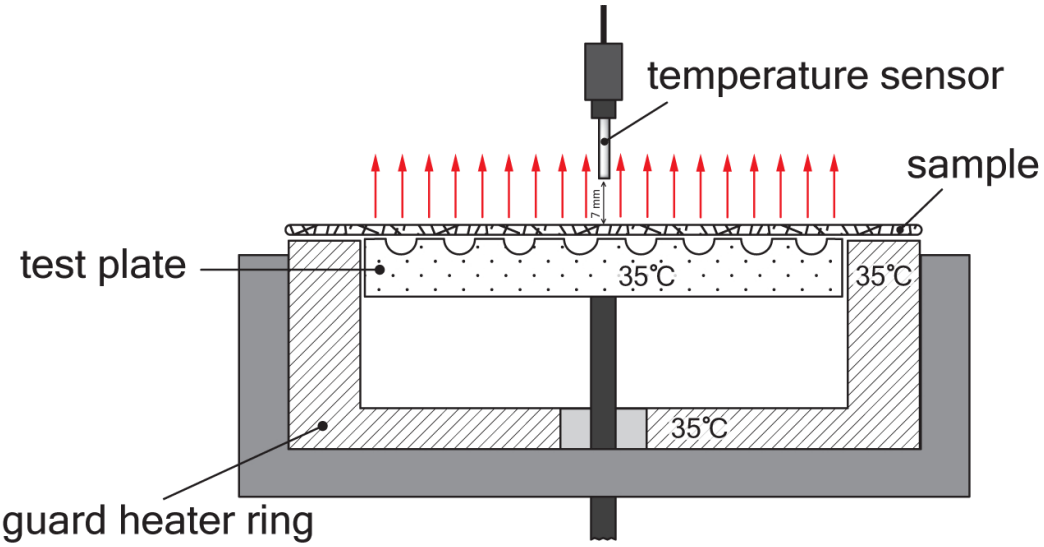

Figure 5.12: Schematic guarded hot plate according to BS EN ISO 11092 [4], [29], [42], [76], [78].

At the end of the measurement the machine provides directly values of R_{ct} (m^2·K/W) (Equation 5.13) that is the thermo total resistance of the sample and the air layer and of R_{cf} (m^2·K/W), which represents the intrinsic thermal resistance of the sample (Equation 5.14).

$$R_{ct} = \frac{(T_m - T_a) \cdot A}{H - \Delta H_c} - R_{ct0} \tag{5.13}$$

Where T_m is the predetermined temperature that the equipment must reach, in degree Celsius or Kelvin; T_a is the temperature of the enclosure in which the test takes place, expressed in degree Celsius or Kelvin; A is the area of the test plate in square metres; H in the heating power in watts of the apparatus; ΔH_c is the correction term for heating power; R_{ct0} is the apparatus constant in square metres Kelvin per watt.

R_{ct}values can be recorded when T_m, T_a and H have reached steady – state [42].

$$R_{cf} = R_{ct} - R_{ct0} \tag{5.14}$$

The fabric intrinsic thermal resistance, R_{cf} is calculated subtracting the thermal resistance without the fabric layer, R_{ct0}, to the thermal resistance of the fabric and air layer, R_{ct} [79].

5.2.5 Bending stiffness measurement using *FAST – 2* bending meter

The stiffness of a material is directly proportional to its bending length; in this work bending length is measured using a *FAST – 2* meter. *FAST – 2* is a cantilever bending stiffness measurement equipment. ISO 9073 [55] test standard was used in this research to measure the stiffness properties of the fabric. The operating principle of this apparatus (Figure 5.5) is described in Section 5.1.3 [55], [56], [57].

Specimens for these measurements were prepared in strips of 50 X 200 mm in machine direction and in cross machine direction as showed in Figure 5.10. The samples were conditioned overnight in the standard atmosphere (temperature of $20 \pm 2\,^{\circ}$C and relative humidity $65 \pm 2\,\%$) and the testing were done in the same conditioned atmosphere. Bending length and bending rigidity are quoted separately for machine direction and cross machine direction [55], [56], [57], [76].

Every experiment (Section 5.2.2 – 5.2.5) was performed on 3 samples and the standard error (SE $\pm$) were measured.

5.3 Results and discussion

5.3.1 Optimising the nonwoven manufacturing process

To identify the best process parameters for manufacturing 40 and 120 $g\,m^{-2}$ nonwoven fabrics, three process parameters were varied as shown in Figure 5.9 and tensile test (machine direction) [71] were performed on the samples [71], [72], [75], [76], [77], [80].

The tensile tests performed on 120 $g\,m^{-2}$ samples show increasing values of breaking force with higher concentration of bi – component fibres; 40 % bi – component fibres give the highest strength results (Figure 5.14).This is expected as increasing the binder should increase the strength of the fabric [72], [74], [80]. The thermal bonding time seems to have less influence on either the strength or elongation of the fabrics suggesting 5 minutes as an adequate bonding time for these samples. When the binder concentration is increased from 20 % to 40 %, the elongation decreased a bit and this is an expected result [71], [72], [74], [80], [81], [82]. The highest values of breaking force were obtained with 40 % of bicomponent fibres. In particular, at a bonding temperature of 140 °C, excellent values of both strength and elongation (5 minutes bonding time) were obtained.

For both 40 and 120 $g\,m^{-2}$ nonwovens, 10 % of bi – component fibres give a very weak samples (Figure 5.13 and Figure 5.14) and the strength of the samples increases with increase in the percentage of bi – component fibres. For 40 $g\,m^{-2}$ samples, there is no observable change in the elongation percentage when the percentage of bi – componemt fibres were increased from 20 % to 40 %.

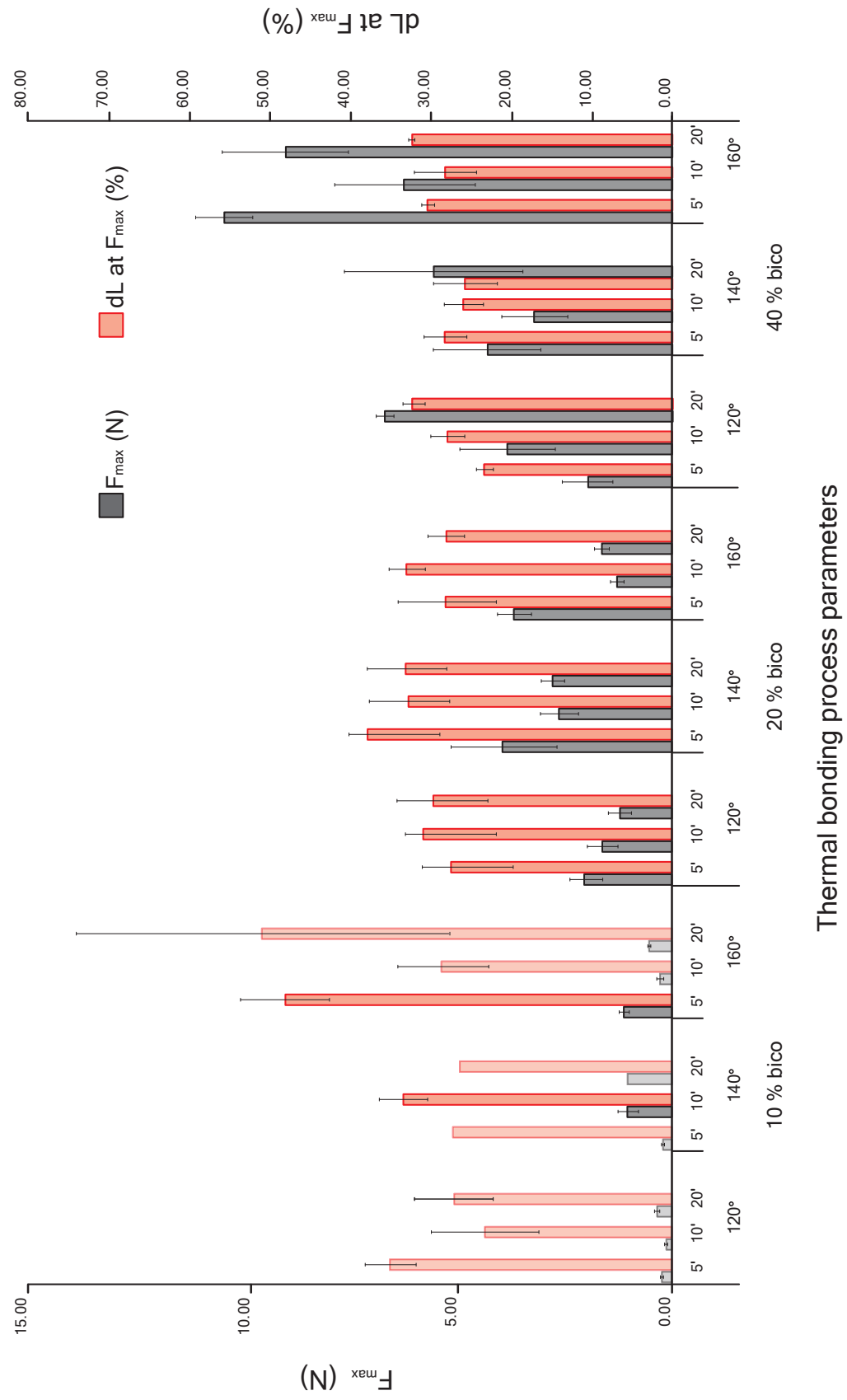

Figure 5.13: Tensile test results of 40 g m^{-2} *G. diversifolia* nonwoven sample (machine direction).

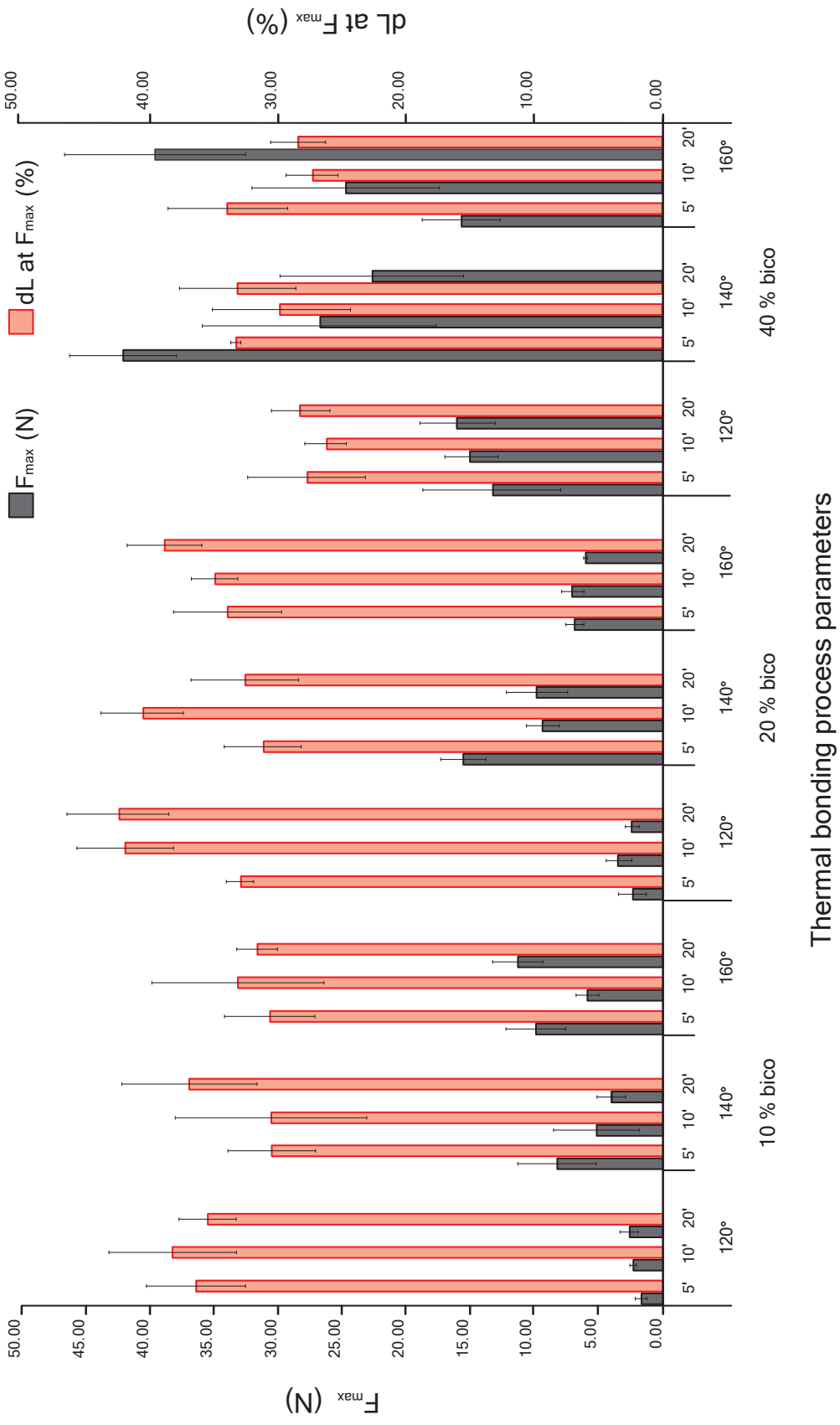

Figure 5.14: Tensile test results of 120 g m^{-2} *G. diversifolia* nonwoven sample (machine direction).

It is clear from Figure 5.13 and Figure 5.14 that for 40 and 120 g m^{-2} fabrics, 40 % of bi – component fibres give the best strength and elongation results. However further experiments were conducted with 20 % bicomponent fibres to protect the inherent properties of the *G. diversifolia* fibres in the fabric; when 40 % (and above) binder fibres are used to bond the web, the fibres act as reinforced plastic and not as a binder [71], [72], [74], [80], [81], [82], [83].

140 °C binding temperature and 5 minutes binding time gives the best strength property for both 40 and 120 g m^{-2} fabrics with 20 % binder concentration with acceptable elongation results; these conditions were used for further nonwoven fabric sample manufacturing with the alkali treated fibres. Lower bonding time also gives a significant energy saving [71], [84].

5.3.2 Three layer fabrics with alkali treated samples

To check the hypothesis proposed in Section 5.1.4 (Figure 5.16), nonwoven fabrics were manufactured with alkali treated *G. diversifolia* fibres (0, 4, and 8 mol dm^{-3} NaOH) using a manufacturing process proposed in section Section 5.3.1 (Figure 5.15). The main purpose of this work is to demonstrate the versatility that could be engineered onto fabrics manufactured with *G. diversifolia* fibres after alkali treatment. Moreover, the application of nonwovens in sportswear is gathering momentum in recent years [85].

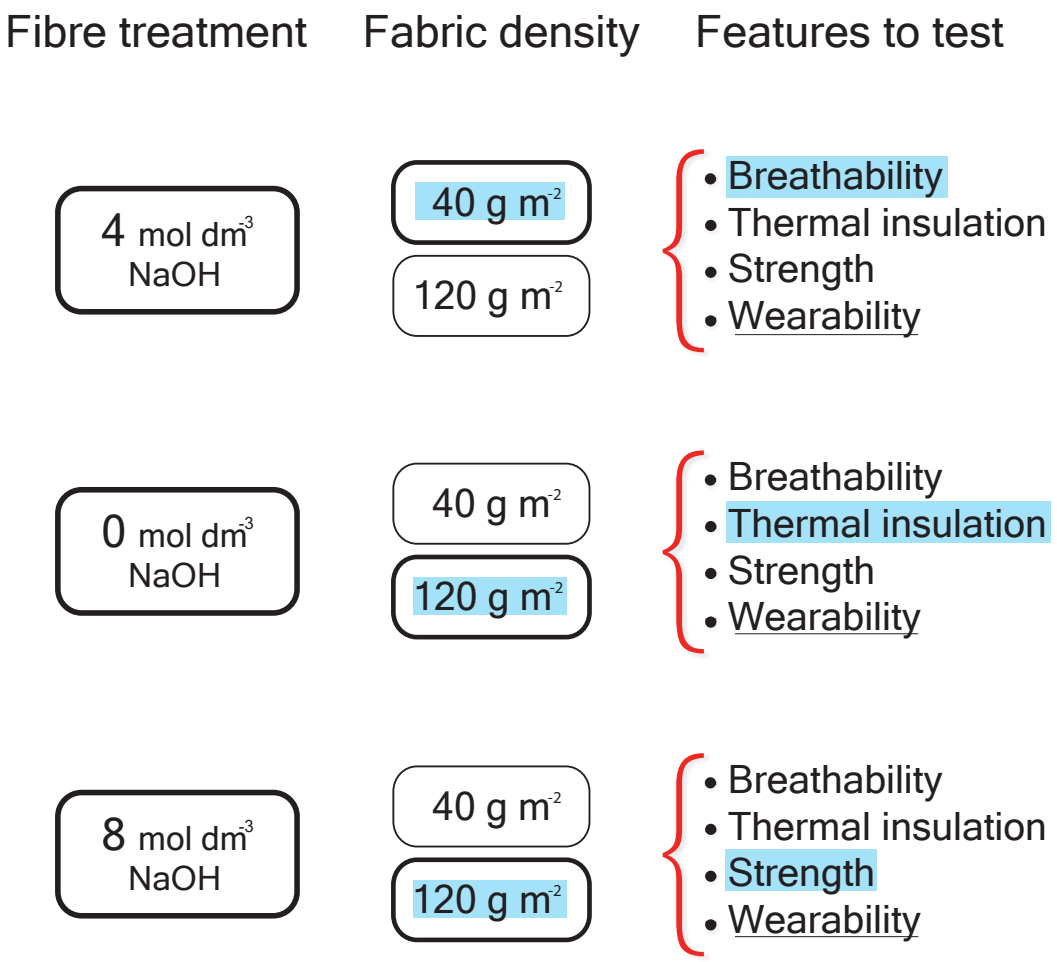

Figure 5.15: Samples of two different area densities (g m^{-2}) manufactured with *G. diversifolia* fibres treated at three different concentrations of sodium hydroxide.

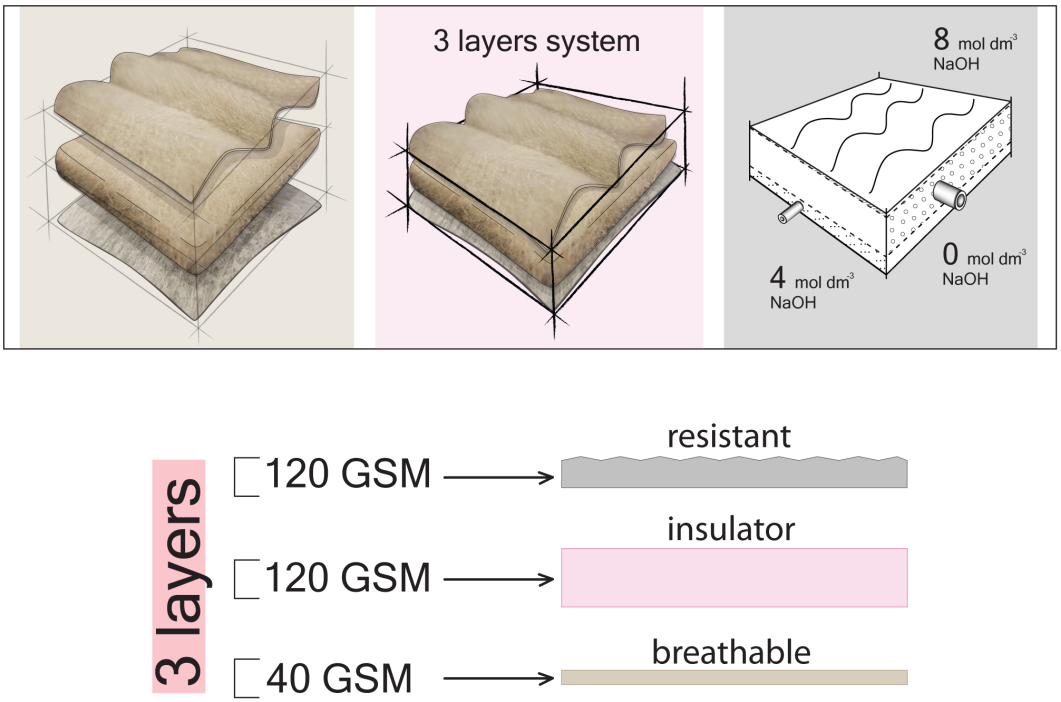

Figure 5.16: System of 3 layers made from the assembly of different nonwoven fabrics made from alkali treated *G. diversifolia* fibres.

5.3.3 Water Vapour Permeability (WVP)

The results of the WVP test are shown in Figure 5.17. It is clear from Figure 5.17 that the 40 g m^{-2} samples show higher water vapour permeability compared to the 120 g m^{-2} samples. The flow rate of water vapour through a fibrous material (porous), is inversely proportional to its thickness. This phenomenon could be explained by Darcy's law (Equation 5.15) [5], [29], [86], [87] and by the Fick's second law described in Section 5.1.3 (Equation 5.2):

$$Q = K\frac{A \times \Delta P}{d \times \mu} \tag{5.15}$$

Where Q (in m^3/ s) is the flow rate through porous medium; A the cross – sectional area (in m^2); ΔP the pressure gradient (in Pa); d the thickness (in m); K the proportionality constant (in m^2) and μ the dynamic viscosity of the

liquid (in Pa·s) [5], [88].

The alkali pre – treated samples were benchmarked against a 100 % PET nonwoven fabric specimen (Primaloft®Sport – 100 g m^{-2}) used in outdoor sportswear as thermal insulator [12]. The *G. diversifolia* nonwoven fabrics have water vapour permeability values greater than that of Primaloft®Sport nonwoven sample however, the WVP value of 120 g m^{-2} nonwoven fabric made of *G. diversifolia* fibre and pre – treated with 0 mol dm^{-3} NaOH (680 g/m^2/day), is very close to that of Primaloft®Sport (671 g/m^2/day). The 100 % PET nonwoven fabric is used as the middle layer in a three layer outdoor sportwear for its efficient thermal insulation properties [12]. The 120 g m^{-2} nonwoven made with Himalayan nettle fibre pre – treated with 0 mol dm^{-3} NaOH was designed to perform the same functions.

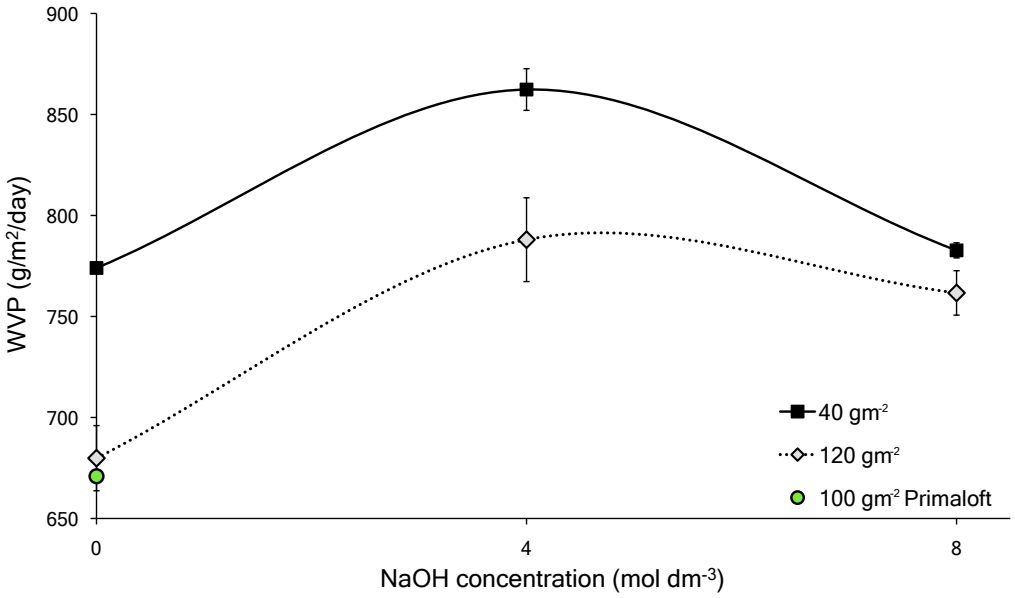

Figure 5.17: Water Vapour Permeability (g/m^2/day) of 40 and 120 g m^{-2} alkali pretreated *G. diversifolia* nonwoven samples and 100 g m^{-2} Primaloft®Sport sample.

For both 40 and 120 g m^{-2} samples, the highest water vapour permeability was obtained for samples pre – treated with 4 mol dm^{-3} NaOH; 0 mol dm^{-3} gives the lowest water vapour permeability. As discussed previously, the porous structure of a fabric affects the passage of water vapour through the fabric *via*

capillary action [5], [29], [86]. The porous structure creates pressure differences (Equation 5.2) and facilitates the movement of water molecules in the free spaces towards areas with lower humidity [16], [31], [37]. In a thicker hydrophilic fabric, the water molecules gets absorbed by the fibres causing fibre swelling; fibre swelling decreases the porosity of the fabric which slows down the flow – rate of water molecules towards the external surface of the fabric [14], [16], [16], [32]. The capillary action is influenced by the fibre cross – section; factors such as the thickness, width of the lumen will affect the capillary action [2], [27]. To create an efficient capillary action, the fibres must function as small pipes. This explain the higher WVP value of samples pre – treated with 4 mol dm^{-3} NaOH; at 4 mol dm^{-3} pre – treatment concentration, the fibres have a narrower cross – section with the lowest cross – sectional area as could be seen from Figure 5.18 [2], [27], [28]. It could be seen from Figure 5.18 that the WVP of fabrics are inversely proportional to the cross – sectional area of the fibres they are made from.

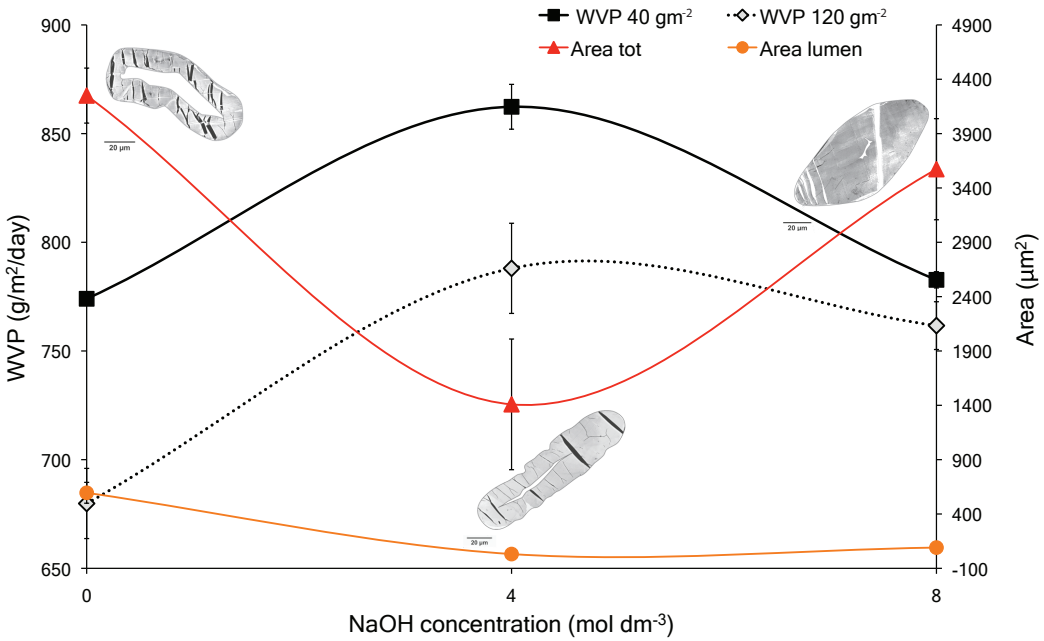

Figure 5.18: Water Vapour Permeability (WVP) and the cross – sectional area (total and lumen area of fibres) of 40 and 120 g m^{-2} nonwoven fabrics manufactured with alkali pre – treated fibres.

The water vapour permeability as well as the breathability of a fabric is affected by the moisture sorption properties of the fibres [16]. The hydrophilic cellulosic fibres readily absorb moisture. The good absorbancy of the cellulosic fibres have the advantage of quickly absorbing the moisture next to the fabric when sweating occurs however, this also creates a damp condition that affects the thermo – physiological comfort of the clothing [1], [2], [16]. To retain water molecules, fibres must have high moisture content and low moisture regain [16], [29], [30], [37].

An efficient breathable fabric must have low moisture content. Low moisture content creates a pressure difference when in contact with moisture *e. g.* next to the skin; this pressure difference starts the capillary action which allows water vapour to move toward the fabric [27]. The efficiency of the capillary action in the fabric is related to the moisture regain of the fibres. Higher the values of moisture regain implies quicker moisture sorption; the diffusivity of moisture also increases with increasing moisture regain. This moisture movement by the process of sorption and desorption creates the hygroscopicity in textile materials [16], [29], [37].

As discussed in Chapters 3 and 4, the Himalayan nettle fibre (like other vegetable fibres) is hydrophobic in the crystalline regions and hydrophilic in the amorphous regions [89]. As Himalayan nettle is a highly crystalline fibre, it has low moisture content and low moisture regain compared to other bast fibres. The nonwoven fabrics manufactured with 4 mol dm^{-3} NaOH pre – treated *G. diversifolia* fibres has been proposed to be the ideal fabric for the base layer of a three layer outdoor clothing; this is because of the fibres' cross – sectional area, its lumen size, and its moisture content and moisture regain values. The combination of lower cross – sectional area, narrow lumen, and low moisture content of the 4 mol dm^{-3} NaOH pre – treated fibres create a capillary action that easily absorbs moisture [2], [27]. This pre – treatment concentration (4 mol dm^{-3}) gives the highest moisture regain value compared to the other two selected pre – treatment concentrations (0 and 8 mol dm^{-3}). The WVP value (Figure 5.17)

confirms that the 4 mol dm^{-3} NaOH pre – treatment concentration gives the fabric highest breathability compared to other treatment concentrations.

The water vapour, from the first layer moves toward the second layer (the insulating layer) which is made with fibre pre – treated with 0 mol dm^{-3} NaOH. Fibres pre – treated with 0 mol dm^{-3} NaOH shows slightly higher moisture content (Figure 5.19) and lower moisture regain (Figure 5.20) compared to 4 mol dm^{-3} NaOH pre – treated fibres. In addition, 0 mol dm^{-3} alkali pre – treated concentration gives the highest cross – sectional area (total and lumen area of fibres) amongst all treatment concentrations (Figure 5.18), providing the best condition to trap air and thereby providing insulation. The WVP values of the fabrics manufactured with 0 mol dm^{-3} NaOH are the lowest as could be seen from Figure 5.18. At this pre – treatment concentration, the fibres are highly crystalline (Chapter 4) which explains the lower moisture regain (MR) and moisture content (MC) of these fibres. The lower MR and MC values of the 0 mol dm^{-3} pre-treated fibres make this pre – treatment concentration ideal for the insulating (middle) layer [4]. The WVP of the 120 g m^{-2} fabric manufactured with 0 mol dm^{-3} NaOH pre – treated fibres are close to that of Primaloft®Sport material. Primaloft®Sport material is used in many three layer outdoor clothing as the middle insulating layer [12].

The nonwovens manufactured with 8 mol dm^{-3} NaOH pre – treated fibres show good WVP values (Figure 5.17) along with very high moisture content (Figure 5.19). These fibres show low crystallinity (Chapter 4) compared to other pre – treatment concentrations and therefore will have more sorption capacity. The test results confirm that fabrics manufactured with 8 mol dm^{-3} NaOH pre – treated fibres show high moisture regain and highest moisture content; properties that are considered ideal for the outer protective layer [88].

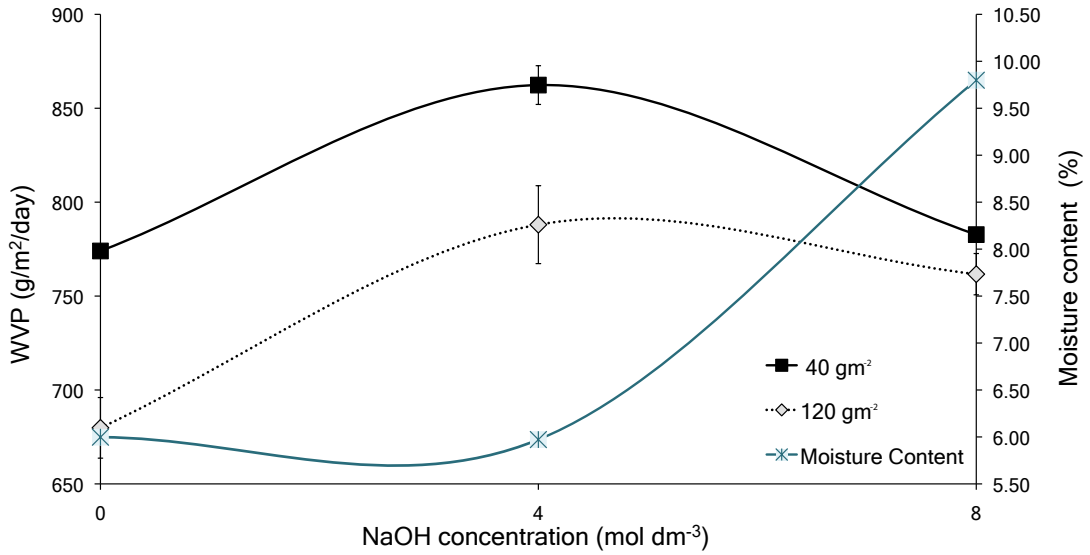

Figure 5.19: Water Vapour Permeability (WVP) and moisture content of 40 and 120 g m^{-2} nonwoven fabrics manufactured with NaOH treated *G. diversifolia* fibres.

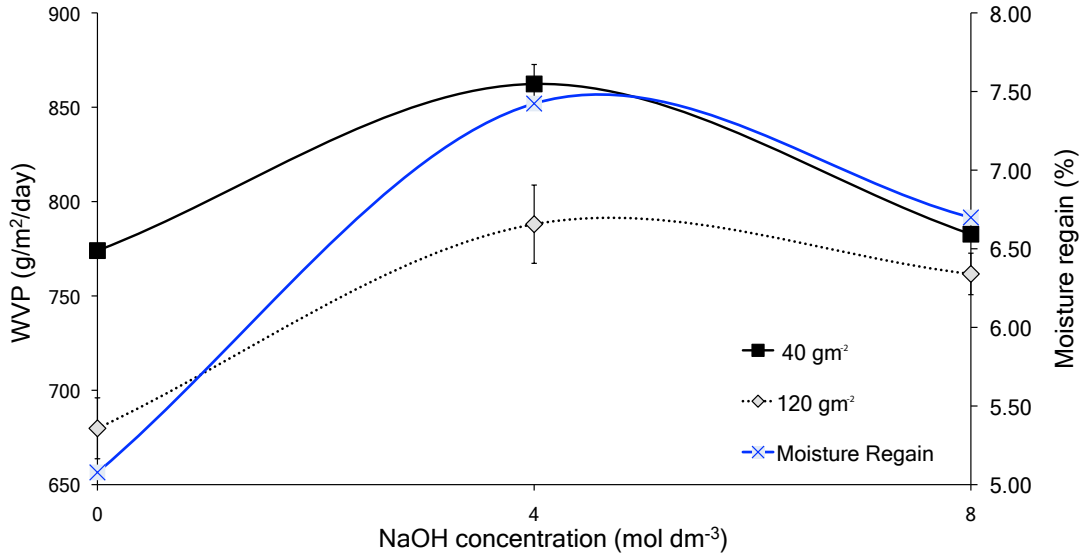

Figure 5.20: Water Vapour Permeability (WVP) and moisture regain of 40 and 120 g m^{-2} nonwoven fabrics manufactured with alkali treated *G. diversifolia* fibres.

5.3.4 Thermal resistance (R_{ct})

From the thermal resistance test, it is possible to measure the intrinsic thermal resistance of the fabric test specimen (R_{cf}) and the, total thermal resistance of the test specimen and the air layer (R_{ct}). This test is performed in a special hood in which atmospheric conditions (humidity, temperature and air speed) are controlled [42].

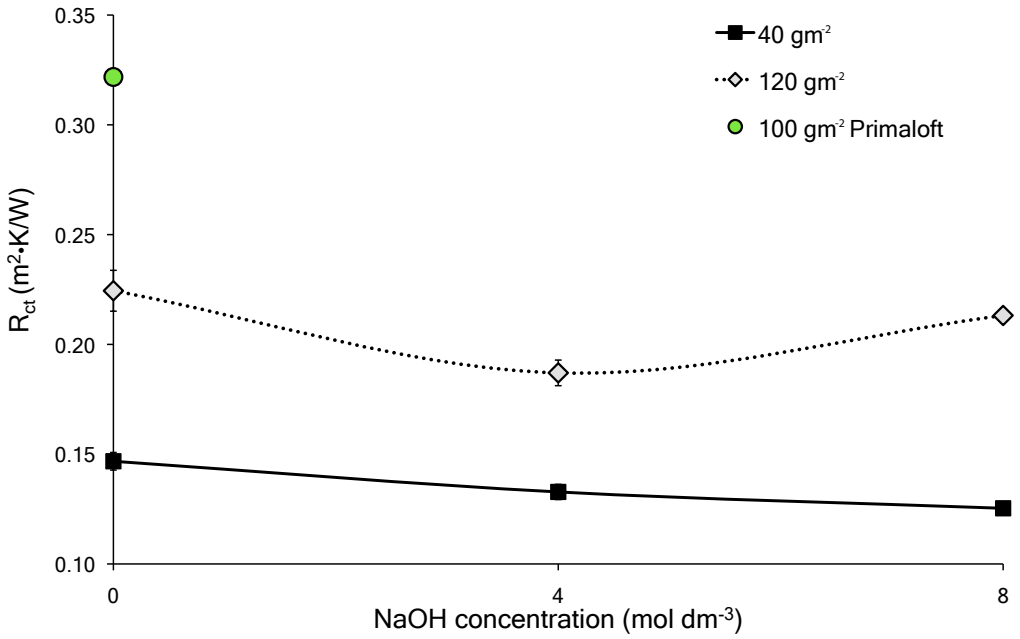

Figure 5.21: Thermal resistance (R_{ct}) of 40 and 120 g m^{-2} alkali pre – treated *G. diversifolia* nonwoven samples and 100 g m^{-2} Primaloft®Sport sample.

The 120 g m^{-2} samples show higher thermal resistance (R_{ct}) than 40 g m^{-2} samples (Figure 5.21). This is expected, as the thermal resistance of a fabric is directly proportional to its thickness (Equation 5.8; Section 5.1.3) [4], [52], [44]. As discussed previously, good thermal insulation of a fabric results from its ability to trap air in its structure. Fibres play an important role in thermal insulation; hollow fibres are better thermal insulators as they could trap air in their structure [44], [48], [90]. The cross – sectional and the lumen area of the fibres pre – treated with 0 mol dm^{-3} NaOH is higher than

the fibres pre – treated with 4 and 8 mol dm^{-3} NaOH (Figure 5.22) allowing a greater thermal resistance. The fabrics manufactured from fibres pre – treated with 4 mol dm^{-3} NaOH have the least thermal resistance amongst the three different treatment concentration; this is expected as fibres pre – treated with 4 mol dm^{-3} have the lowest cross – sectional and lumen area.

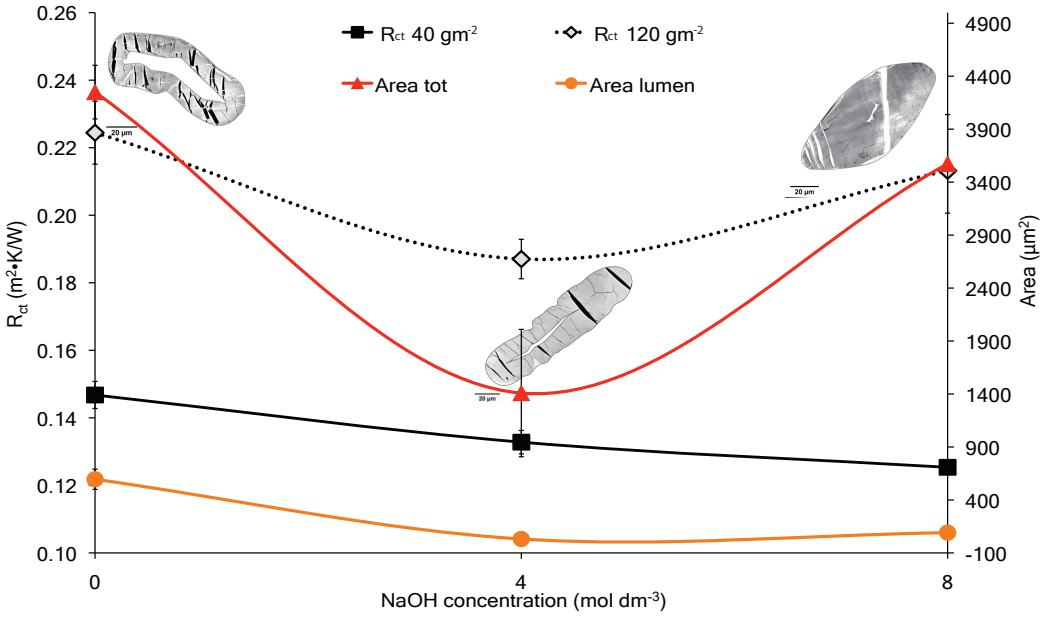

Figure 5.22: Thermal resistance (R_{ct}) and the cross – sectional area (total and lumen area of fibres) of 40 and 120 g m^{-2} nonwoven fabrics manufactured with alkali pre – treated fibres.

It could be seen from Figure 5.23 that the thermal resistance of the alkali treated nonwoven fabrics are inversely proportional to the moisture regain of the constituent fibres. It is in agreement with existing literature where there is clear evidence that increase in moisture regain results in an increase in thermal conductivity and water vapour permeability [91], [92], [93], [94], [95], [96]. Schneider *et al.* [97] demonstrated in their work by analysis of different fabrics (mainly cotton) that thermal conductivity is a function of the moisture regain. Water is 23 times more conductive than air thus increasing the moisture regain increases the conductivity [96], [97], [98], [99], [100].

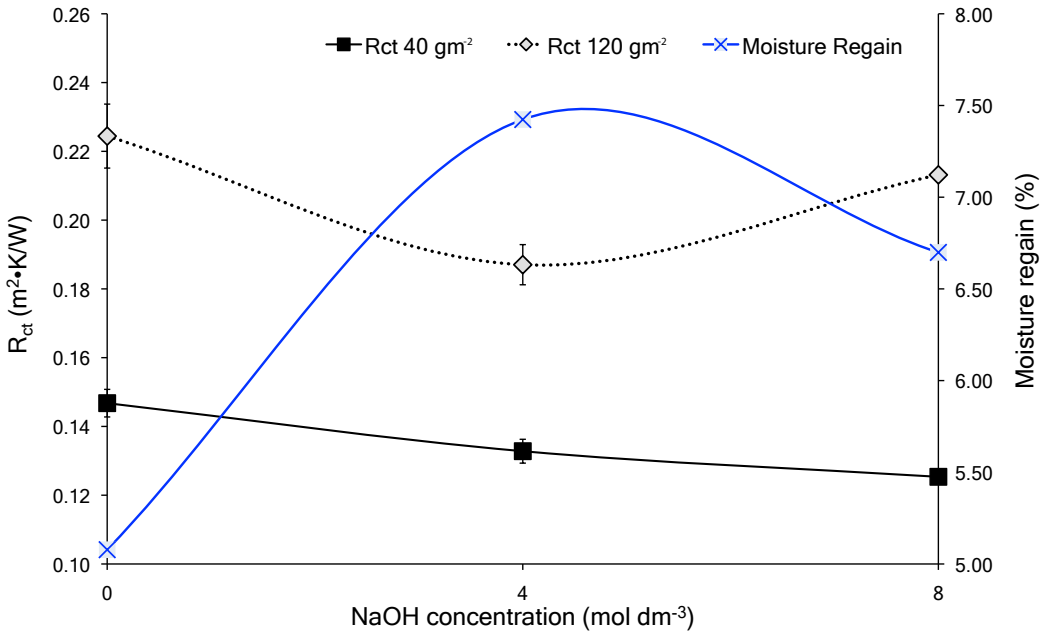

Figure 5.23: Thermal resistance (R_{ct}) of 40 and 120 g m^{-2} alkali pre – treated non-woven fabrics and the moisture regain of alkali treated fibres.

Table 5.2 list different classes of insulating textile materials based on the thermal resistance value [4]; a higher class implies a better insulating material [4]. All 120 g m^{-2} samples give a class 3 material (Table 5.3) implying that these samples are sufficiently insulated to provide protection from cold; all 40 g m^{-2} samples give a class 2 material suggesting the materials could be used as the base layer (next to the skin).

Table 5.3: Classification of the insulating property of 40 and 120 g m^{-2} alkali pre – treated *G. diversifolia* nonwovens and the 100 g m^{-2} Primaloft®Sport sample based on the thermal resistance (R_{ct}).

Class	1	2	3	over
R_{ct} (m^2·K/W)	0.06 – 0.12	0.12 – 0.18	0.18 – 0.25	
40 GSM				
0 mol dm^{-3}		0.15		
4 mol dm^{-3}		0.13		
8 mol dm^{-3}		0.13		
120 GSM				
0 mol dm^{-3}			0.22	
4 mol dm^{-3}			0.19	
8 mol dm^{-3}			0.21	
100 GSM				
Primaloft®Sport				0.32

5.3.5 Breaking force (F_{max}) and elongation (dL at F_{max})

The 120 g m^{-2} samples show higher breaking force compared to the 40 g m^{-2} samples (Figures 5.24 and 5.25); this is an expected result as higher area density implies greater sum of resistances which consists of friction between the materials (the fibres), the number of bonding points, and the amount of fibres [76], [101].

The nonwoven fabrics made with fibres pre – treated with 4 and 8 mol dm^{-3} NaOH gives higher tensile strength compared to fibres treated with 0 mol dm^{-3} NaOH. It is well known that alkali treatment improves the strength of cellulosic fibres [100], [102], [103], [104], [105]. It could also be seen from Figure 5.24 and Figure 5.25 that the samples are anisotropic *i. e.* the strength in MD is higher than the strength in CD; this is an expected behaviour for parallel laid carded samples [71].

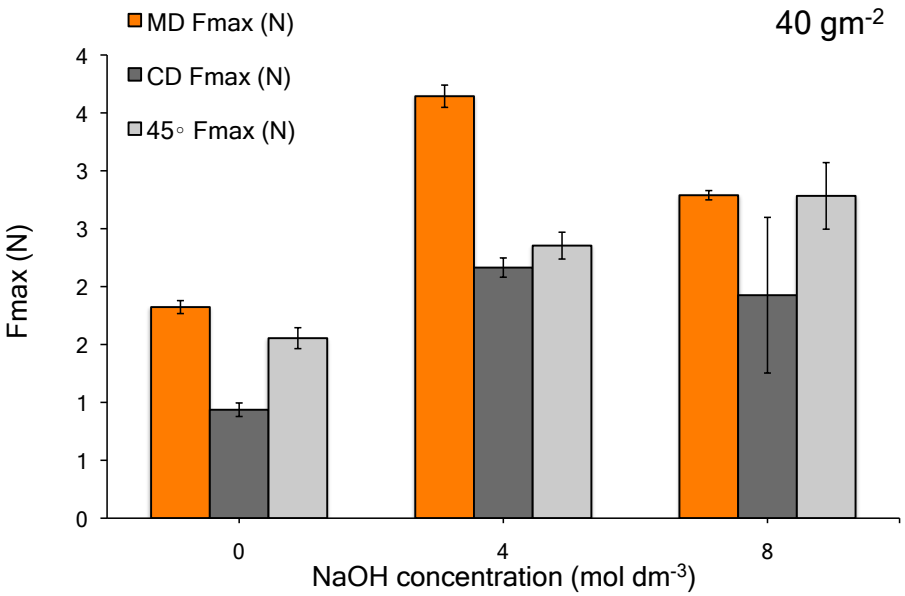

Figure 5.24: Breaking force (F_{max}) in machine direction (MD), cross direction (CD) and at 45 ° of 40 g m^{-2} alkali treated nonwoven samples.

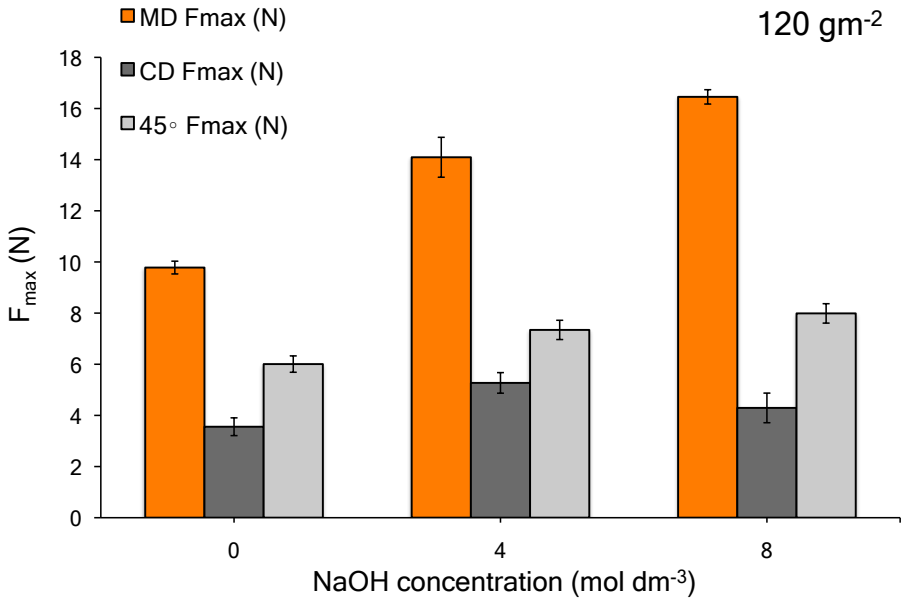

Figure 5.25: Breaking force (F_{max}) in machine direction (MD), cross direction (CD) and at 45 ° of 120 g m^{-2} alkali treated nonwoven samples.

The 120 g m^{-2} samples show slightly higher elongation (%) compared to the 40 g m^{-2} samples with the exception of 4 mol dm^{-3} alkali pre – treatment

concentration in machine direction (Figure 5.26 and Figure 5.27) [71], [106], [107].

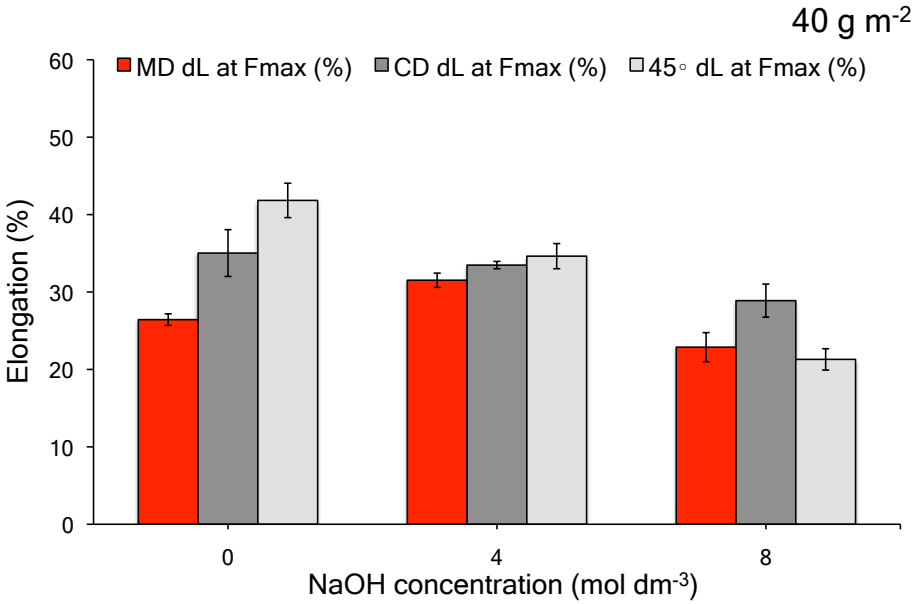

Figure 5.26: Elongation % (dL at F_{max}) in machine direction (MD), cross direction (CD) and at 45 ° of 40 g m^{-2} alkali treated nonwoven samples.

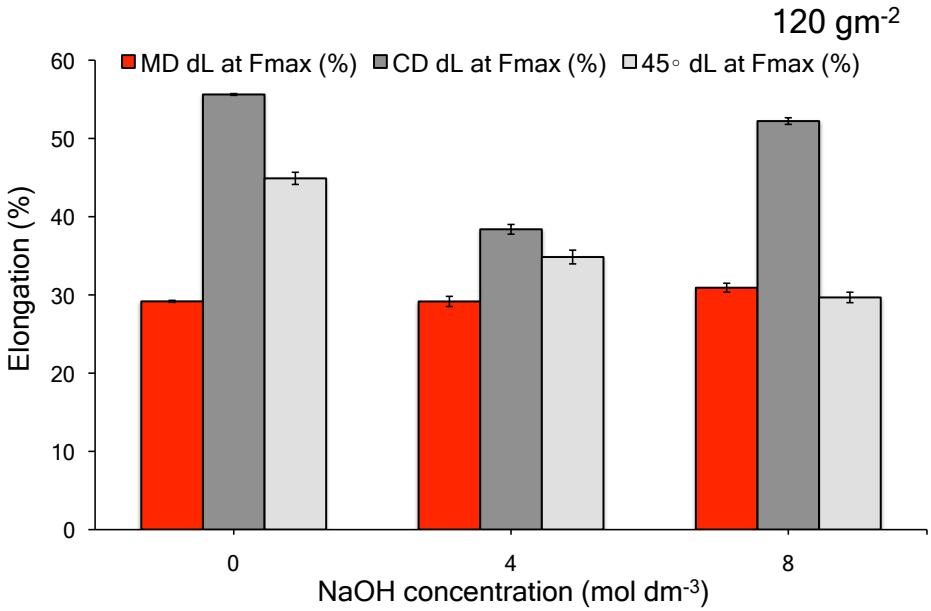

Figure 5.27: Elongation % (dL at F_{max}) in machine direction (MD), cross direction (CD) and at 45 ° of 120 g m^{-2} alkali treated nonwoven samples.

5.3.6 Bending length and flexural rigidity

Bending length measures the stiffness of a fabric that is associated with the draping quality. Flexural rigidity measures the stiffness associated with the handle of the fabric [58].

It could be seen from Figure 5.28 and Figure 5.29 that the bending lengths of 120 g m^{-2} samples are higher compared to the 40 g m^{-2} nonwoven samples. The effect of area density is more prominent with the flexural rigidity results; it could be seen from Figure 5.30 and Figure 5.31 that the 120 g m^{-2} samples show, on an average, approximately 8 times higher flexural rigidity in MD and approximately 5 times higher flexural rigidity in CD, compared to the 40 g m^{-2} samples. This implies that increasing the area density from 40 to 120 g m^{-2} significantly affects the handle of the fabric.

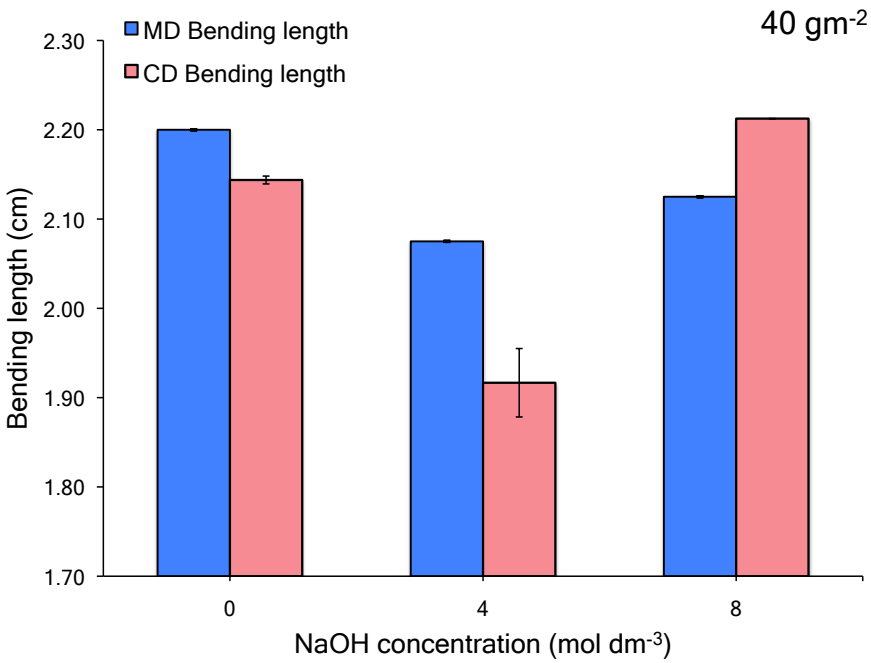

Figure 5.28: Bending length of 40 g m^{-2} nonwoven fabrics pre – treated with NaOH in machine (MD) and cross direction (CD).

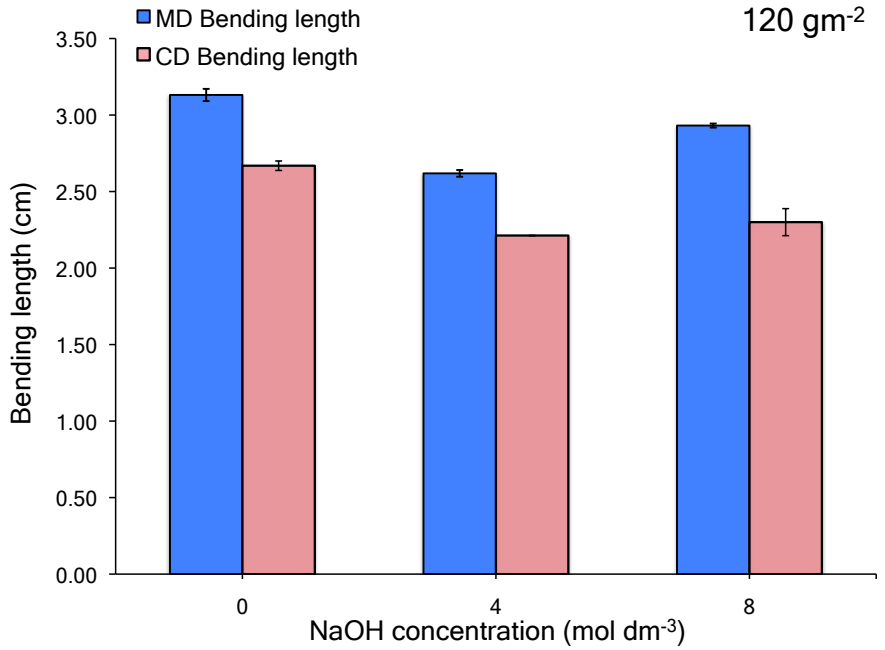

Figure 5.29: Bending length of 40 g m^{-2} nonwoven fabrics pre – treated with NaOH in machine (MD) and cross direction (CD).

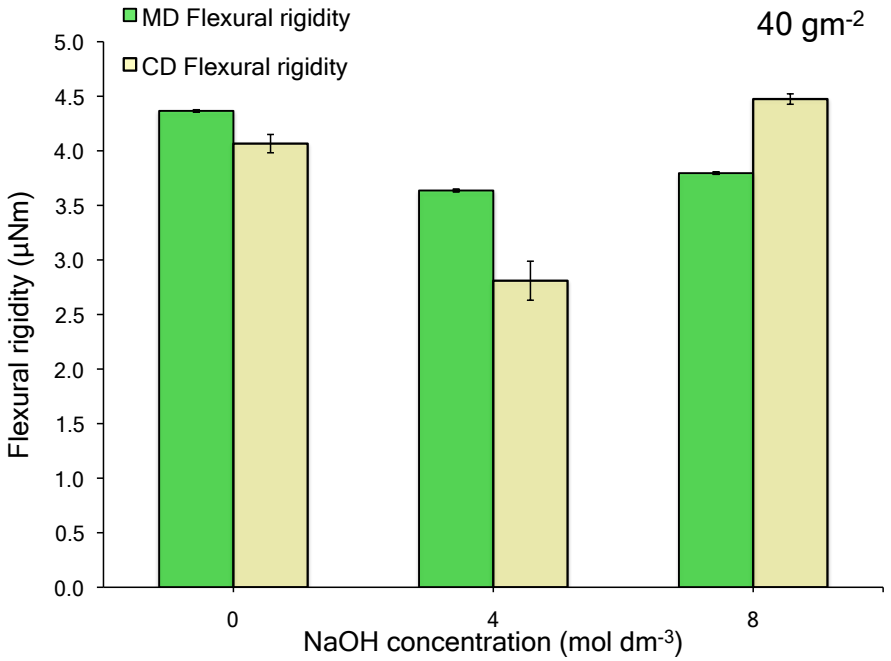

Figure 5.30: Flexural rigidity of 40 g m^{-2} nonwoven fabrics pre – treated with NaOH in machine (MD) and cross direction (CD).

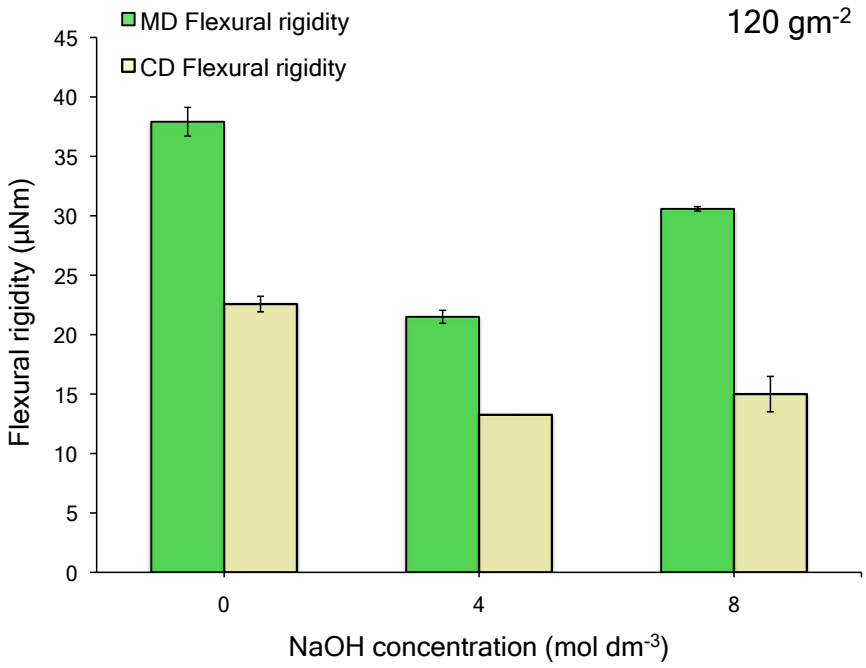

Figure 5.31: Flexural rigidity of 120 g m^{-2} nonwoven fabrics pre – treated with NaOH in machine (MD) and cross direction (CD).

It could be seen from Figure 5.32 to Figure 5.33 that the bending length and flexural rigidity of the nonwoven fabrics correlates with the cross – sectional area (total) of the fibres; as the cross – sectional area increases, the bending length and flexural rigidity also increases. This is in agreement with the work done by Yüksekkaya *et al.* [59]. They observed a second degree relation between the fabric's flexural rigidity and the diameter of the fibres which they are made from; increasing the fibre diameter increases the stiffness of the fabric [59]. 4 mol dm^{-3} alkali pre – treated fibres have the lowest cross – sectional area (total) and therefore fabrics manufactured using these fibres show the lowest bending length and flexural rigidity.

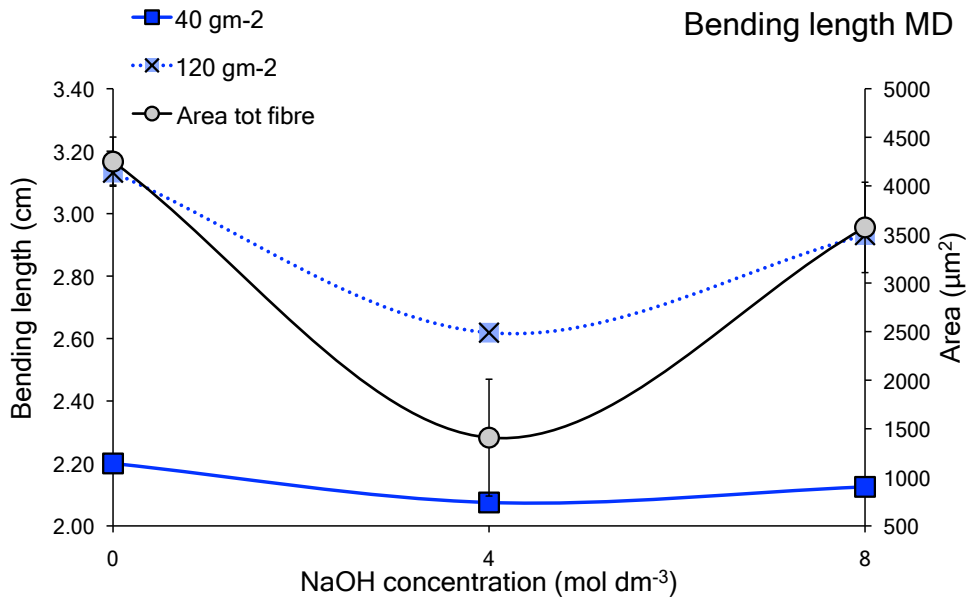

Figure 5.32: Bending length (machine direction) of 40 and 120 g m^{-2} alkali pre – treated nonwoven fabrics and the total cross – sectional area (μm^2) of the alkali treated fibres.

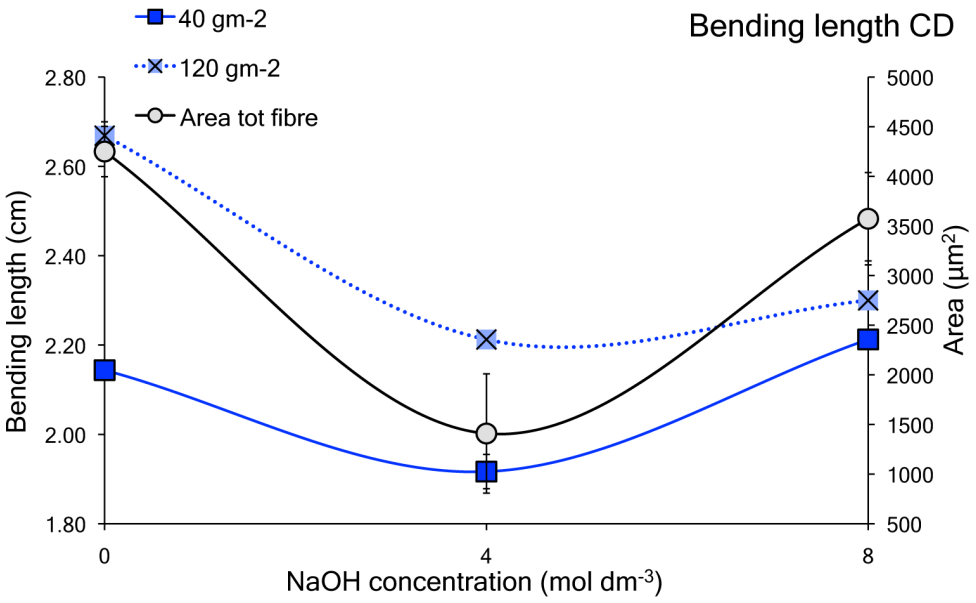

Figure 5.33: Bending length (cross direction) of 40 and 120 g m^{-2} alkali pre – treated nonwoven fabrics and the total cross – sectional area (μm^2) of the alkali treated fibres.

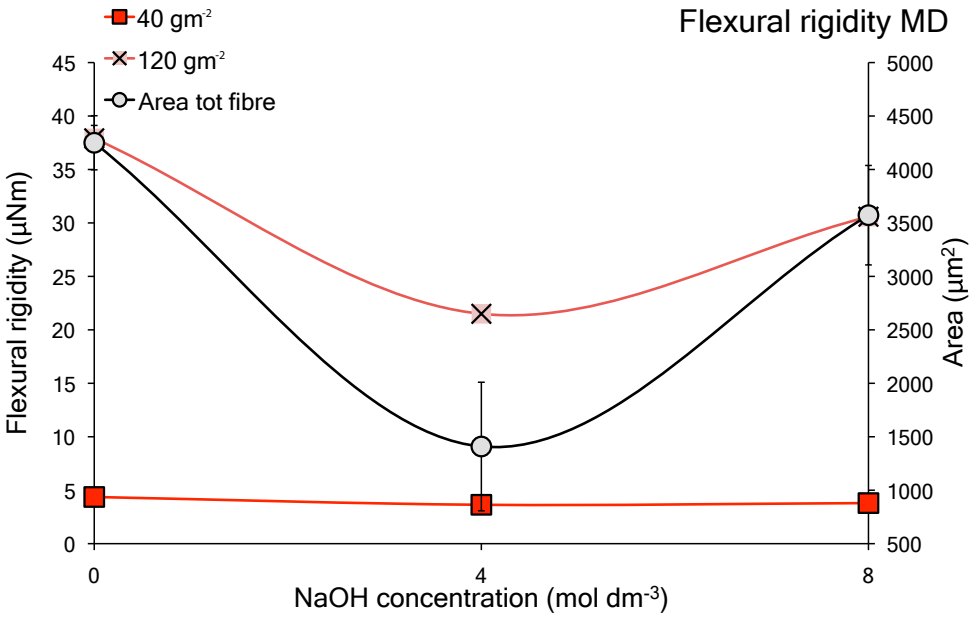

Figure 5.34: Flexural rigidity (machine direction) of 40 and 120 g m^{-2} alkali pre – treated nonwoven fabrics and the total cross – sectional area (μm^2) of the alkali treated fibres.

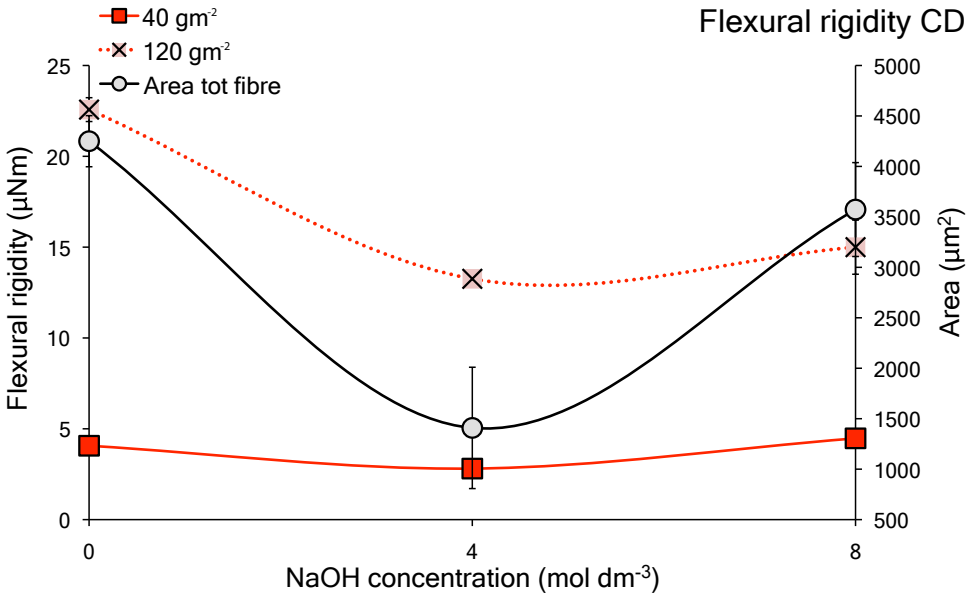

Figure 5.35: Flexural rigidity (cross direction) of 40 and 120 g m^{-2} alkali pre – treated nonwoven fabrics and the total cross – sectional area (μm^2) of the alkali treated fibres.

5.4 Conclusions

Literature suggests that a layered system, either consisting of a single component or by using multiple components, is the best way to ensure a state of comfort in garments used for outdoor sports [1], [2], [3], [4]. Based on the results from Chapter 4, it was proposed that the fibres treated with 4 mol dm^{-3} NaOH would ideal for the base layer, 0 mol dm^{-3} NaOH would ideal for the middle layer, and fibres treated with 8 mol dm^{-3} NaOH would ideal for the shell layer. To check the proposed hypothesis (Section 5.1.4) and to verify if Himalayan nettle fibres could meet the high performance requirements of outdoor clothing, fabrics were made and tested using a nonwoven manufacturing process (carding followed by thermal bonding). It was proposed that the area density of the base layer should be 40 g m^{-2} and the middle and outer layers should have an area density of 120 g m^{-2} (Section 5.1.5).

Experimental results reveal that 40 g m^{-2} nonwoven fabric pre – treated with 4 mol dm^{-3} NaOH shows the highest water vapour permeability amongst all the measured fabrics (Figure 5.17) with acceptable thermal resistance that is required for the base fabric (class II) (Table 5.3). This fabric also has the lowest flexural rigidity compared to all other measured fabrics, implying a superior handle of this fabric (Section 5.3.5); this is an important property for the base layer fabric as it is in direct contact with the skin (Section 5.3.3).

The thermal resistance (R_{ct}) of the 120 g m^{-2} nonwoven fabric pre – treated with 0 mol dm^{-3} NaOH is classified as a class III (Table 5.3) insulating material, implying that these samples are sufficiently insulated to provide protection from cold and this makes it ideal for the middle layer. The WVP values are low (Figure 5.17) and closed to the values of Primaloft®Sport material used in outdoor clothing as a middle insulating layer (Section 5.3.3).

The nonwovens manufactured with 8 mol dm^{-3} NaOH pre – treated fibres show good WVP due to the fibres' high moisture regain and moisture content (Figure 5.19 and 5.19); properties that are ideal for the outer protec-

tive layer. The thermo resistance of this sample is in class III providing effective protection from the cold (Table 5.3).

The nonwoven fabric manufactured with 0 mol dm^{-3} NaOH pre – treated fibres has less tensile strength than fabrics made with 4 and 8 mol dm^{-3} NaOH pre – treated fibres (Section 5.3.5). The 0 mol dm^{-3} NaOH pre – treated fabric sample will be used as the insulating layer and therefore it does not need to have great tensile strength.

This research show that by modifying the Himalayan nettle fibres by NaOH treatment, different functionalities could be achieved; the NaOH pre – treatment changes the physical and chemical properties of the fibre and therefore the resultant fibres could satisfy different functions. The results from this chapter confirms the proposed hypothesis that the fibres treated with 4 mol dm^{-3} NaOH would be ideal for the base layer, 0 mol dm^{-3} NaOH would be ideal for the middle layer, and fibres treated with 8 mol dm^{-3} NaOH would be ideal for the outer layer.

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Chapter 6

Final conclusions and recommendations for future research

6.1 Himalayan nettle (*G. diversifolia*) fibre between Sustainability and High Performance fibres

This research identifies a possible new kind of future textile the Eco + High Performance Fibres, *i. e.* the intersection of two current trends in textile industry: sustainable materials and high performance products. The former corresponds to the growing need for sustainable textile with low impact on the environment; the latter represents the innovation, the answer to the high – end consumer demands. In their intersection the *Girardinia diversifolia* fibre can be seen as a possible solution; this particular plant has on one hand some rather interesting physical and technical characteristics and, on the other social and environmental sustainability; its production makes it particularly attractive from the point of view of technological – social research.

Compared to other Eco – Sustainable artificial fibres, *G. diversifolia* fibre has the advantage of being totally natural: nettle plants do not require chemical treatments to grow and develop; the extraction of the fibre takes place with totally natural methods (with no use of harmful substances for the environment and for humans). Even compared with its closest relative, the common nettle (*Urtica dioica*) which grows in Europe and Asia, *G. diversifolia* fibre presents many advantages in terms of sustainability:

- Its cultivation does not require waste water;
- Its growing season is much longer than *U. dioica*;
- The companies currently working for the industrial and economic development of the areas where the fibre grows, guarantee adequate contractual conditions to workers and trade only with buyers interested in a eco – sustainable product.

G. diversifolia fibre so far is only used for craft goods; its suitability for the textile industry has not been investigated. Certain characteristics make *G. diversifolia* fibre interesting for industrial purposes [1], including biodegradability, socially responsible production, fibre length, resistance to wrinkling, potential thermal insulating properties, antimicrobial, antibacterial and fire retardant.

Herein, an investigation of the fibre was undertaken. For the first time an analysis of the raw fibre (as extracted from the plant bark) and scoured fibre, is provided to describe its physical characteristics and mechanical properties. Alkali treatment with sodium hydroxide (NaOH) in concentrations from 0 to 8 $\mathrm{mol\,dm^{-3}}$ was performed in order to understand if the change and new orientation of the cellulose molecular chains could affect / improve the physical characteristics and the mechanical capabilities of the fibre. The most interesting results were observed by an original comparison of the TCI, LOI and HBI trends, obtained with ATR – FTIR spectroscopy, of the fibres treated with various concentrations of NaOH and the results of mechanical tests and fibre cross – sectional changes. The data obtained from the analysis of *G. diversifolia* fibre

demonstrated that at increasing NaOH concentrations, the molecular structure changes from Cellulose I to Cellulose II, developing different physical features and mechanical properties in the process.

This ability for fibre properties to change with a chemical treatment demonstrates an interesting fibre property: versatility. This is one of the most promising results: treating the fibre with different concentrations of sodium hydroxide may provide characteristics suitable for different fields in the high performance textiles. The problem of high – performance textiles is that they consist mainly of fibres derived from non – renewable resources, therefore they cannot be disposed or recycled easily. In addition, the high – performance fabrics are made from an assemblage of many synthetic fibres, which effectively meet different functions and which makes their recycling complex. To demonstrate *G. diversifolia* fibre versatility and its potential in the textile, according to the Pareto's law [2], the fibre should be used in a niche area where the price is justified by the performance provided. The field of outdoor sportswear, where a high level of comfort is required, has been chosen to demonstrate the performance of *G. diversifolia* fibre. Comfort in this case means resistant material, great wearability and ability to keep the body dry and at constant temperature. A multilayer fabric was hypothesised consisting of three nonwoven layers made with *G. diversifolia* fibre treated at three different concentrations of NaOH and with a different molecular structure. The nonwoven technology has been employed since its effectiveness achieved excellent results in ever more fields, it is a fast technology as it requires no special preliminary treatment and it allows an immediate comparison of the fibre characteristics. The nonwoven samples were tested as part of a system of three – layer fabric. The results reflected the fibre properties in the fabrics and they appear promising to expect performance and eco – friendly materials by *G. diversifolia* fibre. This chapter summarises the values that describe raw *G. diversifolia* and *U. dioica* fibre, and the comparison of the molecular structure with the physical and mechanical properties of the fibres treated with NaOH; the results obtained by the tests of *G. diversifolia*

nonwovens demonstrate the fibre ability to maintain a state of comfort in the sports field. Examples of how the fibre can perform in the final products are provided as a motivation for further investigations and research inspired by this study.

6.2 Final conclusions

In Chapter 3 a comparative investigation on raw and scoured fibres of both *G. diversifolia* and *U. dioica* was performed. The analysis shows that both the above nettle fibres have a larger cross – section than the other common bast fibres, close to the size of hemp and jute. The cross – section increases considerably when the fibres are scoured because of the swelling effect. In particular the Himalayan nettle fibre has a wider lumen. *G. diversifolia* is the longest bast fibre known so far. The moist and shady climate of the Nepalese forests allows to obtain longer fibres than those extracted from *G. diversifolia* plants cultivated in Europe.

The Himalayan nettle fibre is superior in mechanical properties compared to European nettle and other bast fibres. Scouring decreases strength of both nettle fibres. As well the Young's modulus is higher for raw nettle fibres and decreases substantially in the scoured being inversely proportional to the cross – section fibres. *G. diversifolia* fibre has a Young's modulus higher than the *U. dioica*.

The moisture content of raw and scoured *G. diversifolia* fibre is lower than those of the *U. dioica* fibre and other bast fibres. A low moisture content (less than 10 %) indicates good dielectric insulating capacity of the material. The moisture regain of both fibres, *G. diversifolia* and *U. dioica*, is in a range of values that does not influence their physical stability. In particular the low moisture regain of *G. diversifolia* fibre means a higher stability of the fibre to humidity. The moisture regain of the scoured nettle fibres slightly decreases with scouring process.

The mechanical and physical properties of the fibres are affected by the molecular chains orientation. The study, by ATR – FTIR, of the TCI, LOI and HBI trends, describes qualitative molecular arrangements. When these trends are compared with mechanical properties *G. diversifolia* fibre shows, by TCI and LOI, to be highly crystallinity and to have greater strength and low capability to absorb moisture. *U. dioica* fibre is less crystalline and its amorphous regions interacts most readily with water. The trend of HBI is lower for Himalayan nettle fibre than European one. Scouring process, however, allows a slightly increase of HBI trend for Himalayan nettle fibre, while a sharp increase occurs for the European nettle fibre. Both nettle fibres are highly orientated in the molecular chains, even when scoured, as shows by the birefringence analysis.

The first analysis of both raw nettle fibres showed that they are highly resistant, in particular *G. diversifolia* fibre, thanks to their high crystallinity.

In Chapter 4 an investigation was carried on the *G. diversifolia* and *U. dioica* treated at increasing concentration on sodium hydroxide (NaOH). It was observed that the treatment reduces the fibre crystallinity at high NaOH concentration allowing the transformation from Cellulose I to Cellulose II. The transformation of cellulose I to cellulose II occurs between 2 and 4 mol dm^{-3} NaOH when a significant LOI reduction and appreciable changes in HBI take place. The complete transformation into cellulose II takes place above 5 mol dm^{-3} NaOH, where no further crystallinity change was observed.

The ATR – FTIR analysis has showed that during the alkali treatment the values of HBI increase with the elongation of the fibre, while LOI decreases.

By DSC analysis it observed that the endothermic peak trend grows in accordance with the HBI values at higher concentration of NaOH. At 6 mol dm^{-3} and 4 mol dm^{-3} NaOH, respectively for *G. diversifolia* and *U. dioica*, loss in cellulose crystallite length occurs. Both nettle fibres have high thermal resistance as decomposition peaks are in the range of 360 – 380 °C, higher than the most common bast fibres.

From the birefringence (Δn) analysis it is demonstrate the both nettle

fibres are very oriented more than perfectly oriented cellulose. *U. dioica* has an orientation factor f greater compared to *G. diversifolia* fibre.

The alkaline treatment with NaOH influences the mechanical properties of the nettle fibres. Tensile strength values decrease compared to the values reported for the raw fibre, due to the decrease in crystallinity. Although *G. diversifolia* shows values very close to raw fibre because, being highly crystalline, NaOH acts with more difficulty. The elongation increases at high NaOH concentration, according with the HBI trend, conferring greater flexibility to the fibre. The Young's modulus analysis, in fact, remarks the trend of the LOI and is inverse to that of HBI. At increasing NaOH concentration, fibres lose crystallinity reducing the fibre stiffness.

The moisture regain is proportional to the percentage of the amorphous region of the fibre. *G. diversifolia* and *U. dioica* moisture regain is opposite to the LOI trend, increasing with NaOH concentrations. The percentages of moisture regain of both fibres, are in a range that does not compromise the fibres physical stability when exposed to moisture. *G. diversifolia* fibre is less influenced by humidity than *U. dioica* fibre. *G. diversifolia* has also low moisture content indicating that the fibre is an effective dielectric insulating.

Alkaline treatments gives to the fibre a more regular cross – sectional shape. *G. diversifolia* and *U. dioica* increase its cross – section at 5 mol dm^{-3} and 3 mol dm^{-3} NaOH respectively, when the Cellulose conversion from I to II is ongoing.

The *G. diversifolia* versatility is one of the most promising results which makes it a very promising sustainable candidate for niche area of textile. Treated with different NaOH concentration, the fibre develops characteristics capable of satisfying different functions as in the high performance activities where a combination of waterproofness, breathability and resistant to abrasion is required.

In the Chapter 5 the characteristics of the *G. diversifolia* treated at different NaOH concentrations are analysed to demonstrate how it can meet the needs of comfort that provides a three – layer fabric made mostly of synthetic

and high performance fibres. The three layers provide respectively breathability and wearability, thermal resistance, resistance and protection from external factors. Based on the results from Chapter 4, it was proposed that the fibres treated with 4 mol dm^{-3} NaOH would be ideal for the base layer, 0 mol dm^{-3} NaOH would be ideal for the middle layer, and fibres treated with 8 mol dm^{-3} NaOH would be ideal for the outer, shell layer. The verification of the proposed hypothesis was tested by processing the pre – treated Himalayan nettle fibres in nonwovens with an area density of 40 g dm^{-2} for base layer and 120 g dm^{-2} for the middle and outer layers.

The 40 g dm^{-2} nonwoven fabric pre – treated with 4 mol dm^{-3} NaOH showed the highest results of water vapour permeability amongst all the measured fabrics and a thermal resistance required for the base fabric (class II). The lowest flexural rigidity of this fabric compared to all other measured fabrics, implying a superior handle, important factor being the base layer in direct contact with the skin.

The 120 g dm^{-2} nonwoven fabric pre – treated with 0 mol dm^{-3} NaOH are classified in a class III of thermal resistance (R_{ct}) ideal for the middle layer to provide protection from cold. The WVP values are low as those of the materials used in outdoor clothing as a middle insulating layer. The flexural rigidity of this sample is high but not relevant in an intermediate layer.

The nonwovens manufactured with 8 mol dm^{-3} NaOH pre – treated fibres show suitable permeability (WVP) because of the high moisture regain and content of the fibres. These properties, ideal for a shell layer, allow to the fabric not to attract the environment humidity and leave the sweat evaporate outside. The fabric also in class III of thermo – resistance, provides effective protection from the cold.

The strength of the tested nonwoven fabric is low for those manufactured with pre – treated 0 mol dm^{-3} NaOH and higher for others made with 4 and 8 mol dm^{-3} NaOH. The pre – treated 0 mol dm^{-3} NaOH fibres are used to make the insulating middle layer that does not need great resistance. The base

layer and shell layer on the contrary, require a higher resistance, as to obtain respectively ergonomic cuts and complex protection structures, the manufacturing of the fabrics requires more stress.

This research shows that, modifying the Himalayan nettle fibres with NaOH treatment, different functionalities could be achieved. In particular the same fibre, treated with different concentrations of NaOH, can meet different levels of comfort required in 3 layers outdoor sport clothing. As expected in this Chapter 5 was verified the hypothesis that the properties of the treated Himalayan nettle fibres are reflected in the the nonwovens made from it. Fibres treated at 4 mol dm^{-3} NaOH allow to obtain breathable fabric, at 0 mol dm^{-3} NaOH insulated and at 8 mol dm^{-3} shell, as summarizing in the Figure 6.1. The potential to satisfy different functions with a single fibre makes the Giant Himalayan nettle fibre a sustainable candidate in the high performance field.

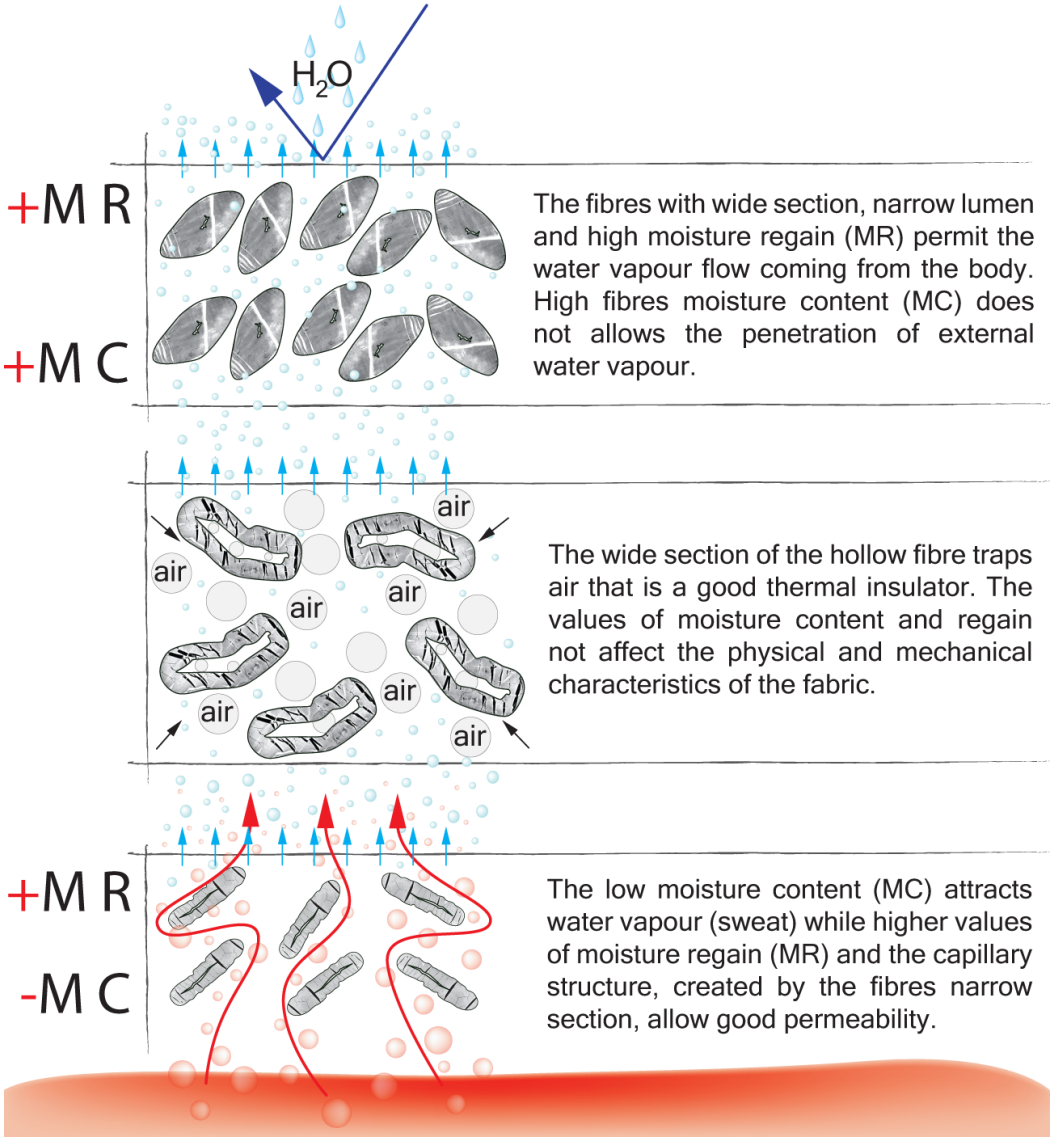

Figure 6.1: Summary of a three layered system made with nonwovens manufactured of 0, 4 and 8 mol dm^{-3} NaOH pre – treated *G. diversifolia* fibres.

6.3 Suggestions for further work

G. diversifolia fibre has not been widely investigated; literature is very limited and its use, until now, has been exclusively in handicraft. Recently, thanks to the interest of some NGOs, has been re – evaluated and its features have appeared attractive to textile. The research herein has provided *G. di-*

versifolia fibre characterisation investigating physical and mechanical properties in order to verify its suitability as industrial fibre. No future investigations are proposed with regard to the extraction, mercerisation, scouring and fibre treatment in a sustainable way as these areas are already underway by biological and textiles researchers of Nepalese and Canadian universities [3]. For completeness of investigation to this research, which adds knowledge to the academic and industrial community, additional mechanical tests are recommended. Being known that the natural fibres, such as cotton, linen and other bast fibres increase strength when wet, while others like wool, silk and viscose lose it, despite *G. diversifolia* fibre showed a good stability to moisture, it is considered useful to compare the strength and the elongation of wet and dry nettle fibres of both species studied [4].

From a previous study carried out by the author on *G. diversifolia* fibre [5], a preliminary analysis regarding exhaustion (E) of dye adsorbed onto the fibre demonstrated a high capacity of the fibre to be dyed. These results do not seem to agree with the highly crystalline molecular structure of *G. diversifolia* fibre, for which the high values of E can be attributed to the structure of the fibre itself. An analysis in this direction looks interesting considering also the molecular and morphological fibre changes that occur during NaOH treatments.

The high resistance of the fibre at high temperatures was supposed observing the decomposition point during DSC analysis. Further investigation of high temperature resistance and flame retardant of the fibre by limiting oxygen index (LOI) test are suggested, in particular with reference to its applications in technical clothing, in house building and automotive field.

The opportunity of a wider investigation, considering the length and strength of the fibre, involves its processing, both in the case of spinning (then weaving and knitting) that of nonwoven technologies. Enterprises [6], [7], [8], [9] green oriented expressed interest about the performance of the yarn in blend denim with cotton and others. Fibres such as *U. dioica*, must be used in high percentages to be enough resistant in weaving process. The tests for light

denim show that a low percentage of European nettle fibre does not work while it is assumed that using Himalayan nettle fibre, as its high strength, excellent results can obtained [6], [10]. Spinning attempts with a blend of *G. diversifolia* and other fibres is already underway by some companies [3].

It is suggested to consider the characteristics of *G. diversifolia* fibre when treated with different NaOH concentrations, in order to obtain a highly resistant yarn for complex machining as honeycomb for protective layers. In the growing and increasingly eclectic nonwoven area, research directions are manifold: testing the *G. diversifolia* fibre performance using various nonwoven technologies; studying the structure and parameters to be obtained nonwovens for specific purposes; exploring possible blends with other fibres and binders. Referring in particular to this work, a thorough study is recommended for thermal insulating fabrics, as the *G. diversifolia* nonwoven showed excellent ability. The analysis of the texture of a lightweight and breathable nonwoven is also interesting, pursuing compatibility with the body: softness, ergonomics (elasticity) and hypoallergenicity. As suggested by the work of Feredova *et al.* [11], a study of the crystallinity of the *G. diversifolia* fibres extracted from the thermally bonded nonwoven and a comparison of them with the mechanical properties of the fabric should be performed. By the potential of *G. diversifolia* fibre demonstrated so far, and the wide spectrum of investigations and directions that it is possible to undertake, some examples of high performance applicability and lateral thinking research are proposed in the following sections.

6.4 Proposals for Himalayan nettle fibres product design application

Here are some examples of product designs that emphasises performance and sustainable qualities of Himalayan nettle fibre and that are the consequence or the incentive to further depth studies.

6.4.1 Proposal 1: Eco – sustainable insole

The ecological value of the *G. diversifolia* fibre is demonstrated by the use of every part of the plant. *G. diversifolia* fibre extraction involves minimal waste: residues resulting from dried fibres may be used to build an insole composed by an inner layer of dry waste (for thermal insulation) and an outer layer made of *G. diversifolia* fibre treated with a NaOH concentration such as to confer a very thin cross – section able to attract and permeate moisture quickly in order to obtain a very breathable fabric [12], [13]. A design of what this might look like is shown in Figure 6.2.



Figure 6.2: Proposal for applications of *G. diversifolia* fibre – thermal insulation – Insole.

6.4.2 Proposal 2: Nordic walking shoe

About 6 million people in Europe practice Nordic walking [14]. Trekking shoes ensure internal comfort, non – permeability, and protection from cuts

and abrasion. Indeed, when a person moves into cold climates, it is important to avoid the presence of internal humidity in the shoes since at extremely cold temperatures sweat may freeze reducing the insulating properties of the shoes [15]. In fact, even with a good pair of boots, but wrong socks, one could be at risk [16]; this is why dried organics, such as hay and moss, have always been used as insulation before the advent of modern, synthetic fibres [17]. Technical shoes for trekking should ensure not only internal comfort for the foot, but also non – permeability and protection from cuts and abrasions [13], [18], [19].

G. diversifolia fibre could be used for all these functions: for the inside fabric, using the fibres treated at a concentrations that confer a large cross – section (*i. e.* leaving the hollow fibre, capable of retaining air and therefore being an excellent insulator). For the outside one the fibres must possess a strong resistance to be worked using the most modern technologies (processing 3D – Honeycomb) (see Schoeller®[20]) to make the shoe abrasion resistant and waterproof. A design of what this might look like is shown in Figure 6.3.

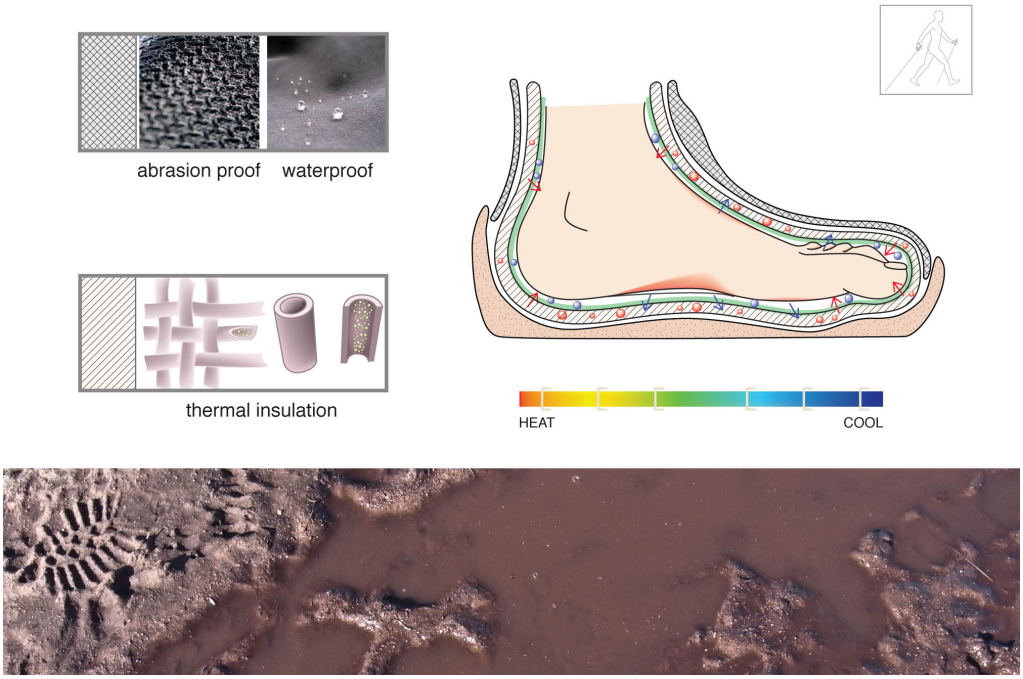

Figure 6.3: Proposal for applications of *G. diversifolia* fibre – thermal insulation, abrasion resistance and waterproof – Technical Shoe.

6.4.3 Proposal 3: Runner t – shirt

During sport activities, the body increases its inner temperature and to compensate start to sweat [21]. To ensure the right comfort, the material that is in direct contact with the skin must be able to evaporate water whilst remaining dry. In practice, this material should be capable of transporting moisture away from the skin to the outer layer and quickly disperse it [22], [23].

For an ergonomic and thermo – physiological wear comfort a material could be designed with two different types of fibre, one not affected by moisture and another absorbent and permeating. Working together with seamless technology it is then possible to obtain clothing that have alternative breathable and insulated areas [24]. This type of material would be the ideal clothing for sports such as running and fitness (respectively practiced by 36.2 and 37.9 million people in the USA alone [23]), and also for sports where there are physical contrast such as football, rugby, basketball and martial arts exploiting the exceptional strength of *G. diversifolia* fibre. A design of what this might look like is shown in Figure 6.4.

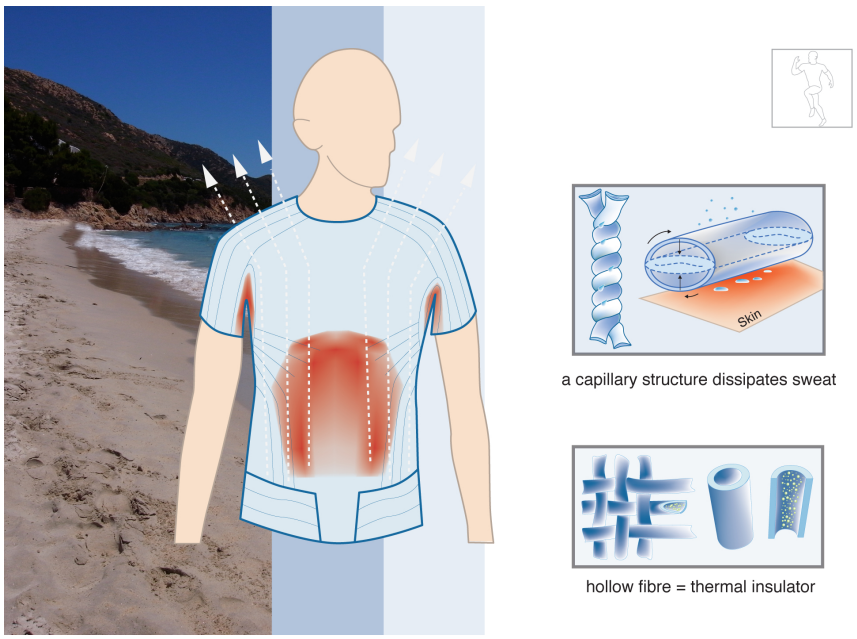

Figure 6.4: Proposal for applications of *G. diversifolia* fibre – breathability and wearability – T – shirt.

6.5 Lateral thinking research

This research aims to demonstrate that it is possible to obtain High – performance products from a single material (the *G. diversifolia* fibre) to fulfil different functions and permit a sustainable disposal. Today, carbon fibres, aramid fibres, fluorine, boron, polyacetal, and other are known as super fibres. Long and thin, these fibres form high – performance fabrics, resistant to abrasion and fatigue. Natural fibres are rarely used when there is an extraordinary demand for resistance, while responding excellently to the need for comfort, hypoallergenicity and eco – compatibility. *G. diversifolia* fibres, long, low sensitivity to moisture, with good strength and elongation at break, originate a new group, parallel to the existing high performance synthetic fibres, named *natural super fibres*, usable in various sectors.

G. diversifolia fibres are very strong and treated at different concentrations of sodium hydroxide increase some of their characteristics rather than others. Working together the fibres treated with different NaOH concentrations, for example with 3D technologies, complex fabrics can be designed for specific applications of technical textiles (Geotech, Buildtech, Indutech, Mobiltech, Medtech, Protech, Sporttech, Clothtech).

G. diversifolia fibre is highly durable, and is the most suitable of the bast fibres to achieve high performance. At this point the research is open to possible directions keeping the aim to demonstrate that this fibre can, thanks to its characteristics, be transformed into an innovative and environmentally friendly product. An interesting option is to obtain from a single material, *G. diversifolia* fibre, a multifunctional product, *e. g.* breathable and insulating at the same time. It controls the humidity acting as the plants themselves, opening and closing depending on the water pressure change in the cells (stomata) and the sensitivity to sunlight [25]. The inspiration for a similar design is represented by imagining a 'living fibre', which acts as if it were still in the stem of the plant, as demonstrated in Figure 6.5, where the fibre reacts to body temperature by

opening and closing becomes breathable or insulating. Today there are about 500 phase changes materials (CPMs) made of natural or synthetic fibres, distinguished by the temperature at which the phase change takes place and for their ability to store or release heat [26]. Similar studies that mimic mechanisms found in nature, about the phase change, are made by some companies, such as Swiss Schoeller Textil of Sevelin [20], with the goal of creating products with eco – sustainable membranes avoiding the use of highly toxic substances such as fluorochemicals (See C_change™membrane [20]).

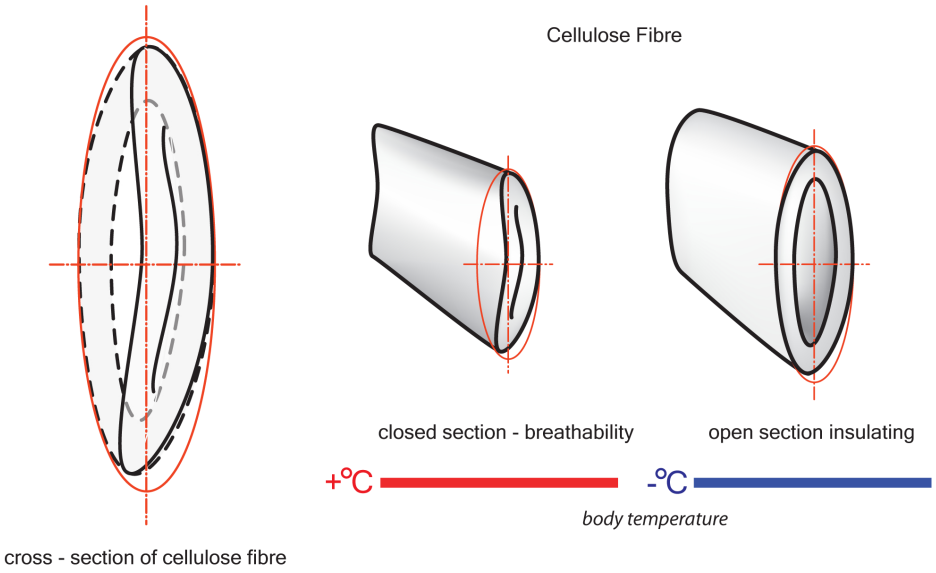

Figure 6.5: The fibre reacts to body temperature by opening and closing becomes breathable or insulating.

Here are proposals to study how to achieve memory shape cellulose fibres. A traditional method is the coating of the fabric with a thermally sensitive finish; the sheath binds to the functional groups outside of the cellulose and reacting to heat or cold, from the external environment, creates a gap, broader or narrower, between it and the fibre where the air or water vapour go through (as demonstrated schematically in Figure 6.6). This technology may not be suitable for textiles that are in contact with the body as increasing the surface of the fibre next to the skin the breathability decreases.

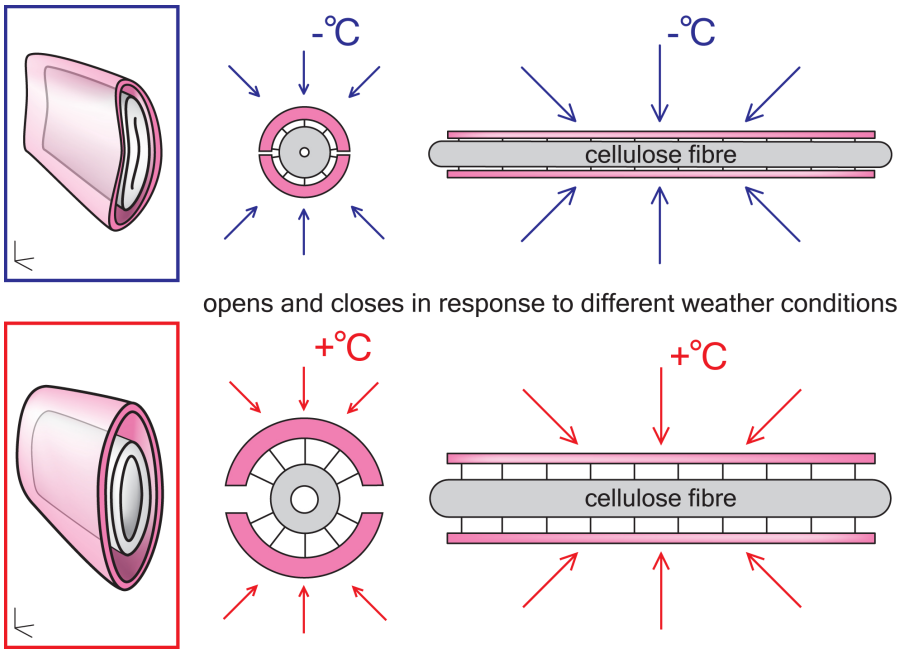

Figure 6.6: The material covering the fabric is thermally sensitive and reacts to environment temperature changes creating a interspace between it and the fibre surface.

An organic highly thermo – sensitive molecular entity could react with the functional inter and intramolecular groups of the cellulose fibre creating a natural and inner movement of the fibre structure when the temperature varies. The cellulose that composes the fibres would be enriched by functional groups (R; Figure 6.7). The developed cellulose – R matrix would have new capacities undergoing a thermo – shape change. This method mimics the nature and is ideal for fabrics next to the skin as maintains their lightness and softness.

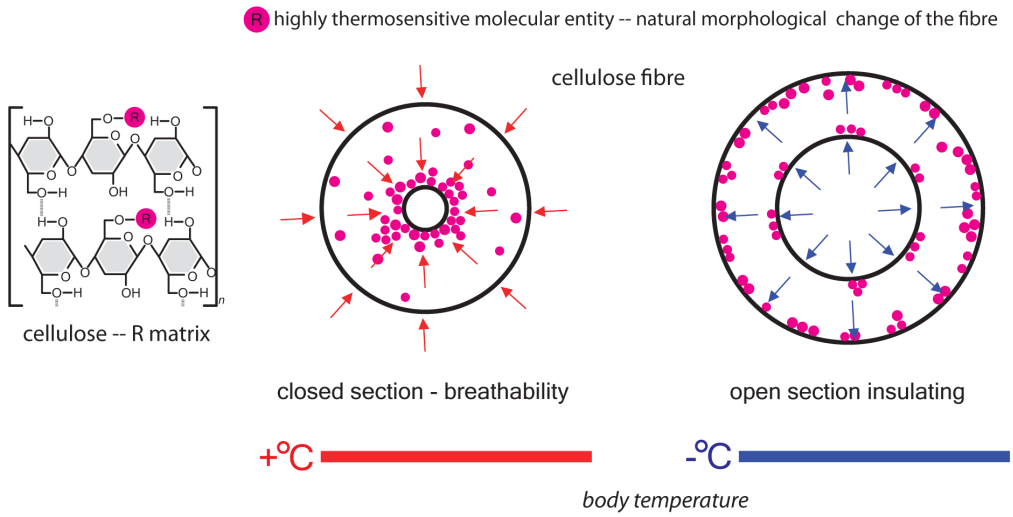

Figure 6.7: The cellulose is converted into cellulose R – matrix, where R is a highly thermo – sensitive molecular entity able to create change in fibre morphology due to the reaction with the heat radiated from the human sweat.

The ability of the *G. diversifolia* fibre to incorporate microcapsules is interesting to investigate. The inserted material may undergo a phase change (as shown for example in Figure 6.8), which solidifies with the cold creating a rigid and protective layer, and melting and softening with heat allowing the passage of air and water vapour. (See the project Comfortemp®[27]).

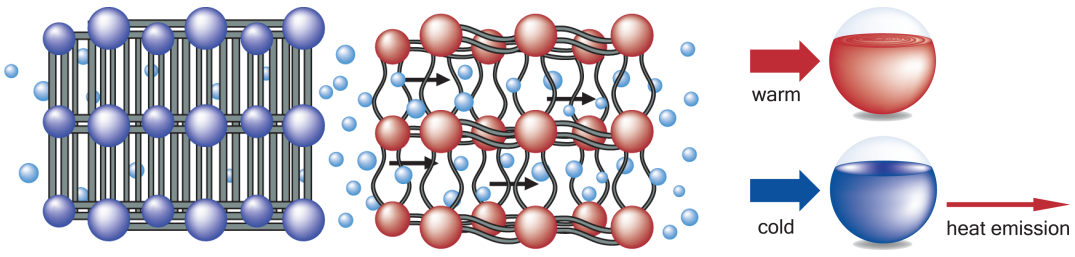

Figure 6.8: Examples of technologies to obtain temperature – sensitive materials: Brownian motion [28] and CPMs paraffin capsules [20], [26].

Experimentation of the *G. diversifolia* ability, and of the fabrics made with it, to host micro capsules is very versatile. Not only phase – change

materials, but also elements that help the fibre to maintain a structure turgid and lubricated as able to rake in a considerable number of water molecules. These element, such as hyaluronic acid, would retain the tonicity and create a filter against the free diffusion into the fabric of particular substances (bacteria, infectious agents). Fabrics with these properties could be used in the medical field, for example, where needed a cushion between a limb prosthetic and the skin. The encapsulated substances could be released on the body or objects depending on use.

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Proceedings

Publications:

'Himalayan nettle (*Girardinia diversifolia*) fibre: investigation of its morphological characteristics and mechanical properties compared with European nettle (*Urtica dioica*) fibre to establish its applicability in high performance textile'

G. Lanzilao, R. S. Blackburn, P. Goswami

A draft article concerning the chapters 3 and 4, is ready to be submitted to the Journal *Biomacromolecules*

Oral presentation and proceeding:

'Properties and Applications of Himalayan Nettle Fibre'

G. Lanzilao, R. S. Blackburn, P. Goswami

13th Asian Textile Conference

November 3th to 6th 2015, Geelong, Victoria, Australia.

'A comparative study of Himalayan nettle (*Girardinia diversifolia*) and European nettle (*Urtica dioica*) as sustainable fibre for high performance garments'

G. Lanzilao, R. S. Blackburn, P. Goswami, H. El – Dessouky

14th National & 1st International Recent Developments, Textile Technology and Chemistry Symposium

May 8th to 10th 2013, Bursa, Turkey.

Award: School of Design – University of Leeds Scholarship.

Poster:

'Himalayan Nettle (*Girardinia diversifolia*) as Sustainable Fibres for High Performance Garments'

G. Lanzilao, R. Blackburn, P. Goswami

EDANA^{TM} – NONWOVENS^{TM} INNOVATION ACADEMY

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Award: EDANA^{TM} Scholarship.