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A proposed methodology for the assessment of beauty and personal care products in a consumer relevant environment

Phoebe-Claire Bramley

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Abstract

Consumer preferences of beauty and personal care (BPC) products are driven by the sensations they evoke both during and after-use. However, the underlying factors that trigger these sensations and how they can be anticipated early in the product development process remain unclear.

This study investigates methodologies for measuring variables associated with the application of BPC products and their influence on tactile perception. This involved studying the effect of products with various rheological characteristics on the frictional behaviour of human skin, to inform on the development of a synthetic skin test-bed. Results throughout were related to sensory attribute scores obtained from a sensory panel which was conducted prior to this PhD by Unilever.

It was found that amongst the attributes rated during product application, *Spreadability* and *Wetness* were significantly negatively correlated to the instantaneous viscosity maximum and yield stress of the products, whilst *Creaminess* was significantly positively correlated to their thixotropic characteristics. Additionally, significant positive correlations were identified between measurements related to the frictional behaviour and the adhesion of skin, and attributes *Sticky Tacky* and *Greasiness*, rated immediately and 5 minutes after application. It was also found that the attribute *Drag* was significantly negatively correlated to the viscosity and the adhesion of the BPC products. Furthermore, the synthetic skin test-bed developed was successful in replicating the frictional behaviour of human volar forearm skin immediately and 5 minutes after the application of three distinct BPC products. Lastly, a deeper understanding was achieved of the different roles played by finger-pad characteristics and volar forearm skin deformation on the perception of various attributes.

These findings suggest that the methodology developed could be used in the preliminary screening of BPC products, although more testing is required to validate it.

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Nomenclature

BPC: Beauty and personal care (products)

COV: Coefficient of variation

DCOF: Dynamic coefficient of friction

IVM: Instantaneous viscosity maximum

OCT: Optical coherence tomography

SC: Stratum corneum

SFE: Surface free energy

SS: Synthetic Skin

WASR: Water absorption swelling rate

Statement of Originality

I, the author, confirm that the Thesis is my own work. I am aware of the University's Guidance on the Use of Unfair Means (www.sheffield.ac.uk/ssid/unfair-means). This work has not been previously been presented for an award at this, or any other, university.

Chapter 1

Introduction

1.1 Background and thesis structure

This project is in partnership with Unilever, a multinational consumer goods company with brands in foods, home care, and beauty and personal care. For this project the focus will be on their beauty and personal care brands, and more specifically moisturisers.

Beauty and personal care (BPC) products are, in most cases, used daily for both healthy and non-healthy skin [1]. They enable the restoration of the intercellular lipids which are part of the skin's natural barrier, improve skin hydration and further protect the skin by forming a hydrolipid film on its surface [2]. Although they can play a crucial role in maintaining skin health or treating various skin conditions [3], they are also often bought off the shelf by the average consumer with no specific medical knowledge [1]. Hence, consumer preferences are widely driven by perceived sensory attributes linked to the products rather than their functions alone [1]. This can be particularly problematic in the cases of sun cream or barrier creams which prevent occupational skin diseases, where products may be dismissed if the skin feeling they provoke is unpopular [4]. Understanding what drives the success of a product in terms of sensory perception as well as function therefore constitutes an essential part in the development of skin care products.

Much of the work done so far at Unilever has involved many human participants, including fully trained panellists and home tests where products are rated depending on the skin feelings they elicit. Furthermore, Unilever products are sold world-wide, which adds complexity to predicting the success of a product due to cultural and environmental differences. Factors such as temperature, humidity, occupation and skin

types could all affect the perception of a product. Additionally, obstacles such as a global pandemic in 2020 have further underlined the need for a simplified screening method which could be used to study the effect of different ingredients on the perception of skin cream. In this context, replacing human skin assessments with adaptable and reusable synthetic skin test-beds could prove highly beneficial.

Several synthetic skin test-beds have been developed by researchers and others already exist on the market. However these often require complex and time-consuming manufacturing methods or incur significant costs. The skin care industry is fast paced and highly competitive, and the high throughput of products developed requires efficient and repeatable testing methodologies.

During this PhD, research was done to understand how the study of skin tribology could enable the development of a method for predicting sensory attributes associated to the application of beauty and personal care (BPC) products. In Chapter 2 a closer look was taken at the literature to understand the anatomy and physiology of skin, and how these characteristics contribute to its frictional behaviour in various environments. The current existing methods for assessing the effect of measured variables to the sensory perception of these products was also reviewed.

In Chapter 3, a deeper understanding of the BPC products was achieved, by looking at the differences in compositions between them and performing rheology measurements. The results were compared to attribute scores obtained during the application of the product on the skin, to assess for any significant correlations. The effect of the different BPC products on the frictional behaviour of human volar forearm skin was also assessed, using friction measurements against a solid probe. The measurements were performed immediately after and 5 minutes after application. The mean dynamic coefficient of friction and the mean coefficient of variation obtained were related to sensory panel scores for attributes rated immediately and after minutes after product application.

In Chapter 4 various experiments were undertaken to explore the effect of different test-bed design elements on their frictional behaviour. The use of glycerin and silicone deadener, as well as the combination of layers and the addition of texture to the test-beds were all found to affect friction and were selected for use in the final test-bed

design. These findings were presented at the World Tribology Congress 2021, details of which are given in the next section.

In Chapter 5, the frictional behaviour and mechanical properties of the refined test-bed designs were tested in the presence of different BPC products immediately and 5 minutes after application. The frictional responses and mechanical properties were compared to that of human volar forearm skin, which revealed a strong linear relationship for one of the test-bed designs. These findings were presented at two conferences: STLE 2023 and UK Colloids 2023, details of which are given in the next section. The frictional behaviour of this test-bed was also assessed against human finger-pads, which indicated significant differences in frictional behaviour and trends when compared to measurements obtained against a solid probe. Finally, the frictional behaviour and adhesion of the test-beds were tested in a Unilever laboratory against a human finger-pad, by adapting their existing protocol. The results were compared to the findings from previous chapters and related to the sensory panel attribute scores.

Hence, the development of a novel reusable synthetic test-bed with a highly repeatable and simple manufacturing process, enabled the identification of trends using measurements related to friction behaviour and adhesion, following the application of three distinct BPC products.

The aims and objectives for this thesis will be detailed subsequent to the literature review. Following these chapters, the methods and results of preliminary experiments will be discussed and plans for the next steps of the project will be outlined.

The attribute scores for the different BPC products, obtained through a sensory panel, were provided by Unilever. All other aspects of the research, including the design and execution of experiments, development and testing of synthetic skin test-beds, rheological and tribological measurements, data analysis, and interpretation, were carried out independently either at the University of Sheffield or using equipment in the Unilever laboratories.

1.2 Research outputs and publication plan

This research has been presented at three international conferences: the World Tribology Congress (*“A proposed methodology for the assessment of consumer expe-*

rience of personal care products in consumer-relevant environments”, poster, Lyon, France, 2022), STLE (“*Development of a Synthetic Skin Test Bed for the Assessment of Beauty and Personal Care Product Consumer Experience*”, oral presentation, Long Beach, USA, 2023) and UK Colloids (“*Development of a Synthetic Skin Test Bed for the Assessment of Beauty and Personal Care Product Consumer Experience*”, oral presentation, Liverpool, UK, 2023).

Two journal publications are currently planned. The first will primarily draw on the findings from Chapter 3, focusing on the characteristics of BPC products and their influence on the frictional behaviour of human skin, in relation to associated sensory perception. The second publication will be based on the findings from Chapters 4 and 5, investigating the key characteristics to consider when developing a synthetic skin model for assessing BPC products.

The journals targetted with be The International Journal of Cosmetic Science, Biosurface and Biotribology, and Skin Research and Technology.

Chapter 2

Literature review, collation of relevant approaches used by industry, aim and objectives

2.1 Introduction

The following chapter will give a background on the anatomical and physiological properties of human skin as well as the composition and effect of BPC products, with a focus on moisturisers. Studies related to the tribology of skin will be reviewed for their relevance in the interactions between BPC products and skin during and following application. The development and use of synthetic test-beds to be used in the instrumentation of such studies will also be addressed. Finally, the use of rheological and tribological measurements for the prediction of the sensory perception of BPC products will be discussed.

The aim for this chapter was to obtain an improved understanding on the characteristics of human skin influencing its frictional behaviour in the presence of BPC products, to allow for the development of a synthetic skin test-bed. Furthermore, the methods previously used for identifying trends related to the sensory perception of BPC products were reviewed to inform on the design of a suitable protocol.

2.2 Skin

2.2.1 Anatomy

Skin makes up approximately 16% of human body weight [5] and exists as a physical and chemical barrier between our internal organs and the external environment. It is made up of three distinct layers, the epidermis the dermis and hypodermis from the

most superficial to the deepest, respectively. The epidermis is avascular with a varying thickness of 60 to 100 μm . It is made up of four layers: the stratum basale, stratum spinosum, stratum granulosum, stratum lucidum and stratum corneum (from deepest to most superficial respectively)[6]. The Stratum corneum (SC) plays an important role in the tribology of skin as it is the only direct point of contact for skin. It is water resistant and made up of dead cells closely packed together and bonded by sebum [7]. This forms a chemical barrier to hold moisture in and keep foreign bodies out. The thickness of the SC is in the region of 10 μm , but can be thicker such as on the soles, palms and fingers [8]. The stratum basale forms a border between the two uppermost layers of the skin : the dermis and epidermis [6]. Cells are formed on the basal layer and in the space of two to four weeks migrate towards the surface of the skin. As they reach the surface they become flattened. The surface of skin contains a multitude of furrows and wrinkles of varying depths (20-100 μm) which enable its extensibility [7]. The junction between the epidermis and dermis is characterised by an undulating pattern called the dermal papilla [6], as shown in Figure 2.1 below. This structure helps anchor the epidermis to the dermis. The ridges found on human hands and feet arise from this layer [6].

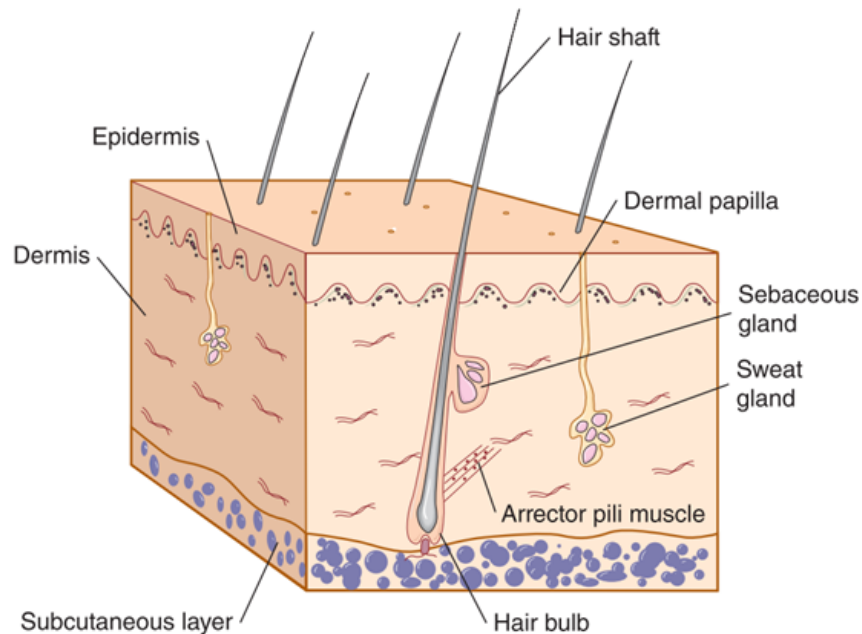


Figure 2.1: The three layers of skin, adapted from Peate [6].

The dermis is the thickest layer of the three, on average 1 to 4 mm [9], and is responsible in the most part for the skin's mechanical properties. It also acts as struc-

tural and nutritional support for the epidermis [10]. It is made up of collagen, elastin and extracellular matrix giving it strength and elasticity [5, 11]. Type I Collagen is secreted by fibroblasts in the Extra Cellular Matrix (ECM). It is an essential protein which provides structure to the ECM [12]. It also contains blood vessels, hair follicles, sweat glands and nerve endings [10]. Lastly the hypodermis, or subcutaneous layer, is a layer of fat providing insulation and padding [5, 13]. It's thickness depends on the area of the body, for example it is relatively thin on the forehead, which can influence contact mechanics [14].

2.2.2 Function

Amongst skin's many functions, sensation, absorption and secretion [6] are the most relevant to this study. Sensation is enabled by a range of sensory receptors embedded into the skin [6]. There are several types of receptors. Mechanoreceptors respond to mechanical stimulation such as the deformation of skin or vibrations. Skin also contains nociceptive units for thermal and mechanical pain, and thermoceptive units for temperature [15]. There is a higher density of receptors in areas such as the lips, fingertips, and genitalia [6]. Mechanoreceptors are divided into two categories. The slow adapting receptors (SA) depict constant mechanical stimuli such as pressure and skin stretch, whereas the rapidly adapting receptors (RA) detect brief mechanical stimuli such as vibrations and transient contact indentation [16]. These are then further divided into Type I and Type II receptors. Type I receptors are closer to the skin's surface and hence have a relatively small receptive field, whereas Type II receptors are embedded deeper into the skin and thus have a larger receptive field [16]. RA II receptors are most sensitive to vibrations.

The function of secretion plays an important role in everyday life. The substances secreted by skin come in the form of sweat and sebum. Sweat is secreted by the eccrine sweat glands and plays a role in thermoregulation [17]. Sebum is secreted by sebaceous glands, which are usually paired with hair follicles [17]. Porter [17] published an article detailing the three functions of sebaceous glands. The first is to coat the hair shaft to form a water repellent barrier on the head. The second is believed to be mixing with sweat in warm conditions, to retain sweat onto the skin and encourage the formation of a sweat sheet rather than droplets that will leave the skin surface. The last function is to repel rain from the exposed parts of the body such as the face.

Lastly, the skin can absorb oxygen, carbon dioxide [6], and fat soluble vitamins such as vitamins A, D, E, and K.

2.2.3 Mechanical properties

Skin can be described as an anisotropic, non-linear and viscoelastic material [18, 19]. Its properties have been approximated in vivo by the means of extension, torsion, suction and indentation [19]. However, an added complexity to skin is that each of the layers making up the skin has different mechanical properties [13]. Hence, the skin's overall mechanical properties will depend on the thickness of each layer. When subjected to stress, skin acts as a homogeneous material combined system. At low levels of strain, the load is carried by the elastin fibres. As the strain increases the collagen fibres begin to straighten, eventually stiffening the skin[20]. Furthermore, when a constant strain is applied to skin, the stress within the skin will diminish over time [20]. With regards to the skin's anisotropic property, Langer was able to identify the direction in which the fibres are aligned in the skin over the entire human body [21]. These lines, known as Langer's lines, are pictured in Figures 2.2 and 2.3. If the skin is stretched in the direction of these lines it will not deform as much as when it is stretched transversally to that direction.

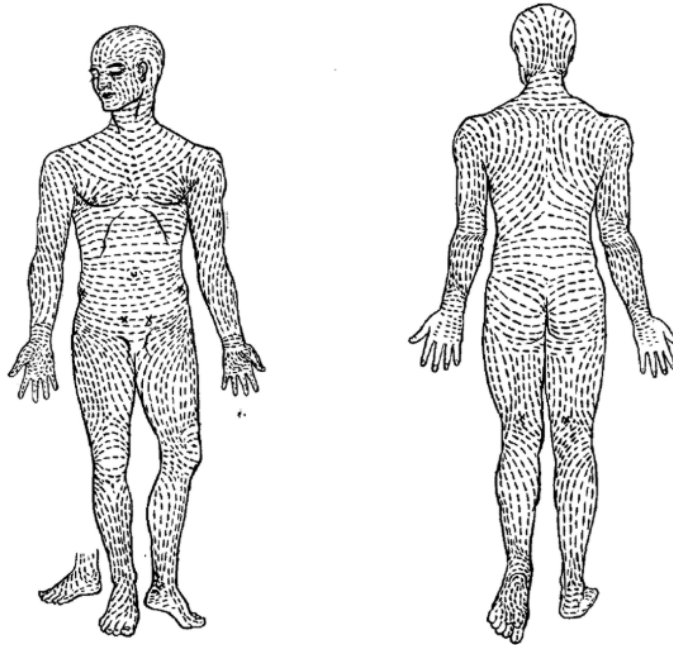


Figure 2.2: Langer's lines adapted from the Joodaki et al. review paper [20].

Another factor affecting the mechanical properties of skin is the level of hydration. To keep the skin to a sufficient hydration state, water flows naturally from the deeper layers of the skin (dermis and epidermis) to the more superficial layers (SC) which have a low water content. This process is called Transepidermal Water Loss (TEWL) [22]. Depending on the exterior conditions, more or less of this water is maintained inside the SC for hydration of the skin. However, a damaged SC barrier will lead to water leaking out meaning the TEWL is increased. Hence, the measurement of TEWL is often used as a mean to evaluate the hydration of skin or the performance of a moisturizer [23].

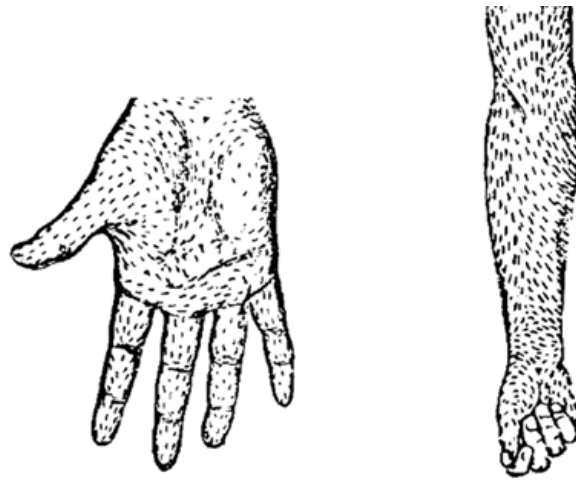


Figure 2.3: Langer's lines for the hand and the volar forearm adapted from the Langer study [21].

2.2.4 Skin surface properties

The epidermis is made up of keratinised stratified squamous epithelium which contains four types of cells: Keratinocytes, Melanocytes, Langerhans cells and Merkel cells. Keratinocytes produce a protein called Keratin which is responsible for the water-resistant properties of skin. It prevents water from entering or leaving the skin [6]. Merkel cells can form a synaptic contact with Merkel discs for the perception of touch [6]. Lastly, Melanocytes protect the skin from UV light by producing Melanin, and Langerhans cells are a part of the immune system [6]. The Keratinocytes migrate through the layers of the epidermis, eventually becoming corneocytes in the Stratum Corneum (SC). The SC is the most superficial layer of the epidermis and it is composed of disk-shaped corneocytes [22], keratin and lipids. Lipids make up approximately 10 to 15% of the overall mass of the SC [22].

The main function fulfilled by Keratins in the SC is forming a protective layer between the body and its environment. However, the properties of Keratin can change depending on the environmental conditions. Water molecules can form bonds with many of the side-chains of keratin proteins. These bonds have been linked to the decreasing elastic modulus and photoacoustic properties of the SC with increasing relative humidity [22].

Human skin topography and SC thickness are dependent on the anatomical region, as demonstrated in the study by Maiti et al. [24], of which the results are pictured in Figure 2.4. Finger pads and palms are characterised by concentric ridges whereas forearms have furrows and wrinkles, contributing to the skin's extensibility [7, 13]. These features are related to the skin's roughness which typically has Ra values of 10 to 30 μ m and Rz values of 30 to 140 μ m [13]. Additionally, Maiti et al. [24] found that the SC was thicker for load-bearing sites, such as fingertips and heels.

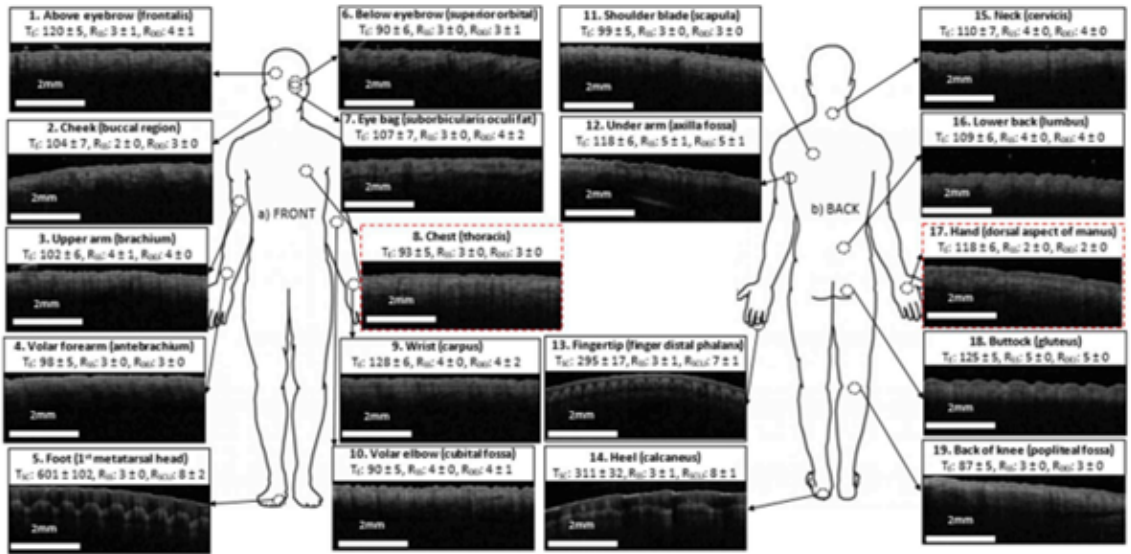


Figure 2.4: Representation of the skin thickness and surface roughness adapted from the study by Maiti et al. [24], with T_{SC} : SC thickness, T_E : Epidermis thickness, R_{SS} : skin surface roughness and the red dotted outline box representing dorsal skin sites.

2.2.5 Physico-chemical properties of skin: surface free energy

The surface free energy (SFE) of a surface exists due to incomplete bonds of surface atoms on a solid material[25]. The value of this energy, which is expressed in mN/m

or dynes/cm, depends on the strength of the bonds between molecules in the bulk of the material. Hence metals tend to have higher surface free energy than polymers [25]. The surface free energy of skin is considered a highly influential factor in the adhesion and wettability of substances coming into contact with it[26], hence it is of particular interest in this study.

The SFE of a material is derived from the stationary contact angle measurements using the sessile drop test method. This experiment consists of depositing a droplet of water (or a different liquid with known surface tension), and measuring the contact angle between the droplet and the material’s surface as illustrated in Figure 2.5a below. Typically, materials are describe as either “hydrophobic” or “hydrophilic” depending on the measured contact angle. These terms relate to the wetting properties of a material, i.e. the extent to which a droplet will spread across it’s surface. According to Law [27], it is common knowledge in the scientific community that a material being considered as hydrophobic means that the contact angle measured on its surface is greater than 90° , whereas a material is said to be hydrophilic when its contact angle is smaller than 90° . However, in reality these properties should be regarded as more of a spectrum and will likely vary depending on factors such as surface texture or the presence of contaminants. In fact, Law [27] found that the sliding angle α (in Figure 2.5b), which is the plate angle at which the droplet starts to slide, was a more accurate measurement for defining the hydrophobicity of a material. Kalin et al. [28] also argued that wetting and surface energy are not equal parameters, as the former depends on both the solid and the liquid whereas the latter is only related to a material’s surface properties. Additionally the authors suggested that a distinction should be made between adhesive-wetting and spreading-wetting. Hence defining the physico-chemical properties of skin is more complex than qualifying it as either hydrophobic or hydrophilic.

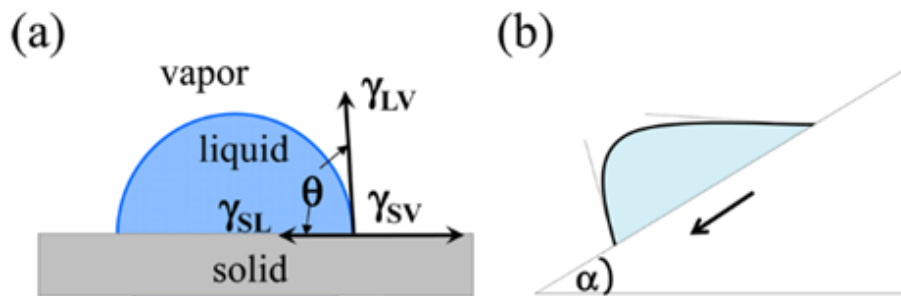


Figure 2.5: Stationary contact angle (a) and sliding angle (b) measured in the study by Law [27].

The proteins, water soluble components and lipids found in the SC are all responsible for the surface energy of human skin [29]. The concentration of each of these components has been found to influence the surface energy. For example, an increase in the amount of surface lipids and in particular sebum, tends to cause an increase in surface free energy [30].

The SFE of skin typically lies between 27 and 38.2 mN/m [31], with the lower value measured on dry skin after it was washed with a delipidizing solvent. In a study by Mavon et al. [30], it was found that for a normal hydrophilicity level, the skin needed medium to high levels of sebum to be present on the skin’s surface. Human skin with low sebum levels showed a lack of hydrophilic components which the authors associated with a deficiency in free fatty acids resulting in a reduced ability to bind water molecules. Hence, the authors concluded that skins exhibiting low sebum levels should be described as “dry skin”. In addition to surface lipids, J.Krawczyk[29] found that temperature also appears to have an effect on surface free energy. In effect, not only the total surface free energy but also the functional groups present on skin will influence it’s wettability and consequently the spreadability of products coming into contact with the skin [29]. The two materials that have been found to most closely replicate the physico-chemical properties of skin in terms of surface free energy and functional groups are Polymethyl methacrylate(PMMA) and Nylon 6 with SFE values of 39.21 mN/m and 40.26 mN/m respectively [29].

2.2.6 Water absorbing capacity

According to Rawlings et al. [32] there are the three mechanisms involved in the maintenance and diffusion of water in skin:

- Intercellular lamellar lipids which act as a semi-permeable barrier to restrict the passage of water out of the skin,
- Corneocytes and corneodesmosomes which give the outer layers of the SC a porous structure therefore enabling the diffusion of water into the skin,
- Natural moisturizing factors (NMF) which enable the absorption of water, present both in and outside of cells.

The SC is mostly made up of keratin-filled corneocytes. These act as a barrier for hydrophilic compounds whilst allowing for the transport of polar molecules such as water [33]. When the skin is exposed to a humid environment the corneocytes can swell up to 50% in height. The swelling of the SC is thought to increase its flexibility through the polar molecules binding onto the keratin chains and thus reducing the interactions between them [34]. Polar molecules, such as Urea and Glycerol, are naturally occurring in the skin and are referred to as “Natural Moisturizing Factors” (NMF), and are often added into skin care products as humectants. These will be discussed in a later section.

2.2.7 Skin ageing

Preventative methods for skin ageing and associated skin conditions represent a significant portion of the skin care market[35, 36]. Understanding the process of skin ageing, as well as its effect on the properties of skin and how this will affect the perception of products, is essential for enabling the adaptation of a test-bed to older skin for a more tailored skin care development. There are two pathways to skin ageing. Intrinsic ageing encompasses the biological changes that happen throughout the course of life of which humans have no control over. Extrinsic ageing are the external factors which cause skin ageing, such as UV radiation, also known as photoaging [12]. In both cases similar mechanisms are observed.

Intrinsic ageing is in great part prompted by hormonal changes which start as early as the mid-twenties with the decline of sex hormones [12]. It has been found in particular that oestrogen and androgen deficiencies are linked to wrinkles, dryness and loss of elasticity [12]. However, 80% of facial skin ageing is said to be caused by UV exposure [12]. UV radiation causes a decrease in collagen production which in turn leads to a loss of mechanical tension caused by the accumulation of collagen

fragments [12]. It has also been found to induce the expression of fibroblast elastase thus damaging elastic fibres and leading to wrinkle formation on the skin [12].

In order to understand the effect of skin ageing on the mechanical properties of skin, Kim et al.[37] conducted a study involving 58 Korean female volunteers between the ages of 25 and 64. The purpose of the study was to evaluate the difference in biomechanical properties of the neck, cheeks and volar forearm related to ageing. With the neck and face both being exposed to the sun, some differences were expected when comparing them with the forearm skin. The neck skin elasticity was found to be more correlated with age compared to the two others. However, the sebum secretion levels and desquamation index of the cheek, which indicates the skin's level of dryness, were more correlated with age than for the neck. Lastly, TEWL was only correlated with age for the neck. Hence it seems that the larger exposure and more movement means the neck is more likely to show effects of skin ageing. This reinforces the theory that sun exposure has a greater effect on the mechanical properties of skin than intrinsic ageing . As sun exposure varies widely amongst people depending on occupation (working outdoors versus indoors) and climate, choosing an anatomical site less likely to be exposed to sun regularly such as the volar forearm seems of most relevance for experiments on skin.

Along with physiological changes to skin, ageing has additionally been found to affect tactile perception. Trautmann et al. [38] studied the effect of age on the preferences for skin creams. The age groups selected for the experiments were 18-25, 30-45 and 50-56. The skin creams assessed had similar viscosities but different textures due to the addition of round beads in some of the emulsions. In addition to the sensory testing, the authors performed haptic perception tests but using sandpaper samples of variant roughness, and special tactile discrimination using the Landolt ring method. The results showed that younger women disliked creams containing larger particles whereas the older women described all creams as “smooth”, “velvety silky” and “soft”. The authors also found some correlations between age and a decline in haptic perception and tactile discrimination. However, it is worth noting that there was a significant source of individual variations which the authors associated to factors such as occupation and hobbies where haptic perception and tactile discrimination are better stimulated.

2.3 Beauty and personal care products

This study will focus on the use of moisturisers. There are four categories of active ingredients commonly found in these products:

- Emollients increase skin hydration by trapping moisture inside the skin. They are oils which provide light occlusion and can easily be spread onto the skin [39]. This film also acts as lubrication [40]. The most commonly used emollients in the skin care industry are esters and silicones due to their hydrophobic properties [31].
- Occlusives also prevent water loss by forming a barrier. Petroleum jelly, a commonly used occlusive, has been found to reduce TEWL by 98% [39]. In comparison, emollients typically reduce water loss by a maximum of 30% [39].
- Humectants hydrate the skin by forming hydrogen bonds on the skin's surface which attract water from the environment [41] as well as from the dermis and epidermis to the SC [42].
- Rheology modifiers usually increase the product's viscosity, making it easier to handle and also change the product's aesthetics which in turn affects the perceived quality of the product [43].
- Emulsifiers prevent the separation of dissimilar ingredients, such as oil and water, in an emulsion [44].

It could be assumed that the main determinant for success of beauty and personal care products in the market is linked to their function. However, the feel of a product during application and skin sensations post-application both play a major role in consumer preferences [45]. One of the challenges which comes with characterising the tactile sensation associated with skin creams is that their composition changes throughout and following the application process. Ingredients such as water will evaporate whereas others will interact with the skin [23], meaning product perception will inevitably change throughout the process. There is also the fact that the product remains on the skin for an extended period unlike soap or shampoo which is rinsed off. Hence, the effect and residues of skin cream can in some cases be experienced hours after application.

2.3.1 Short-term effects of moisturiser application on skin

Moisturisers are mainly used for preserving the integrity of the skin barrier. When this barrier becomes compromised, the skin will become dry which is often accompanied by an itchy sensation [46]. According to a study performed by Marie Loden [47], the skin’s hydration levels begin to increase immediately after the application of emollients. The author used an evaporimeter to measure the evaporation of water from the skin’s surface 5 minutes after product application, and found that a higher concentration of water in the product led to more water being absorbed into the skin. However, the amount that was absorbed was less than the amount of water evaporated from the product. Blichmann et al. [48] also found that the evaporation of water from the product was notable immediately after application of the product. They suggested that the majority of the water evaporation occurred in the first 15 minutes following product application, and that the next 360 minutes at least could be described as the “lipidization phase”. This next phase, according to the authors, is dominated by the oil content of the product and is associated with an increase in skin hydration. They also hypothesised that the physiological effects arise from the lipids penetrating into the superficial layers of the epidermis and that the water phase only acts as a vehicle. They also reported an initial reduction in skin temperature immediately after product application.

Wiechers et al. [49] studied the effects of BPC product ingredients on the mechanical properties of skin. They found that changes in measured skin capacitance following the application of water onto the skin was significantly correlated to changes in the mechanical properties of skin. However, this same trend was not observed when testing isolated ingredients from various BPC products. The authors therefore suggest that physiological changes in skin from BPC products arise from different mechanisms. The findings from the study showed that the ingredients which caused improvements in skin moisturisation showed no effects on the skin’s mechanical properties. Furthermore, the ingredients that did have an effect on the skin’s mechanical properties for up to and beyond 6 hours did not cause an increase in skin moisturisation. Hence this work suggests that only molecule known to affect both properties is water, although these effects are only short term.

2.4 Tribology of human skin

Applying skin cream involves skin-on-skin interactions, separated by a layer of product, between the hands and different parts of the body. The friction caused by rubbing the cream on, the lubricating effect of the cream or sebum and the wear of dead skin cells could all play a role in the perception of skin cream during consumer experience. Hence, understanding the tribology of skin and the factors affecting it is of prime interest.

According to Bowden and Tabor [50] the total frictional force arises from two different mechanisms: deformation (F_{DEF}), which is the force required to deform the bulk of the more compliant material [51], and adhesion (F_{ADH}) which is affected by the molecular bonds between the asperity junctions of the two interacting surfaces. Hence, the total frictional force can be described as stated in equation 2.1. This two term model of friction is often used when looking at the friction of human skin [52].

$$F_{TOT} = F_{ADH} + F_{DEF} \quad (2.1)$$

A review published in 2020 by Meng et al.[53] found skin tribology to be one of the most researched areas in bio tribology alongside joint and oral tribology, with skin creams and other cosmetics as one of the main investigations. Although ethically complex, in vivo studies involving the skin remain a widespread approach. However, a direct comparison of coefficients of friction remains a challenge as not all studies use comparable methods. In this section, the influence of experimental methods and conditions on the coefficient of friction (COF) of skin will be summarised.

2.4.1 Influence of the anatomical region

For this study, the anatomical regions of interest will be the finger-pad, and volar forearm skin. It is important that differences between anatomical regions are carefully considered when developing synthetic skin test-beds, as the contact mechanics may differ which could influence the frictional response. There are a few reasons as to why the anatomical region may have an influence on the coefficient of friction of skin. Firstly, the thickness of skin differs depending on anatomical site, which is mostly due to the thickness of the subcutaneous layer. In addition, sebaceous glands are not equally distributed thus causing some areas of the body to be hairier than others. Sweat glands are also generally concentrated in certain areas which could

enhance lubrication. Hence it has been hypothesized that the coefficient of friction would differ depending on region. Hendriks et al.[8] found no significant difference in COF value between the cheek and forearm, but the COF measured on forearms showed more variation. As mentioned by the authors this could be due to potential beard stubble on the cheek, or the difference in the dermis and epidermis thicknesses. Veijgen et al.[54] also found lower COF for regions with more hair. This could either be due to the lubrication of the hair by the sebaceous glands, or by the reduction in real contact area due to the probe coming into contact with the hair rather than the skin.

Moreover, Liu et al. [11] evaluated the effect of the thickness of the Stratum Corneum (SC) across the fingers and palms on friction. They found SC thickness to be linearly proportional to skin distensibility meaning that anatomical areas with thicker SC lead to a greater contact area in loaded contacts which in turn influenced the friction. Another finding was that the index finger and thumb both had a thicker SC than other fingers and was twice as thick as on the palm. Veijgen et al. [54] additionally found that the finger-pad seemed to exhibit the most variation in terms of skin hydration and skin temperature, which could be due to greater variations in the thickness of the SC. The authors also found that the COF was higher for the finger-pad and volar (inner) forearm than for the dorsal (outside) forearm and dorsum hand. Lastly, the noise in the friction data was also greater for the finger pad, which the authors associated with the finger-pad ridges. Hence anatomical region should be carefully considered when running experiments as values of friction can significantly differ.

2.4.2 Influence of load

Several studies have highlighted the dependence of the COF of skin on load. Bostan et al.[55] found evidence of a slight drop in the COF with increasing load in for ex-vivo human skin. Chen et al.[56] found that when loads used were low, the resulting bulk modulus values were high due to being influenced only by the epidermis. However, as loads were increased, eventually the dermis started to contribute to the bulk modulus which lowered the values. These findings highlight the potential influence of the dermis and subcutaneous tissue on friction values.

Other studies have focused on the relationship between load and the COF of human finger-pads. Several authors have found that the COF of a human finger increases with increasing load[57, 58] but exhibits a two-part response. This is due to the deformation of the finger-pad under 1N [58] yielding a non-linear response. For loads above 1N the frictional response has been found to be linear[57, 58, 59] for the fingertip against materials such as metals and TPU. Liu et al. [11] also observed that skin friction showed a greater amount of variation for hand regions at low loads versus high loads, which also highlights that the skin’s structural properties are less likely to have an effect at higher loads.

Lastly, Inoue[60] evaluated the dependence of the COF on load for a human finger against a commercially available synthetic skin. Results showed that the dependence between the two varied widely for every measurement (both intra- and inter- personal variations were observed). Much of these variations were associated to the moisture content of the finger pad as well as the inevitable changes in contact area during experiments due to changes in finger-pad position.

2.4.3 Influence of ethnicity, sex and age

It is of notable interest to understand differences in frictional behaviour of skin for various demographics as this may be advantageous for the tailoring of products to specific consumers. However, the number of studies related to these three factors were limited, and results inconclusive.

Sivamani et al.[61] studied the effect of ethnicity, sex and age on the tribological properties of cream treated skin. Results suggested that the coefficient of friction of cream treated volar forearm skin and electrical impedance were independent of those three factors. These results could be explained by the anatomical site tested as it is relatively unexposed to UV radiation meaning that age would not affect the properties as much. Veijgen et al.[54] found significant differences in the COF at various anatomical locations between men and women, however these differences were found to be mostly all explained by differences in hydration rather than differences in sex. The hydration level of the finger-pads were found to be significantly higher for men, whilst the hydration of the forearm was significantly higher for women [54].

Jacquet et al. [62] found a downward trend of the initial slope of the stress-strain curve of skin with age. This corresponds to the function of elastin fibres only and is in accordance with the effect of age mentioned in section 2.2.7. However, the COF of skin was not measured in this study.

2.4.4 Influence of probe type material and surface roughness

Many experiments have been conducted using traditional materials as probes, such as steel, aluminium and PTFE, due to contact mechanics being more predictable. However, there exists a variety of tribometer set-ups. Due to experiments being performed on a human being, certain set-ups involving traditional tribometers, are not always achievable. For example, many use probes which slide across the skin from one point to the other [63] such as the one pictured in Figure 2.6a, whilst other studies use rotating probes which rotate with their axis parallel to the skin [64, 54] as pictured in Figure 2.6b. Often lower COF are observed for the latter, seeing as there is no continual deformation of skin [54]. Handheld probes[64, 8, 54], such as the one used in Figure 2.6d and also pictured in Figure 2.6c, offer the advantage of reaching a variety of anatomical regions (such as the cheek), as well as allowing the participant to control the force applied and to end the test at any point should they experience any discomfort.

However, a few limits associated with these probes have not yet been discussed. Firstly, although the gravity spring in the probe developed by Hendriks et al.[8] enables a constant force, it seems the device must be held perfectly perpendicular to the skin. Discrepancies could occur due to fatigue and thus a relaxation of the arm which would lead to unequal load distribution on the skin. Moreover, in the study led by Hendriks et al. [8], where the probe used consisted of a rotating ring (as shown in Figure 2.6c), the tests were limited to 30 seconds as any longer would incur pain and skin irritation. This is likely to be due to skin being more sensitive to shear deformation [64].

The probe roughness has also been found to influence the COF. For example, Hendriks et al.[8] found that the COF of human cheek skin decreased with increasing probe surface roughness when $R_a < 10 \mu\text{m}$. Veijgen et al. [54] also found differences in the COF of human volar forearm depending on the probe used which the authors associated to surface roughness (with $R_a < 10 \mu\text{m}$ for all materials). However, it is

believed that beyond a certain roughness value the COF will decrease due to the asperities carrying the load which will reduce the contact area [57].

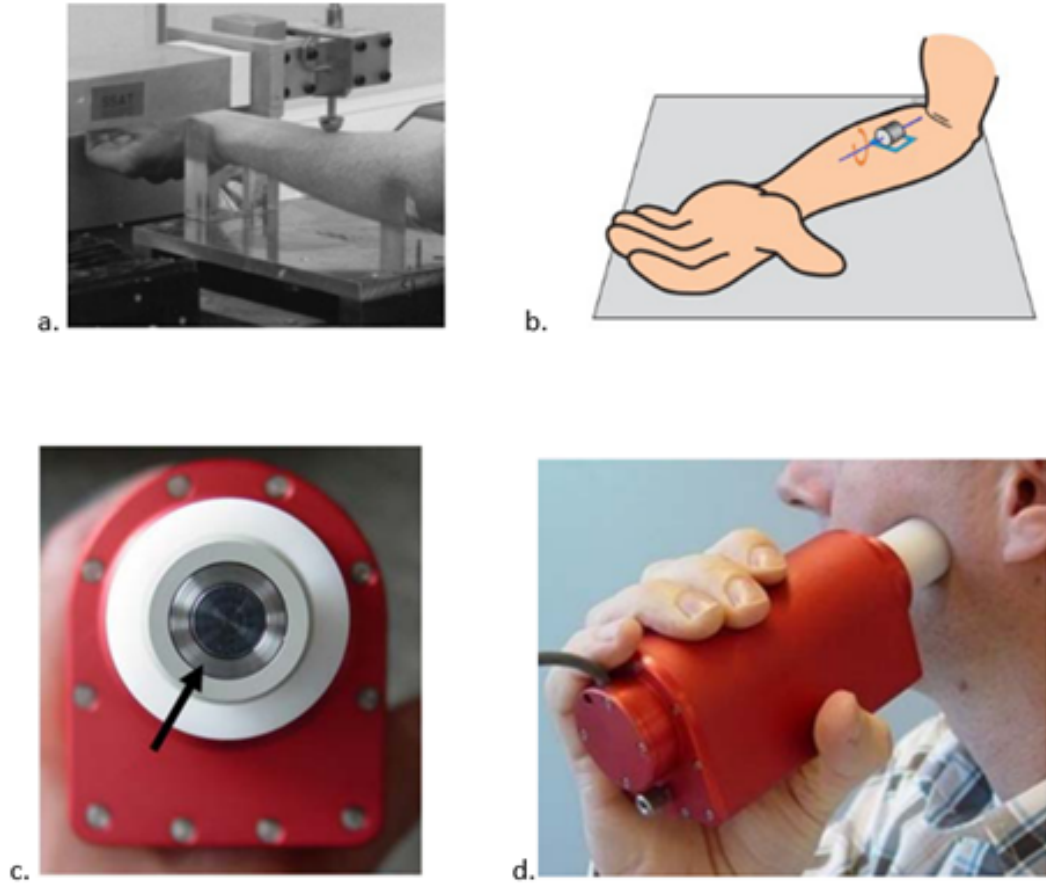


Figure 2.6: a. Tribometer set-up with arm rest used by Adams et al.[63] , b. loading direction of the probe used by Masen et al.[64] , c. and d. Probe used by Hendriks et al.[8].

Lastly, the material used, is also believed to influence friction. Tomlinson et al.[58] found a higher variation in results for the plastics than for the metals. For elastomers, a higher COF was measured, and stick-slip was observed past a certain load. This was attributed to the viscoelastic nature of such materials. Moreover, Hendriks et al. [8] found that PTFE was the only material in their study to give different values of COF against skin. This finding was justified by the relatively low surface energy of PTFE compared to other materials and thus a potential reduction in adhesive forces. Additionally, Tomlinson et al. [65] found that the COF obtained for a human finger against steel was lower than for a polypropylene or brass substrate, despite the similar surface roughness values. The author attributed the findings to

hydrophilic/hydrophobic interactions between the finger and materials. The effect of surface energy seems even more significant in moist conditions as found by Adams et al.[63]. In this study the use of a glass probe (hydrophilic) against wet human skin yielded lower COF values than when using a polypropylene probe (hydrophobic). These results were ascribed to the formation of a more stable water layer thus increasing lubrication and reducing friction.

2.4.5 Influence of skin hydration and TEWL

When conducting experiments on humans, results tend to show high levels of variations. These have mostly been associated with differing levels of hydration. Skin hydration is usually measured via Corneometry [66]. Corneometers enable the measurement of the dielectric constant of skin which changes according to how much water is contained[67]. Heinrich et al.[66] established three distinct skin types based on corneometric values: very dry (<30 AU), dry (30-40 AU) and normal (>40 AU).

Several factors have been found to influence skin hydration levels. For example, conducting tests in environments with higher humidity was found to increase the COF, which Hendriks et al.[8] found to be positively correlated to skin hydration values (25 to 50 AU versus 50 to 75 AU in dry and humid conditions respectively). The increase in COF with hydration was found by several other authors[54, 68] although Veijgen et al.[54] found that the skin hydration of the fingerpad was the only one correlated with relative humidity. Moreover, the authors found that for all anatomical regions the skin hydration was correlated with skin temperature and that the differences observed between men and women seemed to be linked to differences in skin hydration. The increase in COF with increasing skin hydration has been associated by the increased compliance of the skin, therefore increasing the real contact area and adhesion with the contacting surface [54].

Knowing the effect of skin hydration on friction is of particular interest as skin creams can largely influence the skin's hydration levels. This was demonstrated by Masen et al. [64] who conducted a study on the effect of different lubricants on the friction between face coverings and skin for hospital staff with shifts exceeding eight hours. Results showed that humectants tended to increase the COF to higher values than other types of products, as they attract water from the dermis into the epidermis which leads to an increase in skin hydration. The resulting increase in friction has been associated to the larger contact area due to the plasticization

effect of water which, as well as increasing compliance also appears to smooth the skin [13]. Moreover, Heinrich et al.[66] found that skin type (very dry, dry or normal) has an influence on the efficacy of skin creams which is likely to influence consumer preferences.

As mentioned in the previous chapter, the measurement of TEWL is used to assess the condition of the skin's lipid barrier. Skin with an abnormally increased TEWL is often referred to as atopic[69]. Lodèn et al.[47] evaluated the effect of capacitance (relating to skin hydration) and TEWL on the friction of atopic and normal skin. It was found that TEWL was significantly correlated to friction and to capacitance only on the back skin of atopic patients, whilst capacitance was significantly correlated to friction for the back and arm skin in atopic patients and for the hand and back in normal patients.

Lastly, studies have shown that when water is applied directly to the skin the friction also tends to increase [65, 13]. The first reason for this could be that the water forms bonds with the keratin present in the SC, which allows for the absorption of water into the skin's most superficial layer [65]. This in turn has been found to reduce the SC's Elastic modulus which increases the contact area during loading [65]. Additionally the swelling of the skin caused by the absorption of water reduces the surface roughness which could also be a contributing factor [65]. Capillary adhesion and viscous shearing of liquid bridges are other potential causes but the extent of their influence is yet to be demonstrated [65, 13].

2.4.6 Influence of BPC product ingredients.

The observed increase in COF of skin following skin cream treatment has in a majority been attributed to the increase in hydration of skin [47]. However, the effects of the residue of ingredients on the skin surface may also influence the COF of skin. Gore et al.[68] studied the effect of two different emollients commonly found in skin cream on friction: Stearic Acid (SA) and a mineral oil, isohexadecane (IHD). The product were applied to participants' volar forearms manually at 90 rpm after which the friction was measured against a smooth Teflon disk of 16 mm in diameter at 90 rpm for 5 minutes. According to the authors this speed is typically used when spreading emulsions on skin. The highest and lowest friction was obtained for emulsions with the 100% of SA and IDH respectively. Interestingly, the authors also found an initial decrease in friction at the start of the measurements followed by an overall increase

in friction over the 5 minutes for all emulsions tested. Masen et al.[64] also found different results based on ingredients. Silicone based as well as water and glycerin based skin creams caused a rapid increase in friction to values higher the one obtained for dry skin (1% and 29% higher respectively). However, a significantly lower rate of increase was found for Petrolatum after initially reducing the friction to 47% of the dry value.

2.4.7 Summary of the skin friction studies

Tables 2.1, 2.2 and 2.3 below give a summary of the different techniques and contact parameters used for the study of skin tribology. The wide range of reported COF and friction forces gives further evidence on the dependence of the measured frictional behaviour of skin on the methodology as well as on inter-personal variations.

Table 2.1: Summary of the methods and COF values from literature for probe against skin measurements.

Authors	Substrate	Probe	Normal load	Speed	Sliding distance	CoF
Bostan et al. [55]	Ex vivo skin, ex vivo dermis, epidermis, TE skin, SynDaver	Stainless steel sphere, 4mm diameter.	5,10, 30 mN	0.5mm/s	10 mm	Approx. 0.1 - 1.2
Chen et al. [19]	SS: hydrophilic network carboxyl chitosan (CC) and hydrophobic network PDMS (SS covered with wound dressing), human volar forearm	Nylon 1010 sphere, 10mm diameter.	500 mN	0.25mm/s	10 mm	(All approximations) 0.15 to 0.75 (Human), 0.25 to 0.8 (SS)
Nachman et al. [70]	SS: Silicones modified with strongly hydrophilic alpha-oelin sulphonate with texture from skin replica.	Steel sphere, 4mm diameter.	30 mN	0.5 mm/s	10 mm	(All approximations) 0.6 (ASM dry), 1.3-1.4 (ASM moist), 0.7 (dry skin), 1-2.5 (moist skin)
Bhushan et al. [71]	1) Drakon Skin (Smooth-On inc) silicone rubber. 2) Gelatin plastecized by glycerol, polysaccharides and a mixture of lipids, and crosslinked with formaldehyde. 3) Ex-vivo rat skin	Etched silicon probe Si, tip 10nm radius.	120 nN	10 μ m/s	30 μ m	(All approximations) 0.05(SS1), 0.138 (SS1 +cream) , 0.154 (SS2), 0.303 (SS2 + cream) , 0.087 (Rat skin), 0.176 (Rat skin + cream)

Table 2.2: Summary of the methods and COF values from literature for probe against skin measurements (cont.).

Authors	Substrate	Probe	Normal load	Speed	Sliding distance	CoF
Morales - Hurtado et al. [72]	1) Lorica 2) Silicone Skin L7350 3) Epidermal skin equivalent (ESE) made of PDMS and PVA hydrogel crosslinked with glutaraldehyde.	NBR, PA, PE spherical tipped pins, 10mm diameter.	2N, 4 N	50 mm/s	Approx. 62 mm (circular)	Highest approx. 1.6 (NBR on L7350), lowest approx. 0.3 (PE on ESE)
Franklin et al. [73]	SynTissue (Syndaver Labs)	Flat stainless steel ring	0.31, 0.94, 1.56 N	2.56 rev/s (96mm/s)	Missing	(Approximations) 3 - 12 (Dry) 0.2 - 3.25 (Moist)
Zahouani et al. [74]	Tissue engineered Dermal Equivalents (DE) and human volar forearms.	Steel spherical indenter, 4.5 mm diameter.	5 to 100 mN	50 μ m/s	1 mm	(Friction force) 18 mN (human), 2.4 mN (DE)

Table 2.3: Summary of the methods and COF values from literature for probe against skin measurements (cont.).

Authors	Substrate	Probe	Normal load	Speed	Sliding distance	CoF
Veijden et al. [54]	Human skin (volar forearm)	Stainless steel, PE, PTFE, cylindrical handheld probe, 20 mm diameter, 10mm wide.	0.5 - 2 N	1,2,5, 10 mm/s	Missing	Approx. 0.2 - 1.3
Adams et al. [14]	Human skin (volar forearm)	PP and glass spherical probes, diameter 12 and 25mm.	0.1 - 4 N	8 mm/s	Missing	(Approximations) 4.8-7 (wet PP), 0.6 (dry PP), 1.6-2.5 (wet glass) , 0.5 (dry glass)
Hendriks and Franklin [8]	Human skin (volar forearm)	Handheld rotating poyvinylchloride ring.	0.625 N	100 mm/s	30 s	(Friction force) Approx. 1.5 - 2N (dry climate) and approx. 2.75 - 3.75 N (humid climate)

2.5 Synthetic skin test-beds

As previously highlighted, there are a number of limits when it comes to studying the tribology of skin on human subjects. As well as the inter-personal and intra-personal variations [19] mentioned above, the cost incurred from testing on human participants can be significant. Recruitment, obtaining ethical approval and repeating experiments all require a considerable amount of time which is often a limiting factor in a fast-paced consumer goods company such as Unilever. Moreover, uncontrollable outside factors such as the recent global pandemic (2020) can also have a significant negative impact on research, particularly involving the use of human participants. Hence, the development of mechanical and physiologically accurate skin substitutes are of strong relevance. In fact, several synthetic skin (SS) test-beds already exist on the market. Examples of some SS test-beds commonly found in tribology studies are VitroSkinTM and SynDaverTM[41, 55, 75]. Many of these models were developed to match the mechanical properties of skin, however it is not generally specified whether the tribological properties of skin under both dry and moist conditions are also similar. Moreover, the commercial skin test-beds often come at a significant price and, once removed from packaging must be kept in specific conditions, e.g. soaked in deionized water, and their reusability has not yet been explored. Hence, they may not be suitable alternatives for the beauty and personal care industry. It was therefore of interest to investigate alternative methods developed by several authors for the manufacture of synthetic skin test-beds.

2.5.1 Synthetic skin test-bed development

In a review by Dabrowska et al. [76] the use of elastomers, in particular silicones, emerged as a widely favoured option for simulating the physical properties of human skin, in most part due to the potential for incorporating ingredients into the mixture and therefore tailoring their properties. Other options included gelatinous substances, due to their natural hydrophilicity. Examples of such synthetic skin test-beds features the one developed by Chen et al.[19], who trialled different ratios of hydrophilic network car-boxyl chitosan(CC) and hydrophobic network PDMS. Combining the mechanical properties of PDMS with a water absorbent component was also explored by Morales-Hurtado et al. [9] with the addition of a PVA hydrogel into their SS. On the other hand, Nachman and Franklin[70] focused on the multi-layered aspect of skin and its potential influence. For this purpose, they developed a skin model made up of a silicone modified with hydrophilic alpha-oelin sulphonate for the top layer and

a polyurethane gel for the under layer which is shown in Figure 2.7. Lastly, Bhushan et al.[71] used a mixture of gelatin plasticized by glycerol, polysaccharides and lipids, cross-linked with formaldehyde.

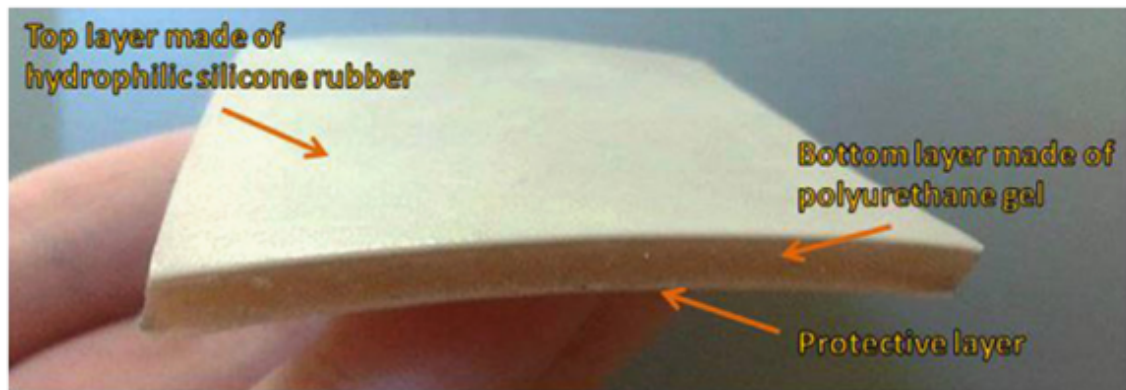


Figure 2.7: Bi-layered artificial skin model developed by Nachman and Franklin [70] with upper layer $100 \pm 10 \mu\text{m}$ and lower layer $2.8 \pm 0.2 \text{ mm}$.

Although the use of Glycerol, also referred to as Glycerin, in the case of the Bhushan et al. test-bed [71] was initially suggested by Lir et al. [77] as a plasticizer to increase the test bed's elasticity, mixing it within a silicone matrix has also been explored as a mean of controllable drug-delivery by Mazurek et al. [78]. In this study the authors found that by applying a high shear force, the two immiscible mixtures could be combined to form a "two-phase elastomeric composite". The authors identified that upon submersion in water, the glycerol was released from within the composite and replaced with water. Hence, it could be a valuable ingredient when developing a synthetic skin test-bed with water absorption capacities.

2.5.2 Synthetic test-bed hydration

As mentioned previously, skin hydration levels have a significant influence on COF values. Hence, when developing an artificial skin model, it is important to evaluate its water uptake capacities. It is known that humidity is correlated with skin hydration values. Therefore, obtaining a skin which will respond to humidity and moisture the same way human skin does is desirable. In order to achieve a 'hydrated state', Chen et al.[19] soaked the developed SS for two weeks and weighed it throughout to determine the swelling rate. Nachman et al.[70] tested two different methods. For the first one the SS was left in a humid room and for the second it was submerged in distilled water for 24 hours, both over a period of 24 hours. Chen et al. found

the water absorption rate of the SS to be between 80% and close to 200% after two weeks depending on the curing agent to PDMS ratio used [19], whilst Nachman et al. reported a water capacity of 120% for the SS submerged and 25% for the SS left in a humid room [70].

Bostan et al. [55] found that for ex-vivo skin, ex-vivo epidermis, ex-vivo dermis, synthetic skin (SynDaverTM), and tissue engineered (TE) skin, the friction increased for increasing hydration. They also found that beyond a certain hydration level, friction started to decrease. The dermis and synthetic skin appeared to have a slightly reduced water holding capacity, as the COF dropped at lower hydration values which the authors associated with the liquid being squeezed out in the contact.

Nachman and Franklin [70] observed that the values obtained from the Corneometer on the ASM were much lower than for human skin even though the water uptake was much higher than the average human skin. The authors reflected that this could be due to the influence of the lower layers for the human skin hydration value, seeing as the SC is normally thinner. Hence reducing the thickness of the artificial skin's top layer could reduce the time needed for the hydration of the SS and give similar hydration values to human skin. However, according to the authors this would likely impact the manufacturing process and durability of the SS.

The SS developed by Bushan et al.[71] and Nachman et al. [70] both yielded encouraging results with the COF increasing in moist conditions. The results from the latter study are illustrated in Figure 2.8 below. It is evident from the graphs that larger variations in COF occurred for human skin than for the SS, particularly in moist conditions. However, it is important to note that although obtaining results such as the ones for the SS would mean the model is predictable and repeatable, it could also mean results will not entirely represent real life events. The peaks observed in the COF of human skin could be pivotal in understanding the tactile perception of a product, for example by provoking a “sticky” feeling.

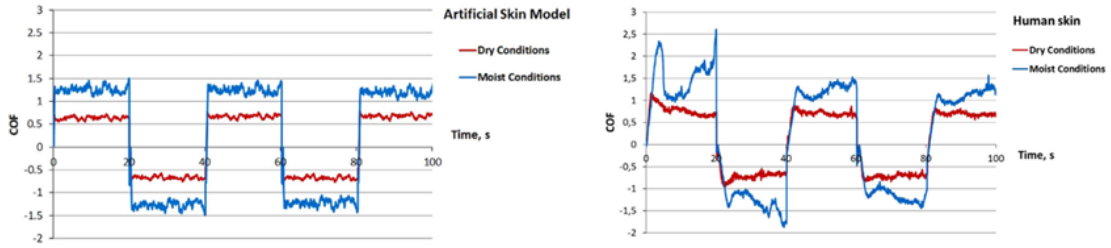


Figure 2.8: Graphs showing the COF over time for the SS developed by Nachman and Franklin [70] (left) and human skin (right) in dry and moist conditions, at a normal load of 30mN.

In the study led by Bhushan et al.[71], results showed that after treatment with the a moisturiser, the COF increased for the ASM as well as for the ex-vivo rat skin used as a control. The authors also observed a decrease in the root mean square and peak-to-valley measurements of roughness for both skins after cream treatment. Moreover, the contact angle decreased meaning the cream treatment improved the hydrophilic properties of the skins. The results in the study are encouraging, as the synthetic skin showed a similar response to ex-vivo rat skin after skin-cream treatment.

2.5.3 Synthetic test-bed surface energy

From the previous section on the surface properties of skin, the surface free energy was discussed as a potential influencing factor for the wettability and adhesion of BPC products. The surface free energy of silicones has been measured at around 20-23 mN/m [79] compared with the 27-38 mN/m range for human skin, hence increasing its surface energy may be of interest for use as synthetic skin test-bed for assessing BPC products.

A method often used to increase the surface energy of polymers is plasma treatment. This method works by bombarded ions and electrons from a plasma at significant speed onto a surface which creates a simultaneous deposition/removal process of particles on a material's surface[80]. The plasma treatment process has gained popularity in the food packaging industry[80, 81] for surface decontamination but also has applications in the biomedical industry due to its potential in optimising biomaterials for colonization by cells and microorganisms [82]. The process also enables the modification of the surface properties without affecting the bulk mechanical properties [81]. Most relevant to this study however has been the use of plasma treatment for improving the wettability and adhesion of silicone elastomers. Lai et al [83] found that

oxygen plasma treatment at 10 W successfully increased the silicone’s hydrophilicity as a function of treatment time. The contact angle went from 11deg after 10 minutes to 51deg after 20 minutes. The study additionally revealed that the plasma treatment improved the peel strength of the silicone rubber, thus highlighting the effect of surface energy on adhesion. However, its application remains limited due to the transient effect of the treatment. Several studies have highlighted the rapid recovery of the material’s hydrophobicity following storage in ambient conditions [83, 81].

Additionally, an alternative to the sessile drop test method for measuring the surface energy of a surface, is the use of solvents with specific gradually increasing surface tensions [84]. The mixtures can be made in accordance with the International Standard ISO8296 for the determination of wetting tension of plastic films and sheeting. The ability for the surface to retain these solvents i.e. if the solvent wets the surface, will inform on its surface energy. These solvents were used in a study by Pankaj et al. looking at the application of plasma treatment in the food packing industry [80] (Figure 2.9).

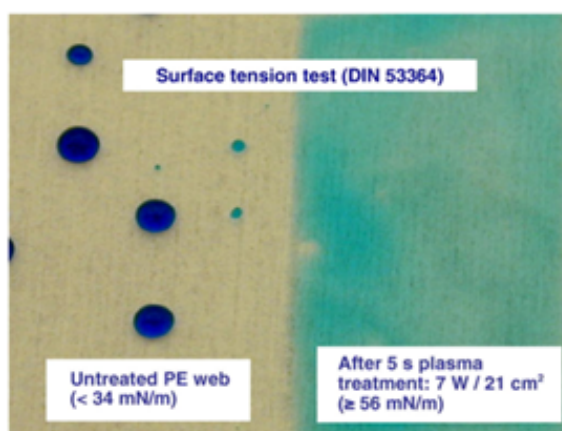


Figure 2.9: Use of solvents for the measurement of surface free energy, adapted from Pankaj et al.[80].

2.6 Sensory perception of BPC products

The current gold standard for assessing the sensory perception of beauty and personal care products are sensory panels [38, 31, 45, 68, 85, 86] . These consist of training a number of individuals to recognise and rate sensory attributes while using BPC products. Unilever have derived several methods to predict the success of a skin care product on the market. These techniques have been split into two categories:

- Subjective/hedonic assessment, which entails finding out whether consumers like or dislike a product,
- Objective/analytical; or finding out what the perceived differences between products are.

For the hedonic assessment, Unilever use Home Use Testing (HUT). This technique involves the participant being given a product to try at home over a set period. The environment in which the product is used is not controlled meaning results can show a lot of variability. The objective assessment, however, involves training a panel of participants for approximately 6 months, for them to recognise certain attributes which will help them differentiate products. These types of tests will be referred to as sensory tests. Some examples of sensory attributes used are bounciness, firmness, stickiness, spreadability and greasiness. For every product, attributes are rated on a scale of 0 to 100. The attributes selected for sensory panel tests were separated into categories depending on the point at which they occur during the consumer experience. Results from several sensory tests were evaluated and the main drivers for consumer preferences were derived: two of the main drivers of preference were found to be the skin feel immediately after application, the skin feel during application (in-use) and the skin feel 15 minutes post-application.

However, methods such as the ones employed by Unilever can be costly and time-consuming hence the focus has been on finding alternative protocols. In literature, much of the instrumental work in sensory perception of consumer products stems from the food industry[87, 88], where texture and mouthfeel are dominating factors for product acceptability[88].

2.6.1 Relating BPC product characteristics to sensory perception

Brummer et al.[89] defined the primary and secondary skin feelings as the sensation which occurs during and towards the end product application, respectively. They found that the primary feeling was related to the product's yield stress and dynamic viscosity which were found by measuring the shear stress and dynamic viscosity at the onset of flow by means of a stress ramp test, and that the secondary feeling was related to the stationary viscosity for a shear rate of 500s^{-1} , which is said to correspond to the shear rate applied whilst rubbing skin cream into skin. The latter was also confirmed in a study by Bekker et al. [90]. Lee et al. [91] also studied

potential correlations between panel ratings and the mechanical properties of facial moisturisers. They found that attributes relating to the appearance and pick-up of a product were correlated to rheological measurements. On the other hand, rub-out attributes correlated to both rheological and tribological measurements and residue attributes only correlated to tribological results.

In order to evaluate the sensory perception of the product, Brummer et al.[89] used a scale which gave products ratings from “poor sensory assessment” to “good sensory assessment”. They used these rating and correlated them to yield stress, maximum viscosity and stationary viscosity to define “optimal skin feeling” window for primary and secondary skin feelings. However, this method relies on a somewhat subjective perception of BPC products which limits its application in terms of a wider population. On the other hand, Lee et al. [91] used a method whereby attributes were defined and allocated to different phases of application e.g. the “pick-up” and “rub-out” phase, and each attribute was rated out of 100. They found that the $\log(\text{IVM})$ could be used to predict all attribute ratings during the appearance and pick-up phase, and that a combination of $\log(\text{IVM})$ and DCOF measurements could be used to predict the attributes *Wetness*, *Spreadability* and *Thickness* rated during the *Rub-out* phase. Lastly the attribute *Grease* was found to only be correlated to the dynamic coefficient of friction (DCOF).

Gore et al.[68] also measured the properties of different emulsions in order to relate them to results from sensory perception tests. The composition of the oil phase was modified using different ratios of two emollients: SA, a solid fatty saturated acid, and IDH a mineral oil. The authors used a photomicroscope to analyse the emulsion’s microstructure, a particle size analyser for the measurement of droplet size and a stress-controlled rheometer for evaluating flow behaviour. Results showed no difference between emulsions in terms of microstructure apart from droplet size increasing with increasing SA percentage. In terms of viscosity, at the higher rate used (1000 s^{-1} , representing the spreading of the emulsion) all emulsions had the same viscosity apart from the 2.5:7.5 IHD:SA emulsion which had a significantly higher viscosity. However, the only significant correlation found was between the spreadability scores obtained through the sensory panel and the viscosity at 0.1 s^{-1} which corresponds to the consistency of the emulsions at rest.

Another widely used technique for relating BPC product characteristics to sensory perception is the TA.XT texture analyser. A common set up seen in literature runs a weighted plate covered with a sheet of PMMA at a constant speed (3mm/s) over a static plate covered with in a sheet of polypropylene [31, 45, 68] in order to evaluate the spreadability of emulsions. Although the two materials have been found to have a similar surface energy to human skin, it is unclear why two different materials were chosen to mimic the skin-on-skin contact in this study. However, the method outlined was found to correlate with difficulty of spreading ratings given by panellists after spreading an emulsion over their volar forearms[31, 45]. Moreover, a series of other tests have been performed which yielded encouraging results. For example, the integrity of shape of the emulsion was found to be best predicted by the combination of the loss modulus, relaxation time and work of extrusion. Additionally, some results have suggested that stringiness could be predicted by the breaking length of filaments and penetration force by the extrusion and compression work[45]. Moreover, a study performed by Texture Technologies [92] used the TA.XT texture analyser to compare CVS dry skin creams and skin cleansers to the market leader's products. The product spreadability was evaluated using a cone probe which was lowered into a female cone (refer to Figure 2.10) thus forcing the product to spread out between to two surfaces. Other tests performed included a penetration test with rounded probe. Results showed the market leader's product required less force to remove the product from the container and to spread it.



Figure 2.10: TA-425 TTC Spreadability Fixture by Texture Technologies [92].

These studies confirm the potential for product texture analysis and rheological measurements to be used for differentiating products and predicting the sensory perception of attributes during product application. However, many other factors such as temperature, load and speed, would need to be considered in order to obtain an accurate predictive model. Furthermore, the results of these tests only reflect the performance of products during use and do not include the effects on skin post-application, which have been found to be the most impactful in consumer preferences.

2.6.2 Influence of friction on tactile perception

Tactile perceptions refers to the sense of touch, which is driven by the mechanoreceptors in the skin. It is mostly influenced by the frictional behaviour of skin and in particular of the human finger-pad [93]. According to the Duplex Theory proposed by David Katz [16, 94], the perception of texture depends on spatial cues for coarse textures and temporal cues for fine textures. Spatial cues depend on the height, frequency, and shape of elements whereas temporal cues relate to the friction induced vibrations (FIVs) perceived when sliding the finger across a surface. There are two types of mechanoreceptors at the interface between the epidermis and dermis. Merkel discs are stimulated by static contact at frequencies and most sensitive to frequen-

cies in the 5-15 Hz range. Meissner corpuscles are triggered by dynamic contacts at frequencies between 20-50 Hz[95]. The Pacinian corpuscles and Ruffini endings are found in the dermis. The Pacinian corpuscles are most sensitive to frequencies in the range of 200-250Hz and have a poor spatial resolution. Ruffini endings are also known as stretch receptors, as they pick up the tangential forces in the skin[95]. Sensations are thought to be dependent on the sliding direction due to the non-uniform distribution of the mechanoreceptors and the finger pad shape[15].

Massimiani et al.[16] conducted a study to understand the influence of friction and vibrations on the perceived features and hedonistic feedback of different textures. Following categorization tests with 20 assessors, four categories were defined to describe the different samples used: Smooth, rough, adhesive, and textured. The authors found no correlation between the COF and results from the sensory perception tests. However, FIVs seemed to play a major role in topographical distinction. The “smooth” samples had a frequency peak in the RA II’s most sensitive range at around 250 Hz whereas the “textured” samples frequency peak was outside of that range at around 95 Hz. The “rough” and “adhesive” samples showed a very similar frequency spectrum with frequency peaks at almost identical frequencies, although the amplitude of vibration was higher for the rough samples. These results thus demonstrated the role of frequency and amplitude in the discrimination of texture.

The main friction mechanism occurring between two smooth surfaces is adhesion [57]. This explains why softer materials will yield higher COF as the contact area is often larger. For a human finger against rough materials, it has been observed that the COF initially decreases, probably due to the reduction in contact area as the finger only touches the asperities [96]. However, as the roughness increases the asperities tend to interlock with the finger-pad ridges and the COF increases with increasing roughness due to the deformation of the skin [57]. In a study led by Tomlinson et al.[58], friction tests performed with a human finger against different materials showed an increase in COF with increasing roughness up to approximately $R_q = 26\mu\text{m}$, after which the COF started to plateau. Another observation was that the width of machine ridges seemed to have a larger effect on friction than their height did.

Zhou et al.[93] studied the influence of sliding speed on tactile perception and tribological properties. The authors conducted a perception test where fifteen participants were asked to differentiate sandpapers. At the lower speed there was a higher recognition rate for finger-pad contact versus fingertip contact. At the higher speed, the recognition rate was similar for both types of contact. The tribology tests also showed an increase in vibration signals magnitude for the higher speed, which would explain the increased perception. All frequency peaks were in the range of 0-1000Hz. The friction force showed larger variations at lower speeds for both types of contact, corresponding to stick-slip behaviour. However, the number of peaks was higher for the fingertip contact which would have significantly interfered with the tactile perception. Hence this could explain the difference in recognition rates between the two contacts at the lower speed.

Hence these findings highlight the influence of finger-pad deformation on tactile perception, as well as the pivotal role of finger-pad ridges. Hence, applying these principles, several authors derived methods for relating differences in skin frictional behaviour to the sensory perception of BPC products.

2.6.3 The role of friction in the sensory perception of BPC products

Understanding the tactile perception of BPC products requires the instrumentation of one of the contacting surfaces, often in the form of a finger-pad stroking a substrate secured to a force-plate. While some studies have preferred the use of animal skin, a number of studies have turned to the use of synthetic skin test-beds for convenience. Many authors have focussed on changes in frictional behaviour upon the application of BPC products, to deepen the understanding of various BPC product characteristics on the perception of sensory attributes.

Wang et al. [97] measured the friction of a finger-pad against the SS developed by Morales-Hurtado, described previously [98]. The authors studied the effect of different liquid crystal content on the induced frictional behaviour. The authors found that the friction during and after product application followed the Stribeck-Curve lubrication regimes. During application, the contact was dominated by a full-film lubrication. At this stage the authors hypothesized that the perception of the product would be driven by their viscosity, which was highlighted by the results mentioned in the previous section. In the instances after application the authors suggested that that the

lubrication regime would go from mixed lubrication to boundary lubrication and the effect of the products on skin would start to influence the tactile perception. Lastly, in hydrodynamic lubrication, the authors suggested that an increase in skin hydration and adhesion would lead to a *sticky* feeling, while a reduction in friction would lead to a more “smooth” sensation. While the authors did not validate these findings using sensory panels, Eudier et al. [99] did find the perceived stickiness of various BPC products to be positively correlated to the pull-off force, and hence adhesion, measured by a solid hemispherical probe against the commercially available Bioskin™ 3 minutes after product application. These findings were validated by a sensory panel which rated the perceived stickiness of various products on their forearm as well as on the Bioskin™ using their hand.

Skedung et al. [75] measured friction for a human index finger against the commercially available artificial skin model Vitro-Skin™ on a force plate with tangential and horizontal load cells. The authors used normal loads between 0.5 and 1N, which was reported as the optimum load for assessing a tactile stimulus, and a sliding speed of 14 mm/s. Three skin creams were used for the experiments, each with different types of hydrating mechanisms. The creams were spread using a gloved finger so as to obtain 3 mg/cm². The friction was measured immediately (0 minutes), 10 minutes and 60 minutes after application. After a reduction in COF for the three formulations immediately after application compared with the untreated control, after 10 minutes the friction was found to reach similar values to the untreated SS for two of the formulations (X and Y). A slower increase was observed for the third (Z). These differences highlighted the fact that the humectant was not solely responsible for this increase in COF as it was present in Z but not in Y. The authors also associated an increase in the perceived smoothness of the skin to an increase in hydration. The increase in the perception of *smoothness* following the cream treatment of pig skin was also found by Ding et al. [100], which the authors associated to a decrease in the friction induced vibration signal peak amplitudes.

Vitro-Skin™ was also used in a study by Tang et al. [41] to depict the different tactile sensations recognised by consumers when applying skin cream. Using the commercial artificial finger BioTac SynTouch LLC as a probe, the authors measured the frequency of vibrations for the perception of fineness, the coefficient of friction to describe the perceived stickiness, adhesive force to understand the perceived greasiness and vertical deviations for perceived roughness. A sensory panel was used in parallel to rate

these attributes on a scale of 0 to 100, for cream-treated samples of varying cream film thicknesses. The authors found that an increase in vibration frequency, COF and adhesion all lead to an increase in perceived fineness, stickiness and greasiness respectively, and a reduction in vertical deviations lead to an increase in perceived smoothness. However, these tests were only repeated for the same product, with varying amounts applied to the skin.

Lastly, Horiuchi et al. [101] also used an experimental set-up where in the case of both the probe and substrate, human skin was replaced by synthetic skin. The authors used a silicone-rubber with a Young's modulus value of 0.5 MPa to manufacture a hemispherical probe of 16 mm in diameter, a flat smooth test-bed and a flat grooved test-bed. The authors used the measurements of static, kinetic friction and vibration to predict the attributes *smoothness*, *silky feeling*, *velvety feeling*, *softness* and *skin adhesion ability*. The attributes were rated by a trained panel for comparison. Through a multiple regression analysis, strong correlations were found for various combinations of the measured variables and each attributes. However, these were not found to be significant, which the authors attributed to the potential non-linearity of human skin's response to a stimuli.

2.6.4 Summary of the methods related to the sensory perception of BPC products

Tables 2.4, 2.5 and 2.6 summarise the methods used in the studies related to the assessment of the effects of BPC products on the frictional behaviour of skin. The variety in methodologies highlights the absence of a standard protocol for relating the measurements of friction to the sensory perception of BPC products.

Various materials have been employed in studies assessing BPC products, including polypropylene [31], PMMA [68], and silicone rubber [101]. While these materials can mimic certain characteristics of human skin, none offer a comprehensive solution that combines multiple skin-like properties, such as water absorption and mechanical behaviour, into a single test-bed. Other synthetic skin test-beds developed, like the one proposed by Morales-Hurtado et al. [18], require intricate and time-intensive fabrication processes, including 48 hours of curing under controlled temperature conditions, which limits their practicality and reproducibility in fast-moving industrial settings.

Table 2.4: Summary of methods used in the assessment of the effects of BPC product on the frictional behaviour of skin.

Author	Title	Test-bed	Probe	Normal load	Speed	Sliding distance	Product amount
Ali et al. [102]	Tactile friction of topical creams and emulsions: Friction measurements on excised skin and VitroSkin TM using ForceBoard TM	VitroSkin TM and pig skin (ear)	Human finger	0.5 N	Missing	40 mm	2.5 mg/cm ²
Sivamani et al. [61]	Tribological testing of skin products: gender, age, and ethnicity on the volar forearm	Proximal and distal parts of human forearms	Copper cylindrical friction/electrical probe (diameter 16mm)	20 g	0.4 mm/s	10 mm	0.5 mg/cm ² - 3 mg/cm ²
Horiuchi et al. [101]	Relationship between tactile sensation and friction signals in cosmetic foundation	Flat silicone rubber (Thickness : 5mm).	Spherical silicone rubber (diameter 16mm)	0.2 N	20 mm/s	Missing	Missing
Tang et al. [103]	Adhesion, friction and wear characterization of skin and skin cream using atomic force microscope	Rat skin	AFM	25-250 nN	20 μ m/s	10 μ m/s	1 - 3 mg/cm ²
Ding et al. [100]	Tactile Perception of Skin and Skin Cream by friction induced vibrations	Pig skin, VitroSkin, PMMA	Synthetic finger	50mN - 200mN	20mm/s - 30mm/s	30 mm	1.2 mg/cm ²

Table 2.5: Summary of methods used in the assessment of the effects of BPC product on the frictional behaviour of skin (cont.)

Author	Title	Test-bed	Probe	Normal Load	Speed	Sliding distance	Product amount
Skedung et al. [75]	Tactile friction of topical formulations	Vitro-Skin	Human finger	0.5-1 N	14 mm/s	Missing	5 mg/cm ²
Savary et al. [31]	Impact of emollients on the spreading properties of cosmetic products: A combined sensory and instrumental characterization	Polypropylene	PMMA plate	207.9 g	3 mm/s	6 cm	4 drops of 40 μ L each
Gore et al. [68]	Spreading behavior of cosmetic emulsions: impact of the oil phase	Polypropylene and human volar forearm	PMMA plate/ Teflon disk probe (16 mm dia)	Missing	90 rpm	Continuous for 5 minutes	40 μ L
Tang et al. [41]	Tactile Perception of Skin and Skin Cream	VitroSkin	BioTac, Syn-touch artificial finger	1.5 N	10 mm/s	30 mm	1.2 mg/cm ² - 5 mg/cm ²
Wang et al. [97]	Study on the development of wax emulsion with liquid crystal structure and its moisturizing and frictional interactions with skin	Synthetic Skin [98]	Human finger	0.4 5N	Missing	Missing	5 mg/cm ²

Table 2.6: Summary of methods used in the assessment of the effects of BPC product on the frictional behaviour of skin (cont.)

Author	Title	Test-bed	Probe	Normal load	Speed	Sliding distance	Product amount
Tang et al. [10]	Friction, adhesion and durability and influence of humidity on adhesion and surface charging of skin and various skin creams using atomic force microscopy	Rat dorsal skin	Etched silicon probe	25– 250 nN	20 μ m/s	10 μ m/s	0.2 mg/cm ²
Masen et al. [64]	Evaluating lubricant performance to reduce COVID-19 PPE-related skin injury	Human cheek skin	Motorised wheel shaped probe	1.1 N	18.4 mm/s	Continuous for 6 seconds	2 mg/cm ²
Gilbert et al. [45]	Predicting sensory texture properties of cosmetic emulsions by physical measurements	Polypropylene	PMMA plate	207.9 g	3 mm/s	120 mm	200 μ L

2.7 Conclusion

The skin is a complex, multifunctional material. Its mechanical behaviour is influenced by the orientation of collagen and elastin fibres, making its deformation direction-dependent. Additionally, the Stratum Corneum's ability to absorb moisture from the environment increases the skin's compliance. Surface properties, such as texture and surface energy, also play a significant role, as they can affect the adhesion and wettability of beauty and personal care (BPC) products during application.

In order to understand the mechanisms involved in the perception of different BPC product, it is essential to instrument the skin-on-skin interactions and hence replicating skin in the form of a synthetic skin Test-bed is especially relevant. Although some studies have proven successful in relating the characteristics of BPC products to the perception of attributes during the application process, the tactile perception after application, when the lubrication enters the mixed or boundary regimes, remains the main driver in consumer preferences.

Several authors have studied changes in skin frictional behaviour following BPC product application on commercially available or newly developed synthetic skin test-beds, and were successful in correlating these measurements to changes in sensory perception. However, the processes involved in the development of the aforementioned SS test-beds are often complex, and commercially available options incur significant costs. Moreover, the variety of the attributes studied remained limited, and their definitions unclear.

There is a clear need to identify which skin characteristics most strongly influence the sensory perception of BPC products, in order to refine the design of a synthetic skin test-bed. Doing so will support the refinement of synthetic skin test-bed designs, rendering them more practical and effective for industrial application.

This thesis will explore the influence of BPC products of varying characteristics on the frictional behaviour of human skin. Combined with the existing sensory capabilities in Unilever, the findings will be used to inform the design of a synthetic skin test-bed for use in the sensory assessment of BPC products.

2.8 Aim and objectives

The aim of the PhD is to develop a methodology for assessing attributes related to the sensory perception of beauty and personal care products.

The objectives are as follows:

Understand the differences between BPC products and their effect on the frictional behaviour of human skin

- Perform rheology measurements on BPC products used for sensory tests provided by Unilever,
- Relate findings to specific product compositions,
- Develop a test protocol based on sensory panel methodology and findings from the literature review,
- Evaluate changes in the frictional behaviour of human skin in the presence of BPC products,
- Assess whether assessed variables can be used to predict attribute scores.

Understand the effect of synthetic skin test-bed design element on its frictional behaviour

- Manufacture synthetic skin models of various properties and perform friction experiments
- Analyse trends and relate findings to human volar forearm measurements.

Develop a methodology for assessing BPC products on a synthetic skin test-bed

- Combine effective design elements into one test-bed and replicate human volar forearm tests
- Relate findings to existing sensory test data provided by Unilever,
- Determine if the method developed is a suitable prototype screening tool in an industry setting.

Chapter 3

Beauty and personal care product characterisation and effect on the frictional behaviour of skin

3.1 Introduction

Consumers' perceptions of sensory attributes related to beauty and personal care (BPC) products begin even before the product is applied to the skin. Previous studies have demonstrated that characterising BPC products through rheological measurements can predict sensory attribute scores related to the initial pick-up of the product by the consumer [91]. Furthermore, it has been shown that combining tribological and rheological measurements allows prediction of sensory attributes rated during product application on the skin [91, 89].

The gold standard for evaluating the sensory perception of BPC products involves trained sensory panels. These panels, consisting of several trained panellists, evaluate a range of sensory attributes using a predefined scale. In this study, panel sensory data for five BPC products, along with a list of attribute definitions, were provided by Unilever. During panel testing, sensory attributes are evaluated at specific stages of product use and scored on a scale of 0 to 100.

Specifically, Unilever's evaluation procedure categorizes "in-use skin feel" attributes as those rated during the first 16 rubs of product application, known as the "rub-out phase." This phase is divided into two parts: the first 8 rubs and the subsequent 8 rubs. "After-use" attributes are assessed immediately after application and again 5 minutes later. These after-use scores are believed to be influenced by changes in the

frictional properties of the skin [89, 91, 97]. Because skin creams are not rinsed off, the residue left on the skin has a prolonged impact on sensory perception.

The research question addressed in this chapter is how differences in product composition, intended function, and rheological properties influence the frictional behaviour of human skin. The goal is to understand how these factors correlate with sensory perception.

Rheology measurements were conducted using adapted methodologies from the studies cited in the literature review chapter. Similarly, the tribological experiments were informed by Unilever’s sensory panel protocols and supplemented with methods from literature. Five products were evaluated using rheological measurements. Of these, three were selected for tribological testing to measure changes in skin friction immediately and 5 minutes after application. The protocol used for the friction measurements was designed to replicate the conditions of sensory panel testing as closely as possible. Friction experiments were carried out on the volar forearm of 12 human participants at both time intervals. Testing on human subjects was important for gaining a clearer understanding of how different products influence the frictional behaviour of skin. Additionally, these results served as a critical benchmark for future comparisons with the developed synthetic skin test-beds.

3.2 BPC product sensory evaluation at Unilever

The sensory evaluation at Unilever is conducted in a temperature and humidity controlled laboratory. Panellists are trained over a period of several months to apply the BPC products, as well as recognise and rate attributes. Firstly, 0.2 ml of product is dispensed onto the panellists’ fingers. The individuals then spread the product onto their skin using gentle oval motions, for a maximum of 120 seconds. Several attributes are evaluated at different points in the application process. The “in-use skin feel” attributes, are evaluated during the first 8 rubs, and the next 8 rubs. Details of the attribute definitions are given in Table 3.1 below. The “after-use skin feel” attributes detailed in table 3.2, are evaluated immediately and 5 minutes after the end of the product application. It should be noted that although panellists are trained to recognise and rate attributes objectively, there is an inevitable bias, as the attributes are defined by Unilever.

Table 3.1: In-use attributes and definitions, provided by Unilever.

Stage	Attribute	Definition
In-use (first 8 rubs)	Wetness	Amount of water perceived while rubbing.
	Creaminess	A substance that spreads smoothly and evenly glides between the fingertips.
	Spreadability	The ease of moving the product across the skin.
	Thickness	Amount of product felt between the fingertips and skin.
In-use (next 8 rubs)	Oily	The perception of a slippery, fatty substance. Light/Slick.
	Greasy	The perception of a slippery, fatty substance. Heavy/Thick.

Table 3.2: After-use attributes and definitions, provided by Unilever.

Stage	Attribute	Definition
After-use (Immediately and after 5 minutes)	Drag	The difficulty of moving fingers across the skin.
	Oily	The perception of a light, slick, fatty film remaining on the skin.
	Greasy	The perception of a thick, slippery, fatty film remaining on the skin.
	Waxy	The perception of a heavy and somewhat draggy dry coating remaining on the skin
	Silky Smooth	A surface textural assessment rating the degree of ease to which fingers glide across the surface of the skin due to a silicone-type feel
	Powdery Smooth	The degree to which fingers glide across the surface of the skin due to a light, dry talky-type feel.
	Sticky Tacky	The degree to which a finger adheres to residual products and skin pulls up from the forearm (while tapping downward on the skin

3.3 Beauty and personal care product characterisation

The products assessed by the panel and tested in this chapter are listed in Table 3.3 below. They will be referred to using the product code for the remainder of the thesis. The five ingredients found at the highest concentrations as well as their function in brackets have also been included in the table, as these will likely have the most impact on product characteristics. As mentioned in Chapter 2, occlusives form a barrier which prevents water loss from the skin. Emollients are oils which are considered partial occlusives [39] are less dense and more easily spreadable. Lastly, humectants attract water molecules from the deeper layers or from the air into the

more superficial layers of the skin.

Typically, body lotions such as Product A and Product B are more easily spreadable as they are applied to larger surface areas. Table 3.3 shows that both of these products contain water at the highest concentration. However, A contains an occlusive as the second highest concentration as opposed to a humectant for B. Hence it would be expected for B to have a more pronounced hydrating effect than A. In contrast, with the occlusive Petrolatum being present at the second highest concentration, it should be expected that A would have a slightly thicker consistency. However, a hydrating effect should also be observed for A as it also contains Glycerin as a humectant, as the fourth ingredient. On the other hand, C is a pure occlusive which would imply more difficulty in spreading and forming a stronger layer of product on the skin rather than any immediate hydrating effect. Hence it can be assumed that the product characteristics would be expected to dominate the contact for an extended period of time. Product D, like C, has an occlusive as its primary ingredient. However, with water as a second ingredient it can be expected to have a thinner consistency and be more spreadable than C. Lastly, Product E is the only product specifically marketed as a facial moisturiser. In this case, the two ingredients found in the second and third highest concentration after water are emollients, strengthening the skin barrier effect whilst still maintaining a light feel and increase in skin hydration with the presence of humectants as the fourth and fifth ingredients.

Table 3.3: List of the ingredients at the five highest concentrations for the BPC products used.

Product code	Ingredients at the five highest concentrations
Product A	Water (Solvent) Petrolatum (Occlusive) Mineral oil (Emollient) Glycerin (Humectant) Stearic Acid (Emollient)
Product B	Water (Solvent) Glycerin (Humectant) Stearic Acid (Emollient) Isopropyl Myristate (Emollient) Mineral Oil (Emollient)
Product C	Petrolatum (Occlusive)
Product D	White Petrolatum (Occlusive) Water (Solvent) Caprylic/Capric Triglyceride (Emollient) Stearic Acid (Emollient) Glycerin (Humectant)
Product E	Water (Solvent) Palmitic Acid (Emollient) Stearic Acid (Emollient) Niacinamide (Humectant) Glycerin (Humectant)

3.3.1 Methodology for the rheology measurements

Rheology measurements were taken for the products in the Texas Instruments Rheometer (Discovery HR 10, TA Instruments). The Cone-plate geometry was used with a 2 °angle. The truncation gap, which is the gap between the tip on the cone and the plate, was set to 53 μm . For cone-plate geometries the truncation gap to use is specified by the manufacturers. Figure 3.1 shows the set-up used for the rheology experiments. Three repeats of each test were performed per product and the resulting data sets were averaged point by point to give a single data set per product. The methods outlined in this section were adapted from the study led by Lee et al. [91].

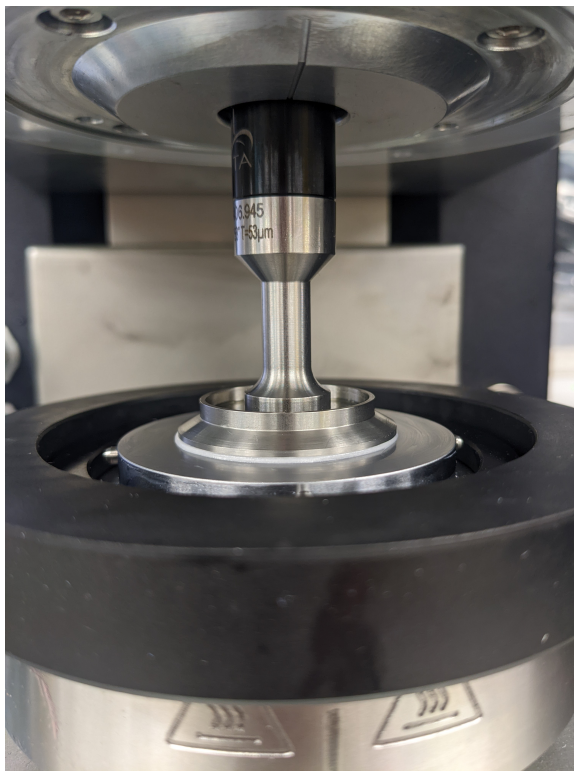


Figure 3.1: Cone and plate set up used for the rheology assessment of BPC products.

A stress ramp test was performed to measure the yield stress and instantaneous viscosity maximum (IVM) of the different BPC products. The shear stress was linearly increased from 0 to 100 Pa and the viscosity was measured every second. For product C the stress was increased to 1000 Pa as the yield stress was not reached after 100 Pa. A pre-shear was also applied for one second at 30s^{-1} to replicate the shear undergone by the product as it is removed from its container. The tests were performed at a temperature of 25°C as at the start of the application the assumption is that the skin cream is still at room temperature and has not yet been heated up by the skin or the application process. To achieve this, once the product has been deposited onto the Rheometer, the products were “soaked” for 120 seconds at 25°C before the start of the test. The values for IVM were taken as the maximum viscosity over the test period and the yield stress was taken as the stress at maximum viscosity.

The stationary viscosity was measured by applying a shear rate of 500 s^{-1} over a period of 120 seconds. The BPC product was first soaked for 120 seconds at a temperature of 38°C as it is assumed that towards the end of the product application

the heat from the human skin has been transferred to the product. The values for stationary viscosity were taken as the viscosity after 30 seconds. A shear rate sweep was also performed as a separate test, whereby the shear rate was increased from 0.1 to 30s^{-1} with 10 points per decade and 10 seconds per point, and then decreased from 30s^{-1} to 0.1s^{-1} .

3.3.2 Results from the rheology measurements

Results of the stress ramp test performed at 25°C are shown in Figure 3.2 below. The product with the highest yield stress and IVM was C. For Product B, which is a body lotion, the measured dynamic viscosity maximum was 724.5 Pa.s which was the lowest of all products. Product E had the lowest yield stress meaning it required the least amount of stress at the onset of flow. The values for all products are detailed in Table 3.4 below.

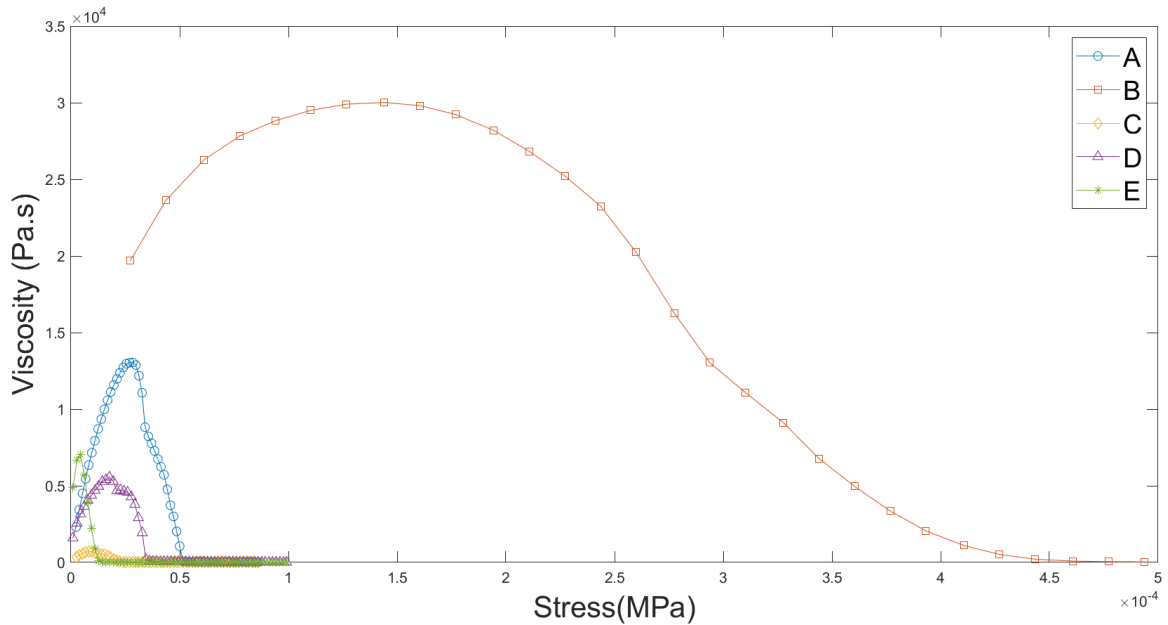


Figure 3.2: Results of a stress ramp test for different BPC products at 25°C .

Table 3.4: Yield stress and instantaneous viscosity maximum (IVM) at 25°C for BPC products.

Product	Yield Stress at 25°C (Pa)	IVM at 25°C (Pa.s)
A	28.4	13731.0
B	8.14	775.4
C	144.2	30014.0
D	17.7	5564.7
E	4.37	7061.4

The results for the shear peak hold tests are pictured in Figure 3.3 below and the viscosity after 30 seconds are reported in Table 3.5.

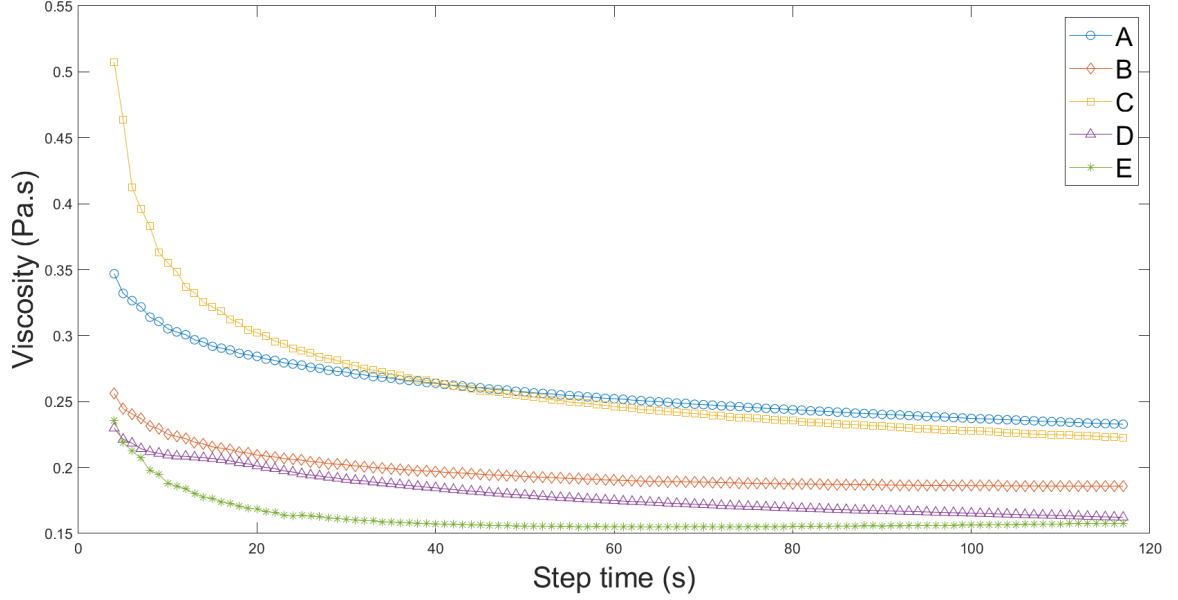


Figure 3.3: Results of the shear peak hold test for different BPC products at a shear rate of 500s^{-1} at 38°C .

Table 3.5: Mean viscosity for a constant shear rate of 500s^{-1} and at 38°C .

Product	Viscosity at 30 s (Pa.s)
A	0.272
B	0.202
C	0.279
D	0.191
E	0.161

Lastly, Figure 3.4 shows the results from the shear sweep test. Shear sweep tests highlight the thixotropic nature of certain BPC products, which is characterised by a time-dependent and reversible decrease in viscosity [104]. C exhibited the most thixotropic characteristics which is illustrated by a larger area between its upshear and downshear curves. On the other hand, there was almost no difference in viscosity for B between upshear and downshear.

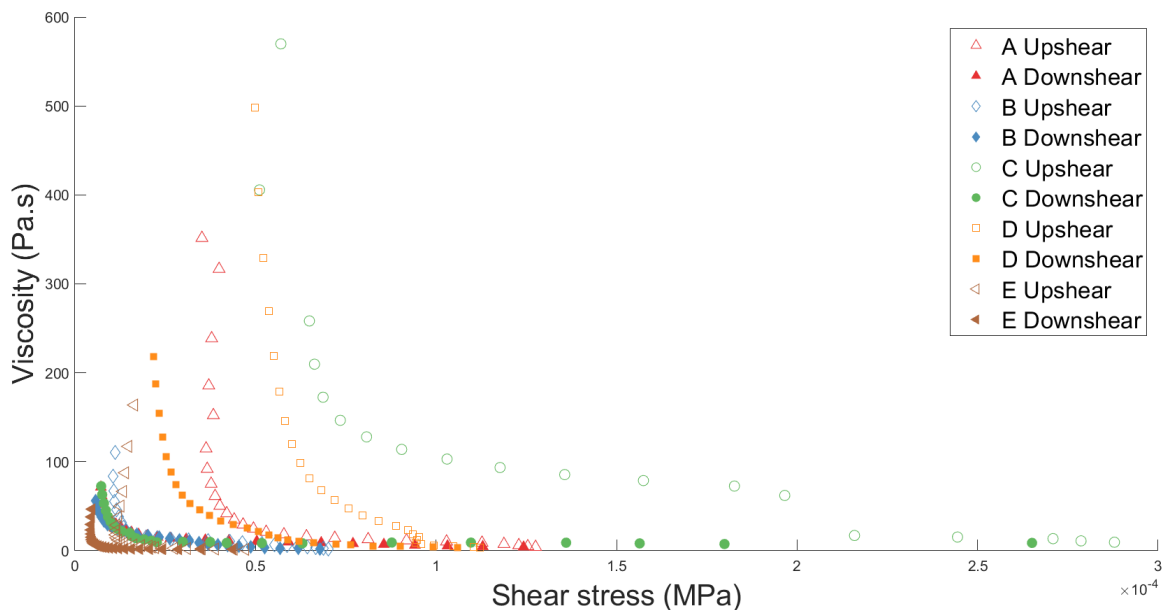


Figure 3.4: Results from the shear sweep test.

From the results of these initial measurements, a selection of three BPC products, A, B and C, was made for the next set of experiments. Their significantly different dynamic viscosities and differences in terms of composition was deemed as essential for facilitating the identification of trends in the data when focusing on the “after-use skin feel”. Product C is made up of a single ingredient and its only function is to occlude the skin i.e. to stop water from seeping out. It can therefore be assumed that its composition will exhibit little to no variation in the minutes following application. In contrast, Product B has a humectant at the second highest concentration which, combined with its water content, should incur more rapid changes in the skin’s hydration level. Water evaporation is also expected almost immediately after application which may influence the sensory perception. Product A integrates characteristics from the two prior products with both occlusive and humectant ingredients at high concentrations after water. Lastly, although not essential, the commercial availability of these products in the UK was of practical relevance.

3.4 Frictional behaviour of the BPC products

3.4.1 Methodology for the friction measurements

A total of 12 participants, 4 females and 8 males, were recruited for this study. The participants were asked for their age and ethnicities, which are listed in Table

3.6. Ethical approval was obtained from the University’s Research Ethics committee prior to commencing the experiments (Application 056989, refer to the Appendix).

Table 3.6: Participant ethnicities, age and sex.

Ethnicity	Age (years)	Sex
English, Welsh, Scottish, Northern Irish or British (White)	29	M
English, Welsh, Scottish, Northern Irish or British (White)	25	F
English, Welsh, Scottish, Northern Irish or British (White)	25	M
English, Welsh, Scottish, Northern Irish or British (White)	23	F
Any other White background	27	F
Any other Asian background (Asian or Asian British)	29	F
English, Welsh, Scottish, Northern Irish or British (White)	28	M
Arab	35	M
English, Welsh, Scottish, Northern Irish or British (White)	28	M
African	25	M
Chinese (Asian or Asian British)	27	M
White and Asian (Mixed or multiple ethnic groups)	28	M

The three products used for these tests were the Product C, an occlusive, Product A, with occlusive and humectant properties, and Product B containing mainly humectants. Refer to Table 3.3 for further details on the specific product compositions and functions. The volar forearm was chosen as its lack of hair and limited exposure to sunlight allow for a reduction in variations between participants [61]. Moreover, the relatively flat surface allows for a large testing area, it is easily accessible and offers enough manoeuvrability and control for participants to move it across a probe at a controlled speed and load.

Participants were asked to refrain from using any BPC products other than soap on their forearms for 24 hours before testing. Prior to the first measurements, the participants’ volar forearms were wiped clean using a paper towel impregnated with rubbing alcohol, and left to dry at room temperature. In the interest of identifying any outliers and improving the understanding of skin behaviour, the skin moisture levels were measured using a Moistsense device (Moritex Europe). This device measures the capacitance of skin and converts it to a scale of 1 to 99 in arbitrary units (au), with lower values corresponding to dryer skin. Three moisture readings were taken at different points on the volar forearm. An area of 75 cm² was then drawn onto the volar forearms of each participants, as depicted in Figure 3.5. The friction was measured on the dry volar forearm, as explained in the next section. 0.2 +/- 0.01 g

of product was then weighed out and spread evenly over the entire drawn area for 60 seconds with a gloved finger so as to not contaminate the product. This resulted in a product distribution of 2.6 mg/cm^2 , which is within the range of product quantities used in literature [75, 64, 102, 41]. Weighing out the product was preferred as a more controlled and repeatable method over volumetric measurements. At the end of the 60 seconds a 5 minute timer was set. The friction was first measured immediately after the 60 seconds of application and the second measurement was taken 5 minutes after the end of the 60 second product application. The two pictures in Figure 3.5 show the visual difference in the appearance of the product treated skin. After 5 minutes the product is no longer identifiable, due to a combination of evaporation and absorption of ingredients.



Figure 3.5: Marked test zone on a participant's volar forearm immediately (left) and 5 minutes (right) after Product B was applied.

The friction was measured using a probe of 16 mm in diameter manufactured from PMMA and screwed onto a force plate, as shown in Figure 3.6. PMMA had been used in previous studies related to the sensory perception of BPC products due to its surface energy being similar to human skin [68, 45, 31]. A more detailed experimental approach to evaluating the effect of probe material will also be described in Chapter 4. Participants were asked to rub their volar forearm back and forth six times (with one direction equivalent to one time) in a linear trajectory against the probe, whilst

maintaining a target normal load between 0.5 and 1N. This has been reported as the range of forces typically used for tactile exploration [96, 75], and is therefore more likely to match the loads used by panellists when rating the sensory attributes for the various BPC products.

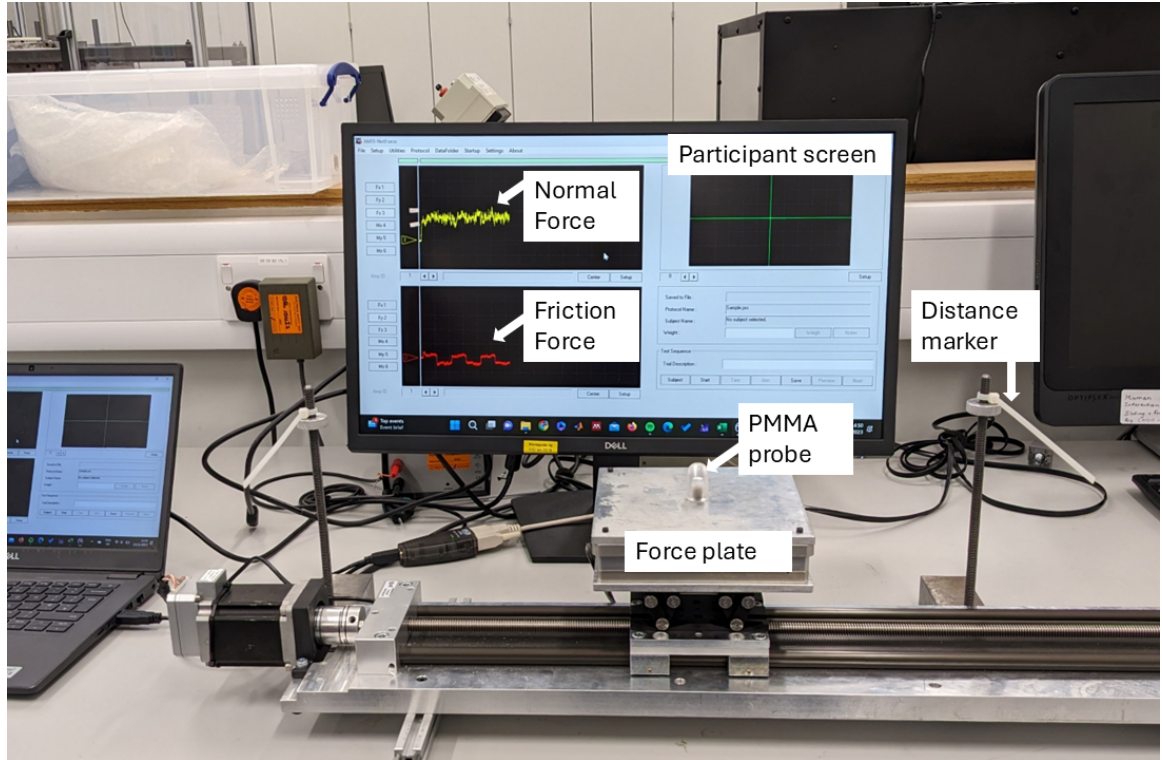


Figure 3.6: Test set-up for measuring the coefficient of friction of human volar forearm skin.

The normal load appeared on a screen in front of the participants in real time as demonstrated in Figure 3.7, which allowed them to adapt the force accordingly. The participants were also given distance targets, as shown in Figure 3.6 and 3.7, whereby the direction of sliding would change upon contact with the fist on one side and the elbow on the other. These were placed so as to achieve a total sliding distance of 130 mm in each direction. During the sliding, verbal guidance was given by means of counting out loud to 13 seconds, in order to achieve a target speed of 10 mm/s. This speed was used in a similar study by Tang et al. [41] and has been reported as the lower limit of speeds typically used for tactile exploration [96]. It was also deemed appropriate as it is likely that higher speeds would render maintaining the normal load within the desired range more challenging. The participants were also given an

opportunity to practice the sliding speed and load before their forearms were cleaned for the experiment.

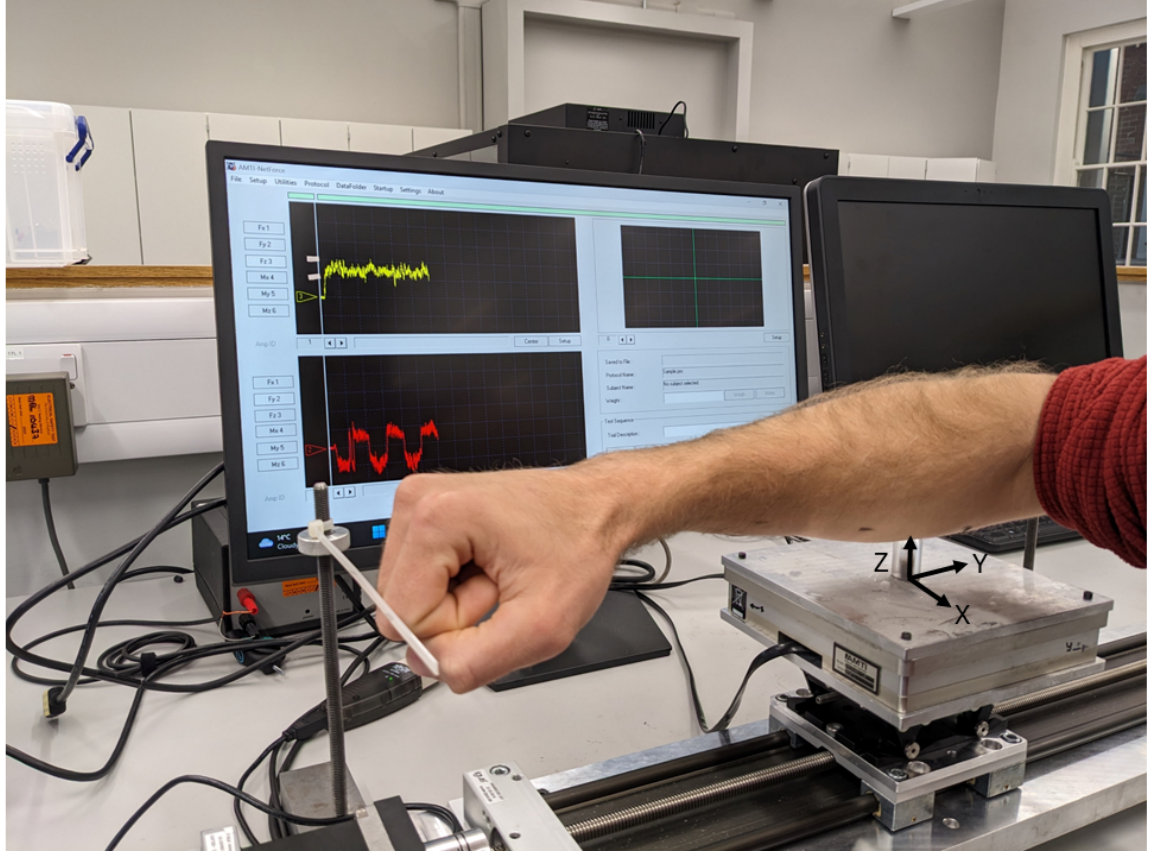


Figure 3.7: Forearm position during the tests with X,Y and Z axes.

The force plate recorded the horizontal friction force in the X and Y directions, as pictured in Figure 3.7, as well as the vertical normal force in the Z direction at a sample rate of 0.02. The resultant friction force was calculated by using Equation 3.1. Each data point was then divided by the normal force at the corresponding point in time which gave a dataset for the COF over time.

$$F_f = \sqrt{F_y^2 + F_x^2} \quad (3.1)$$

3.4.2 Results from the friction measurements

An example of the resulting data for normal force, friction force and COF over time is pictured in Figure 3.8. Each change in direction is marked by the sudden drop in friction force. At the start of each slide the COF transitions from static to dynamic. To ensure only the dynamic COF (DCOF) was considered for the calculation of the

overall mean, the portion of each “plateau” where true sliding was occurring was extracted from the overall data and averaged. The selected data is highlighted by the red data points in Figure 3.8. As the sliding speed was not recorded, the selection was made in Matlab where 6 seconds worth of data points were selected every 13 seconds with a 6 second delay at the start. The decision to exclude the data either side of this portion was made to ensure any abrupt changes in velocity would not affect the mean DCOF, as well as the deformation of the skin from changing direction which could be recorded as friction whilst no sliding was occurring. The measured DCOF for each participant and for the different BPC products are given in Table 1 in the Appendix.

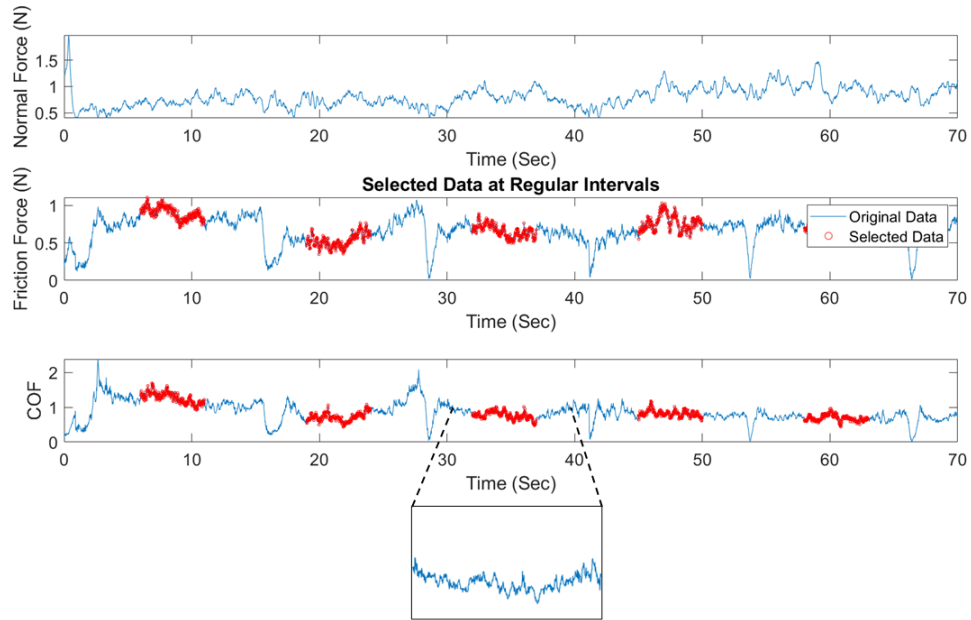


Figure 3.8: Example of the raw friction and normal force data along with calculated COF over time for a human volar forearm 5 minutes after the application of a BPC product. The box shows a close-up of the variation in DCOF during one slide.

The average moisture level for each participant was plotted against the mean DCOF measured on their dry forearm in order to assess any correlation. The resulting plot is pictured in Figure 3.9 below. While hydration levels have been previously found to be positively correlated to the DCOF of skin [54], in this case there was no significant correlation between the two variables ($R^2 = 0.290, F(1, 10) = 4.088, p = 0.071, n = 12$). However, this result was not unexpected as it is likely that the DCOF of human skin will be influenced by many different factors.

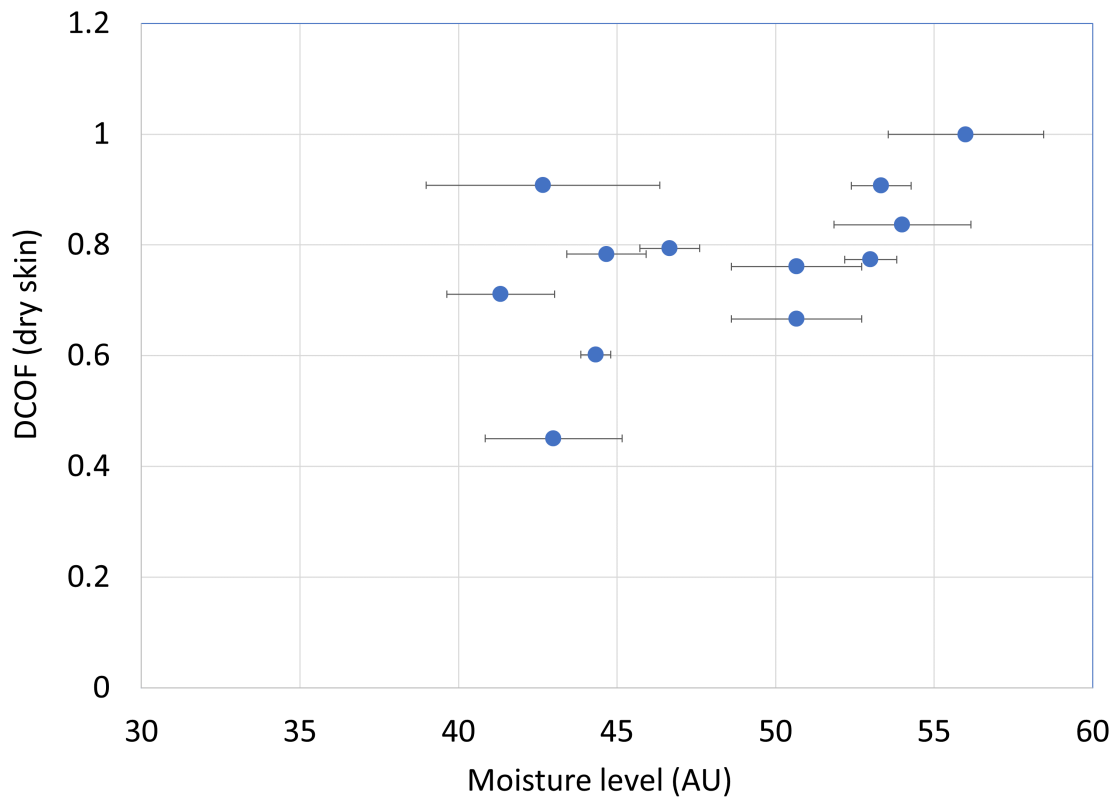


Figure 3.9: Moisture levels across participants.

The overall mean DCOF for the different BPC products immediately and 5 minutes after application are plotted in Figure 3.10, along with standard deviations. Results from a paired t-test, details of which are given in Table 3.7, showed that the mean DCOF was significantly greater after 5 minutes for both A and B ($p < 0.05$). There were however no significant differences between the DCOF immediately and after 5 minutes for C. The results also showed that all DCOF values measured on the skin after product application were significantly lower than the values measured on the dry skin (Table 3.8) ($p < 0.001$) with the exception of B after 5 minutes which showed no significant differences.

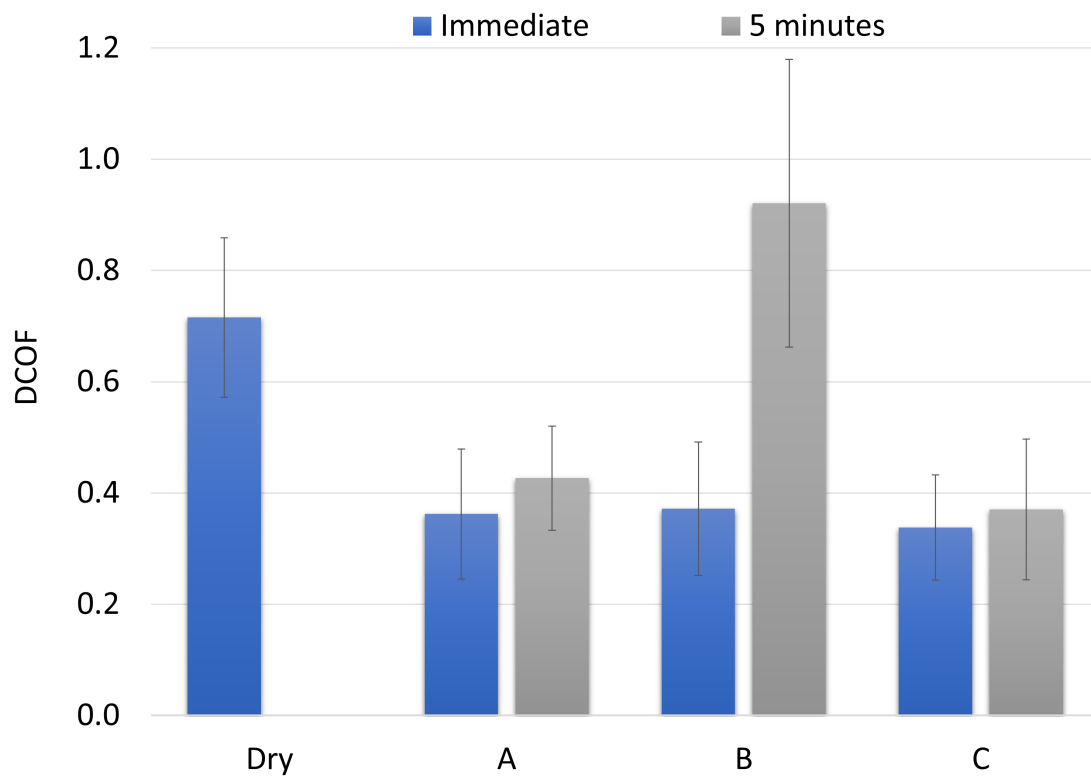


Figure 3.10: Mean dynamic COF with standard deviations on dry and product treated human volar forearms (n=12) immediately and 5 minutes following product application.

Table 3.7: Results from a paired t-test to evaluate the significance of the differences in mean DCOF immediately and 5 minutes after application.

	Mean Difference	t	df	Significance	
				One-Sided p	Two-Sided p
A (Imm) - A (5 min)	-0.064	-2.305	11	0.021	0.042
B (Imm) - B (5 min)	-0.549	-7.175	11	<0.001	<0.001
C (Imm)- C (5 min)	-0.032	-0.939	11	0.184	0.368

Table 3.8: Results from a paired t-test to evaluate the significance of the differences in mean DCOF for the dry versus treated skin.

	Mean Difference	t	df	Significance	
				One-Sided p	Two-Sided p
Dry - A (Imm)	0.404	6.614	11	<0.001	<0.001
Dry - B (Imm)	0.394	6.522	11	<0.001	<0.001
Dry - C (Imm)	0.428	7.826	11	<0.001	<0.001
Dry - A (5 min)	0.339	6.063	11	<0.001	<0.001
Dry - B (5 min)	-0.155	-1.556	11	0.074	0.148
Dry - C (5 min)	0.396	11.862	11	<0.001	<0.001

Table 3.9: Results from a paired t-test to evaluate the significance of the differences in mean DCOF between products immediately and 5 minutes after application.

	Mean Difference	t	df	Significance	
				One-Sided p	Two-Sided p
B (5 min) - A (5 min)	0.494	6.259	11	0.000	0.000
B (5 min) - C (5 min)	0.551	5.526	11	0.000	0.000
A (5 min) - C (5 min)	0.056	1.089	11	0.150	0.299
B (Imm) - A (Imm)	0.010	0.317	11	0.378	0.757
B (Imm) - C (Imm)	0.033	0.665	11	0.260	0.520
A (Imm) - C (Imm)	0.024	0.486	11	0.318	0.637

The box highlighted in Figure 3.8 shows the variations in the DCOF data. To obtain a better understanding of these variations and to understand whether they were caused by stick-slip friction, the coefficient of variation (COV) was calculated by dividing the standard deviation within the data-set selected by the method outlined above, by the mean COF of that same data set. Stick-slip is characterised by an oscillating sliding motion which tends to occur when the static COF is greater than the DCOF [52]. Derler and Rotaru [52] reported that a COV lower than 10% corresponded

to stationary sliding whereas stick-slip could be characterised by a COV greater than 25% [52]. The different COV for the participant data as well as the standard deviation between participants is shown in Figure 3.11. In all cases the mean COV lies above 10% but below 25% which according to Derler and Rotaru would be considered non-stationary sliding, meaning that the higher variations in friction force observed are more likely due to variations in tissue response than stick-slip. However, taking a closer look at the individual COV data, pictured in Figure 3.12, revealed that the COV was above 25% for a number of participants. This is especially true for the friction measurements taken immediately after application, with the most found for Product A ($n=5$) and the highest lying above 40% for the same product.

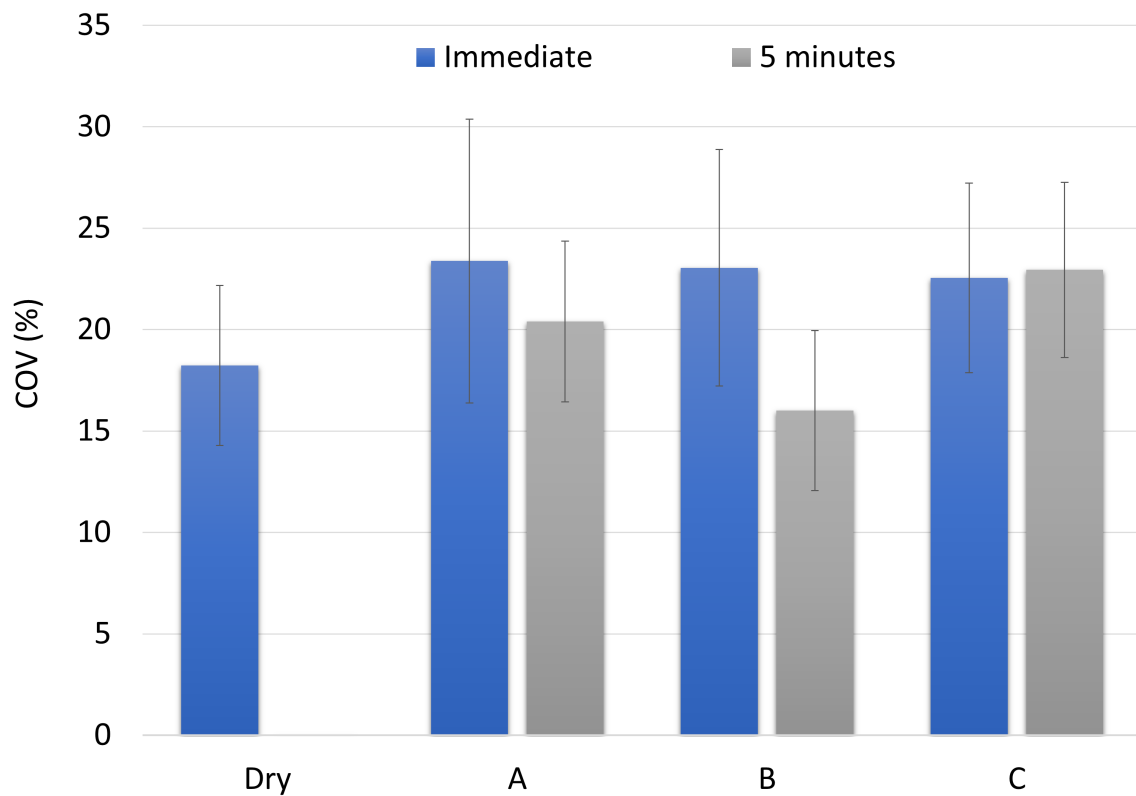


Figure 3.11: Mean coefficient of variation (COV) with standard deviations on dry and product treated human volar forearms ($n=12$) immediately and 5 minutes following product application.

Results from a paired t-test, details of which are given in Table 3.10, showed that the decrease in COV after 5 minutes was significant for A and B ($p < 0.05$). However, there were no significant differences between means for C. Additionally, the only

significant difference between the dry and the product treated skin was found for B after 5 minutes (Table 3.11) for which the COV was lower ($p < 0.05$).

Table 3.10: Results from a paired t-test to evaluate the significance of the differences in mean COV immediately and 5 minutes after application.

	Mean Difference	t	df	Significance	
				One-Sided p	Two-Sided p
A (Imm) - A (5 min)	0.030	1.926	11	0.040	0.080
B (Imm) - B (5 min)	0.070	5.119	11	<0.001	<0.001
C (Imm)- C (5 min)	-0.004	-0.345	11	0.368	0.736

Table 3.11: Results from a paired t-test to evaluate the significance of the differences in mean COV for the dry versus treated skin.

	Mean Difference	t	df	Significance	
				One-Sided p	Two-Sided p
Dry - A (Imm)	-0.040	-1.484	11	0.083	0.166
Dry - B (Imm)	-0.036	-1.638	11	0.065	0.130
Dry - C (Imm)	-0.031	-1.391	11	0.096	0.192
Dry - A (5 min)	-0.010	-0.508	11	0.311	0.621
Dry - B (5 min)	0.034	1.941	11	0.039	0.078
Dry - C (5 min)	-0.035	-1.605	11	0.068	0.137

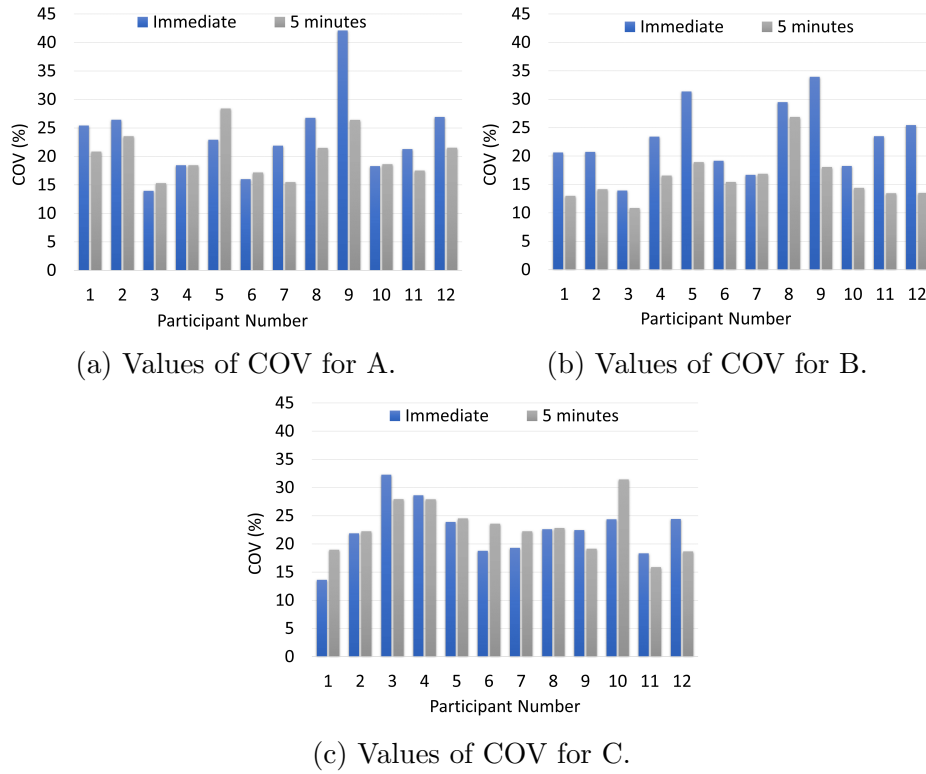


Figure 3.12: Values of COV for each participant(n=12) immediately and 5 minutes after product application.

3.5 Correlations with panel scores

3.5.1 Rub-out phase

The following sensory panel data was provided by Unilever. The sensory panel consisted of 15 panellists who were trained over a period of six months to recognise and rate attributes on a scale of 0 to 100. Figures 3.13 and 3.14 below show the scores for the attributes evaluated during the first 8 rubs i.e. at the start of the application and during the next 8 rubs i.e. towards the end of the application, respectively.

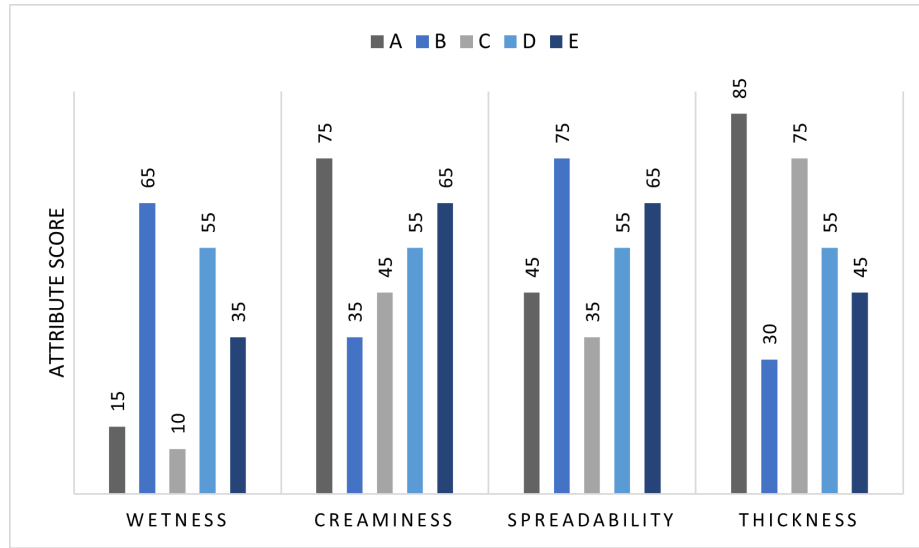


Figure 3.13: Panel scores for attributes during the first 8 rubs of the cream application (n=15).

It is to be noted that all panellists were in agreement with the scores for every attribute, with the exception of the attribute *Oily* for product D, illustrated by the error bar in Figure 3.14.

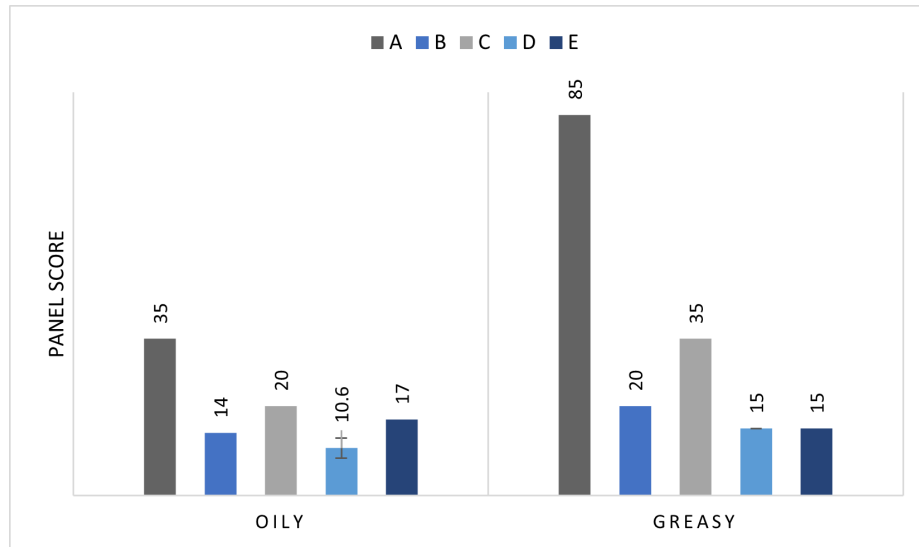


Figure 3.14: Panel scores and standard deviation for attributes during the next 8 rubs of the product application (n=15).

In order to establish potential correlations, different rheological measurements were plotted against attribute scores. The measurements studied were: yield stress, dynamic viscosity maximum, stationary viscosity at 30 seconds, 60 seconds and 120

seconds, viscosity at 0.1s^{-1} , 1s^{-1} and 10s^{-1} . Strong linear relationships were found for the log of yield stress and IVM, as highlighted by the plots in Figure 3.15 and 3.16 respectively, as well as for the viscosity at different shear rates obtained from the shear sweep tests.

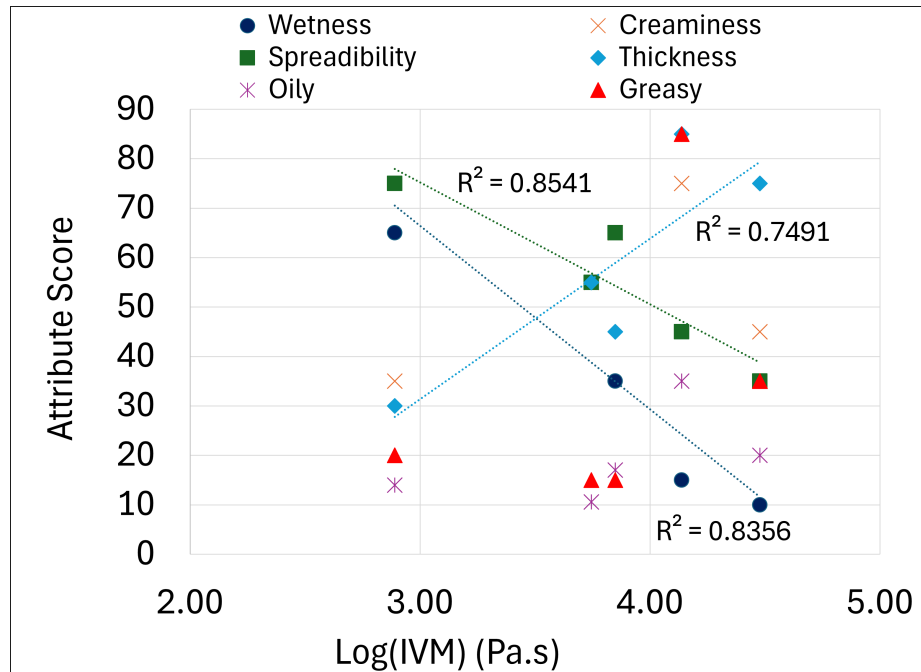


Figure 3.15: Log(IVM) versus attribute scores for the five BPC products.

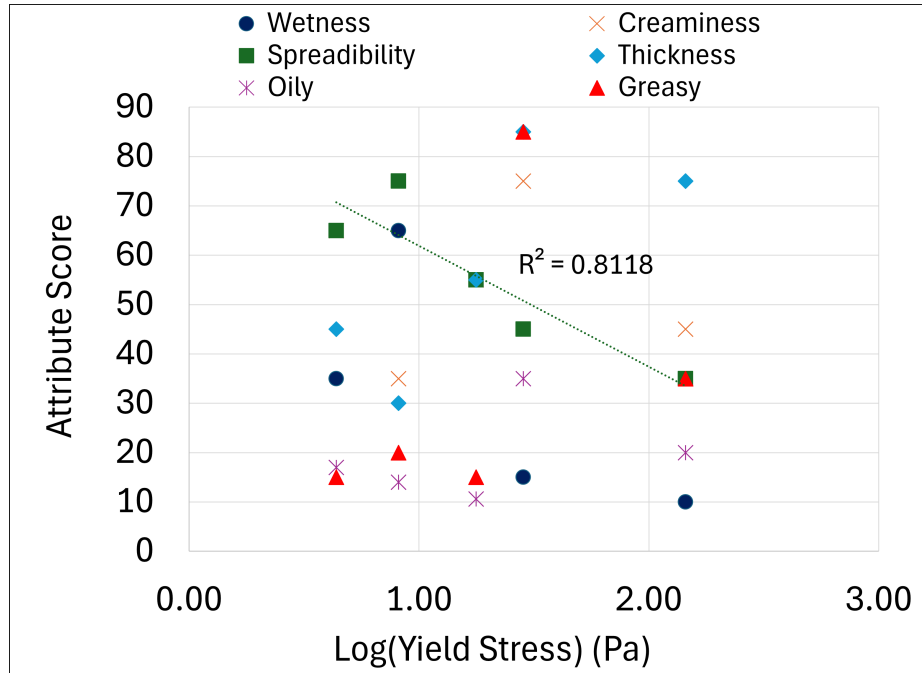


Figure 3.16: Log(Yield Stress) versus attribute scores for the five BPC products.

The software SPSS was used to run a simple linear regression analysis, to test if Log(IVM) significantly predicted the panel scores for attributes *Thickness*, *Wetness* and *Spreadability*. The overall regression was statistically significant for *Wetness* and *Spreadability* ($p < 0.05$) but not for *Thickness* ($p = 0.058$). Details of the results can be found in Table 3.12. A simple linear regression analysis was also run to check for correlations between the log(Yield Stress) and scores for the attribute *Spreadability*. It revealed that the log(Yield Stress) significantly predicted *Spreadability* ($p < 0.05$), as detailed in Table 3.13. A multiple regression analysis also revealed that Log(IVM) and Log(Yield Stress) significantly predicted attribute score for *Spreadability* ($p < 0.05$) with individual predictors Log(IVM) ($t = -4.356, p < 0.05$) and Log(Yield Stress) ($t = -3.763, p < 0.05$) both significant at the 95% confidence interval (Table 3.14).

Table 3.12: Results from the linear regression analysis with predictors (constant) and Log(IVM) (n=5), and the attribute score as dependent variable (n=15).

Attribute		Sum of Squares	df	Mean Square	F	Sig.
Wetness	Regression	1938.5	1	1938.5	15.2	0.030
	Residual	381.5	3	127.2		
	Total	2320.0	4			
Spreadability	Regression	854.1	1	854.1	17.6	0.025
	Residual	145.9	3	48.6		
	Total	1000.0	4			
Thickness	Regression	1483.3	1	1483.3	9.0	0.058
	Residual	496.7	3	165.6		
	Total	1980.0	4			

Table 3.13: Results from the linear regression analysis with predictors (constant) and log(Yield Stress)(n=5), and the attribute score as dependent variable (n=15).

Attribute		Sum of Squares	df	Mean Square	F	Sig.
Spreadability	Regression	810.620	1	810.620	12.841	0.037
	Residual	189.380	3	63.127		
	Total	1000.000	4			

Table 3.14: Results from the multiple regression analysis with predictors (constant), log(Yield Stress) and log(IVM)(n=5), and the attribute score as dependent variable (n=15).

Attribute		Sum of Squares	df	Mean Square	F	Sig.
Spreadability	Regression	981.942	2	490.971	54.376	0.018
	Residual	18.058	2	9.029		
	Total	1000.000	4			

Lastly, a multiple regression was run to predict attribute score from the Viscosity at shear rates 0.1s^{-1} , 1s^{-1} and 10s^{-1} . The results highlighted in Table 3.15 showed a significant correlation with the attribute *Creaminess* ($p < 0.05$). The individual predictors, Viscosity at 0.1s^{-1} ($t = 43.419, p < 0.05$), 1s^{-1} ($t = 41.726, p < 0.05$), and 10s^{-1} ($t = -46.517, p < 0.05$) were all significant at the 95% confidence interval.

Table 3.15: Results from the multiple regression analysis with predictors (constant) and Viscosity at 0.1 s^{-1} , 1 s^{-1} and 10 s^{-1} ($n=5$), and the attribute score as dependent variable ($n=15$).

Attribute		Sum of Squares	df	Mean Square	F	Sig.
Creaminess	Regression	999.580	3	333.193	793.496	0.026
	Residual	0.420	1	0.420		
	Total	1000.000	4			

3.5.2 Immediately and 5 minutes after application

The attribute scores were plotted against the DCOF obtained for all three products to identify any trends. With the dataset being limited to three data points, it was difficult to establish any significant relationships between attribute scores and DCOF values. However, it is clear from the graphs in Figure 3.17 that the small differences in attribute scores immediately after application versus the larger differences in DCOF values means that the two variables are unlikely to be related. For the DCOF measured 5 minutes after applications for the four attributes reported in Figure 3.17 there was a stronger linear relationship with attribute scores for *Greasy* and *Oily*, however more data points are necessary to form any strong conclusions.

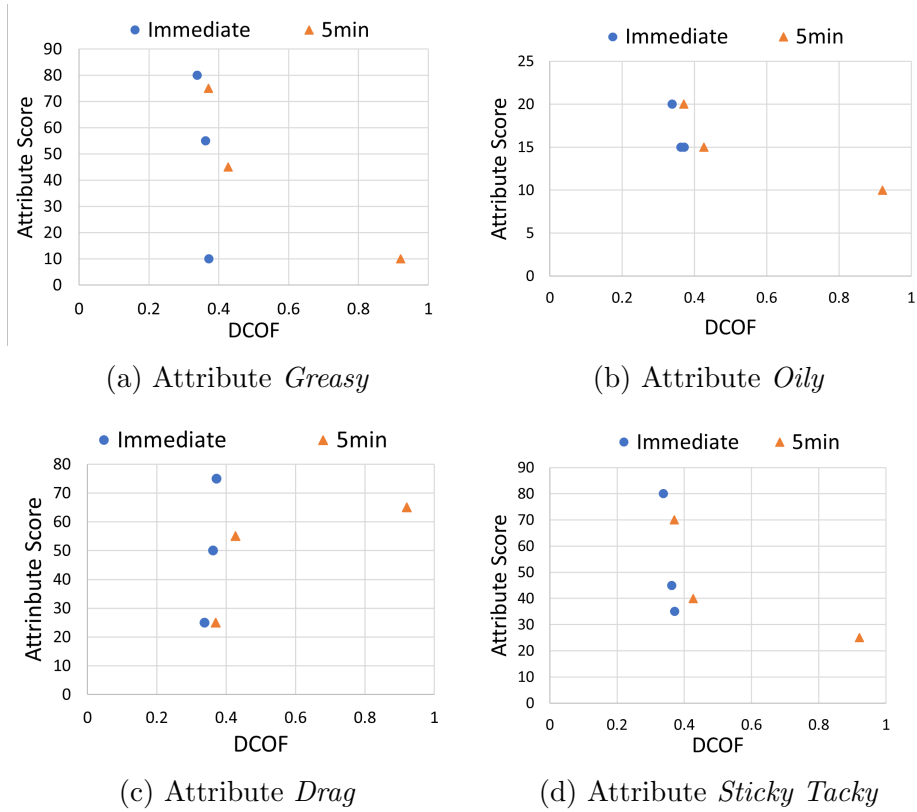


Figure 3.17: Human mean DCOF versus attribute scores.

Plotting the mean COV against attribute scores, as pictured in Figure 3.18, also revealed a strong linear relationship for the attribute *Sticky tacky* 5 minutes after application. However, a linear regression analysis revealed that this correlation was not significant, also likely due to the limited number of data points.

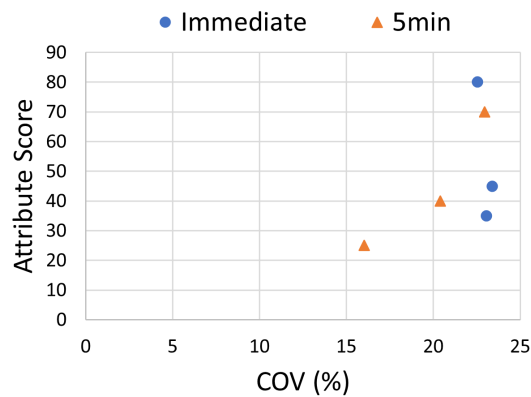


Figure 3.18: Mean coefficient of variation (COV) versus attribute scores for *Sticky Tacky*.

3.6 Discussion

In this study it was found that $\log(\text{IVM})$ measured at 25°C could predict the scores for attributes *Wetness* and *Spreadability*, and $\log(\text{Yield stress})$ could also be used to predict scores for the attribute *Spreadability*. The yield stress is the stress at which the product starts to flow. Hence a lower yield stress would be expected to incite the perception of a product as being more easily spreadable. Viscosity is the measurement of a product’s resistance to flow and the IVM is the viscosity measured at yield stress. Hence it is the maximum viscosity achieved by the product. According to the results from this study, the lower a product’s maximum viscosity, the more wet the product may be perceived. This can be explained by the fact that water is characterised by a high flow rate and very low viscosity and the attribute *wetness* is defined by the amount of water perceived while rubbing. The shear ramp test also revealed a correlation between viscosity at different shear rates and scores for the attribute *Creaminess*. Interestingly, no correlations were found between any of the individual predictors and this attribute through a simple linear regression analysis. Hence, only the combination of different shear rates, i.e. the thixotropic nature of the products, seems to impact the perception of *Creaminess*. Thixotropy is defined as the continuous decrease in viscosity with time when a sample is subjected to stress and subsequently the increase in viscosity with time when the stress is removed [104]. *Creaminess* is defined in Table 3.1 as “A substance that spreads smoothly and evenly glides between the fingertips”. Hence, the changes in viscosity when stress is applied to a product during application leads to changes in the perceived *creaminess*.

In the study led by Lee et al. [91], the authors used a similar approach whereby attributes were defined and allocated to different phases of application e.g. the “pick-up” and “rub-out” phase, and rated out of 100. The authors found that the $\log(\text{IVM})$ could be used to predict all attribute ratings during the appearance and pick-up phase. However, the authors found that both the rheology ($\log(\text{IVM})$) and tribology (DCOF) measurements influenced the ratings of attributes evaluated during the “rub-out” phase, such as *Wetness*, *Spreadability* and *Thickness*. Hence, this could explain why in the case presented above a significant correlation could not be established between *Thickness* and the rheology measurements alone.

There are also other limitations to consider. Of the attributes rated during the “rub-out” phase, the four first ones, including *Thickness* were rated during the first

8 rubs of the application whereas *Oily* and *Greasy* were rated during the next 8 rubs. It is unclear why the number 8 was picked and whether this was enough to differentiate between the start of the application and the end of the application as well as how much the skin temperature will have had an effect on the products at that point. Furthermore, the rate at which the product was applied was not specified in the Unilever protocol. In an ordinary setting, the rate at which a product would typically be applied to the skin depends on its intended use. Body lotions will more commonly be subjected to higher shear rates in order to cover larger areas more efficiently [90]. On the other hand, skin on the face is generally more sensitive than it is on the arms or legs, which could influence the stress applied to a face cream such as Product E. The shear rate of 500s^{-1} that was selected for the shear peak hold is said to correspond to the shear rate that body lotions are ordinarily subjected to [90]. Hence it may be relevant to adapt this method to suit various BPC products.

Having established clear correlations between the rheological properties of the skin creams and specific sensory attributes experienced during application, it is essential to consider how these product characteristics influence sensory perception beyond initial contact. Several authors have linked the changes in tactile perception of products following application to friction measurements [91, 41, 101], however the attributes used and the time intervals at which the friction was measured were varied.

From the results outlined at the start of this chapter, B had the highest concentration of humectants which meant it was expected to have the most immediate effect on the skin's hydration levels. Moreover, from the rheology measurements it was found to have the lowest yield stress and instantaneous viscosity maximum compared to the other BPC products tested. The sensory panel data also revealed it was perceived as the most spreadable product and the least thick. This indicates that during the 60 seconds application, the product will spread more rapidly across the skin compared with other BPC products, and coat it in a thinner layer, thus leaving more time for the water to evaporate from the product as it is warmed by the skin. Hence, after 5 minutes the water would be expected to be almost entirely removed from the contact, as highlighted in the study by Loden [47]. This reduced lubricating effect combined with the product's hydrating properties, would therefore be expected to increase the DCOF above that of dry skin. Although the increase in DCOF compared to the mean DCOF of dry skin observed in the results was not statistically significant, the DCOF

for B after 5 minutes was significantly higher than the two other products which is likely to be associated with the mechanisms stated.

On the other hand, C contains no water and consists of a single ingredient that acts as an occlusive. Hence, rather than increasing the skin's hydration levels by attracting water molecules, it acts as a protective layer preventing the water within the skin's lower layers from getting out of the skin. It is therefore expected that it's composition will remain relatively unchanged for longer. This was confirmed by the friction measurements, as there were no significant differences found between the DCOF immediately and 5 minutes following product application.

Lastly, A which contains a humectant at a high concentration whilst also containing water and an occlusive at the two highest concentrations, is expected to follow a similar trend as C with little changes between the immediate and 5 minute data. However, changes over time are also expected with the evaporation of the water and increase in skin hydration from the humectant which was also confirmed by these experiments, with a significant increase found for A, although the difference was less pronounced than for B.

Results from the friction measurements on the human volar forearm skin of 12 participants revealed some potential linear relationships between the DCOF measured and the sensory panel attribute scores. However, in the case of the DCOF measured immediately after application, judging by the relatively small differences between the DCOF values, these linear relationships are likely attributable to natural variations arising from the methodology and physiological differences, rather than any significant underlying correlation. In reality it is likely that immediately after application the contact will be in a mixed-lubrication regime, as proposed in the study by Wang et al. [97]. This is also highlighted in Figure 3.5 showed at the start of this chapter, as the BPC product is still visible on the skin immediately after application. Hence it is reasonable to assume that the perception of the product at that point will be influenced by both the characteristics of the product and the friction behaviour of the skin. Nevertheless, the sample size of three products meant that combining independent variables in a multiple regression analysis could not produce reliable estimates.

Figures 3.17 and 3.18 suggest potential linear relationships between the DCOF and the attribute scores for *Greasy* and *Oily* after 5 minutes, as well as between the coefficient of variation (COV) after 5 minutes and the *Sticky Tacky* attribute scores. However, additional data points are required to determine the statistical significance of these trends.

While not definitive, these findings support the idea that monitoring changes in skin’s frictional behaviour after applying BPC products may offer valuable insights into associated sensory perceptions. Furthermore, although prior work has examined changes in vibration frequency [41], the connection between variations in COV and sensory perception remains relatively unexplored and warrants further investigation.

Lastly, it should be expected that the use of a solid probe could affect correlations with sensory panel outcomes. During sensory panel tests, panellists evaluate the product using their finger. Although the surface energy of PMMA is close to that of skin, which may impact the adhesion of the product onto the probe, the harder material could mean more product is pushed out of the contact during sliding. Although the same force would be applied in both cases, the deformation of the finger would be expected to increase the contact area which in turn would lead to a decreased contact pressure, the finger-pad would therefore in theory be “carried” by the product to a greater extent. In contrast, with a higher contact pressure, the solid probe could be penetrating the product layer and pushing it away from the skin as the sliding begins, therefore affecting the measured DCOF. This aspect of the study will be explored further in Chapter 5.

3.7 Conclusions

In this chapter, the characteristics of various BPC products were evaluated as well as their frictional behaviour against the volar forearm skin of 12 human participants. Rheological parameters, ingredient function and friction measurements were used to establish and understand correlations between sensory attribute scores and measured variables. The findings are summarised below:

- The rheological assessment of the BPC products demonstrated that parameters such as yield stress and instantaneous viscosity maximum (IVM) could be used to predict the attribute scores for *Spreadability* and *Wetness*.

- A multiple regression analysis highlighted the complexity of the attribute *Creaminess* which appears to be influenced more by the thixotropic behaviour of a product than by any single rheological parameter.
- The DCOF of human volar forearm skin significantly increased after 5 minutes for A and B but not for C.
- The most significant difference between immediate and 5 minute measurements was found for B.
- The relatively small changes in mean DCOF values immediately after application between BPC products highlighted the presence of a mixed lubrication regime and reinforced the theory of a combined impact of product characteristics and DCOF on sensory perception.
- Although potential negative linear relationships were found for attributes *Greasy* and *Oily* after 5 minutes and the mean DCOF, more data points are needed to establish significant correlations.
- An increase in data points may also reveal a significant positive correlation between the COV, used to characterise stick-slip friction, and the scores for the attribute *Sticky Tacky*.

The results from these initial tests provided an improved understanding of the response of human skin to different BPC products. These results helped inform the design of the synthetic skin test-beds which will be outlined in the next chapter.

Chapter 4

Assessment of key design elements for a synthetic skin test bed

4.1 Introduction

Chapter 3 explored the characteristics of different BPC product in terms of composition function and rheology as well as their effect on the frictional behaviour of human skin. These findings were then related to the sensory perception of the products during the application process. Using the refined perspective achieved from the previous chapters, the emphasis transitioned to the development of a synthetic skin test-bed to replicate the behaviour of human volar forearm skin subsequent to the application of BPC products.

The BPC product industry is fast evolving and requires efficient testing protocols with repeatable outcomes. Although sensory panels are effective, they are often time consuming and costly as the number of panellists required and the training duration for a significant outcome is high. Therefore, relating physiological measurements to sensory attribute ratings and understanding the physical mechanisms leading the perception of these attributes is especially relevant. While measuring changes in human skin is feasible, there are a number of interpersonal variations which could affect sensory perception, such as skin type and moisture levels, hence obtaining an accurate representation of the population is challenging. This brings to the solution of the development of a synthetic skin test-bed. Albeit a number of synthetic skins already exist on the market, such a VitroSkin[™], they are expensive and are often limited to a single use. Other studies have aimed at developing their own through a variety of different processes [70, 9, 75, 41] but these often result in complex and lengthy procedures rendering them unsuitable for manufacturing in an industry setting.

The research question investigated in this chapter is how a synthetic skin test-bed can be developed to reliably replicate the frictional and sensory behaviour of human volar forearm skin following the application of BPC products, and what the key factors that influence its performance and industry suitability are. The study is presented in five distinct parts, each examining how different features influence the behaviour of a synthetic skin test-bed. To ensure that the influence of each characteristic on test-bed performance could be clearly understood, test-beds were systematically manufactured both with and without the specific feature in question. This controlled approach allowed for repeatable testing and direct comparisons, making it possible to attribute observed differences in behaviour to the presence or absence of that particular characteristic with greater confidence.

4.2 Synthetic skin test-bed manufacture

Based on the results from the literature search detailed in Chapter 2, a design specification was compiled, as presented in Table 4.1, outlining the requirements and features which will constitute the main focus for the development of the synthetic skin test-bed. It is worth noting that although the features were studied individually, there is an element of interdependence between them. For example, greater precision in terms of hydration properties may affect the reusability of the test-bed, and a more anatomically accurate structure may involve a complex manufacturing process. Hence, certain compromises must be made in the final synthetic test-bed design.

Silicone was selected as a base for the test-beds as it is readily available and has a long shelf life. Its properties can also be tuned by adding different substances such as silicone deadener [76], and in many cases it is stable as its properties will not change with exposure to light or to atmospheric pressure, meaning it can be reused between experiments thus making it a suitable candidate for larger scale testing in industry. The type of silicone used was an addition cure silicone rubber (Polycraft Silskin 10 Special Effects, MB Fibreglass). A Room Vulcanizing Temperature (RTV) silicone was preferred due to the efficient manufacturing process, as it can be cured at room temperature without the need for specific equipment.

Table 4.1: Design specification for the synthetic skin test-beds.

	Requirements	Features
Function	Replace human subjects for the sensory assessment of BPC products	
Performance	Match the DCOF trends for the different BPC products as measured on human volar forearm skin	- Replicate human volar forearm skin texture - Replicate the bulk mechanical properties of human volar forearm skin - Replicate the water absorption capacities of human skin - Replicate the surface energy of human volar forearm skin
Manufacturing	- Efficient - Reproduceable results	- Room temperature vulcanising (RTV) silicone - Acrylic two-part moulds, with a design suitable for laser-cutting
Environment	Reusable	Products can be washed off without affecting the test-bed properties
Materials	- Stable - Widely available - Cost effective	Commercially available RTV silicone

Two different moulds were designed for the manufacture of the SS test-beds. The first mould was made out of Aluminium with a 1 mm deep rectangular groove, as pictured in Figure 4.1. Two frames, of 2 mm and 5 mm in thickness, were 3D printed to be screwed onto the base plate to control the test-bed thickness. The second mould consisted of a smooth flat base laser cut out of a Perspex Acrylic sheet, with a frame

of 6mm in thickness.

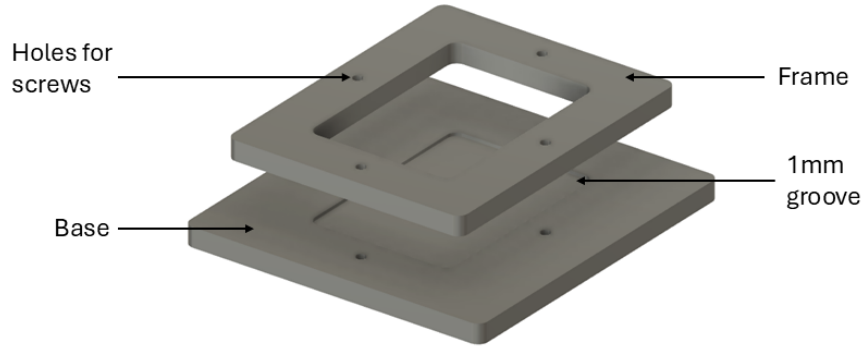


Figure 4.1: Mould manufactured for the bi-layered synthetic skin test-beds.

These moulds were designed for the manufacture of a test-bed with dimensions of 63x45x3 mm and 63x45x6 mm. The dimensions of the base and frame are pictured in Figures 4.2 and 4.3 respectively. These dimensions were chosen to allow for testing in the University Mechanical Tester (UMT), whilst allowing for a sliding distance of 30 mm, which was used in a similar study by Tang et al. [41]. The additional test-bed length also allowed for enough space between the end of test-bed and the probe, so as to not lift the edges of the test-bed during the change in direction of the probe. Further details of the testing methodology will given in a subsequent section.

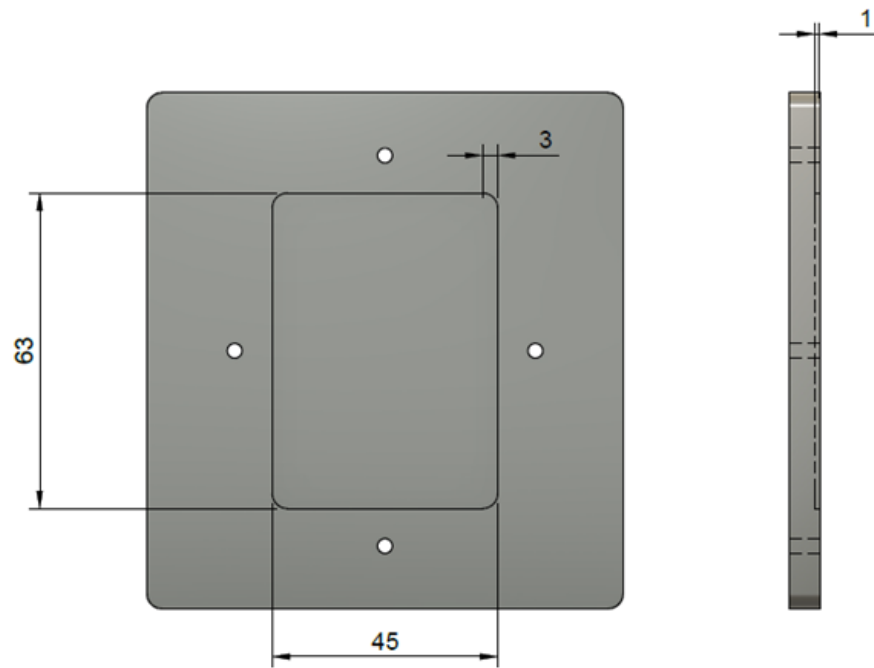


Figure 4.2: Dimensions for the mould base with 1mm groove for the bi-layered synthetic skin test-beds.

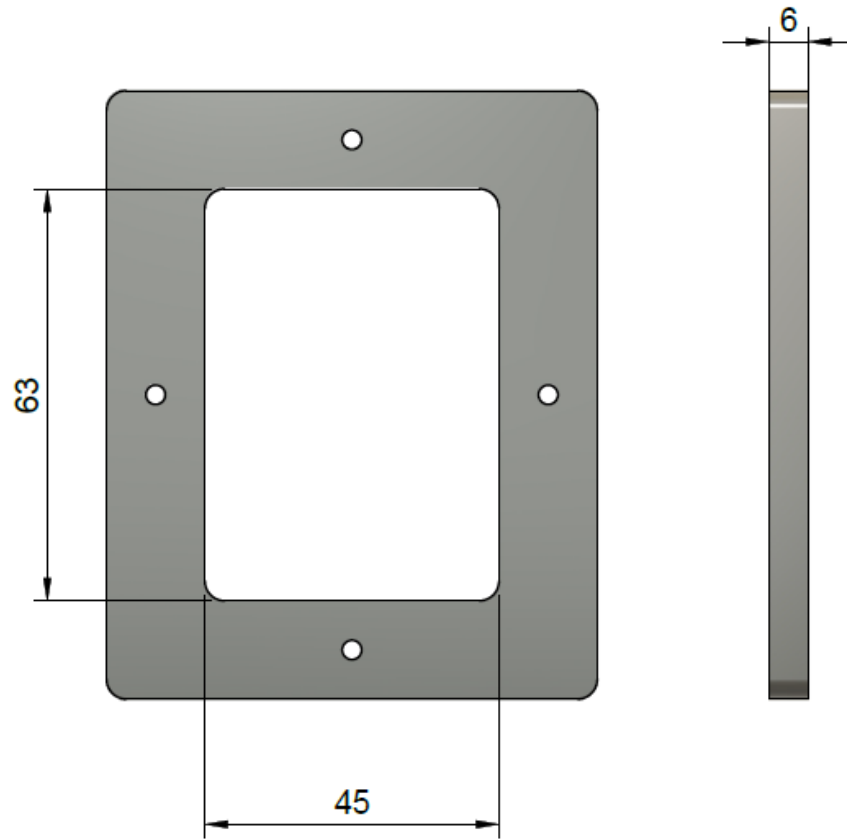


Figure 4.3: Dimensions for the mould frame for the synthetic skin test-beds.

The Aluminium mould was used to make bi-layered models to simulate an epidermis/dermis structure. These will be discussed in more detail in the following sections. All silicone mixtures were placed inside the degasser, shown in Figure 4.4 below, for 4 minutes in total, or until air bubbles no longer formed at the surface, before they were poured into the moulds.



Figure 4.4: Degasser used to remove air bubbles from silicones.

When pouring the silicone into the mould, extra care was taken to place the container as close as possible to the mould to avoid any air getting trapped inside the mixture. The excess silicone was scraped off using a flat mixing stick. For the bi-layered test-beds, the 1mm thick silicone was left to cure for a minimum of 1 hour. An example SS test-bed “epidermis” is pictured in Figure 4.5 below.



Figure 4.5: 1 mm thick upper layer made out of Silskin 10 special Effects RTV silicone (MB Fibreglass).

An example of a bi-layered test-bed is pictured in Figure 4.6 below. As both layers were made out of silicone they naturally adhered to each other during the curing

process, thus preventing the need for glue.

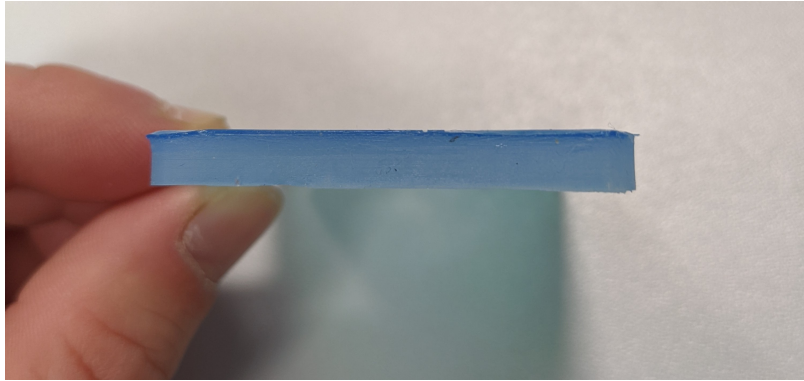


Figure 4.6: Lateral view of a bi-layered model with 1mm thick upper layer and 5 mm thick dermis.

Texture was added to some of the SS test-beds by creating replicas of human forearm texture to line the moulds. This was done using SILFLO (Medelink), a two component silicone with a high flow rate facilitating the capture of surface microstructures. One drop of catalyst was added for every gram of SILFLO used and the mixture was then spread onto the skin using a plastic mixing stick to form a layer as demonstrated in Figure 4.7 below. Once cured, the replica was peeled off the skin and placed at the bottom of the mould. Vaseline®Petroleum Jelly (Unilever, London, UK) was spread onto the replica to ensure it could be peeled off of the SS test-bed surface after curing.



Figure 4.7: Method used for obtaining a replica of volar forearm texture using SILFLO.

Figure 4.8 below shows the texture of the replica which was scanned in an optical profilometer.

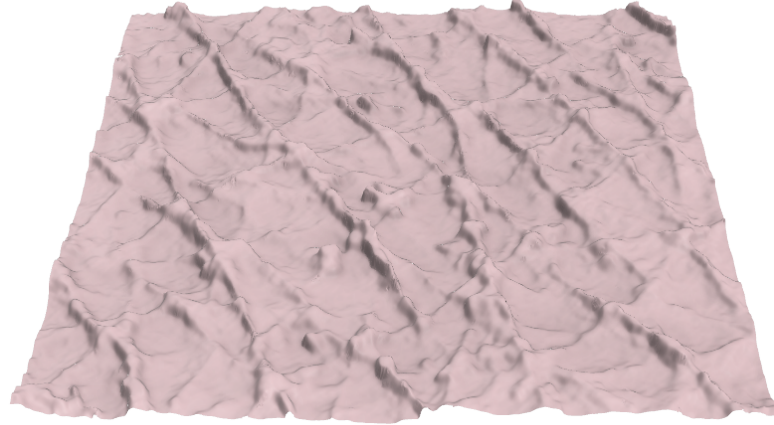


Figure 4.8: 3D model of the surface texture of a SILFLO replica of human volar forearm skin.

4.2.1 Statistical analysis

The statistical analysis was conducted using the SPSS software. The differences in means were tested for statistical significance using either independent t-tests (differences between test-bed or products) or paired t-tests (differences between immediately and 5 minutes after application on the same test-bed). For the independent t-tests, equal variances were assumed for a Levene's significance value above 0.05. The values were reported accordingly. The statistical significance of correlations between attribute scores and measured variables were determined using a Linear Regression Analysis.

4.3 Effect of hydration

4.3.1 Methodology

The three different test-beds used for the following experiments are outlined in Table 4.2. The test-beds were made using an addition cure silicone (Polycraft Silskin 10 Special Effects Addition Cure Silicone Rubber, MB Fibreglass), glycerin was added at 20 wt% of the base and catalyst mixture to enable water absorption.

Table 4.2: Test-bed manufactured for the hydration experiments.

Test-bed name	Test-bed description	Composition
TB1	Textured addition cure silicone with glycerin (3mm)	Single layer of addition cure silicone (base + catalyst at 1:1 ratio) with added glycerin at 20wt% and forearm texture.
TB2	Smooth bi-layered silicone with glycerin (3mm)	Top layer (1mm) : addition cure silicone (base + catalyst at 1:1 ratio) with added glycerin at 20 wt% Bottom layer (2mm) : addition cure silicone
TB3	Smooth bi-layered silicone with glycerin (6mm)	Top layer (1mm) : addition cure silicone (base + catalyst at 1:1 ratio) with added glycerin at 20 wt% Bottom layer (5mm) : addition cure silicone

4.3.1.1 Water uptake

A “hydrated state” was obtained by submerging the SS test-beds in water over a period of time. A water uptake experiment was carried out in order to evaluate the rate at which water was absorbed for test-beds TB1, TB2 and TB3 (Table 4.2). The three test-beds were submerged for 98 hours and weighed throughout to measure the weight difference. All test beds were made using the same glycerine weight to base silicone weight percentage but differed in thickness. For the two bi-layered models the glycerine was only added to the upper layer which was 1 mm in thickness for both.

The water absorption swelling rate (WASR) was calculated using equation 4.1, according to the method outlined by Chen et al. [19], and was reported as a percentage.

$$WASR = \frac{W_{Swollen} - W_{Dry}}{W_{Dry}} \quad (4.1)$$

An optical profilometer (InfiniteFocusSL, Bruker Alicona) was also used for evaluating changes in surface texture following the “hydration” of SS test beds. Three measurements were taken for each, with different areas of the test-bed used for each measurement.

4.3.1.2 Friction

Two of the test-beds TB1 were manufactured to assess the effect of hydration on the frictional properties of the SS test-bed with and without product-treatment. The friction tests were performed in a Universal Mechanical Tester (UMT) (Bruker) which allowed for an automated control of sliding speed, normal load and sliding distance. The testing set-up is pictured in Figure 4.9. The test-bed was secured to a base-plate using double sided tape, and the base plate was screwed onto a linear reciprocating platform in the UMT. A PMMA spherical tipped probe with a 16 mm diameter was secured to a force-plate. The platform moved in a reciprocating linear motion at a speed of 10 mm/s over a distance of 20 mm in each direction whilst the probe remained static. The probe was positioned so as to avoid the test-bed extremities lifting during sliding. A normal load of 2N was chosen in order to replicate the method used by Adams et al. [14] in their study on the influence of water on the friction of human skin. A higher normal load than previously used was also preferred for assessing whether water would be squeezed out of the test-beds during the tests, which was observed for different SS test-beds [70]. The sliding velocity was selected based on the study by Veijgen et al. looking at the relationship between skin hydration and friction [54] and the study performed by Tang et al. on the tactile perception of skin cream [41].

The friction was measured on one non-hydrated and one hydrated test-bed. The hydrated state was obtained by submerging the test-bed in water for 48 hours. The hydrated test-bed was removed from the water, dabbed with a paper towel and left to dry for 10 minutes prior to the start of the tests. Product treatment was obtained by weighing out 0.1g of Product C and spreading it across the entire surface of the test-beds for 60 seconds using a gloved finger, to avoid any contamination of the product. This yielded a product distribution of 2.6 mg/cm². The friction was measured immediately, 30 minutes and 1 hour following the end of the product application process. The friction was measured three times per test-bed and the probe was wiped with isopropyl alcohol between each repeat. The probe was also moved left or right before each new measurement to cover a new trajectory (as pictured in Figure 4.9).

The UMT records the friction force, sliding speed, normal load and automatically calculates the COF overtime. The raw data was imported in Matlab and the data points where the sliding speed was below 10mm/s i.e. near the end of the sliding distance where the probe is changing directions, were removed from the data sets.

This ensured that only the dynamic COF (DCOF) was taken into account. The remainder of the data points were averaged to give the mean DCOF for each test.

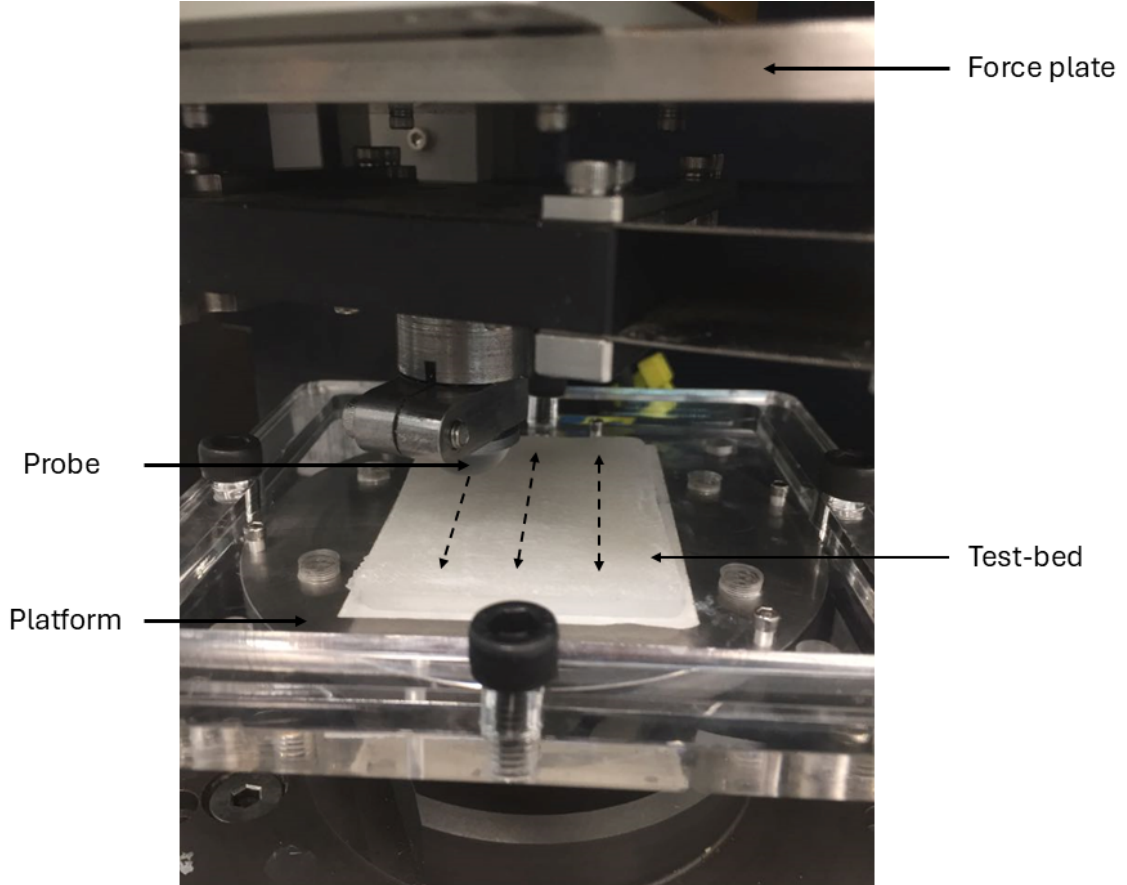


Figure 4.9: Set-up used for measuring test-bed friction in the Universal Mechanical Tester, with dashed arrows showing the sliding direction and trajectory for the three measurements.

4.3.2 Results

4.3.2.1 Water uptake

The different WASR measured for the three test-beds in the water uptake experiment are plotted in Figure 4.10. The graph shows the WASR increases at a similar rate over the first 5 hours for the three test beds. However, the differences between them become more apparent over longer periods of time (68 to 96 hours) with the single layered test-bed absorbing the most water. This is to be expected as there is more surface area for the water to penetrate the test-bed compared to the bi-layered models of which one surface has been cured against a base silicone with no glycerin. Interestingly, there are noticeable discrepancies between the two bi-layered models

which could be due to variations in the distribution of glycerin within the layer. As the two test-beds were made separately and the mixing was done manually, differences in the mixing rate and duration may have impacted the homogeneity of the mixture. The immiscibility of the two substances was highlighted in a study by Mazurek et al. [78] who suggested subjecting the emulsion to high shear forces while mixing.

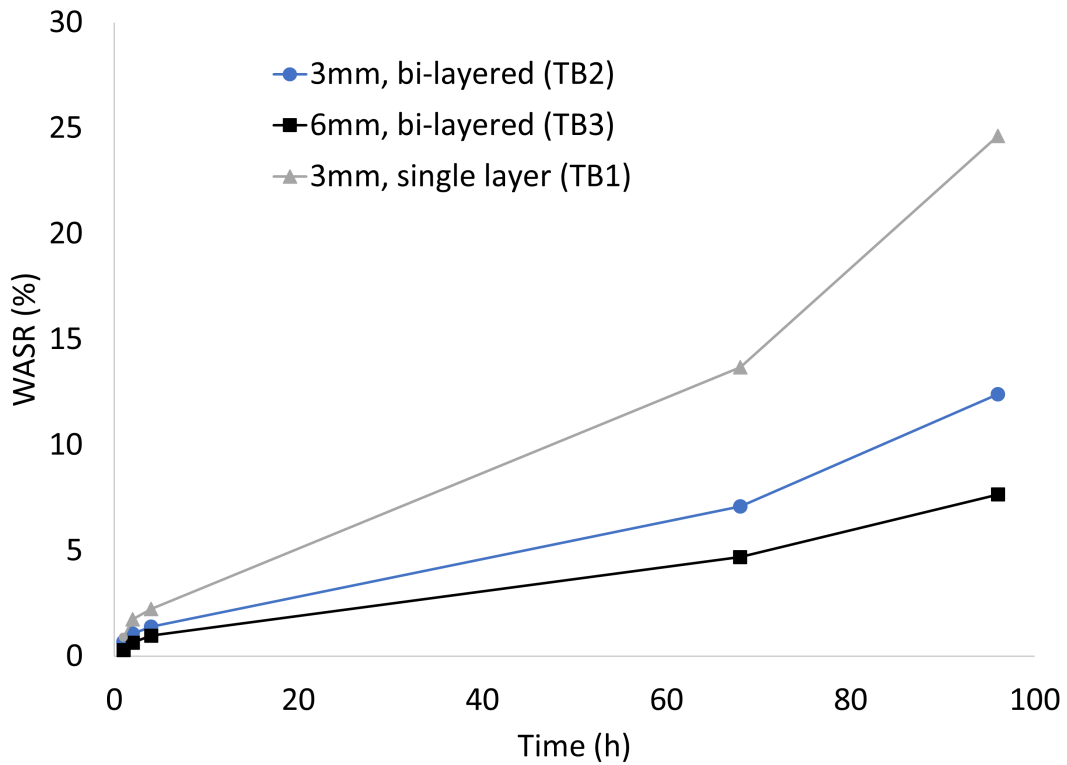


Figure 4.10: Water absorption swelling rate (WASR) over time for different SS test-beds when submerged in water.

The inhomogeneous mixture was also highlighted by the optical profilometry measurements of the hydrated smooth bi-layered test-bed (TB2). Figure 4.11 shows the increased roughness of an SS test bed made with no added texture following submersion in water for 96 hours. It appears that some areas were more prone to swelling than others, most likely due to their position relative to the test-bed surface.

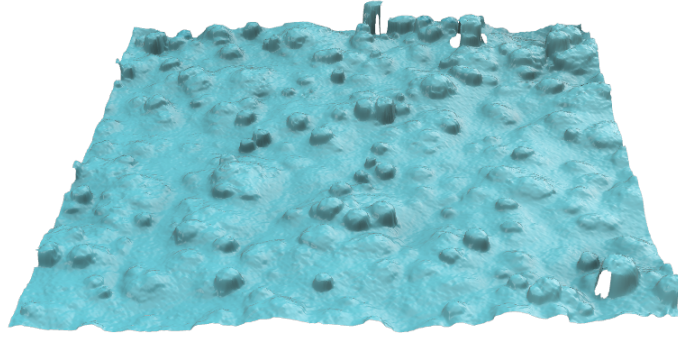


Figure 4.11: Surface texture of a hydrated smooth bi-layered test-bed (TB2).

The changes in surface texture following hydration were also evaluated for the textured test-beds. Figure 4.12 shows a 3D rendering of the surface texture of test-bed TB1 which highlights some swelling in the hydrated state. Interestingly the troughs are still visible and the peaks seem to have mainly swelled in the vertical direction.

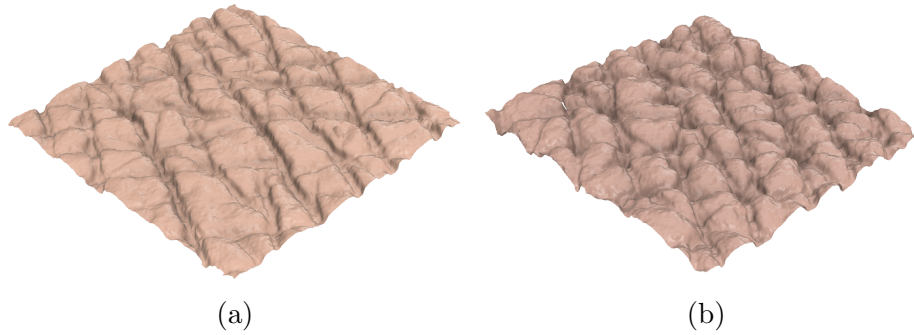


Figure 4.12: Surface texture of a non-hydrated (a) and hydrated (b) textured test-bed with added glycerin (TB1).

This observation is also highlighted in Table 4.3 with the increase in average roughness value (R_a) by approximately $1\mu\text{m}$ and an increase in the average maximum height (R_z) of the profile by $7\mu\text{m}$. This phenomenon is comparable to the corneocytes in a human Stratum Corneum which tend to swell primarily in the vertical direction [33]. Another matter to note is the absence of smaller “bumps” which were present on the test-bed with no texture in Figure 4.11. This could be due to the added troughs which allowed for some swelling to occur in the horizontal direction.

Table 4.3: Average roughness (Ra) and maximum height (Rz) values obtained in the Alicona for a dry and hydrated textured SS test bed.

Substrate	Ra (μm)	Rz (μm)
Dry textured test-bed (TB1)	5.47	33.80
Hydrated textured test-bed (TB1)	6.53	40.03

4.3.2.2 Friction measurements

The mean DCOF for the non-hydrated and hydrated SS test-beds before and after product-treatment is shown in Figure 4.13. The values and standard deviation are also reported in Table 4.4.

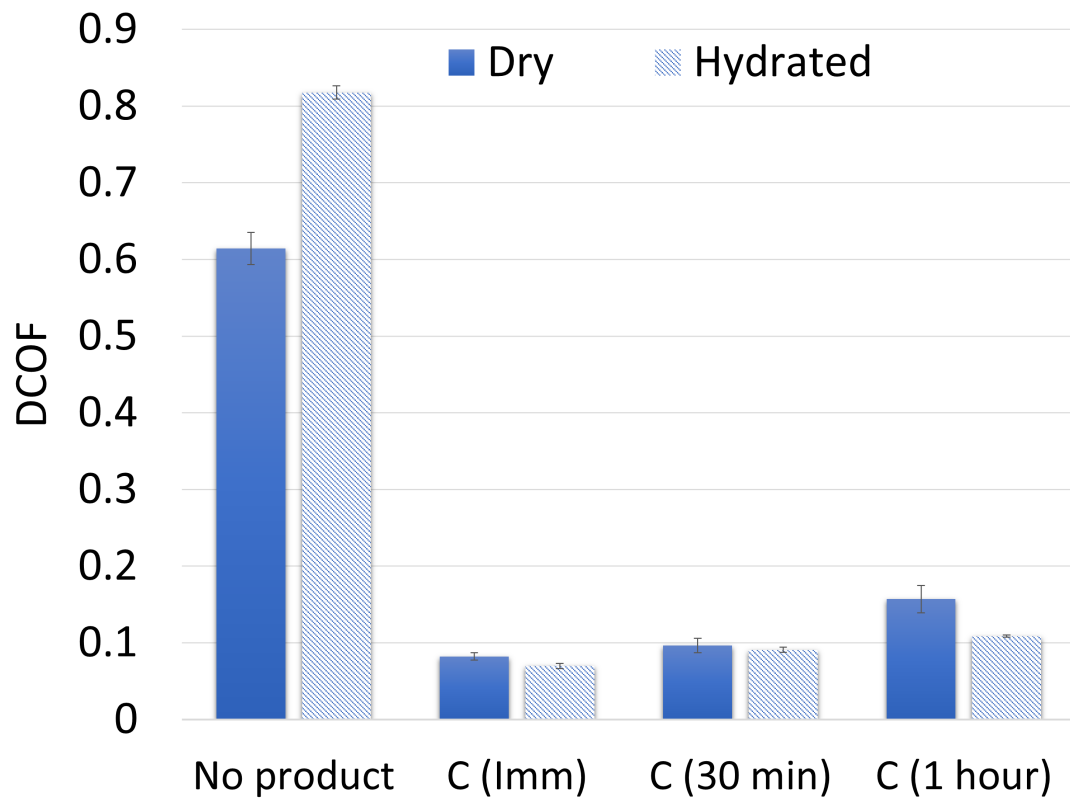


Figure 4.13: Mean dynamic coefficient of friction for hydrated test-beds without C and at different time intervals after C application with standard deviations (n=3).

Table 4.4: Mean DCOF values with standard deviations for the non-hydrated and hydrated test-beds before and after product application.

Product treatment	Test-bed	N	Mean DCOF	Std. Deviation
None	Non-hydrated	3	0.614	0.026
	Hydrated	3	0.818	0.011
C Immediate	Non-hydrated	3	0.082	0.006
	Hydrated	3	0.07	0.004
C 30 min	Non-hydrated	3	0.097	0.011
	Hydrated	3	0.09	0.004
C 1 hour	Non-hydrated	3	0.157	0.022
	Hydrated	3	0.109	0.002

From the results of an independent t-test (reported in Tables 4.4 and 4.5) it was found that prior to product-treatment the mean DCOF of the hydrated test-bed was significantly greater than for the non-hydrated test-bed ($p < 0.001$). In contrast, the DCOF of the non-hydrated test-bed both immediately and 1 hour following the application of C, was significantly greater than for the hydrated test-bed ($p < 0.05$). There were no significant differences between the two 30 minutes after product application.

Table 4.5: Results from the independent t-test for the differences between the non-hydrated and hydrated test-beds (TB1).

Treatment	t	df	Significance	
			One-Sided p	Two-Sided p
None	-12.6	4	<0.001	<0.001
C (Imm)	2.92	4	0.022	0.043
C (30 min)	0.813	4	0.231	0.462
C (1 hour)	3.86	2.03	0.030	0.060

From a paired t-test it was found that the DCOF after application was significantly lower than before application at all time intervals for both the non-hydrated and the hydrated test-beds (Table 4.6). The DCOF for the non-hydrated test-bed 1 hour after application was significantly greater than the DCOF 30 minutes and immediately after application. However there were no significant differences between the 30 minute and the immediate DCOF.

Table 4.6: Mean DCOF and standard deviation for the non-hydrated test-bed TB1 before and after product application (TB1).

Pair	t	df	Significance	
			One-Sided p	Two-Sided p
Dry no C - Dry C Imm	43.765	2	0.000	0.001
Dry C 30 min - Dry C Imm	1.426	2	0.145	0.290
Dry C 1 hour - Dry C 30 min	3.165	2	0.044	0.087
Dry C 1 hour - Dry C Imm	8.132	2	0.007	0.015
Dry no C - Dry C 1 hour	95.116	2	0.000	0.000
Dry no C - Dry C 30 min	24.557	2	0.001	0.002

For the hydrated test-bed the DCOF 1 hour after application was also significantly greater than 30 minutes and immediately after application. And the DCOF 30 minutes after application was significantly greater than the immediate DCOF (Table 4.7).

Table 4.7: Results from the paired t-test for the mean DCOF the hydrated test-bed TB1 before and after product application.

Pair	t	df	Significance	
			One-Sided p	Two-Sided p
Hyd no C - Hyd C Imm	190.143	2	0.000	0.000
Hyd C 30 min - Hyd C Imm	5.966	2	0.013	0.027
Hyd C 1 hour - Hyd C 30 min	12.499	2	0.003	0.006
Hyd C 1 hour - Hyd C Imm	13.413	2	0.003	0.006
Hyd no C - Hyd C 1 hour	103.790	2	0.000	0.000
Hyd no C - Hyd C 30 min	99.048	2	0.000	0.000

4.3.3 Discussion

Glycerin was found to successfully allow for water absorption in the test-beds which in turn led to an increase in friction. These results are in accordance with studies looking at the effect of an increased hydration of human skin on friction [54, 68, 8, 14] which associated this phenomenon with the increased compliance of hydrated skin. However, an inverse trend was found following the application of C. This effect was also observed in a study by Wang et al. [97], where the friction of different emulsions were measured against “semi-moist” and “fully-moist” skin. For one of the emulsions the authors found that the DCOF was reduced for the fully moist skin, which they associated with a saturation of the skin meaning the emulsion mostly acted as a lubricant. However, with C being an occlusive it is unlikely that this effect was entirely due to an increase in hydration for the non-hydrated test-bed. Moreover, images obtained in the optical profilometer gave evidence of some swelling following the hydration of the test-bed. This in turn may have led to the reduction in the width of the furrows and hence more product remaining on the surface of the test-bed and thus increasing the lubrication effect. Lastly, although previous studies have highlighted that water was being squeezed out of synthetic skin test-beds during friction tests [70], this phenomenon is unlikely in this case, as a lower DCOF would have been observed from the first set of measurements.

The water uptake experiment also revealed the potential effect of the uneven distribution of glycerin within the test-bed on the water absorption swelling rate. The results from the surface texture measurements further highlighted some irregular swelling of the smooth surface of one of the test-beds. As discussed earlier these inhomogeneities could be due to the manual mixing technique used in the manufacture of the test-beds. It could therefore be pertinent to control the shear forces and shear rate applied to the silicone mixture during the manufacturing process.

4.4 Probe selection

The aim for the probe was to achieve a set-up as close to a human finger against skin contact whilst avoiding two soft materials interacting, which would inevitably complicate the interaction. Using a solid probe allows for a better understanding of the effects of different test-beds and ensures a more repeatable method. Hence two materials were selected for testing: steel and PMMA. Steel is one of the most common materials found in literature for a probe on skin contact [55, 19, 70, 73]. Meanwhile,

PMMA has been used as a substitute for skin [100, 68, 31] due to its surface energy being close to that of human skin. Surface energy affects wettability and adhesion [26] which could impact how it interacts with the BPC products during experiments. Hence it was of importance to consider the differences between the two probes in order to get an accurate representation of how the different materials could affect the resulting DCOF.

4.4.1 Methodology

Two spherical tipped probes of 16 mm in diameter were manufactured, one out of steel and one out of PMMA. The friction was measured in the UMT using a similar method to the one outlined in the previous section, however the normal load was reduced to 1N at the sliding distance was increased to 30 mm. The speed of 10 mm/s and reciprocating linear motion were maintained as previously stated. According to Skedung et al., normal loads of 0.5N-1N have been found to be the optimal loads used when assessing tactile stimulus [75]. This range of loads will be used for the remainder of the experiments. The sliding distance was increased to 30 mm to allow for more sliding to occur in between changes in direction. This sliding distance was also used in a similar study by Tang et al. [41].

For the following experiments, a smooth test-bed was made using silicone with added Glycerine at 20wt% (TB1 with no added texture). The friction was first measured in dry conditions, after which 3 water droplets of 1 ml in volume each were deposited onto the surface of the test-bed using a pipette, for the friction to be measured again. Each test was repeated 3 times, and the test-bed was dabbed with a paper towel and left to dry for at least 10 minutes between each test. The mean DCOF for each configuration was calculated using the method stated previously.

4.4.2 Results

The mean DCOF for both probes along with the standard deviations are shown in Figure 4.14 below.

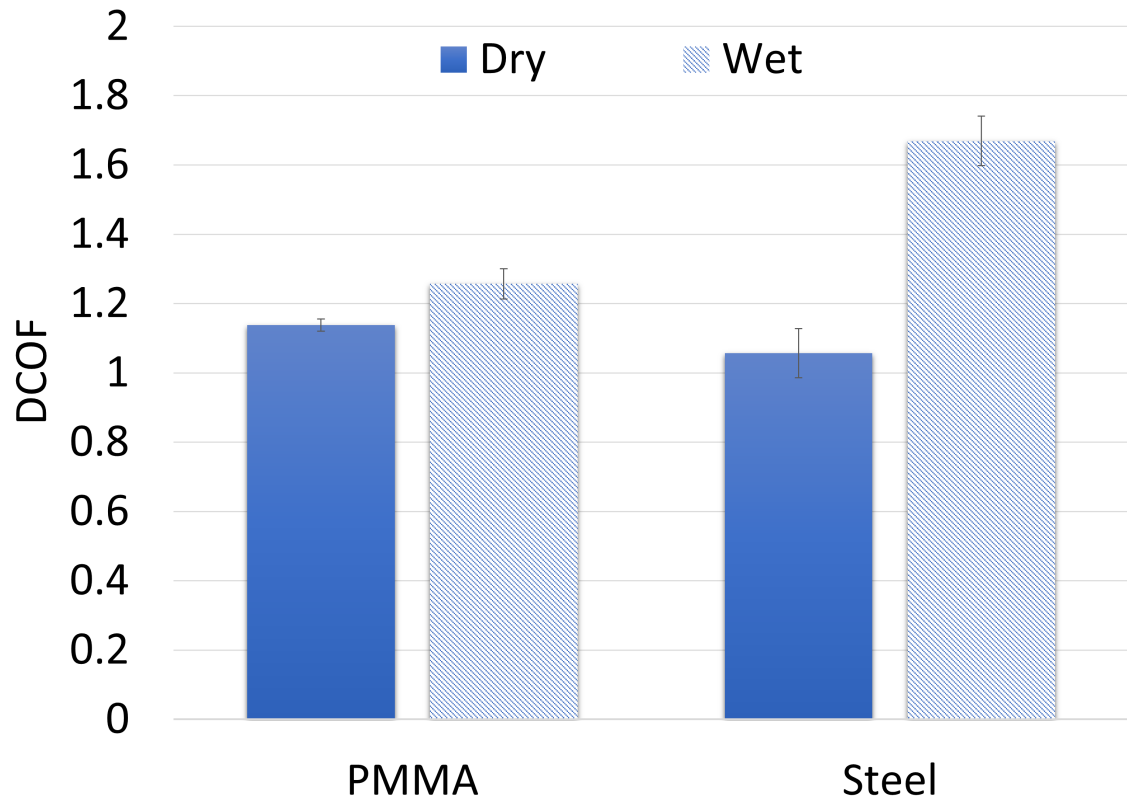


Figure 4.14: Mean DCOF and standard deviations for the PMMA and steel probes in dry and wet conditions ($n=3$).

An independent samples t-test was run with a 95% confidence interval for the differences between means. It was found that the DCOF in the wet conditions was significantly higher for the steel probe than for the PMMA probe ($p < 0.01$). For both probes the DCOF was significantly higher in the wet conditions than in the dry conditions ($p < 0.05$).

Table 4.8: Mean DCOF and standard deviations for the different probes in dry and wet conditions.

Condition	Probe	N	Mean	Std. Deviation
Dry	PMMA	3	1.14	0.023
	Steel	3	1.06	0.086
Wet	PMMA	3	1.26	0.053
	Steel	3	1.67	0.088

Table 4.9: Results from the independent t-test to evaluate the statistical significance of the differences between the probes in dry and wet conditions.

Condition	t	df	Significance	
			One-Sided p	Two-Sided p
Dry	1.578	4	0.095	0.190
Wet	-6.967	4	0.001	0.002

Table 4.10: Results from the independent t-test to evaluate the statistical significance of the differences between the dry and wet conditions for a PMMA and a steel probe.

Probe	t	df	Significance	
			One-Sided p	Two-Sided p
PMMA	-3.581	4	0.012	0.023
Steel	-8.631	4	<0.001	<0.001

4.4.3 Discussion

These experiments highlighted significant differences in the DCOF obtained against a PMMA probe versus a steel probe in wet conditions. The DCOF in the wet conditions was found to be significantly lower for the PMMA probe than for the steel probe. A similar study was undertaken by Adams et al. [14] who found that the DCOF in wet conditions on human skin was lower for a hydrophilic glass probe compared to a hydrophobic Polypropylene probe. In this case, the surface energy of PMMA has been reported as 39 mN/m [68] whereas for it steel it was measured at 30 mN/s [105] which therefore makes the steel probe more hydrophobic than the PMMA probe. Adams et al. [14] associated this reduction in friction for the more hydrophilic probe to the formation of a more stable layer of water molecules on the surface of the probe, thus increasing its lubricating effect.

Following on from these results it was decided that the tests would be performed using the PMMA probe, as the difference in surface energies appears to have a significant effect on friction in wet conditions. This is an important factor to consider as most of the BPC products used in this study contain water at the highest concentration compared to other ingredients.

4.5 Texture and mechanical properties

As properties of ingredients such as glycerin and deadener were explored, as well as the combination of layers of different thicknesses and textures, it was deemed of importance to measure their effect on the mechanical and frictional properties of the test-beds. The test-bed tested in this following section are detailed in Table 4.11.

Table 4.11: Test-beds used for the assessment of the impact of layer combinations on mechanical properties, and the effects of texture and mechanical properties on friction.

Test-bed name	Test-bed description	Composition
TB1	Textured silicone with glycerin (3mm)	Single 3mm layer of addition cure silicone (base + catalyst at 1:1 ratio)
TB2	Smooth bi-layered silicone with glycerin (3mm)	Top layer (1mm) : addition cure silicone (base + catalyst at 1:1 ratio) with added glycerin at 20wt% Bottom layer (2mm) : addition cure silicone
TB3	Smooth bi-layered silicone with glycerin (6mm)	Top layer (1mm) : addition cure silicone (base + catalyst at 1:1 ratio) with added glycerin at 20wt% Bottom layer (5mm) : addition cure silicone
TB4	Silicone with deadener (Smooth/Textured)	Single 6 mm layer of addition cure silicone (base + catalyst at 1:1 ratio) with added deadener at x wt%.

4.5.1 Test-bed mechanical properties

The mechanical properties of the SS test-beds were measured by means of indentation tests in a Force Gauge (IMADA). The assessment of mechanical properties under compressive loading was deemed as the most relevant due a compressive load being applied by a finger onto the skin during BPC product application and sensory evaluation. Using the same PMMA probe as described in the previous sections, a compressive load of up to 5N was applied at a speed of 2mm/s. The test set-up is shown in figure 4.15. A minimum of three measurements were taken per test-bed, ensuring different areas of the test-bed surface were used for every measurement.



Figure 4.15: Force gauge set-up used for the indentation tests.

One of the main challenges with regards to developing a synthetic skin test-bed for use in industry is ensuring the results obtained are repeatable. Hence it is of interest to understand how variations in thickness and layer combinations may affect experimental outcomes. Figure 4.16 below shows the force displacement data for test-beds TB1, TB2 and TB3 : a single-layer of silicone cross-linked with glycerin and two bi-layered test-beds of different thicknesses with added glycerin in the upper-layer. The figure shows the single layered test-bed and the thicker of the two bi-layered test-beds follow a similar trajectory whereas the thinner bi-layered test-bed shows a sharper increase in force and lower displacement. This indicates that although at a normal load of 1N the displacement is below 1mm which corresponds to the thickness of the upper layer, the effect of material underneath the test-beds is still noticeable for a test-bed with an overall thickness of 3 mm.

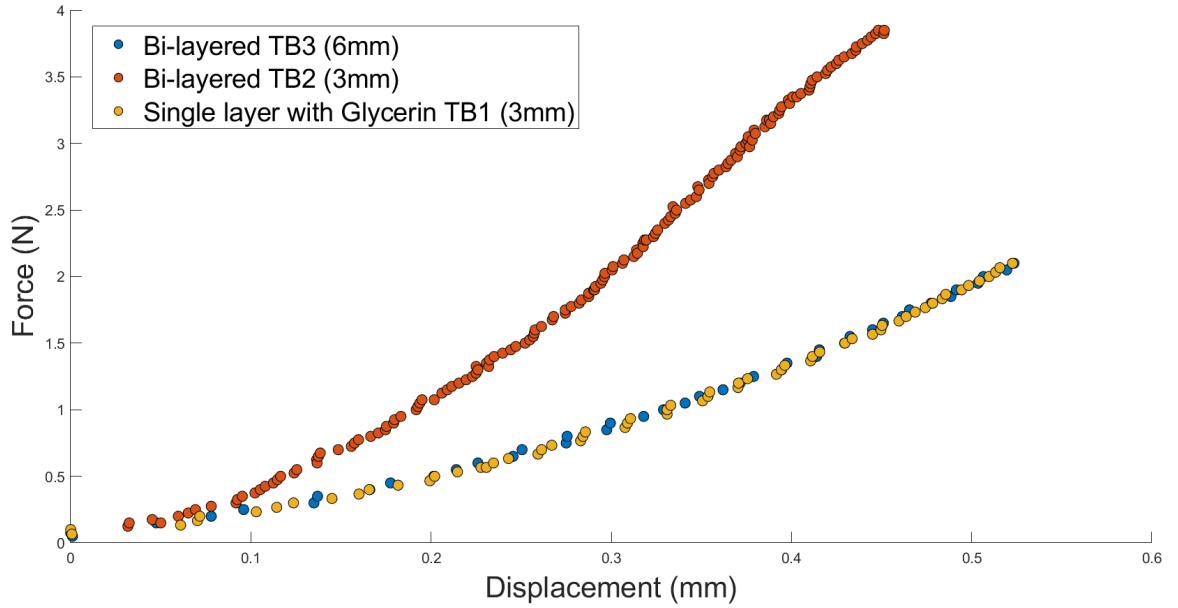


Figure 4.16: Force-displacement data for two bi-layered test-beds with a 1 mm upper layer with added glycerin and lower layers of different thicknesses, and a single layered test-bed with added glycerin.

The use of deadener (Polycraft Silicone Deadener Additive, MB Fibreglass) was also explored to obtain a more compliant and visco-elastic material which would more closely match the properties of human forearm skin. Deadener is a silicone modifier which inhibits cross-linking of the silicone. It can be added up to a maximum of 40 wt% of the total weight of the silicone as adding too much into the mixture could hinder curing. Figure 4.17 shows the effect of different weight percentages of deadener on the behaviour of the test-beds under compressive loading from 1 to 5N. As expected, test-bed containing the most deadener (35wt%) was found to be the most compliant. The graph also shows the differences in the area of the hysteresis loops, which is larger for the test-beds with more deadener. This highlights the increased visco-elasticity of the test-beds with a higher weight percentage of deadener.

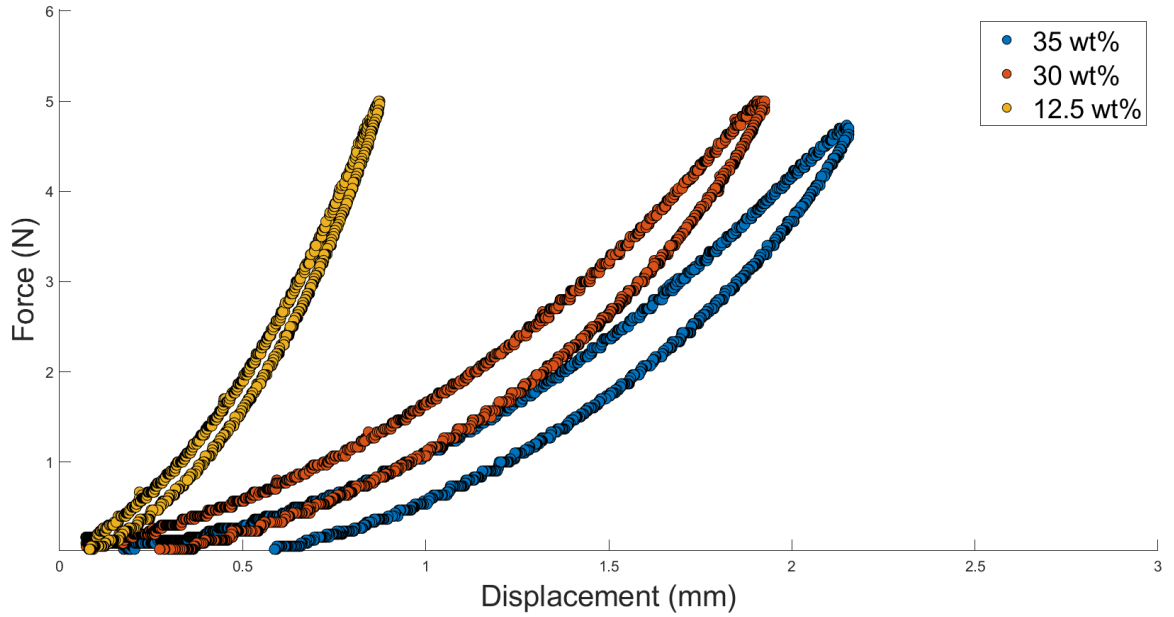


Figure 4.17: Force-displacement data for three SS test-beds (TB4) with different deadener weight percentages.

4.5.2 Test-bed surface texture

Surface roughness measurements were taken using an optical profilometer as outlined previously. Surface texture could have a significant effect on the sensory perception of BPC products hence it is of interest to replicate the surface texture of human volar forearm skin onto the SS test-bed. The resulting measurements of test-bed roughness was also used to evaluate whether the use of the Vaseline Petroleum Jelly (Unilever, London, UK) as a release agent for the SILFLO replica would cause a considerable loss in groove depth. Figure 4.18 shows the surface texture of base silicone. Interestingly, the alignment of the grooves seems to follow a pattern of parallel lines which could influence the directionality dependence of skin deformation.

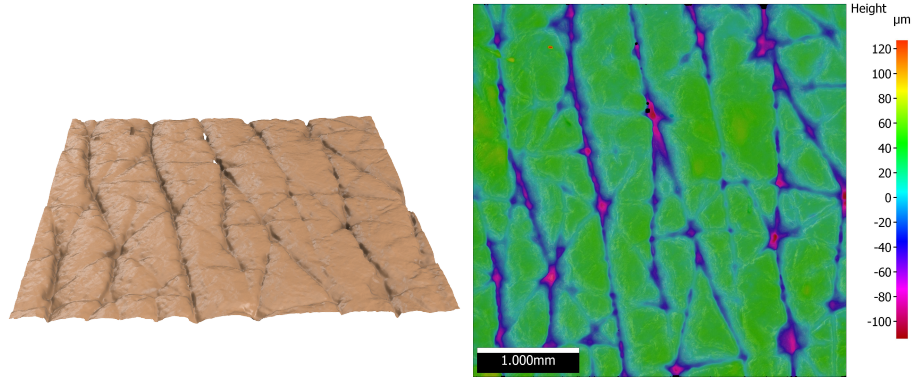


Figure 4.18: Surface texture of a SS test-bed made with Silfo.

The areal average roughness (S_a) and skewness (S_{sk}) of the SS test-bed were measured and compared with the values found for a human volar forearm in literature [99]. The areal average roughness represents the average height of all points measured over an area whereas the skewness indicates whether a surface is made up mostly of valleys (negative values) or peaks (positive value) [106]. The S_a was preferred over R_a for a more comprehensive view of the roughness over a larger area. As the surface texture of human skin is irregular, vital information could be missed by solely comparing two dimensional R_a values. The S_a value measure, as listed in table 4.12, was $1\mu\text{m}$ below the range reported in literature which is most likely due to the thickness of C. However, the skewness value indicates that the surface is still dominated by valleys, which is highlighted in the 2D roughness profile in Figure 4.19. These valleys could play a key role in the tribological behaviour of the SS test bed and therefore it is of importance that they are replicated in the test-bed.

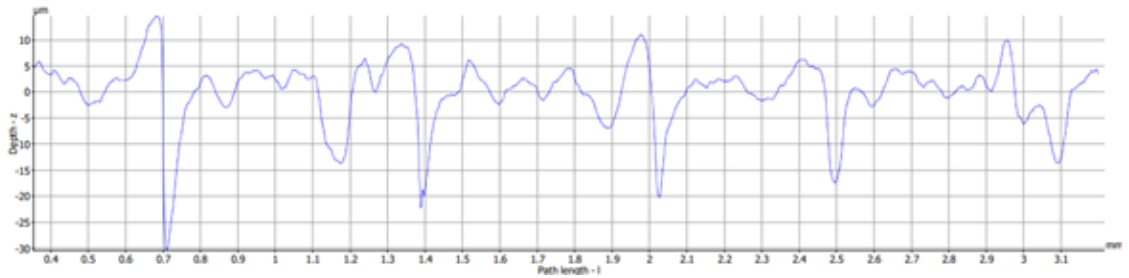


Figure 4.19: 2D profile of the SS test-bed surface texture.

Table 4.12: Areal average roughness (Sa) and Skewness (Ssk) values obtained in the optical profilometer for a textured SS test bed.

Substrate	Sa (μm)	Ssk
Volar Forearm [99]	17.0-20.0	-0.771
SS test bed	16.0	-1.30

4.5.3 Frictional behaviour

This section details the experiments undertaken to assess the effect of adding texture and deadener into the models on the frictional behaviour of the test-beds, in the presence of a BPC product. Four TB4 test-beds were made (Table 4.11), two with 12.5 wt% of deadener and two with 25wt% of deadener. Volar forearm texture was added to one of each pair whilst the other was left smooth. Product D was applied to each test-bed and the friction was measured immediately and 5 minutes after application. The normal load used was 1N, with a sliding speed of 10 mm/s over a sliding distance of 30 mm. The friction was measured in the UMT over 6 cycles, with one cycle representing an out-and-back trajectory. The probe was moved either left or right along the test-bed between the immediate and 5 minutes measurements (as pictured in Figure 4.9) to avoid taking measurements where the product had been removed by the probe. These tests were repeated three times for each test-bed. The test-beds were washed with soap-free face wash (Simple Face Wash, Unilever), dabbed with a paper towel and left to dry for 10 minutes before the D was re-applied between each repeat. The probe was wiped with isopropyl alcohol between tests. Each data point was averaged between the repeats and the resulting data set was plotted.

Figures 4.20 and 4.21 show the averaged data for friction force over time for the different test-beds immediately and 5 minutes after application respectively for the 6 cycles. The differences in friction become apparent from the first few strokes. Both textured test-beds seem to maintain a constant friction force whereas both smooth test-beds show much larger variations during a single stroke. Figure 4.20 also shows an increase in friction over time all test beds which could be attributed to the product gradually being pushed out of the contact by the probe along the linear trajectory.

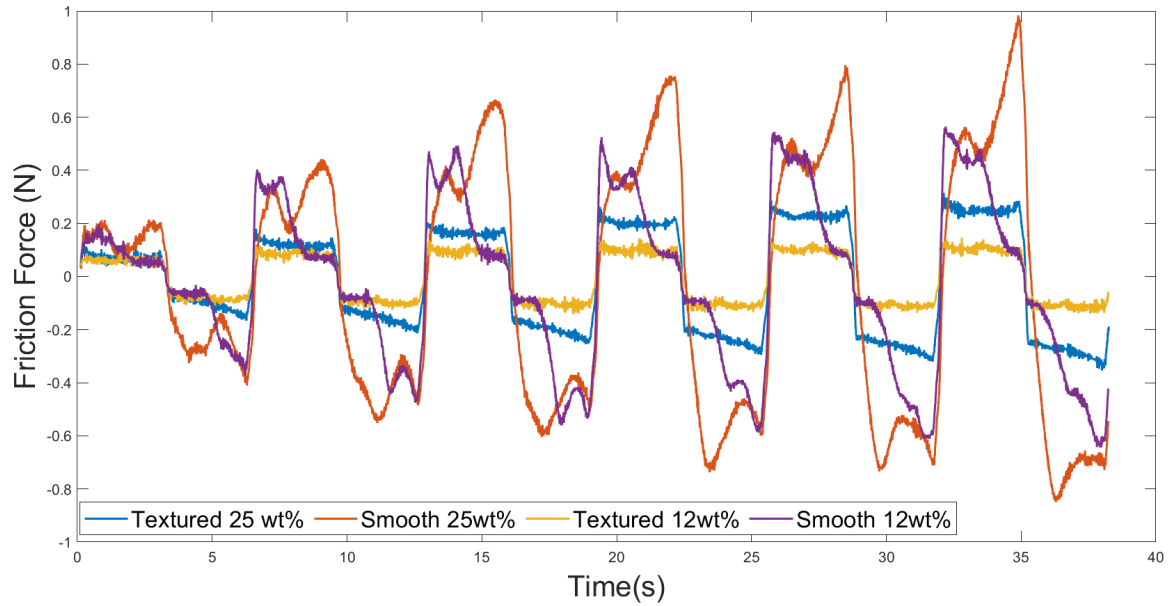


Figure 4.20: Friction force over time for textured and smooth test-beds with different deadener weight percentages immediately after application of D ($n=3$).

After 5 minutes the differences in friction force between each test-bed become more apparent. The friction force still appears constant for both textured models, and the variations in friction also appear to be reduced for both smooth models. However, some evidence of deformation becomes more apparent for the smooth model with 25wt% deadener as the friction curve seems to become more peak-like towards to end of the test which implies a reduction in the amount of sliding by the probe. A larger increase in friction force over time can be observed for the textured test-bed with 25wt% compared to the textured test-bed with less deadener, which could be attributed to the increase in compliance of the test-bed thus increasing the contact area.

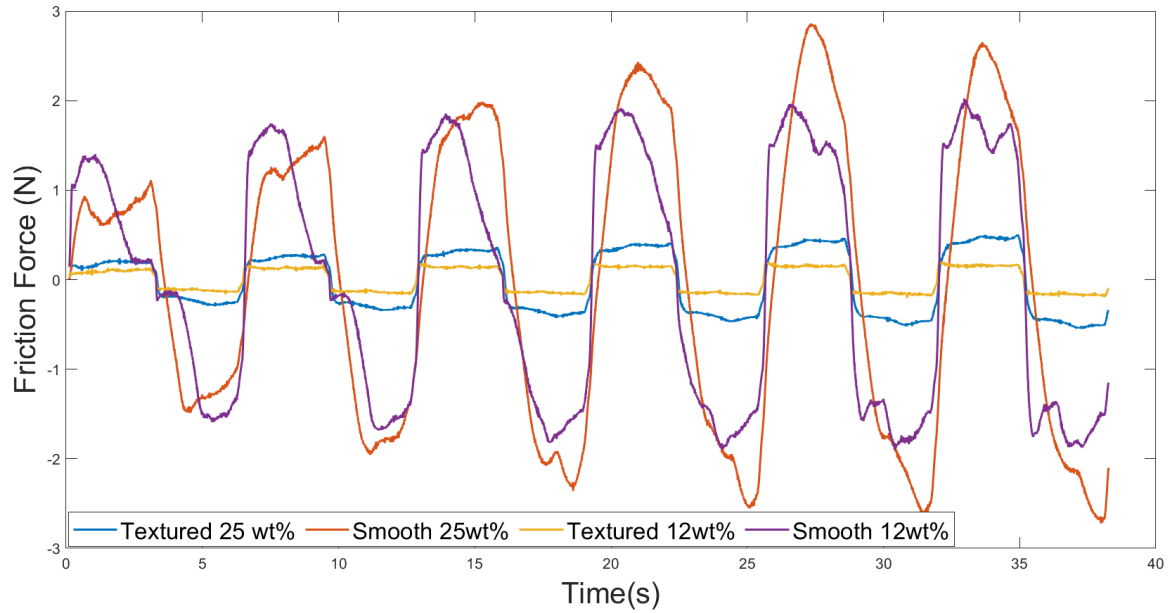


Figure 4.21: Friction force over time for textured and smooth test-beds (TB4) with different deadener weight percentages 5 minutes after application of D (n=3).

The mean DCOF calculated for each test-bed and the standard deviations between each repeat are shown in Figure 4.22 and summarised in Table 4.13. These values were obtained using the method previously outlined. The largest standard deviations were observed for both smooth models. This could be attributed to stick-slip due to an increase in adhesion incurred by the increase in contact area. The highest friction forces were obtained for the smooth test-bed with 25wt% deadener and the lowest for the textured test-bed with 12.5 wt% deadener.

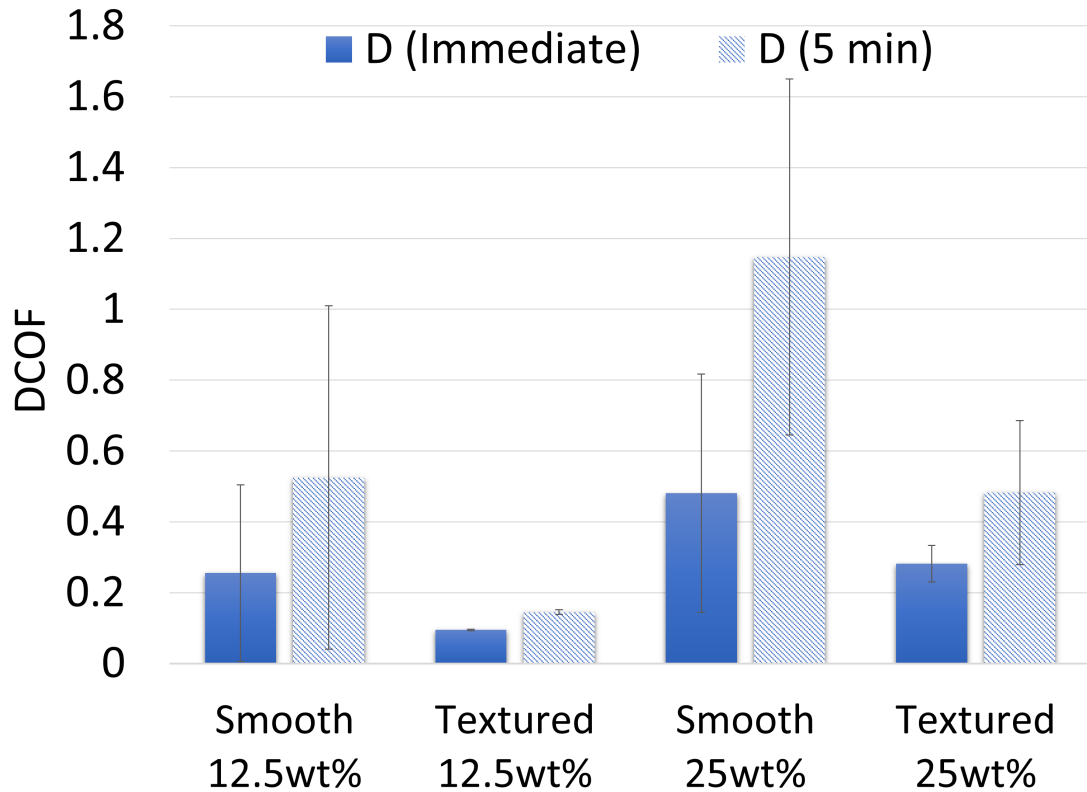


Figure 4.22: Mean dynamic coefficient of friction and standard deviations for textured and smooth test-beds (TB4) with different deadener weight percentages immediately and 5 minutes after application of D (n=3).

Table 4.13: Summary of the number of repeats, mean DCOF and standard deviations for the smooth and textured test-beds (TB4) with different weight percentages of deadener.

Deadener wt%	Time	Test-bed	N	Mean DCOF	Std. Deviation
12.5	Immediate	Smooth	3	0.255	0.305
		Textured	3	0.095	0.003
	5 minutes	Smooth	3	0.525	0.593
		Textured	3	0.145	0.008
25	Immediate	Smooth	3	0.481	0.3675
		Textured	3	0.282	0.063
	5 minutes	Smooth	3	1.15	0.515
		Textured	3	0.483	0.249

Following several independent T tests it was found that the DCOF for all textured test-beds combined regardless of the deadener weight percentage was significantly lower than the DCOF for the smooth test-beds ($p < 0.05$) (Table 4.14).

Table 4.14: Results from an independent t-test for the difference between the total mean DCOF of the textured test-beds (n=6) and the total mean DCOF of the smooth test-beds (n=6)

t	df	Significance	
		One-Sided p	Two-Sided p
2.19	13.9	0.023	0.046

For the test-beds with 25wt% deadener the mean DCOF measured for the smooth test-beds was significantly higher than the mean DCOF of the textured test-beds ($p < 0.05$). There were no significant differences for the test-beds with 12.5 wt% deadener (Table 4.15).

Table 4.15: Results from an independent t-test for the difference between the DCOF of the textured and smooth models for different weight percentages of deadener (n=6).

Deadener wt%	t	df	Significance	
			One-Sided p	Two-Sided p
12	1.48	5.04	0.099	0.199
25	1.84	10	0.048	0.096

There were no significant differences between the immediate and 5 minutes DCOF for both the 12.5 and the 25 wt% deadener (Table 4.16).

Table 4.16: Results from an independent t-test for the difference between the DCOF immediately and 5 minutes after product application at different weight percentages of deadener (n=3).

Deadener wt%	Time	t	df	Significance	
				One-Sided p	Two-Sided p
12	Immediate	0.911	2.000	0.229	0.458
	5 minutes	1.110	2.001	0.191	0.383
25	Immediate	0.927	2.118	0.223	0.447
	5 minutes	2.012	4	0.057	0.115

Lastly, the DCOF measured on the textured test-bed with 12.5 wt% deadener was significantly lower than for the textured test-bed with 25 wt% deadener, immediately after product application ($p < 0.05$) (Table 4.17).

Table 4.17: Results from an independent t-test for the difference between the DCOF of smooth and textured test-beds immediately and 5 minutes after product application (n=3).

Time	Test-bed	t	df	Significance	
				One-Sided p	Two-Sided p
Immediate	Smooth	-0.819	4	0.229	0.459
	Textured	-5.128	2.008	0.018	0.036
5 minutes	Smooth	-1.372	4	0.121	0.242
	Textured	-2.348	2.004	0.072	0.143

Finally, a paired t-test was run to assess the differences in friction immediately and after 5 minutes for each test-bed. The results detailed in Table 4.18 revealed a significant increase in friction after 5 minutes for the textured test-bed with 12.5wt% deadener and for the smooth test-bed with 25 wt% deadener.

Table 4.18: Results from a paired t-test on the mean DCOF for the textured and smooth test-beds of with different weight percentages of deadener immediately and 5 minutes after product application.

Pair	t	df	Significance	
			One-Sided p	Two-Sided p
Textured 12.5wt% Imm - Textured 12.5 wt% 5 min	-12.705	2	0.003	0.006
Textured 25wt% Imm - Textured 25 wt% 5 min	-1.858	2	0.102	0.204
Smooth 12.5wt% Imm - Smooth 12.5 wt% 5 min	-1.621	2	0.123	0.246
Smooth 25wt% Imm - Smooth 25 wt% 5 min	-6.811	2	0.010	0.021

4.5.4 Discussion

Indentation tests revealed that the addition of deadener in the silicone mixture increased the compliance and visco-elasticity of the test-beds. Additionally, it lead to a significant increase in the DCOF of the test-beds against a solid probe following product treatment.

Furthermore, results from profilometry measurements showed that the method used to add texture onto the test-beds was suitable for replicating the human volar forearm skin texture, although there was slight reduction in Sa when compared to human skin which was attributed to the use of Petroleum Jelly as a means to stop the two silicones

from adhering. Texture was found to significantly reduce the DCOF both immediately and 5 minutes following product application for the test-bed with 25wt% deadener. The decrease in friction with increasing roughness of materials was highlighted in several other studies [107, 8], and was associated with a decrease in real contact area thus decreasing the adhesion with contacting materials. Ding et al. [100] also related changes in the friction of pig skin to changes in roughness following hydration. They found that the friction increased with decreasing roughness.

Moreover, Adams et al. [14] observed that for wet human skin, which has an increased compliance compared to dry skin, the topographical features are expected to be flattened under a sliding load applied by a solid probe and therefore act a smooth surface. Hence this could explain the increase in friction measured for the textured test-beds with a higher weight percentage of deadener.

Lastly, although the friction appears to increase for all test-beds 5 minutes after the application of D, these differences were only significant for the textured test-bed with 12.5wt% deadener and for the smooth test-bed with 25wt% hence no real trend can be identified from the data. This can be associated to the large standard deviations found between the means of the three repeats. However, it can be hypothesized that the more pronounced increase in friction for the smooth test-beds is due to the effect of texture on the BPC product lubricating process. As the test-beds did not contain any glycerin, the hydrating effect can be discarded from the mechanisms observed. In fact, the furrows highlighted in the optical profilometer images may act as reservoirs for the product as it is spread across the skin, thus allowing a continuous lubrication process as the probe slides across the test-bed and the product is squeezed out of the furrows. However, as the compliance of the test-bed is increased, these furrow may become stretched and flatten, as mentioned in the Adams et al. study [14], therefore causing these reservoirs to empty more rapidly. This theory can be validated by the fact that for the textured test-bed with 25 wt% deadener, the standard deviation was low for the immediate measurements but high after 5 minutes. For the smooth models this reservoir effect is non-existent therefore giving larger differences as the product dries more rapidly, therefore increasing the likelihood of stick-slip.

4.6 Effect of a bi-layered design

4.6.1 Test-bed mechanical properties and structure

The last combination to be tested was a bi-layered test-bed with a textured 1 mm upper layer and a 5 mm lower layer cross-linked with deadener at 35 wt%. However, considering the SILFLO replica had to be placed within the 1mm deep groove in the aluminium mould, the epidermis was expected to be thinner than 1mm. An example of the combined SS test-bed is shown in figure 4.23 below. Interestingly the addition of a stiffer upper layer caused a wrinkling effect when the SS test-bed was squeezed between two fingers. The two test-beds used for this section are detailed in Table 4.19.

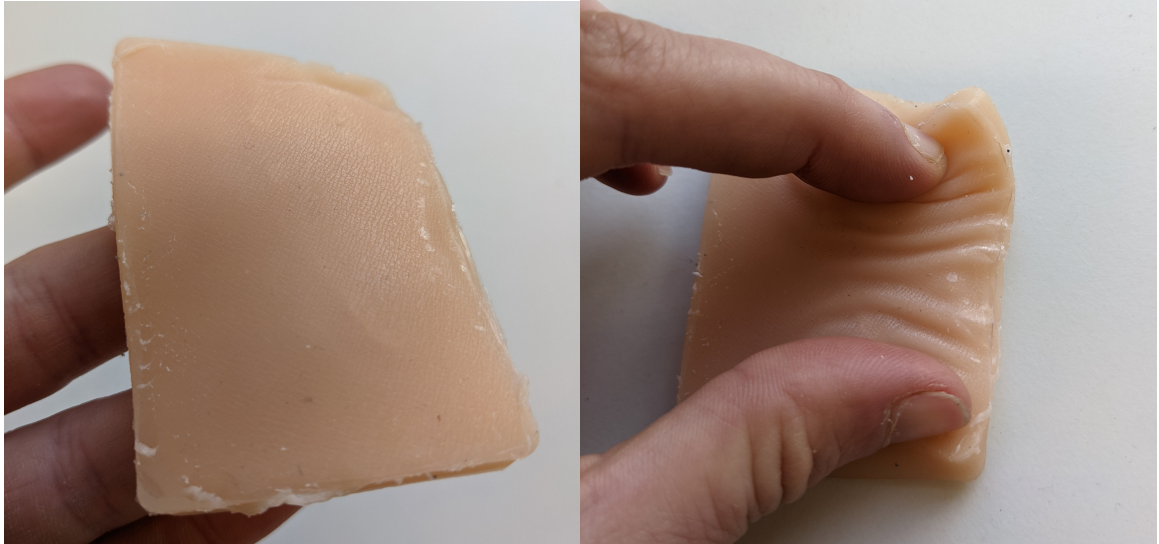


Figure 4.23: A textured bi-layered SS test-bed with 35 wt% deadener in the lower layer, and the observed “wrinkling effect”.

Table 4.19: Test-beds manufactured for evaluating the effect of a bi-layered structure on the test-bed behaviour.

Test-bed name	Test-bed description	Composition
TB5	Smooth silicone with deadener	Single 6 mm layer of addition cure silicone (base + catalyst at 1:1 ratio) with added deadener at 35 wt%.
TB6	Textured bi-layered silicone with deadener	Top layer (<1mm) : addition cure silicone (base + catalyst at 1:1 ratio) with added texture. Bottom layer (5mm) : addition cure silicone with added deadener at 35 wt%.

The silicone used for making the SS test-beds is naturally translucent. Hence the use of pigments (Polycraft Silicone Pigment, MB Fibreglass) was trialled to differentiate between the layers. In the interest of obtaining an accurate measurement of the upper layer thickness, optical coherence tomography (OCT) was used to obtain a cross sectional view of the two layers. OCT uses the principles of light interference to allow for the visualisation of the internal structure of materials of biological systems. Therefore, the upper layer should be translucent enough to allow for the light to travel through, whilst the lower layer should be more opaque to allow for an accurate differentiation of the layers. The set-up is shown in figure 4.24 below.

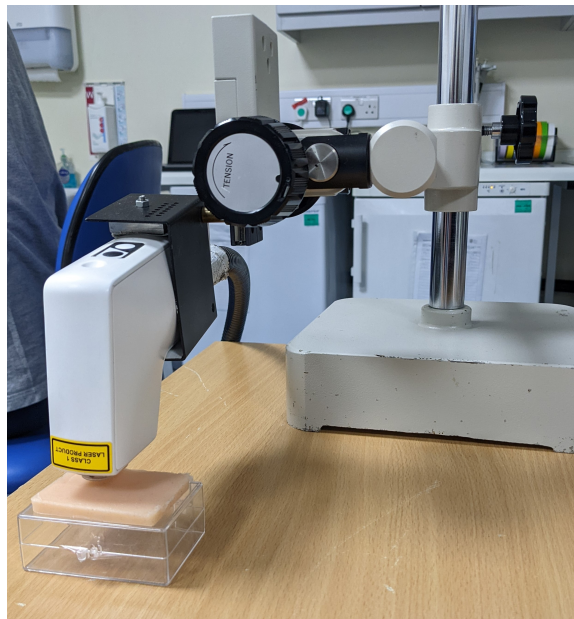


Figure 4.24: OCT set-up.

The scan obtained is shown in figure 4.25 below. The thickness of the upper layer was measured at 0.4 mm.

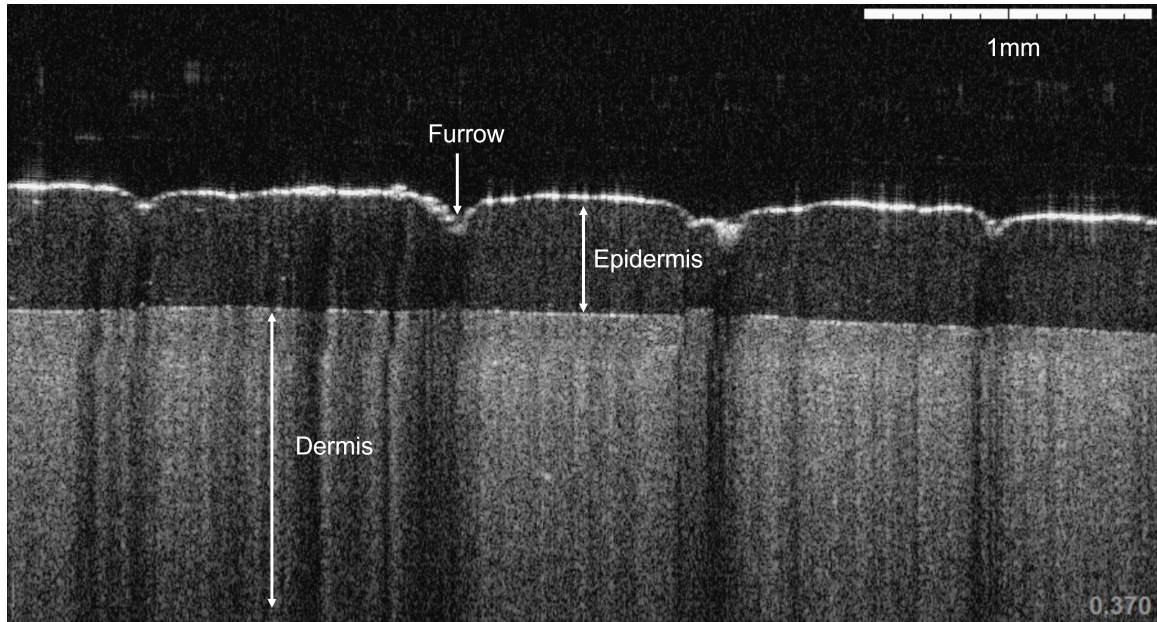


Figure 4.25: Cross sectional area of a textured bi-layered SS test-bed obtained via OCT.

The mechanical properties of the test-beds were assessed using the method outlined in a previous section. Figure 4.26 below shows the force versus displacement curves for the test-beds TB5 and TB6. The force-displacement data for the bi-layered model shows a slightly stiffer behaviour, which becomes more apparent at higher loads.

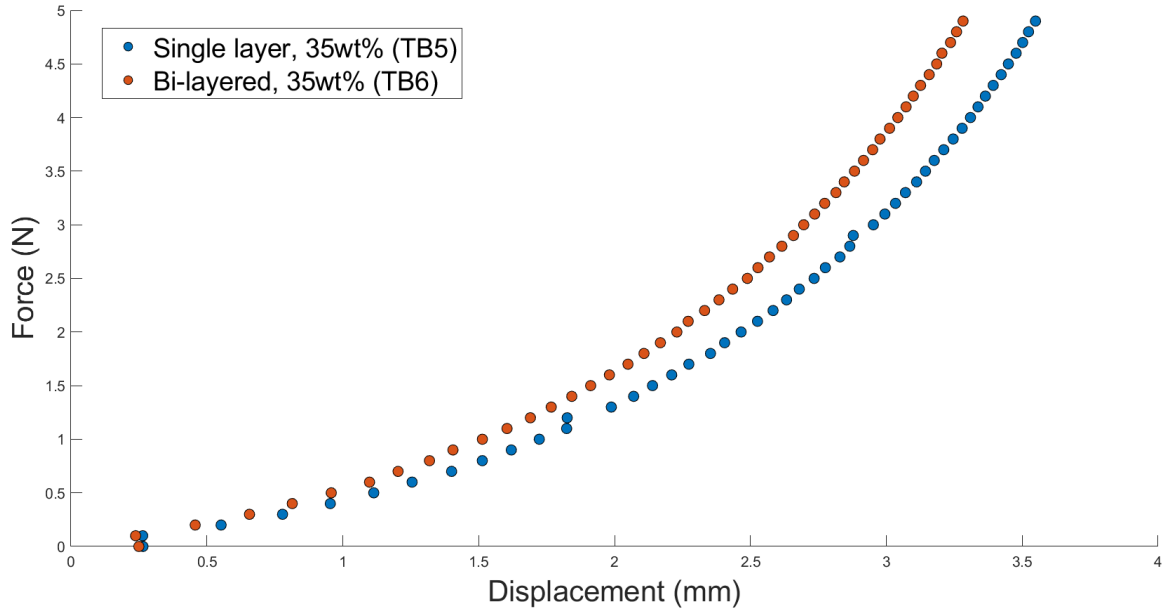


Figure 4.26: Force-displacement data for a bi-layered and single layered SS test-bed.

4.6.2 Friction behaviour

The effect of adding a stiffer upper layer to a SS test-bed cross-linked with deadener was assessed in the UMT at a normal load of 1N and at a speed of 10 mm/s over a distance of 30 mm. The test was repeated three times per test-bed and the probe was moved to different parts of the test-beds between each repeat. The raw friction data over the six cycles for the bi-layered and the single layered test-bed are shown in Figures 4.27 and 4.28 respectively. Both figures show the results from the three repeated runs, which were measured after moving the probe left or right along the width of the test-beds. The peak friction force measured was around 3N for both test-beds although slightly higher for the bi-layered test-bed. No DCOF could be extracted from the data as the deformation was such that no sliding occurred.

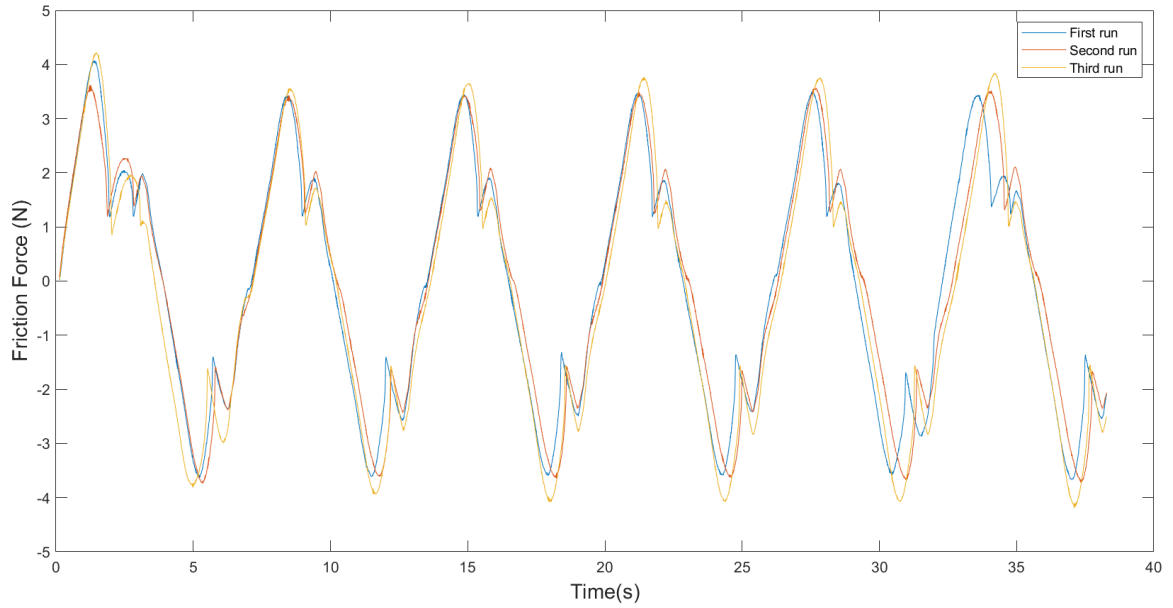


Figure 4.27: Friction force over time for a textured, bi-layered SS test-bed modified with deadener at 35 wt%.

The main difference between the two test-beds seems to be the way in which they deform. In the raw data for the bi-layered test-bed, several peaks are observed during a single slide in contrast to a single peak for the single layered test-bed in the majority of the strokes. This gives further evidence to the “wrinkling effect” previously observed in Figure 4.23. However, more importantly these graphs highlighted the need for longer test-beds to allow for sliding to develop for the more compliant test-beds. Additionally, it was observed that the UMT was unable to maintain a normal load within a suitable range due to the increase in deformation, which highlighted the limitations of this method for testing the SS test-beds.

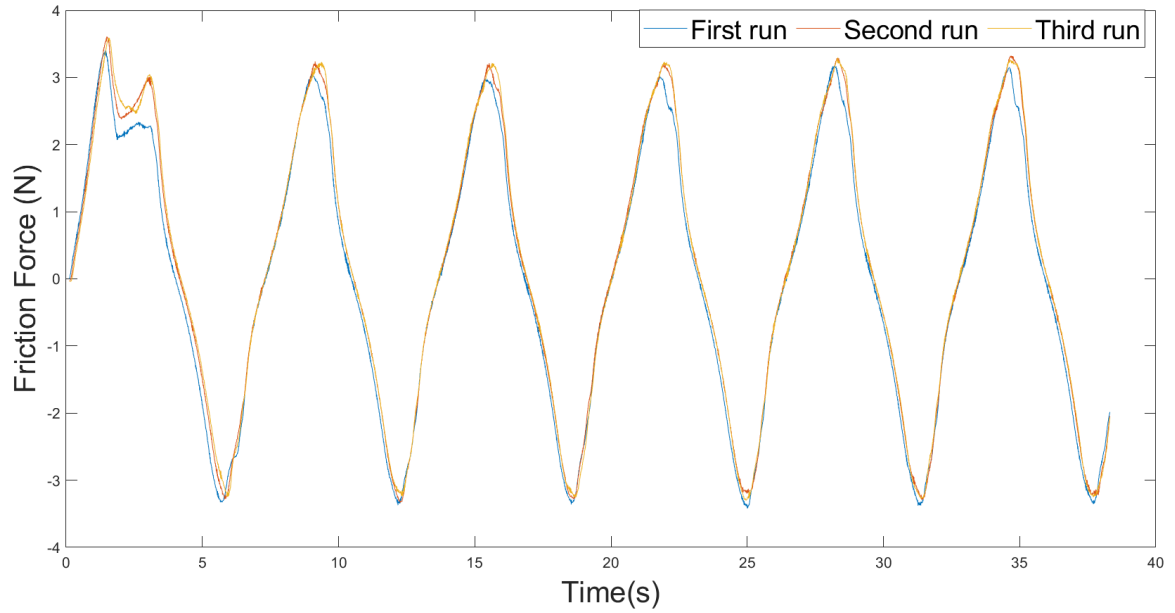


Figure 4.28: Friction force over time for a smooth, single layered SS test-bed modified with deadener at 35 wt%.

4.6.3 Discussion

Adding a stiffer layer to a test-bed cross-linked with deadener at 35wt% was found to increase its bulk stiffness with increasing load. Adding pigment to the lower layer also allowed for the visualisation of both layers using OCT, and for the measurement of the upper layer thickness. This could be used in the future to look at the behaviour of both layers under shear.

Although no measurements of DCOF could be used to compare the two test-beds, these results highlighted the differences in deformation behaviour between the two test-beds, with a “wrinkling effect” observed for the bi-layered test-bed. Changes in deformation patterns could impact the stimulus of mechanoreceptors within the skin and hence is important to consider when developing a test-bed for the assessment of sensory perception.

Lastly, the findings suggested the need to increase the SS test-bed length in order to allow for sliding with increased deformation, and highlighted the limits of using an automated method of testing for highly compliant and visco-elastic materials.

4.7 Plasma treatment

4.7.1 Methodology

Silicones typically have a surface energy of 22mN/m whereas the surface energy of human skin covers a range of 27mN/m to 38.2 mN/m [31]. The use of Oxygen plasma treatment, of which the set-up is pictured in Figure 4.29, was explored as a means to increase the surface energy of the SS test-beds. Three parameters can be controlled when using plasma treatment: duration, power and chamber pressure. These parameters were altered in order to obtain a surface energy between 36 and 38 mN/m. The optimal set of parameters was found to be 90 seconds at 10 W at a pressure of 0.8 mbar. Surface energy is traditionally calculated using the sessile drop test method. However this method can be time consuming and without an automated system to calculate the droplet angle, can lead to user error. Hence the use of inks was explored to track changes in surface energy. These were also used in a study by Pankaj et al. [80] looking at the effect of plasma treatment on polymers.

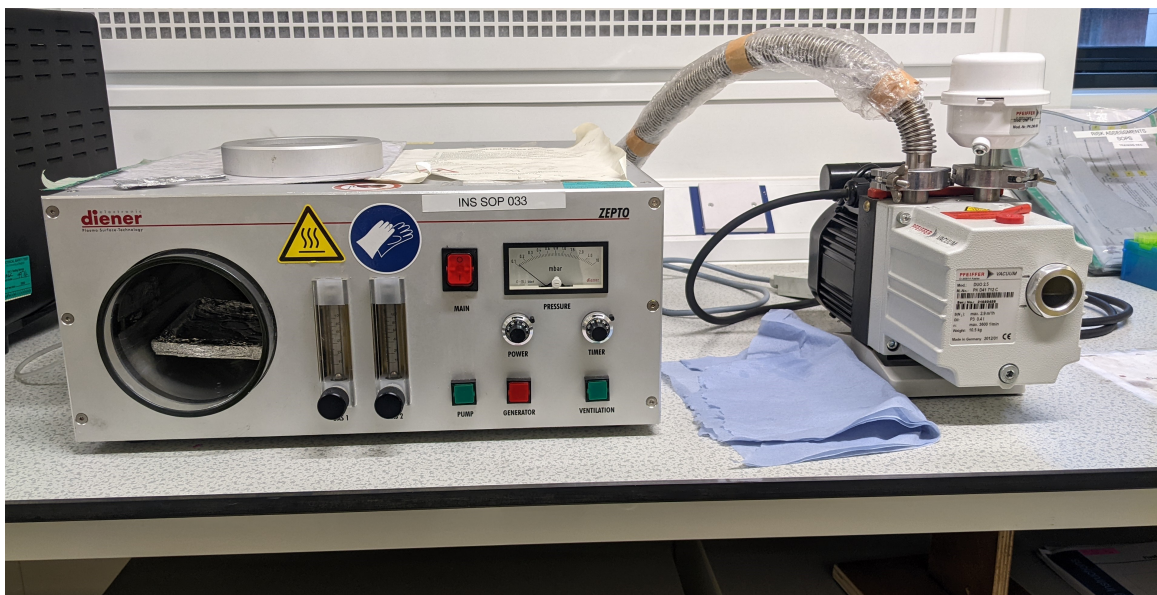


Figure 4.29: Oxygen plasma treatment rig used for the experiments.

The inks (Smartdrop Test Inks, Acxys Technologies) used have discrete surface tensions meaning they will only wet a surface when the surface energy of the substrate is equal or above their specific surface tension. Figure 4.30 demonstrates the changes before and after plasma treatment on two test-beds, one smooth and one textured.

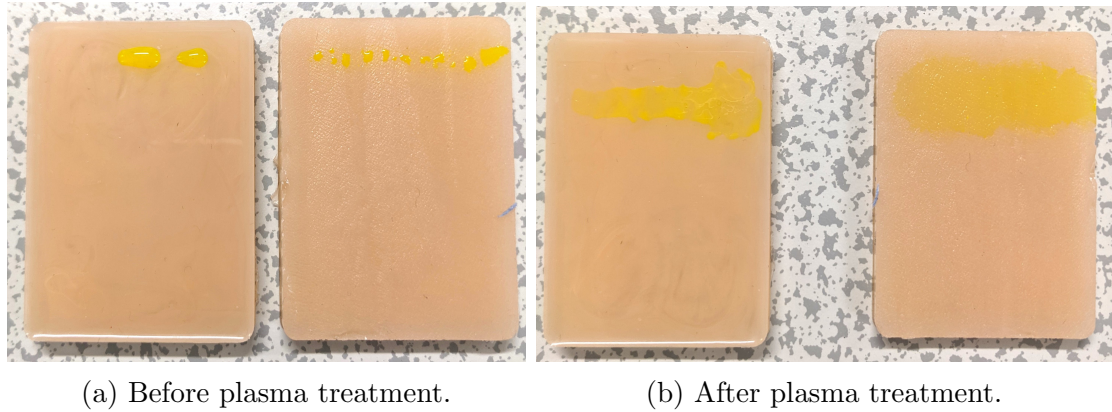


Figure 4.30: Ink on the SS-test-bed

Plasma treatment was tested on three SS test-beds which are detailed in Table 4.20. The friction was measured three times for the same test-bed, using the same contact parameters as described in the above sections. The plasma treatment was only done once per test-bed. The probe was moved to different areas of the test bed for the repeated measurements.

Table 4.20: Test-beds manufactured for evaluating the effect of plasma treatment on the DCOF.

Test-bed name	Test-bed description	Composition
TB5	Smooth silicone with deadener	Single 6 mm layer of addition cure silicone (base + catalyst at 1:1 ratio) with added deadener at 35 wt%.
TB6	Textured bi-layered silicone with deadener	Top layer (<1mm) : addition cure silicone (base + catalyst at 1:1 ratio) with added texture. Bottom layer (5mm) : addition cure silicone with added deadener at 35 wt%.
TB7	Textured silicone	Single 6mm layer of addition cure silicone (base + catalyst at 1:1 ratio) with added texture.

4.7.2 Results

The mean DCOF and standard deviations between different measurements are shown in Figure 4.31 below and summarised in Table 4.21.

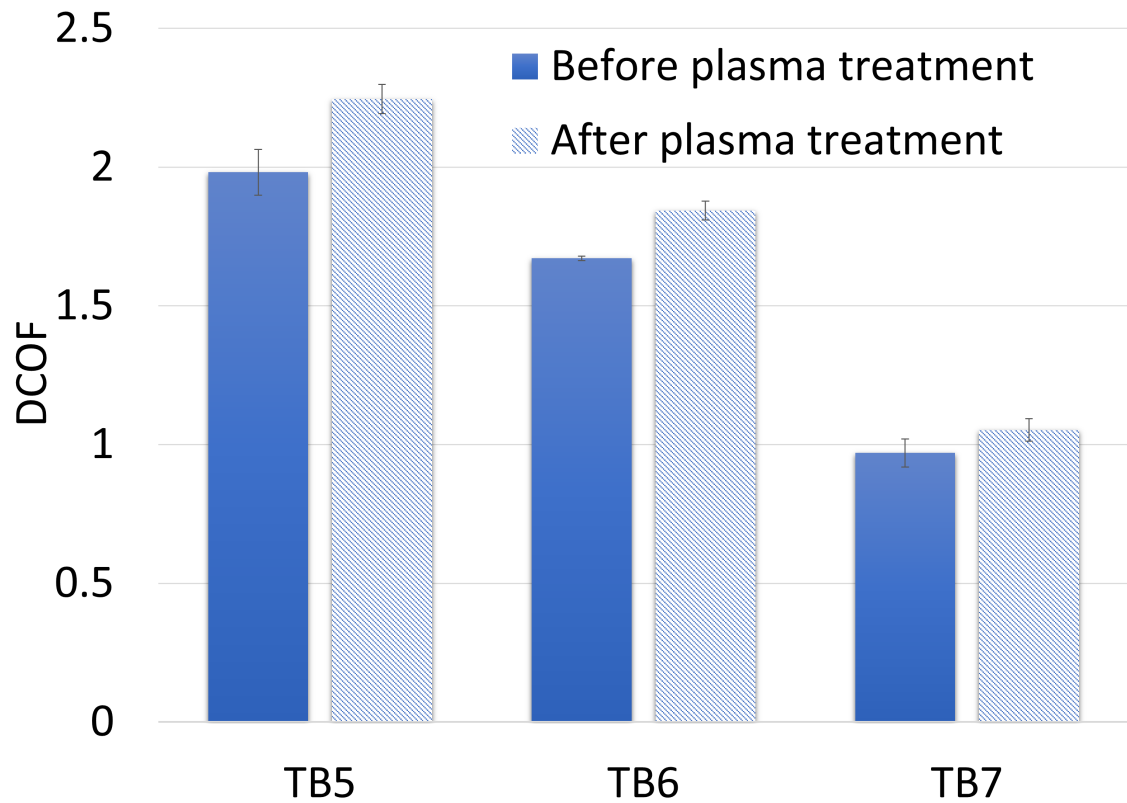


Figure 4.31: Mean DCOF and standard deviations before and after plasma treatment for the three SS test-beds.

The graph shows an increase in DCOF for all three test-beds. A paired t-test was performed (Table 4.22) which revealed that the increase in DCOF after plasma treatment was statistically significant for all test-beds ($p < 0.05$).

Table 4.21: Summary of the mean DCOF and standard deviation before and after plasma treatment for the three test-beds.

Test-bed	Condition	Mean	N	Std. Deviation
TB5	Plasma	2.24	3	0.0643990
	No plasma	1.98	3	0.1012545
TB6	Plasma	1.84	3	0.0409979
	No plasma	1.67	3	0.0098997
TB7	Plasma	1.05	3	0.0495413
	No plasma	0.97	3	0.0621589

Table 4.22: Results from a paired t-test for the difference in DCOF before and after plasma treatment.

Test-bed	Pair	t	df	Significance	
				One-Sided p	Two-Sided p
TB5	Plasma - No plasma	2.98	2	0.048	0.097
TB6	Plasma - No plasma	6.24	2	0.012	0.025
TB7	Plasma - No plasma	11.3	2	0.004	0.008

4.7.3 Discussion

Plasma treatment was considered as a means to increase the surface energy of the test-beds. A higher surface energy increases the wettability and adhesion of a substrate which could influence interactions with BPC products. Results from friction measurements of plasma treated versus non-plasma treated test-beds did reveal an increase in DCOF for all three plasma treated test-beds, and the use of inks was successful in allowing for the direct visualisation of changes in surface energy.

Although these differences were statistically significant, the plasma treatment was not repeated on the same test-bed more than once and hence it is difficult to conclude that plasma treatment will consistently result in an increase in DCOF. However, the tests have shown that changing the surface energy of the test-beds may affect the DCOF and that plasma treatment appears to work on textured surfaces. Nevertheless, as mentioned in Chapter 2, Oxygen plasma treatment is not indefinite and the process starts reversing as soon as the test-bed is exposed to the local atmospheric pressure [83]. That being said, other avenues may be explored, such as higher power and longer treatment times or using different plasmas (e.g. Argon [82]).

4.8 Conclusions

The aim for these preliminary tests was to inform on the influence of each test-bed design element on the test-beds' frictional behaviour in the presence of BPC products.

- Adding glycerin to the silicone mixture was successful in allowing water absorption into the test-beds. The hydration of the test-beds in turn caused an increase in DCOF and significant differences were observed between the DCOF

of hydrated and non-hydrated test-beds following the application of a BPC product (C).

- Friction measurements in dry and wet conditions revealed a significantly higher DCOF for the steel probe than for the PMMA probe in the wet conditions. With the surface energy of PMMA being closer to that of human skin, it was preferred as a material for measurements in the continuation of this study.
- Adding human volar forearm texture to the test-beds significantly decreased the DCOF following the application of a BPC product (D).
- The impact of the material underneath the test-beds of 3 mm in thickness on the measured stiffness at 1N highlighted the need for an increased overall thickness (from 3 mm to 6 mm).
- An increase in compliance, visco-elasticity and DCOF of the test-beds was observed with increasing weight percentage of deadener. The increase in compliance also lead to an increase in friction following product treatment (D).
- Combining a stiffer layer of less than 1 mm in thickness with a highly visco-elastic lower layer in a test-bed influenced the bulk mechanical properties of the test-beds and its deformation behaviour under shear loading.
- Oxygen plasma treatment increased the surface energy of the test-beds which appeared to increase the DCOF. However, more studies are needed to assess its use for the assessment of BPC product tribology.

These results informed on key elements to include in the final protocol. Firstly, it was decided that the probe used would be made out of PMMA, and that the test-bed would be textured. The moulds were also re-designed to double the test-bed length, and to achieve a thickness of 6mm. Deadener would be used to tailor the test-bed visco-elasticity and glycerin added in the superficial layer to allow for water-absorption. A manual method for measuring friction was explored and test-beds were not plasma treated for the reminder of the tests.

Chapter 5

Refining the synthetic skin test-bed design and assessing its capability in an industry setting

5.1 Introduction

In order to predict the perception of a product, it is necessary to understand what mechanisms can influence it. However, testing on human subjects is time consuming and can often result in large discrepancies due to naturally occurring physiological differences. Hence, the knowledge obtained from the previous chapters was used to develop a methodology for assessing the differences in frictional response of BPC products.

In Chapter 3, the frictional behaviour of human volar forearm skin was evaluated for three different products. In Chapter 4, different design elements were studied for their impact on the tribological behaviour of a test-bed in the presence of BPC products. Combining the knowledge obtained through these two chapters helped refine the test-bed design and finalise the testing protocol. In this chapter, four different test-beds were manufactured and tested using the method outlined in Chapter 3. The results were compared to the human data, and statistical tests were run to check for significant linear relationships between the measurements and the attribute scores.

This chapter also examines the use of synthetic test-beds in an industry setting. Four test-beds were manufactured in a Unilever laboratory where they were tested against a human finger-pad. The force plate used allowed for the measurement of adhesion, which has been found to predict various attributes related to the sensory

perception of BPC products in previous studies [41, 99]. The methodology previously used was adapted to suit the testing methods used at Unilever.

As the industry moves towards a fully automated and representative system for assessing differences between BPC products, replicating the tactile exploration process used during sensory assessments is of primordial interest. This means instrumenting a “finger against skin” interaction which will give valuable insight into the consumer experience.

This chapter will explore how effectively synthetic test-beds can replicate the frictional and adhesive interactions of human skin with BPC products under varying test conditions, and to what extent they can be used as reliable, repeatable alternatives to sensory panels in an industrial setting. The methods employed in this chapter expand on those introduced in Chapters 3 and 4, combining bench testing with human volunteer studies to closely reflect current industry protocols. Specifically, the synthetic skin test-bed was used in place of the human volar forearm as described in Chapter 3, in order to validate its ability to replicate real-world conditions. To further explore the interaction between the volar forearm and fingertip during product evaluation, the synthetic test-beds were also assessed against human finger-pads. This marked an initial step towards the development of a fully automated testing methodology.

5.2 Methodology

5.2.1 Synthetic skin test-bed manufacture

Two different moulds were lasercut out of acrylic for the manufacture of 150 x 50 x 6 mm test-beds. A 1 mm in depth 150 x 50 mm groove was CNC milled into the base of one of the moulds to allow for the manufacture of bi-layered test-beds. Addition cure silicone (Silskin 10 Special Effects, FibreGlass, MB) was used as a base for all test-beds. For texture, a replica of volar forearm skin was taken using SILFLO and placed at the bottom of the mould where it was covered with a thin layer of Petroleum Jelly to avoid any adhesion with the test-beds. All silicones mixtures were degassed for 2 minutes or until air bubbles no longer formed at the surface. For the bi-layered models, the silicone was poured in the 1 mm deep groove and a flat mixing stick was used to scrape the excess off. The thin layer was left to set for an hour and the 6 mm in thickness mould frame was then screwed to the base, as pictured on the left of Figure 5.1.

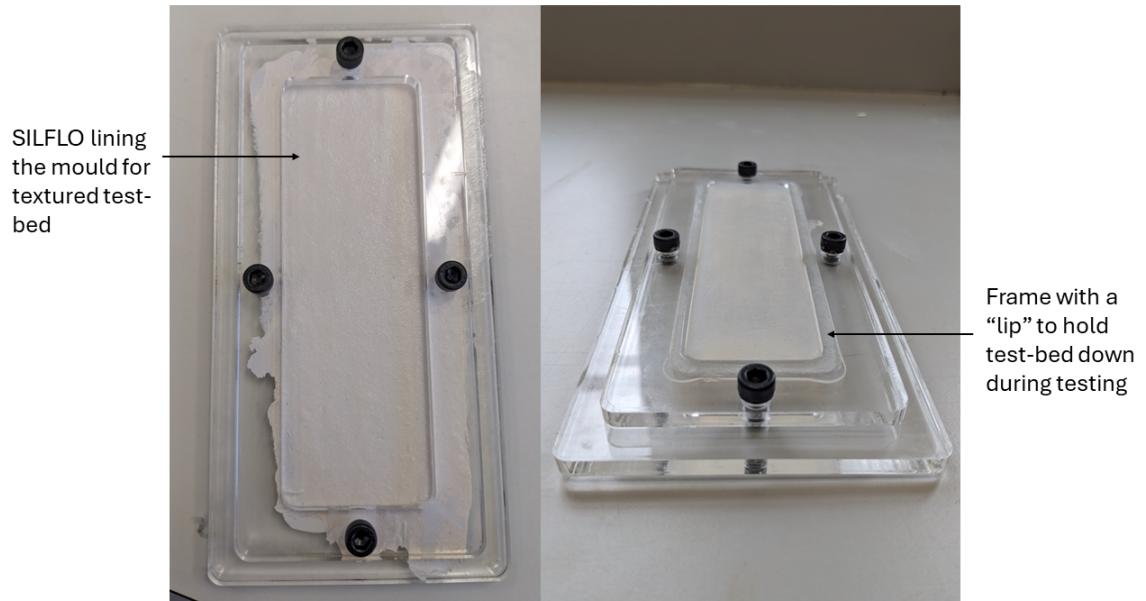


Figure 5.1: Mould lined with SILFLO used to make the textured test-beds (left) and frame used to secure the test-bed during friction measurements (right).

The second layer of silicone was then poured on top and left to cure for 1 hour or up to 24 hours for the silicones modified with deadener. Four different test-beds were made for the tests outlined in this chapter. A detailed description of each test-bed is given in table 5.1 below. For friction experiments, a frame with a “lip” was lasercut to hold the test-beds in place as they were held upside down above the probe (Figure 5.1).

Table 5.1: Name and description of the test-beds tested in this chapter.

Test-bed name	Test-bed description	Composition
TB1	Textured silicone	Single 6mm layer of addition cure silicone (base + catalyst at 1:1 ratio).
TB2	Textured silicone with glycerin	Single 6mm layer of addition cure silicone (base + catalyst at 1:1 ratio) with added glycerin at 40wt% of total silicone weight (base + catalyst)
TB3	Textured bi-layered silicone with deadener	Top layer (<1mm) : addition cure silicone (base + catalyst at 1:1 ratio) Bottom layer (2mm) : addition cure silicone with deadener at 40wt%.
TB4	Textured bi-layered silicone with glycerin and deadener	Top layer (<1mm) : addition cure silicone (base + catalyst at 1:1 ratio) with added glycerin at 40wt% Bottom layer (5mm) : addition cure silicone with deadener at 40wt%

5.2.2 Mechanical properties measurements

The test-bed mechanical properties were assessed using a force-displacement rig. The compressive force was applied with a spherical tipped probe of 16 mm in diameter, which was also used for the friction tests, at a speed of 2mm/s up to a maximum of 2N and removed at the same speed to allow for a measurement of hysteresis. Force-displacement data was obtained for the four test-beds and was re-measured for the bi-layered test-bed with glycerin in the “hydrated” state, which was achieved by submerging the test-bed in water for one hour. Three measurements were taken for each test-bed and the probe was moved to a different point on the test-bed between each measurement.

The mechanical properties of human volar forearms were also assessed for comparison, by applying a compressive load of up to 2N using a manual force-displacement rig. The participants placed their arm in an arm holder, as pictured in Figure 5.2, and were asked to place their hand on a handle whilst not gripping it with any force in order to keep their forearm in a relaxed state. The measurements were done on a total of 8 participants and repeated three times per participant. Details for the participants

are presented in Table 5.2 Ethical approval was obtained from the University’s Research Ethics committee prior to commencing the experiments (Application 050484, refer to the Appendix).

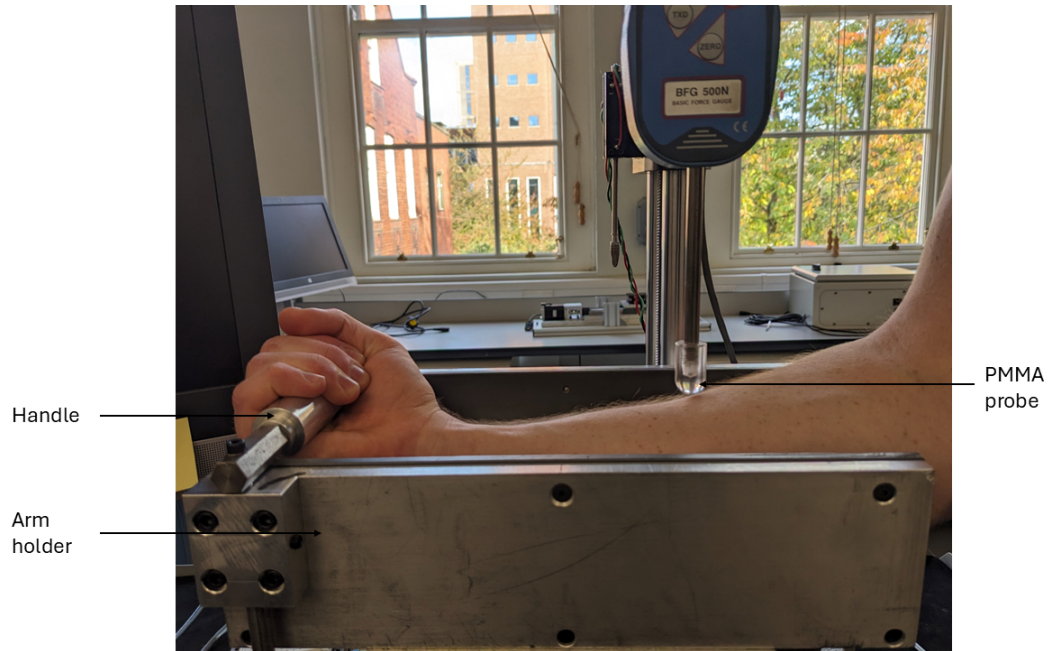


Figure 5.2: Set-up used for the indentation tests of human volar forearms (n=8).

Table 5.2: Participant ethnicities, age and sex for the volar forearm indentation tests.

Participant number	Ethnicity	Age (years)	Sex
1	White and Asian (Mixed or multiple ethnic groups)	22	Male
2	Any other white background	23	Male
3	Chinese (Asian or Asian British)	25	Male
5	English, Welsh, Scottish, Northern Irish or British (White)	22	Female
8	Any other white background	23	Female
10	English, Welsh, Scottish, Northern Irish or British (White)	23	Male
12	Any other white background	22	Female
14	Any other white background	23	Male

5.2.3 Friction measurements

5.2.3.1 Synthetic skin test-bed against a solid probe

The friction was first measured using the solid PMMA probe which was used in Chapter 3 to measure the friction on human volar forearms. This method aimed to replicate the method used in Chapter 3 whereby participants were asked to rub their volar forearm over a stationary probe. The manual method was preferred over the automated method in the UMT outlined in Chapter 4, as for compliant and visco-elastic materials the UMT was unable to maintain a normal load within the desired range. The test-bed was secured within a frame and held above the probe, as pictured in Figure 5.3. The test-bed was manually pressed onto the probe, as pictured in Figure 5.3, until a normal load between 0.5 to 1N was reached. This has previously been reported as the typical range of loads used for the sensory assessment of a surface [75]. It was then moved linearly in a reciprocating motion along its length whilst maintaining a constant load and speed of 10 mm/s. The optimal speeds used for tactile exploration have been reported to range from 10 to 200 mm/s [96], however, as the speed was not recorded by the force plate, using a lower speed was preferred as it was deemed less challenging to control and replicate. This was achieved by counting to 13 seconds and using the extremities of the test-bed as distance markers.

Product treatment was obtained by applying 0.2g of BPC product across the entire surface of the test-beds, for a product distribution of 2.6 mg/cm² which is within the range of product quantities used in literature [75, 64, 102, 41]. Using product mass was preferred over volume as this method allowed for a more repeatable outcome. The product was continuously rubbed in for 60 seconds in a circular motion. At the end of the 60 seconds, a timer was set for 5 minutes. The friction was measured immediately after application and 5 minutes after application. Between repeats the test-beds were washed with water and face wash (Simple Kind to Skin Facewash, Unilever, London, UK), dabbed with a paper towel and left to dry for 10 minutes. The test-beds were then wiped with Isopropyl Alcohol and left to dry for another minute.

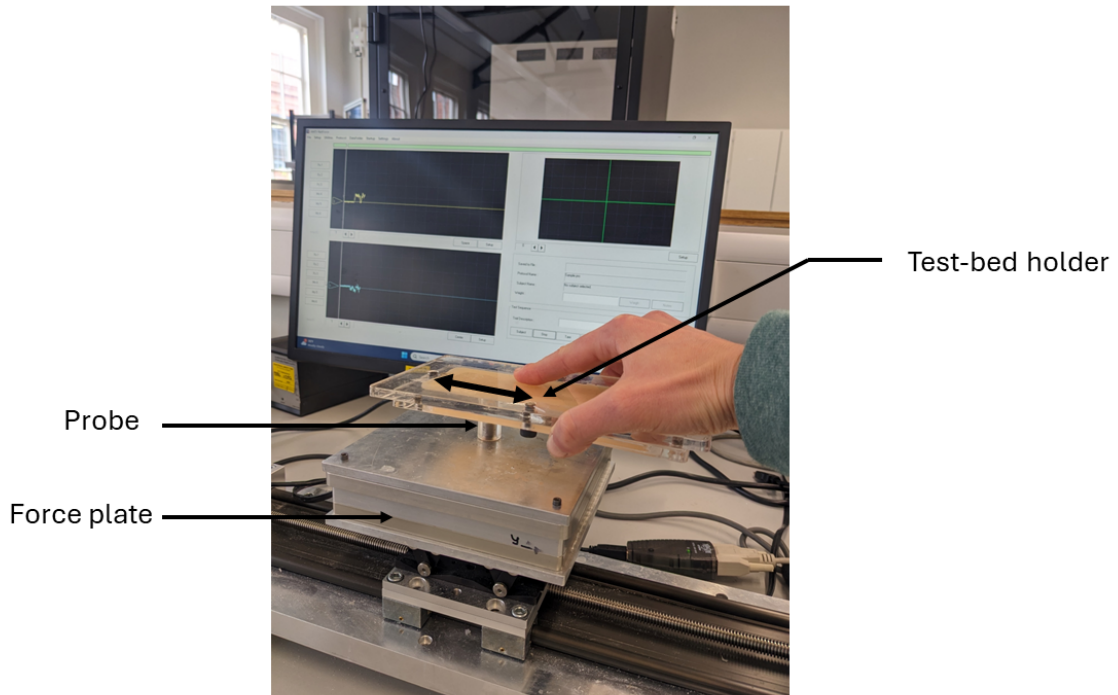


Figure 5.3: Set-up used for the friction measurements with the double arrow showing the sliding direction.

5.2.3.2 Synthetic skin test beds against a human finger-pad

The friction was then measured for the finger-pad of six human participants against the bi-layered test-bed with glycerin (TB4). Details of the participants recruited are provided in Table 5.3. Ethical approval was obtained from the University's Research Ethics committee prior to commencing the experiments (Application 046093, refer to the Appendix). The participants were asked to rub the index finger of their dominant hand in a linear reciprocating motion from side-to-side, as pictured in Figure 5.4. They were asked to maintain a normal load between 0.5 to 1N. The normal load was shown on a screen in front of them throughout the test. They were asked to maintain a constant speed whilst sliding over the length of the test-bed. Verbal guidance was provided by means of counting to 13 seconds after which they were asked to switch sliding direction. This rendered a speed of approximately 10 mm/s. Two test-beds (TB4) were manufactured, to allow for one to dry after it was cleaned whilst the other was tested. The test-beds were secured to the force plate using double sided tape and the participants were asked to rub the substrate laterally in a linear reciprocating motion, using the index finger of their dominant hand, as pictured in Figure 5.4. Before the tests began, the participants were given 10 minutes to acclimatize to the

lab's humidity and temperature levels. Their fingertip was then wiped with isopropyl alcohol and left to dry for another minute. Their finger was also wiped with isopropyl alcohol between the immediate and 5 minute measurements.



Figure 5.4: Human finger sliding against a synthetic skin test-bed for friction measurements with arrow showing the direction of sliding.

Table 5.3: Participant ethnicities, age sex and hand dominance for the finger-pad friction tests.

Participant number	Ethnicity	Age (years)	Sex	Dominant hand
1	English, Welsh, Scottish, Northern Irish or British (White)	21	Male	Right
5	English, Welsh, Scottish, Northern Irish or British (White)	23	Male	Right
6	Any other Asian background (Asian or Asian British)	26	Male	Right
8	Any other Asian background (Asian or Asian British)	23	Male	Right
9	Any other Asian background (Asian or Asian British)	24	Male	Right
10	Chinese (Asian or Asian British)	23	Male	Left

5.2.4 Assessing the capability of the test-bed in an industry setting

The force plate used at Unilever measures the friction force, normal force (including pull-off force) and speed. It's spatial resolution also allows for direct visualisation of where the force is being applied. The tests presented in this chapter were performed for one participant for which the details are given in Table 5.4. Ethical approval was obtained prior to the commencement of these tests.

Table 5.4: Participant ethnicity, age sex and hand dominance for the finger-pad friction and adhesion tests.

Ethnicity	Age (years)	Sex	Dominant hand
English, Welsh, Scottish, Northern Irish or British (White)	26	Female	Right

The methodology was programmed into the software linked to the force plate therefore giving prompts to the user for the different steps, which will be outlined in more detail below. The friction was measured so as to cover a range of loads starting from 0.1N and going up to 5N, and a range of speed going from 30 mm/s to 500 mm/s. The screen above the force-plate provided direct feedback of the sliding speed and normal load by plotting the data points on a graph, as pictured in Figure 5.5. The automated protocol guided the user to change speeds and loads so as to fill each box with the required amount of data points.

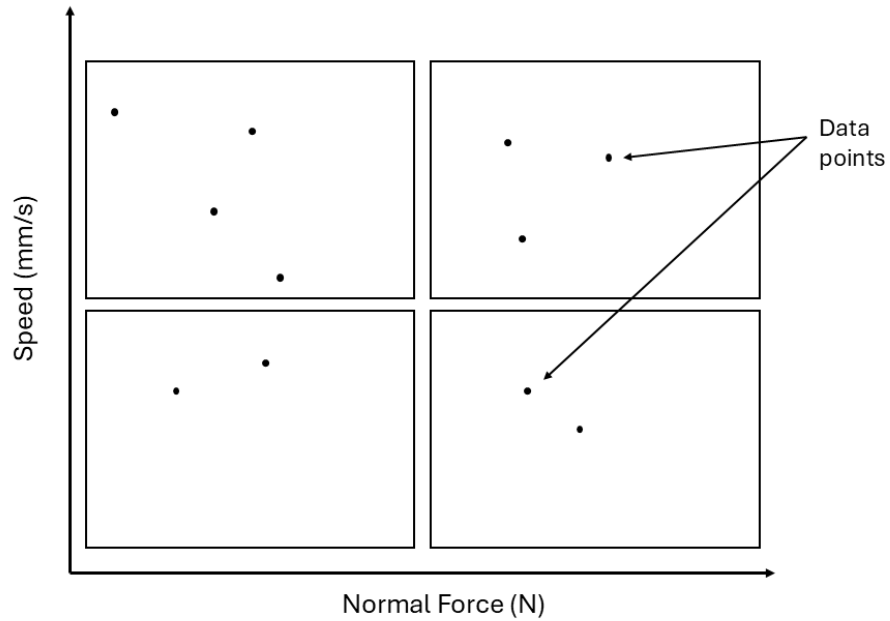


Figure 5.5: Diagram of screen shown to the user during the measurement DCOF and adhesion against test-beds.

The four synthetic test-beds manufactured for these tests are detailed in Table 5.5. Two synthetic test-beds were tested at the same time, and were both secured to the force plate using double sided tape as pictured in Figure 5.6. The protocol was divided into eight steps as follows:

1. 0.2 g of BPC product was spread across the entire surface of Test-bed 1 for 60 seconds.
2. The adhesion was measured immediately after application by pressing the left index finger-pad down onto the test-bed several times whilst applying a range of loads up to 1N at different places along the test-bed's surface. The pull-off force was recorded as adhesion.
3. The “friction immediately after application” was measured by sliding the finger-pad from side-to-side across the length of the test-bed as pictured in Figure 5.6.
4. The BPC product was then applied on Test-bed 2.

5. Steps 1 and 3 were repeated on test-bed 2 using the index finger of the right hand.
6. The “adhesion after 5 minutes” was measured on Test-bed 1 using the left index finger.
7. The “friction after 5 minutes” was measured on Test-bed 1 using the left index finger.
8. Steps 6 and 7 were repeated on test-bed 2 using the index finger of the right hand.

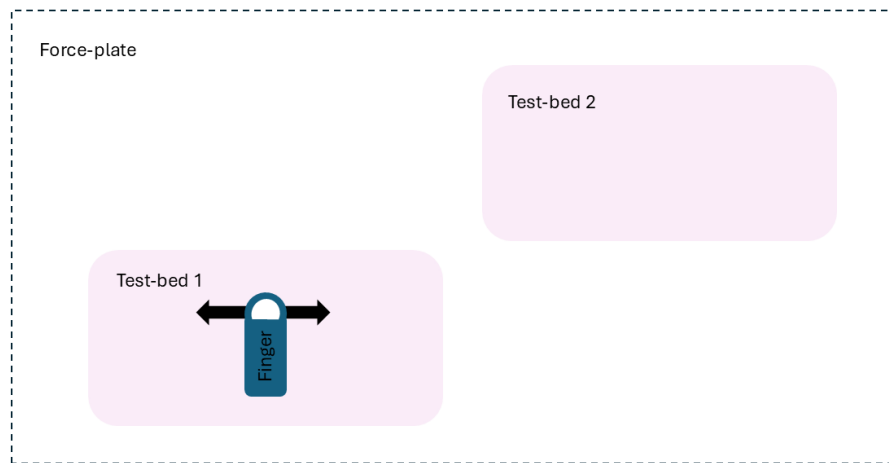


Figure 5.6: Diagram of testing procedure for measuring the DCOF and adhesion against test-beds.

Step 5 was programmed to last 5 minutes in order to obtain friction measurements immediately and 5 minutes after product application. The finger-pad was wiped using paper towels between each step. Between repeats the test-beds were washed with soap-free face wash (Simple Kind to Skin Facewash, Unilever, London, UK) the excess water was dabbed with a paper towel and the test-beds were left to air dry for 10 minutes. They were then wiped with rubbing alcohol (Boots Surgical Spirit, Boots UK limited, Nottingham, UK) and left to dry for another minute. The finger-pad was wiped with rubbing alcohol (Boots Surgical Spirit, Boots UK limited, Nottingham, UK) before each test.

Due to time constraints the tests were only repeated twice for the TB1 and TB5 test-beds. They were repeated four and three times for the TB2 and TB4 respectively. Priority was given to the latter two to achieve the maximum sizes of data set possible, for comparison with results from the tests at the University of Sheffield.

The Unilever experimental set-up includes an automated data analysis pipeline, which fits a trend line to all the data points within the range of speeds and loads described earlier. The data is then automatically interpolated to obtain the adhesion at 1N, and friction at 0.5N and speed of 50mm/s.

Table 5.5: Name and description of the test-beds tested at Unilever.

Test-bed name	Test-bed description	Composition
TB1	Textured silicone	Single 6mm layer of addition cure silicone (base + catalyst at 1:1 ratio).
TB2	Textured silicone with glycerin	Single 6mm layer of addition cure silicone (base + catalyst at 1:1 ratio) with added glycerin at 40wt% of total silicone weight (base + catalyst)
TB4	Textured bi-layered silicone with glycerin and deadener	Top layer (<1mm) : addition cure silicone (base + catalyst at 1:1 ratio) with added glycerin at 40wt% Bottom layer (5mm) : addition cure silicone with deadener at 40wt%
TB5	Smooth single layer silicone with glycerin	Single 6mm layer of addition cure silicone (base + catalyst at 1:1 ratio) with added glycerin at 40wt% of total silicone weight (base + catalyst)

5.3 Results

5.3.1 Mechanical properties

The results from the compressive tests are shown in Figure 5.7 below. The resulting data was obtained by averaging the three repeats for each test-bed. The base silicone(TB1) was the stiffest and most elastic test-bed. Adding glycerin into the silicone mixture (TB2) caused a slight increase in hysteresis and increased compliance.

The test-beds with added deadener were the most compliant and most visco-elastic (TB3 and TB4). There appears to be no differences over this range of loading for both bi-layered test-beds and no difference between the non-hydrated and hydrated bi-layered test-beds.

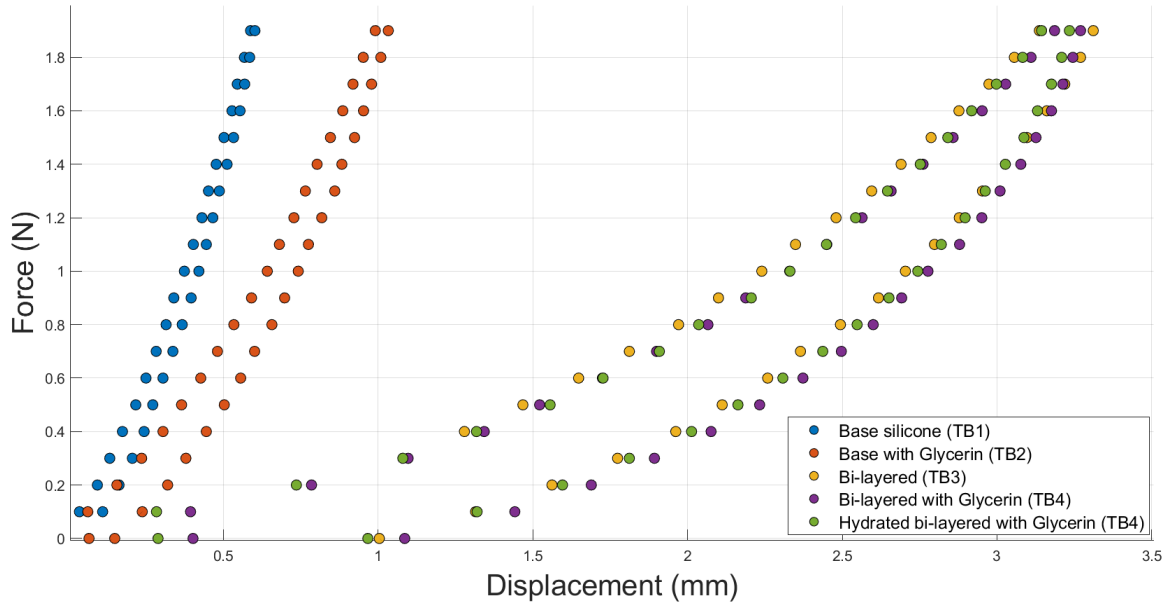


Figure 5.7: Hysteresis loops for the different test-beds obtained at a speed of 2mm/s with a spherical tipped probe of 16 mm in diameter.

All of the participant data is plotted in Figure 5.8 as well and the data for the base silicone and bi-layered test-bed for comparison. The human volar forearm force displacement data covers a large range for the different participants which is to be expected over this range of loads due to the differences in subcutaneous layer thickness and muscle.

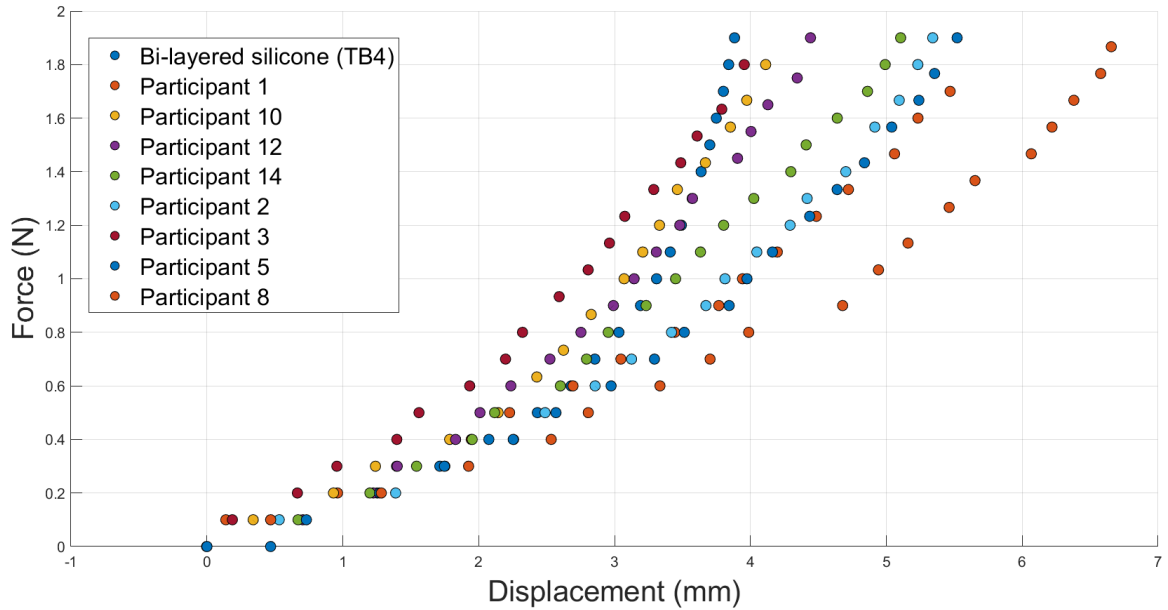


Figure 5.8: Individual force-displacement data obtained on human volar forearms (n=8) and on a bi-layered test-bed with 40 wt% deadener (TB4).

The volar forearm data was averaged across all participants and the resultant force-displacement curve is plotted in Figure 5.9 below along with three other test-beds for comparison. The graph shows that within the range of 0.5 to 1N, which is the range at which the friction measurements are performed, the bi-layered test-bed modified with deadener (TB4) and the average human volar forearm data follow the same trajectory. Differences becomes more evident above 1N, which could be linked to the thickness of the test-bed (6mm) meaning the stiffer material beneath starts to have an effect whereas for the volar forearm, the relatively thick layer of subcutaneous tissues beneath the skin means that the stiffness does not increase appreciably at loads above 1N.

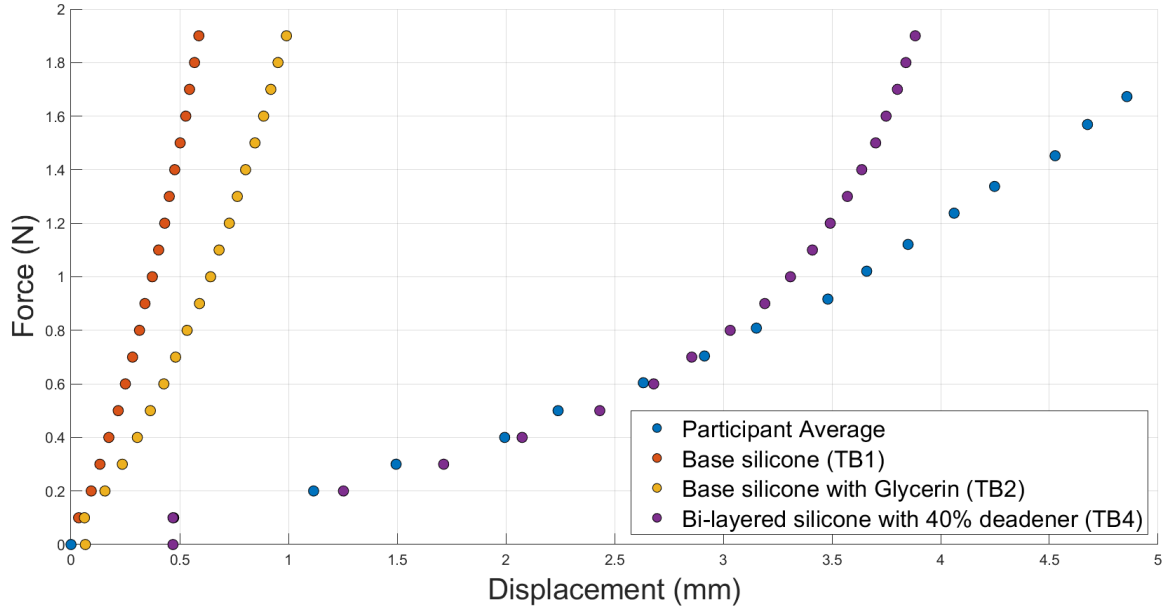


Figure 5.9: Force-displacement data for different test-beds and averaged human volar forearm data (n=8).

Based on the study led by Johnson et al. [51], and under the assumption that skin behaves in a Hertzian manner, the indentation depth can be calculated using equation 5.1 below with r the radius of the probe, W the load and ν poisson's ratio.

$$d = \frac{a^2}{r} = \left(\frac{9}{16r}\right)^{\frac{1}{3}} \left(\frac{1-\nu^2}{E}\right)^{\frac{2}{3}} W^{\frac{2}{3}} \quad (5.1)$$

The equation predicts that the load and displacement are related by a power law equation in the form of equation 5.2 below, where k is a constant.

$$W = kd^{1.5} \quad (5.2)$$

This was confirmed for the bi-layered test-bed and the human volar forearm experimental data between 0.2 and 1N as pictured in Figure 5.10 below. This was also observed the study led by Johnson et al. [51]. Discrepancies are to be expected due to experimental methods and differences between participants for the human data.

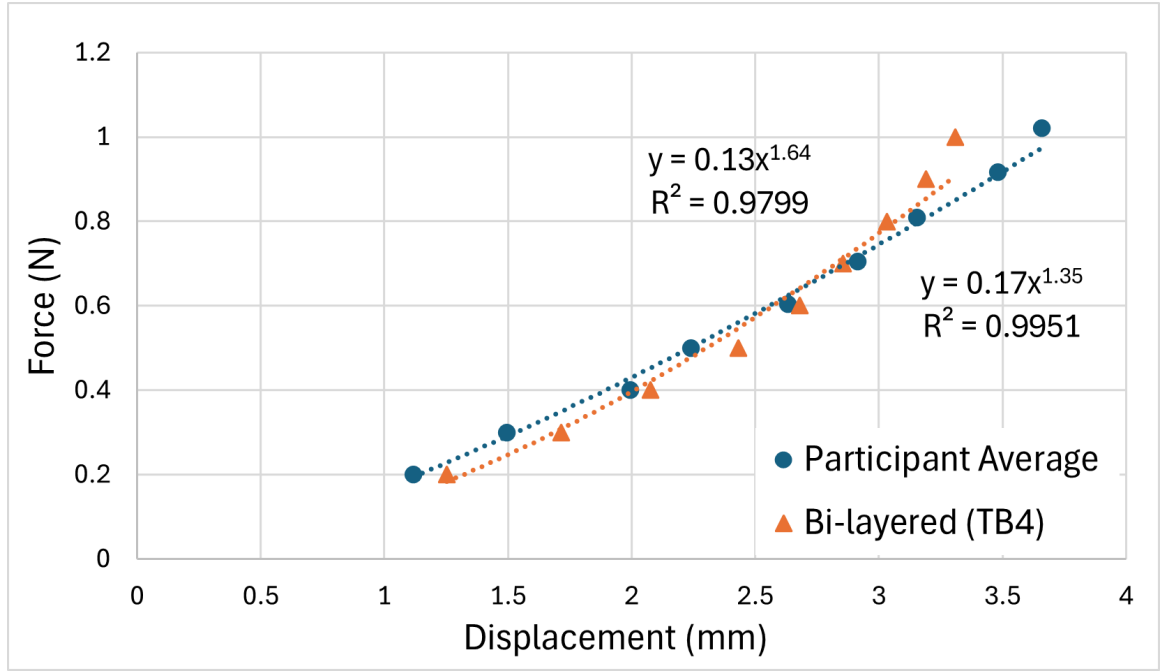


Figure 5.10: Fitted power law for the average force-displacement data of the bi-layered test-bed (TB4) (n=3) and human volar forearms (n=8).

From this equation and using the displacement values from the indentation tests at a load of 1N, the Elastic Modulus E can be calculated, using a value of 0.49 for Poisson's ratio and $r = 0.008\text{m}$ as the radius of the probe. The E modulus values found were 33 kPa and 28 kPa for the test-bed and human skin respectively. According to the study led by Pailler-mattei et al. [108], for an indentation depth below 2mm it is expected that the mechanical properties of the subcutaneous tissues will influence the bulk modulus.

5.3.2 Friction measurements

5.3.2.1 Synthetic skin test-beds against a solid probe

The method used to extract the dynamic COF (DCOF) from the raw data detailed in Chapter 3 was also used for the test-bed friction data. The DCOF was then averaged between each stroke for each repeat, and the average DCOF was averaged between each repeat to obtain one mean DCOF value for every test-bed and BPC product combination. The results were plotted in a bar chart, pictured in Figure 5.11, with each graph corresponding to a test-bed. These were plotted on the same scale for comparison. The mean DCOF obtained for each individual repeat is presented in Table 3 in the Appendix.

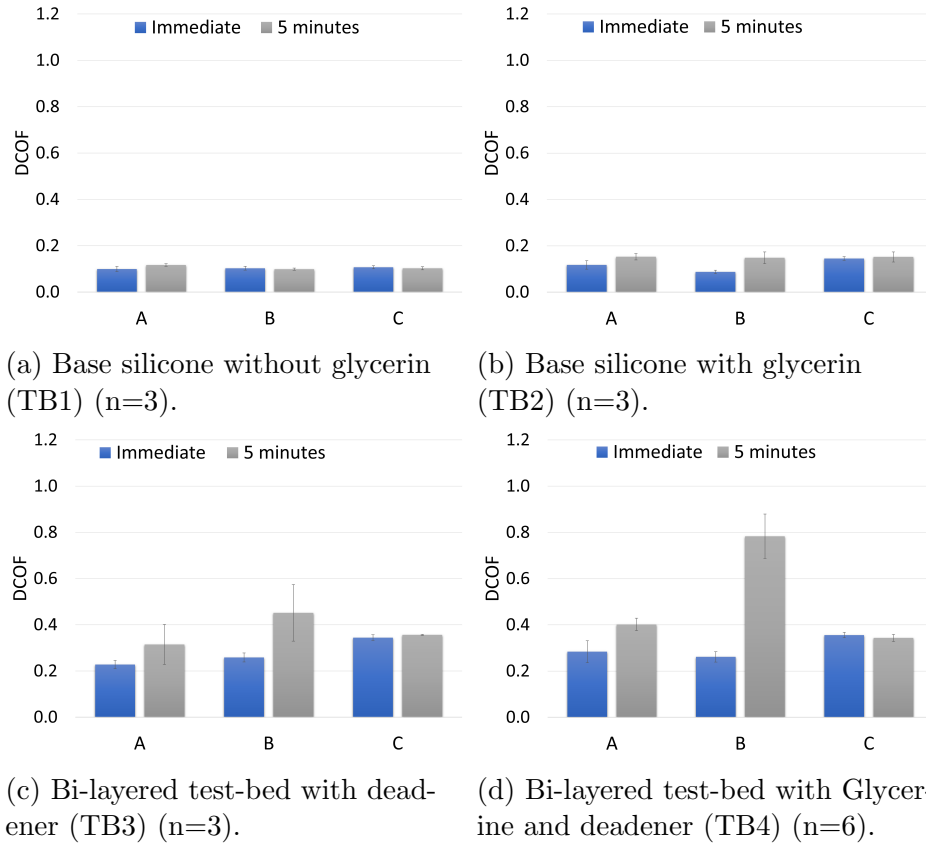


Figure 5.11: Synthetic test-bed mean DCOF immediately and 5 minutes with standard deviations following the application of three BPC products.

The lowest DCOF was obtained for the base silicone (TB1) (Table 5.6). Results from a paired t-test showed the only significant difference for this test-bed was found for the product A between the DCOF immediately and 5 minutes after application ($p < 0.05$). For the bi-layered test-bed with glycerin (TB4), the mean DCOF for A 5 minutes after application was significantly greater than the mean DCOF immediately after application ($p < 0.01$), and the mean DCOF for B 5 minutes after application was significantly greater than the mean DCOF immediately after application ($p < 0.01$). There were no significant differences for any of the products for the silicone with glycerine (TB2) and for the bi-layered test-bed without glycerin (TB3).

Table 5.6: Summary of the number of repeats, mean DCOF and standard deviations for each test-bed and product combination.

Test-bed	Product	Mean DCOF	N	Std. Deviation
Base Silicone (TB1)	A (Imm)	0.100	3	0.013
	A (5 min)	0.117	3	0.007
	B (Imm)	0.103	3	0.010
	B (5 min)	0.099	3	0.006
	C (Imm)	0.107	3	0.008
	C (5 min)	0.103	3	0.008
Silicone with glycerin (TB2)	A (Imm)	0.118	3	0.022
	A (5 min)	0.153	3	0.017
	B (Imm)	0.088	3	0.009
	B (5 min)	0.148	3	0.031
	C (Imm)	0.145	3	0.010
	C (5 min)	0.152	3	0.027
Bi-layered (TB3)	A (Imm)	0.228	3	0.021
	A (5 min)	0.315	3	0.106
	B (Imm)	0.259	3	0.024
	B (5 min)	0.451	3	0.150
	C (Imm)	0.344	3	0.015
	C (5 min)	0.357	3	0.003
Bi-layered with glycerin (TB4)	A (Imm)	0.304	6	0.077
	A (5 min)	0.461	6	0.093
	B (Imm)	0.282	6	0.063
	B (5 min)	0.952	6	0.354
	C (Imm)	0.405	6	0.086
	C (5 min)	0.389	6	0.071

Table 5.7: Results from the paired t-tests for comparing the differences in mean DCOF immediately and 5 minutes after product application for all test-beds and product combinations.

Test-bed	Product	t	df	Significance	
				One-Sided p	Two-Sided p
Base Silicone (TB1)	A (Imm) -	-4.152	2	0.027	0.053
	A (5 min)				
	B (Imm) -	0.429	2	0.355	0.710
	B (5 min)				
	C (Imm) -	0.534	2	0.323	0.647
	C (5 min)				
Base with glycerin (TB2)	A (Imm) -	-2.720	2	0.056	0.113
	A (5 min)				
	B (Imm) -	-2.881	2	0.051	0.102
	B (5 min)				
	C (Imm) -	-0.693	2	0.280	0.560
	C (5 min)				
Bi-layered (TB3)	A (Imm) -	-1.723	2	0.114	0.227
	A (5 min)				
	B (Imm) -	-1.928	2	0.097	0.194
	B (5 min)				
	C (Imm) -	-1.651	2	0.120	0.241
	C (5 min)				
Bi-layered with glycerin (TB4)	A (Imm) -	-5.356	5	0.002	0.003
	A (5 min)				
	B (Imm) -	-5.546	5	0.001	0.003
	B (5 min)				
	C (Imm) -	1.255	5	0.132	0.265
	C (5 min)				

Independent t-tests were also run to establish if the differences between test-beds were significant. The results outlined in Table 5.8 showed that the addition of glycerine to the base silicone caused a significant increase in the DCOF immediately after application between the glycerin test-bed (TB2) and the base silicone (TB1) ($p < 0.01$). The differences in means after 5 minutes were also all significantly increased ($p < 0.05$).

Table 5.8: Results from the independent t-test for the differences in DCOF between the base silicone (TB1) (n=3) and the base silicone with added glycerin (TB2) (n=3).

Product	Time	F	Sig.	t	df	Significance	
						One-Sided p	Two-Sided p
A	Imm	0.915	0.393	-1.196	4	0.149	0.298
	5 min	3.804	0.123	-3.470	4	0.013	0.026
B	Imm	0.034	0.863	1.907	4	0.065	0.129
	5 min	5.400	0.081	-2.710	4	0.027	0.054
C	Imm	0.045	0.842	-5.136	4	0.003	0.007
	5 min	3.881	0.120	-3.049	4	0.019	0.038

Table 5.9 shows no significant differences between the two bi-layered test-beds immediately after product application. However, the addition of glycerine in the bi-layered test-bed did cause a significant increase in DCOF after 5 minutes for the Product A ($p < 0.05$) and for the Product B ($p < 0.05$).

Table 5.9: Results from the independent t-test for the differences in DCOF between the bi-layered test-bed (TB3) (n=3) and the bi-layered test-bed with added glycerin (TB4) (n=6).

Product	Time	F	Sig.	t	df	Significance	
						One-Sided p	Two-Sided p
A	Imm	3.763	0.094	-1.623	7	0.074	0.149
	5 min	0.031	0.866	-2.135	7	0.035	0.070
B	Imm	1.401	0.275	-0.617	7	0.278	0.557
	5 min	3.145	0.119	-2.286	7	0.028	0.056
C	Imm	2.182	0.183	-1.171	7	0.140	0.280
	5 min	2.650	0.148	-0.772	7	0.233	0.465

Lastly, the DCOF values obtained for the bi-layered test-bed with glycerin and deadener (TB4) were the most similar to the values obtained on human volar forearms (detailed in Chapter 3), both in terms of trends and values. Figure 5.12 shows the values obtained on the human volar forearm on the left and values for the bi-layered test-bed TB4 on the right. The standard deviations were greater for the human values than for the test-bed, which highlights the natural variations associated with human testing. These differences could also be influenced by the larger number of repeats (12 for the volar forearm versus 6 for the test-bed).

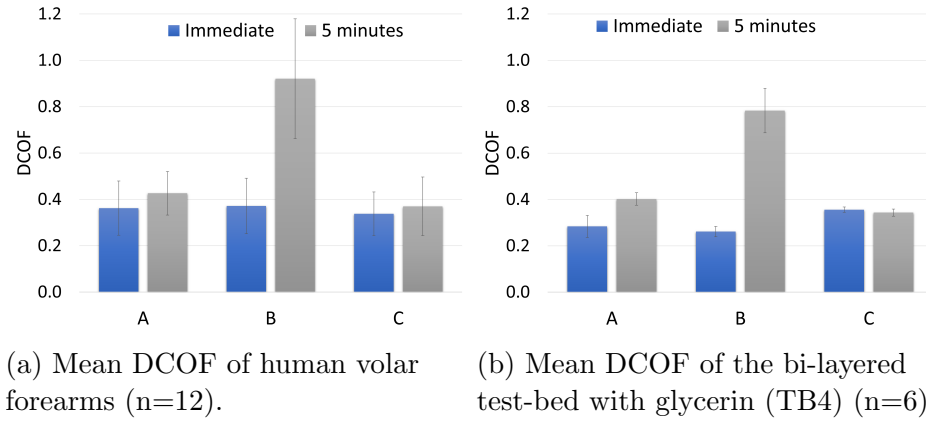


Figure 5.12: Comparison of the mean DCOF and standard deviations on human volar forearm skin and on a bi-layered test-bed (TB4) following the application of three BPC products.

An independent t-test was run to establish how well the DCOF obtained on the bi-layered test-bed TB4 matched that of the human volar forearm. The results, which are summarised in Table 5.10, revealed a significant difference between the test-bed and the forearm for B immediately after application ($p < 0.05$). While this difference could be associated with differences in skin physiology between subjects, as well as the number of repeats, it could also be explained by a lower absorption rate of water into the test-bed compared with the skin.

Table 5.10: Results from the independent t-test for the differences in DCOF between the bi-layered test-bed with added glycerin (TB4) (n=6) and the human volar forearm (n=12) with a solid probe.

Product	Time	F	Sig.	t	df	Significance	
						One-Sided p	Two-Sided p
A	Imm	1.10	0.310	-1.245	14.9	0.116	0.233
	5 min	0.266	0.613	0.711	16	0.244	0.487
B	Imm	15.2	0.001	-2.02	16.0	0.030	0.060
	5 min	1.09	0.311	0.208	16.0	0.419	0.838
C	Imm	0.321	0.579	1.41	16.0	0.088	0.175
	5 min	5.501	0.032	0.401	15.8	0.347	0.693

Lastly, the DCOF results for the SS test-bed TB4 were plotted against the DCOF obtained on the human volar forearm for the three products immediately and after 5 minutes, which is shown in Figure 5.13. The graph further highlights the strong linear relationship between the values.

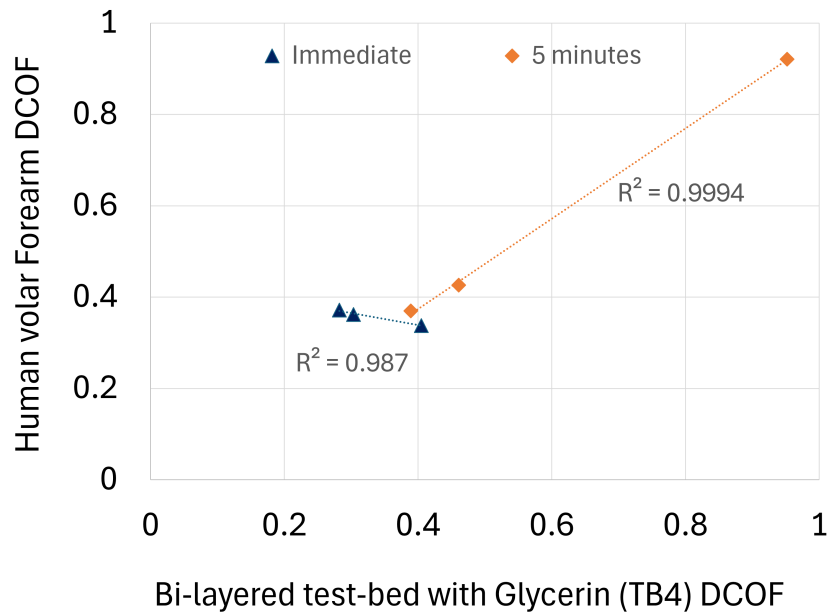


Figure 5.13: Mean DCOF for test-bed TB4 (n=6) versus mean DCOF for human volar forearms (n=6).

5.3.2.2 Synthetic skin test-beds against a human finger

The results from the friction measurements are shown in Figure 5.14. The mean DCOF obtained for each participant is presented in Table 2 in the Appendix. Upon initial observation the DCOF values measured against the human finger-pads appear to be higher for all products immediately after application, and for A and C after 5 minutes.

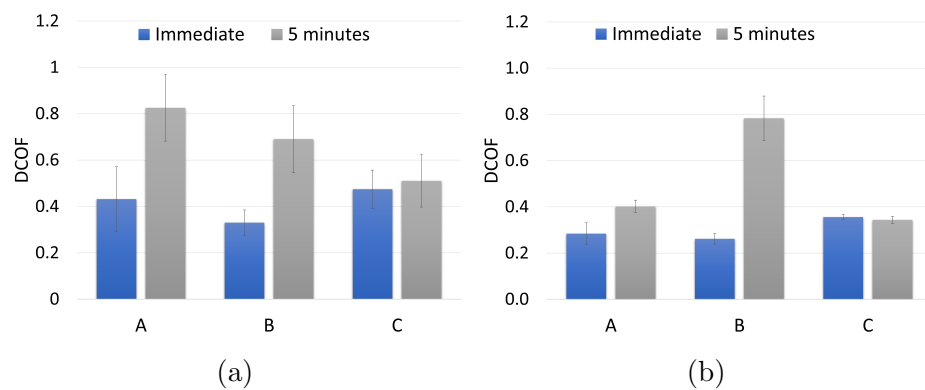


Figure 5.14: Mean DCOF measured for the bi-layered test-bed TB4 against human finger-pads(n=6) (a) and a solid probe (n=6)(b).

A paired t-test was run to assess the differences between the DCOF measured immediately and 5 minutes after product application against the human finger-pads. The values of mean DCOF, standard deviations and results from the t-test are outlined in Tables 5.11 and 5.12). The mean DCOF measured against the finger-pads was found to be significantly greater after 5 minutes for both A and B ($p < 0.01$). However, there were no significant differences for C.

Table 5.11: Mean DCOF, standard deviations and number of repeats for different BPC products measured against a probe and human finger-pads.

Product	Time	Probe	N	Mean DCOF	Std. Deviation
A	Imm	Finger	6	0.432	0.153
		Probe	6	0.304	0.077
	5 min	Finger	6	0.825	0.157
		Probe	6	0.461	0.093
B	Imm	Finger	6	0.330	0.060
		Probe	6	0.282	0.063
	5 min	Finger	6	0.690	0.158
		Probe	6	0.952	0.354
C	Imm	Finger	6	0.474	0.090
		Probe	6	0.405	0.086
	5 min	Finger	6	0.510	0.123
		Probe	6	0.389	0.071

Table 5.12: Results from the paired t-test for the differences in DCOF measured for human finger-pads (n=6) against the bi-layered test-bed TB4 immediately (Imm) and after 5 minutes for each BPC product.

Pair	t	df	Significance	
			One-Sided p	Two-Sided p
A (Imm) - A (5 min)	-35.0	5	<0.001	<0.001
B (Imm) - B (5 min)	-7.01	5	<0.001	0.001
C (Imm) - C (5 min)	-0.895	5	0.206	0.412

An independent t-test was also run, to evaluate the differences in DCOF values obtained against a probe and a human finger-pad. The results (Table 5.13) showed significant differences between the two datasets for A (immediately and after 5 minutes) and for C (after 5 minutes). However, it should be noted that overall the standard deviations were slightly higher for the finger-pad data, especially for B after 5 minutes (0.354 for finger-pad versus 0.158 for the solid probe), probably due to inter-personal variations between participants. It is also likely that differences in the

angle at which the finger was held incurred the largest discrepancies due to changes in the contact area.

Table 5.13: Results from an independent t-test to evaluate the differences between the DCOF measured for TB4 against a solid probe (n=6) and the DCOF measured against human finger-pads (n=6).

Product	Time	F	Sig.	t	df	Significance	
						One-Sided p	Two-Sided p
A	Imm	4.614	0.057	1.833	10	0.048	0.097
	5 min	5.221	0.045	4.895	8.097	<0.001	<0.001
B	Imm	0.023	0.883	1.344	10	0.104	0.209
	5 min	4.991	0.049	-1.653	6.926	0.071	0.143
C	Imm	0.018	0.897	1.364	10	0.101	0.202
	5 min	3.891	0.077	2.082	10	0.032	0.064

5.3.3 Friction and adhesion measurements in an industry setting

Figure 5.15 shows the measured DCOF of the bi-layered test-bed with glycerin against a solid probe (TB4) and against a human finger measured using the adapted Unilever method. The values are also summarised in Table 5.14. The mean DCOF obtained for each repeat is also presented in Table 4 in the appendix.

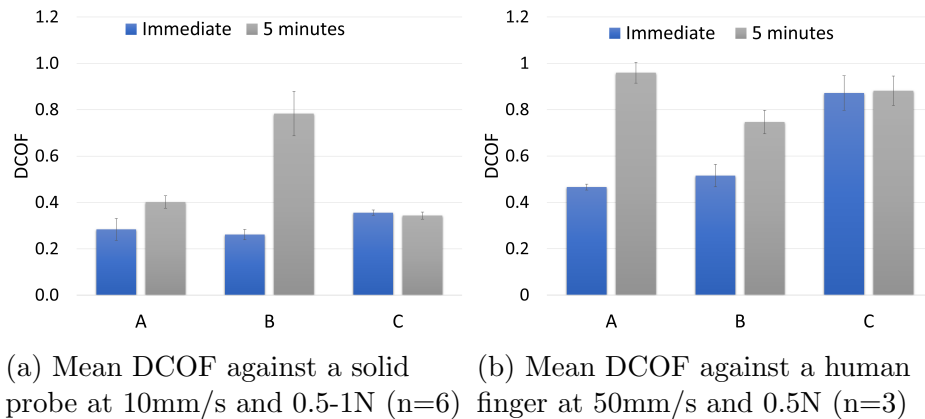


Figure 5.15: Mean DCOF and standard deviations for the TB4 against a solid probe and a finger-pad.

Results from an independent t-test indicated that the DCOF was significantly higher when measured against the finger-pad both immediately and after 5 minutes for all products ($p < 0.01$), except for B after 5 minutes where no significant differences were found (refer to Tables 5.14 and 5.15). The highest DCOF was measured for A

after 5 minutes and the second highest was measured for C both immediately and after 5 minutes. The DCOF for C against the human finger showed no significant difference immediately and after 5 minutes, which was also found for the DCOF measured against a solid probe. For the products A and B, the mean DCOF immediately and after 5 minutes was significantly different ($p < 0.01$).

Table 5.14: Summary of the mean DCOF, number of repeats and standard deviations for a finger-pad and a probe against the bi-layered test-bed TB4.

Time	Product	Test Bed	N	Mean	Std. Deviation
Imm	A	TB4 (Finger)	3	0.466	0.015
		TB4 (Probe)	6	0.304	0.077
	B	TB4 (Finger)	3	0.516	0.060
		TB4 (Probe)	6	0.282	0.061
	C	TB4 (Finger)	3	0.872	0.091
		BLG (Probe)	6	0.405	0.086
5 min	A	TB4 (Finger)	3	0.960	0.055
		TB4 (Probe)	6	0.461	0.093
	B	TB4 (Finger)	3	0.747	0.062
		TB4 (Probe)	6	0.952	0.354
	C	TB4 (Finger)	3	0.882	0.078
		TB4 (Probe)	6	0.389	0.071

It is important to highlight that both the speed at which these tests were performed and the amount of times the finger was slid across the surface were different in the two cases. For the Unilever method, the aim is to cover a range of speeds and loads in order to fit the data and improve the accuracy of the mean DCOF at a certain point which implies a greater amount of sliding.

Table 5.15: Independent t-test results for the mean DCOF measured on the TB4 test-bed against a solid probe and a human finger immediately (imm) and 5 minutes (5 min) after product application (n=3).

Time	Product	F	Sig.	t	df	Significance	
						One-Sided p	Two-Sided p
Imm	A	4.633	0.068	3.515	7	0.005	0.010
	B	0.014	0.910	5.355	7	<0.001	0.001
	C	0.004	0.951	7.538	7	<0.001	<0.001
5 min	A	0.498	0.503	8.437	7	<0.001	<0.001
	B	6.459	0.039	-1.377	5.585	0.111	0.221
	C	0.055	0.821	9.556	7	<0.001	<0.001

Figure 5.16 allows for a visual comparison between the mean DCOF measured for different participants at a speed of 10 mm/s (as outlined in section 5.2.3) and the DCOF measured at a speed of 50 mm/s in the Unilever laboratory. Upon initial inspection both graphs look to follow similar trends although the values appear higher for the speed of 50 mm/s.

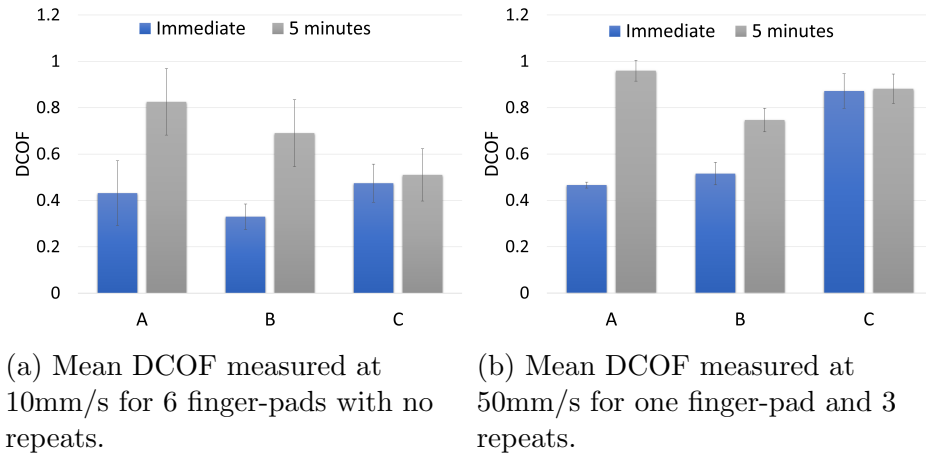


Figure 5.16: Mean DCOF and standard deviations for human finger-pads at different speeds against the bi-layered test-bed TB4.

An independent t-test revealed that the only significant difference between the two speeds was found for B immediately after application and C both immediately and after 5 minutes ($p < 0.05$). However, it should be noted that in one instance the tests were replicated by 6 participants but not repeated for the same participant whereas in the other the tests were repeated by the same person but only 3 times which lead to discrepancies in standard deviations.

Table 5.16: Independent t-test results for the mean DCOF measured for a human finger-pad at 10 mm/s ($n=6$) and at 50 mm/s ($n=3$) against the bi-layered test-bed (TB4).

Time	Product	t	df	Significance	
				One-Sided p	Two-Sided p
Imm	A	0.544	5.2	0.304	0.609
	B	4.39	7	0.002	0.003
	C	6.22	7	<0.001	<0.001
5 min	A	1.88	6.74	0.052	0.104
	B	0.580	7	0.290	0.580
	C	4.68	7	0.001	0.002

Lastly, Figure 5.17 allows for the visualisation of the differences in mean DCOF for the four test-beds tested at Unilever. The first observation is the similarity between the DCOF obtained for the Textured base silicone with glycerin test-bed (TB2) and the DCOF obtained for the textured bi-layered with glycerin test-bed (TB4). The differences between these two test-beds were highlighted in section 5.3.2, with significantly lower DCOF values for the stiffer test-bed (TB2). This was attributed to the differences in mechanical properties. Another observation are the seemingly lower DCOF values immediately after application for the smooth test-bed compared to the textured test-beds, for A and B. This is also different to results from previous chapters which associated texture with lower DCOF values.

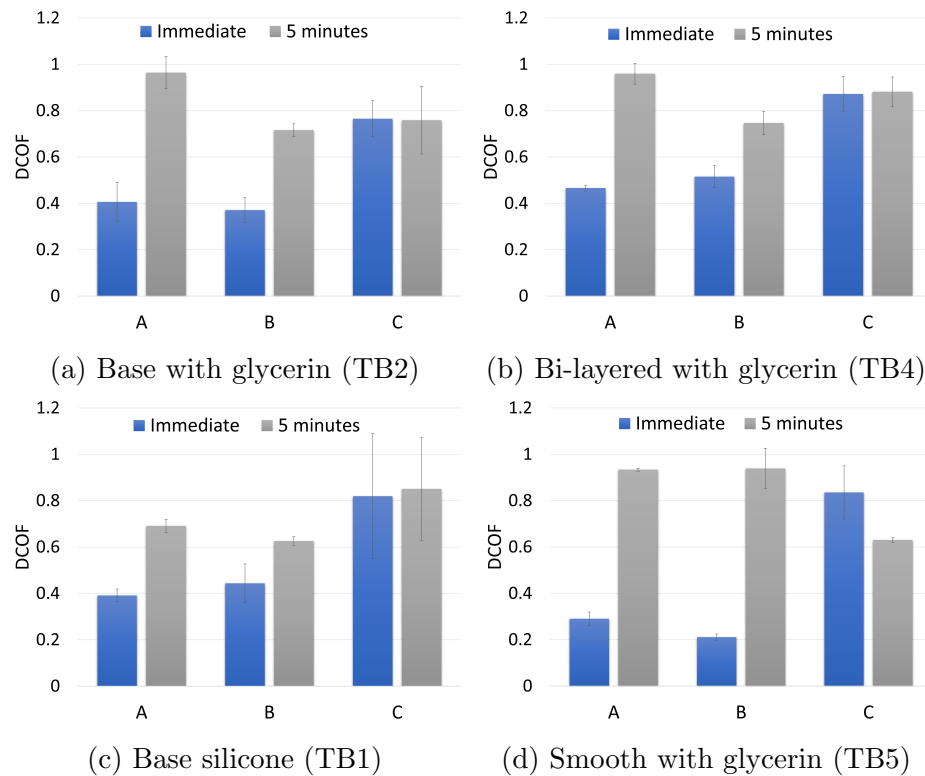


Figure 5.17: Mean DCOF and with standard deviations for a human finger against different test-beds.

Tables 5.17, 5.18 and 5.19 below outline the results of the independent t-tests run for the mean DCOF of different test-beds. For the Base silicone (TB1) and Base silicone with glycerin (TB2) test-beds the mean DCOF was significantly different for B immediately and after 5 minutes and for A after 5 minutes ($p < 0.05$). Looking at the effect of texture, the only significant difference between the textured base silicone with Glycerine (TB2) and smooth base silicone with glycerin (TB5) was found for

B immediately after application ($p < 0.05$). Lastly, the only significant differences found between the silicone with glycerin (TB2) and the bi-layered (TB4) test-beds were found for B immediately and after 5 minutes ($p < 0.05$).

Table 5.17: Independent t-test results for the base silicone (TB1) (n=2) and the base silicone with glycerin (TB2) (n=4) .

Product	Time	F	Sig.	t	df	Significance	
						One-Sided p	Two-Sided p
A	Imm	11.009	0.029	0.255	3.998	0.406	0.811
	5 min	0.908	0.395	4.361	4	0.006	0.012
B	Imm	1.778	0.253	-7.067	4	0.001	0.002
	5 min	1.028	0.368	4.114	4	0.007	0.015
C	Imm	26.635	0.007	-0.198	1.055	0.437	0.874
	5 min	1.973	0.233	-0.496	4	0.323	0.646

Table 5.18: Independent t-test results for the bi-layered test-bed with glycerin (TB4) (n=3) and the textured base silicone test-bed with glycerin (TB2) (n=4)

Product	Time	F	Sig.	t	df	Significance	
						One-Sided p	Two-Sided p
A	Imm	28.062	0.003	-1.200	3.196	0.156	0.311
	5 min	0.437	0.538	0.082	5	0.469	0.938
B	Imm	0.158	0.707	-3.065	5	0.014	0.028
	5 min	0.289	0.614	3.870	5	0.006	0.012
C	Imm	0.001	0.978	-1.548	5	0.091	0.182
	5 min	1.074	0.348	-1.153	5	0.151	0.301

Table 5.19: Independent t-test results for the smooth (TB5) (n=2) and textured base silicone with glycerin (TB2)(n=4) test-beds.

Product	Time	F	Sig.	t	df	Significance	
						One-Sided p	Two-Sided p
A	Imm	10.497	0.032	2.022	3.999	0.057	0.113
	5 min	2.742	0.173	0.504	4	0.321	0.641
B	Imm	4.068	0.114	3.327	4	0.015	0.029
	5 min	0.798	0.422	0.310	4	0.386	0.772
C	Imm	1.714	0.261	-0.722	4	0.255	0.510
	5 min	2.167	0.215	1.015	4	0.184	0.368

The adhesion data was automatically processed and an average value for a normal force of 1N was given for each test-bed immediately and 5 minutes after application.

The values were averaged over the repeats. The results are plotted in Figure 5.18 for each test-bed and for the three products.

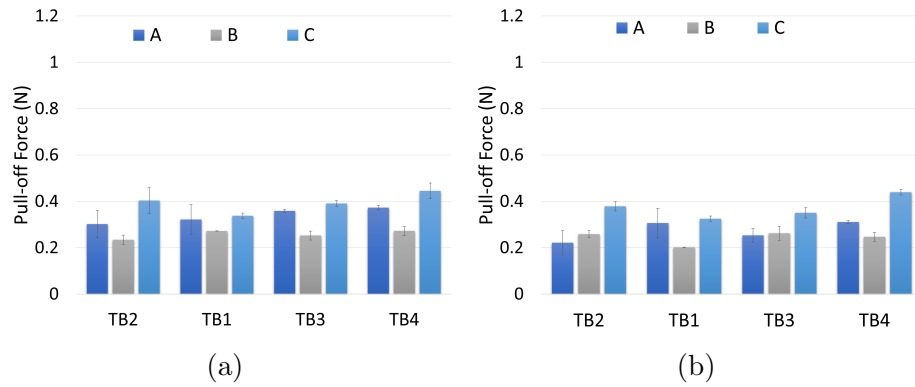


Figure 5.18: Mean adhesive force and standard deviation for the different synthetic skin test-beds immediately (a) and 5 minutes (b) following product application.

It is apparent from the graph that the most adhesive product was C for all test-beds both immediately and after 5 minutes. Furthermore, the differences in means reported in table 5.20 below, show a reduction in adhesion for all test-beds and products after 5 minutes, with the exception of B on the smooth and textured glycerin test-bed. However, the paired t-test revealed no significant differences between the values expected for A for both the smooth base with glycerin (TB5) and bi-layered (TB4) test-beds ($p < 0.05$).

Table 5.20: Results from a paired t-test for the differences in the adhesion measured on the different test-beds immediately and 5 minutes after product application.

Test-Bed	Time	Difference in Means	t	df	Significance	
					One-Sided p	Two-Sided p
Base with glycerin (TB2)	A (Imm) - A (5min)	0.08	2.259	2	0.076	0.152
	B (Imm) - B (5min)	-0.024	-2.407	2	0.069	0.138
	C (Imm) - C (5min)	0.025	0.487	2	0.337	0.674
Base silicone (TB1)	A (Imm) - A (5min)	0.016	0.462	1	0.362	0.725
	B (Imm) - B (5min)	0.07	2.264	1	0.132	0.265
	C (Imm) - C (5min)	0.012	0.381	1	0.384	0.768
Smooth base with glycerin (TB5)	A (Imm) - A (5min)	0.105	6.530	1	0.048	0.097
	B (Imm) - B (5min)	-0.01	-1.679	1	0.171	0.342
	C (Imm) - C (5min)	0.041	0.860	1	0.274	0.548
Bi-layered with glycerin (TB4)	A (Imm) - A (5min)	0.062	2.903	3	0.031	0.062
	B (Imm) - B (5min)	0.026	1.787	3	0.086	0.172
	C (Imm) - C (5min)	0.006	0.189	3	0.431	0.862

5.3.4 Correlations with panel data

The software SPSS was used to run a simple linear regression analysis to check if the average DCOF measured for the SS test-beds could be used to predict the scores for any of the attributes. The analysis was first run for each attribute without differentiating between “Immediate” and “5 minutes”, however no correlations were found. The dataset was then further divided between the DCOF obtained immediately and 5 minutes after application.

For the DCOF obtained for the human finger-pads against the the bi-layered test-bed with glycerin (TB4), the linear regression analysis showed a significant positive

correlation with the attribute scores for *Greasy* immediately after application ($R^2 = 0.995, p < 0.05$), with the values reported in Table 5.21. The two variables are plotted in Figure 5.19b. The results also indicated that the DCOF obtained for a solid probe against TB4 showed a significant positive correlation with the scores for the attribute *Sticky Tacky* immediately after application ($R^2 = 0.997, p < 0.05$) with values reported in Table 5.22. The two variables are plotted in Figure 5.19a.

However, as mentioned in Chapter 3, the accuracy of these correlations remains limited as the range covered by the DCOF and the number of data point are relatively small.

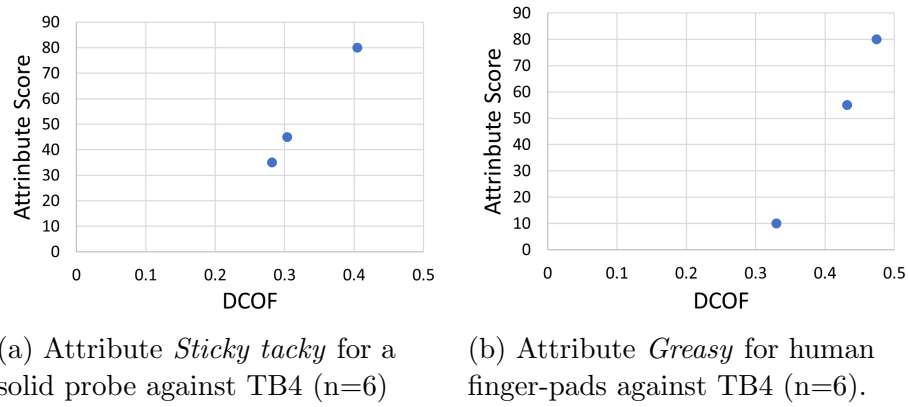


Figure 5.19: DCOF immediately after application versus attribute scores.

Table 5.21: Results from the simple linear regression analysis with predictors (constant) and the mean DCOF of a finger-pad against TB4 (n=6), and the attribute score as dependent variable.

Attribute	Time	Sum of Squares	df	Mean Square	F	Sig.
Greasy	Immediate	Regression	2505.1	1	2505.1	217.5 0.043
		Residual	11.5	1	11.5	
		Total	2516.7	2		
	5 minutes	Regression	600.2	1	600.2	0.396 0.643
		Residual	1516.5	1	1516.5	
		Total	2116.7	2		

Table 5.22: Results from the simple linear regression analysis with predictors (constant) and the mean DCOF of a probe against TB4 (n=6), and the attribute score as dependent variable.

Attribute	Time	Sum of Squares		df	Mean Square	F	Sig.
Sticky-tacky	Immediate	Regression	1116.4	1	1116.4	4115.2	0.01
		Residual	0.271	1	0.271		
		Total	1116.7	2			
	5 minutes	Regression	724.7	1	724.7	2.23	0.376
		Residual	325.3	1	325.3		
		Total	1050.0	2			

In terms of adhesion the most significant correlations were found for the TB4 test-bed, details of which are given in tables 5.23 below. The adhesion measurements immediately and after 5 minutes were found to be predictors for the attribute scores of *Drag*, *Greasy* and *Sticky Tacky* ($p < 0.01$). The direction of the slopes in Figure 5.20 reveal a positive correlation for both *Sticky Tacky* and *Greasy* and a negative correlation with *Drag*. For the attribute *Sticky Tacky* this is logical, as it is defined as “the degree to which a finger adheres to residual products and skin pulls up from the forearm” hence a higher adhesion would be expected to lead the product to be perceived as more *sticky tacky*. *Greasy* refers to the feeling of a “thick, slippery, fatty film remaining on the skin” and hence a higher adhesion with the product would lead to more product remaining on the finger-pad. However, *Drag* refers to “the difficulty of moving fingers across the skin” hence in this case a positive correlation would be expected as an increased adhesion would prevent the fingers from moving well.

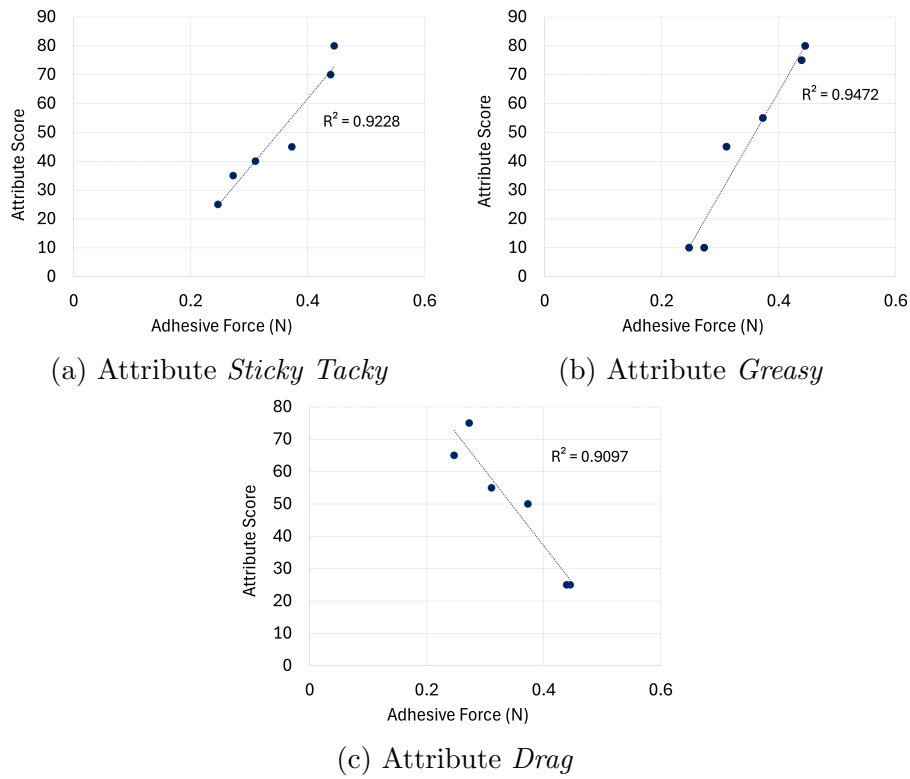


Figure 5.20: Finger-pad adhesion versus attribute scores.

Table 5.23: Results from the simple linear regression analysis with predictors (constant) and the mean adhesive force for test-bed TB4 ($n=6$), and the attribute score as dependent variable.

Attribute		Sum of Squares	df	Mean Square	F	Sig.
Drag	Regression	1929.3	1	1929.3	40.3	0.003
	Residual	191.6	4	48.0		
	Total	2120.833	5			
Greasy	Regression	4424.0	1	4424	71.7	<0.001
	Residual	246.849	4	61.712		
	Total	4671	5			
Sticky Tacky	Regression	2095.6	1	2095.597	47.8	0.002
	Residual	175.2	4	43.8		
	Total	2270.8	5			

For further analysis the results from the adhesion measurements were plotted against viscosity measurements from the experiments detailed in Chapter 3, and linear regression analyses were performed. The results revealed a significant positive linear relationship between the Viscosity values obtained at a shear rate of 1 s^{-1} in the shear sweep tests, and the adhesive force after 5 minutes ($p < 0.05$) as highlighted

in Figure 5.21a. The linear regression analysis also revealed a significant negative correlation between the viscosity at a shear rate of 1 s^{-1} and the attribute scores for *Drag* ($p < 0.05$), results of which are plotted in Figure 5.21b. Results from the linear regression analyses are detailed in Tables 5.24 and 5.25 respectively.

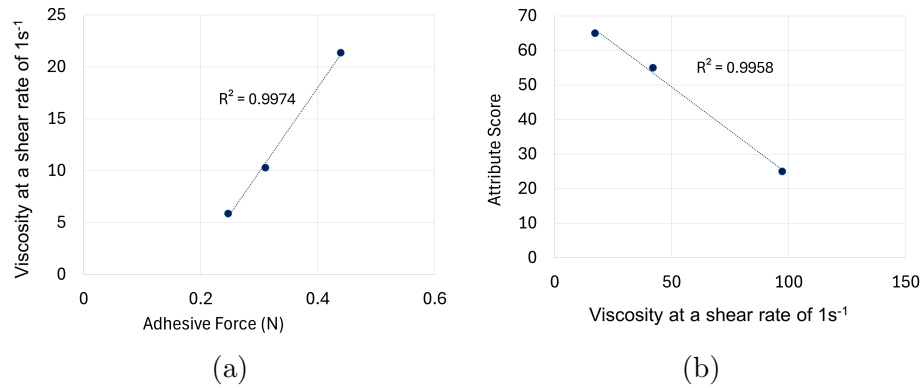


Figure 5.21: Viscosity at a shear rate of 1 s^{-1} related to adhesive force after 5 minutes (a) and attribute scores (b).

Table 5.24: Results from the simple linear regression analysis with predictors (constant) and the viscosity at a shear rate of 1 s^{-1} ($n=3$) and the adhesive force as dependent variable.

Time	Sum of Squares		df	Mean Square	F	Sig.
Immediate	Regression	0.014	1	0.014	9.787	0.197
	Residual	0.001	1	0.001		
	Total	0.015	2			
5 minutes	Regression	0.019	1	0.019	1573.321	0.016
	Residual	0.000	1	0.000		
	Total	0.019	2			

Table 5.25: Results from the simple linear regression analysis with predictors (constant) and the viscosity at a shear rate of 1 s^{-1} ($n=3$) and the attribute score for *Drag* as dependent variable.

Attribute	Time	Sum of Squares		df	Mean Square	F	Sig.
Drag	Immediate	Regression	1192.3	1	1192.3	20.7	0.138
		Residual	57.7	1	57.7		
		Total	1250	2			
	5 minutes	Regression	863.1	1	863.1	239.7	0.041
		Residual	3.6	1	3.6		
		Total	866.7	2			

5.4 Discussion

Synthetic skin test-bed against a solid probe

The results revealed that replicating the bulk mechanical properties and surface topography of volar forearm skin, combined with a capacity to absorb water, successfully replicated the frictional behaviour of human skin for the three BPC products. The addition of glycerin into the silicone mixture caused a significant increase in DCOF 5 minutes after product application of all products for both the bi-layered test-bed TB4 and the base test-bed DCOF compared with the bi-layered and base test-beds without glycerin, respectively. The DCOF also increased significantly for the base with glycerin (TB2) compared to the base without (TB1) immediately and 5 minutes after the application of C. These differences were not observed between the bi-layered test-beds. Hence, it appears that the frictional behaviour of the stiffer test-beds is primarily influenced by the characteristics of the product whereas for the more compliant test-beds, the behaviour is dominated by the deformation of the test-beds.

Bowden and Tabor [50] described the total frictional force as a two component equation consisting of a deformation term (F_{DEF}) and an adhesion term (F_{ADH}), as stated in equation 5.3. This two term model of friction is often used when looking at the friction of human skin [52].

$$F_{TOT} = F_{ADH} + F_{DEF} \quad (5.3)$$

The adhesion term is affected by the molecular bonds between the asperity junctions of the two interacting surfaces, whilst the deformation term arises from the force required to deform the bulk of the more compliant material [51]. According to the review written by Derler et al. [13], in studies looking specifically at skin tribology, the adhesion component has mostly been considered to be the main mechanism of friction.

However, in a well lubricated contact it is expected that the interfacial adhesion between the two interacting surfaces will be reduced and hence the deformation losses will have a greater impact on friction, as highlighted by the Greenwood et al. study [109]. The authors derived an equation for the deformation component of friction, which is given in equation 5.4 below.

$$F_{DEF} = \beta \left(\frac{9}{128r} \right)^{\frac{2}{3}} \left(\frac{1 - \nu^2}{E} \right)^{\frac{1}{3}} W^{\frac{4}{3}} \quad (5.4)$$

The significant increase in DCOF for the test-beds with added glycerin could be highlighting the hydrating effect of the BPC products. The values of DCOF obtained for the bi-layered model without glycerin (TB3) were lower than the ones obtained on human volar forearms which indicates that this rapid hydrating effect could also be occurring on human skin. The increase in hydration of human skin 5 minutes after the application of an emollient has been previously reported in a study by Loden [47]. The author found a positive correlation between the increasing hydration level and the concentration of water in the products. In literature, it has generally been accepted that the increase in DCOF of hydrated skin is associated with the plasticisation of the superficial layer of skin which in turn increases the real contact area [14]. However, the force-displacement data obtained for the two bi-layered test-beds as well as the “hydrated” test-bed showed no obvious changes which implies that a change in the mechanical properties of the superficial layer of the test-beds did not have an effect over this range of loading (0.5-1N). Therefore, it can be hypothesized that the increase in DCOF is the result of an increase in adhesive bonding of the two interacting materials and that the changes in friction cannot solely be attributed to an increase in deformation of the test-bed or skin.

Consequently, the viscoelastic hysteresis loss fraction β for the bi-layered test-bed TB4 was obtained by calculating the area of the hysteresis loop from the data presented in Figure 5.7, which yielded a value of 0.984. Using the E modulus value previously calculated and the equation derived from the Greenwood et al. study [109], the deformation component of the total friction was found to be $F_{DEF} = 0.119$ N. As the friction was measured at a normal load between 0.5 and 1 N, a value of 0.75 N was chosen to estimate the coefficient of friction. This yielded a value of $\mu(0.75) = 0.159$. Referring to the values of mean DCOF reported in Table 5.6, this finding confirms that some adhesion is occurring, and the contact is already no longer fully lubricated for the measurements performed immediately after the 60 seconds application process.

Synthetic skin test-bed against human finger-pads

The DCOF measured for human finger-pads against the bi-layered test-bed (TB4) highlighted some significant differences compared to the mean DCOF measured using

the PMMA probe, with the most significant difference found for A after 5 minutes. There are a number of mechanisms which could explain these disparities.

Firstly, as the finger-pad initially deforms under the normal load, the contact area will likely be increased compared to the solid probe. This increase in contact area as well as added deformation could explain the overall increase in DCOF observed in the results. The lack of deformation of the solid probe means the contact pressure is greater for the same normal load which entails it is more likely to penetrate the layer of BPC product. In the case of C, which has the lowest evaporation rate due to its purely occlusive properties, this difference means that the solid probe is more likely to cut through the product, leaving the interface between the two surfaces relatively product-free. Meanwhile, with a reduced contact pressure, the fingertip is more likely to “sit” on top of the product. Additionally, results from the rheological assessment of the three products outlined in chapter 3 showed that C had the highest yield stress which means it requires the most force to induce its flow. This could explain the higher DCOF values found for the finger-pad versus the solid probe.

The most notable difference however, was found for A after 5 minutes, which yielded the highest DCOF value out of the three products. In both the test-bed and human data measured using a solid probe, the largest DCOF was found for B. This was attributed to its more humectant characteristics which implied that the larger increase in DCOF after 5 minutes in comparison to the two other products was most likely due to an increase in the hydration of the test-bed and human skin. While A does contain water and a humectant at relatively high concentrations, it also contains an occlusive at the second highest concentration which would be expected to remain on the skin longer thus maintaining a lubricating effect after 5 minutes. Furthermore, the yield stress measured for A was less than half that of C, implying that the increase in friction cannot solely be attributed to product accumulating in front of the finger-pad, as this mechanism would have yielded higher values of DCOF for C.

Additionally, the finger-pad ridges may have impacted the DCOF. These have been hypothesised to increase the DCOF through the increase in contact area with the asperities of the interacting surface [107]. In this case, it is likely that the compliance of the bi-layered test-bed (TB4) would mean the texture of the test-bed is stretched and acts as a smooth surface during the rubbing interaction [14]. Hence, the ridges would not be interlocking with the test-bed surface. However, they may be increasing

the contact area with the BPC product still present on the test-bed. The overall increase in DCOF immediately after application could therefore be attributed to an increase in adhesion with the BPC products. Furthermore, the troughs formed by the finger-pad ridges could be causing a suction effect, therefore removing more product from the contact and further reducing its lubricating effect.

Lastly, the heat from the human finger-pad could be contributing to an increase in the evaporation rate of the BPC product, therefore also leading to an increase in DCOF.

Applicability of the synthetic skin test-bed in an industry setting

The force plate used in the Unilever laboratory enabled the measurement of adhesion, as well as the measurement of friction at higher speeds against a human finger-pad. Three of the test-beds previously evaluated were tested using this method, as well as a smooth test-bed for further comparison. The purpose of these tests was to assess the applicability of the developed method in an industry setting, as well as move towards a finalised method which will include a synthetic skin test-bed with a synthetic finger-pad probe. Although the use of a human finger to measure friction was assessed using the original method, the tests conducted in industry allowed for a more in depth analysis and improved understanding of the effect of various factors on friction for the three BPC products.

The first finding was that the friction measured on the bi-layered test-bed with glycerin (TB4) was significantly higher for all products and measurement time when measured against a human finger-pad compared to a solid probe, except for B after 5 minutes. However, in this case the differences cannot be solely attributed to the finger-pad as there were several discrepancies between the two methodologies. Notably the increased speed and the increase in number of strokes. Effectively, these two factors may have increased the amount of product removed from the test-bed therefore increasing the DCOF. This could be caused by accumulation of the product on the finger as well as by the generation of heat which in turn may have triggered a more rapid evaporation of ingredients from the products. The resulting diminished lubricating properties would explain the higher friction immediately after application observed in Figure 5.15 compared to the friction measured with a probe. Additionally, the increase in DCOF with increasing number of strokes has been previously

hypothesized as a result of increased sweat secretion due to a natural mechanism in the finger-pad for optimal grip [96].

However, although the friction appeared overall higher at a speed of 50mm/s versus 10mm/s, these differences were only statistically significant for C immediately and 5 minutes after application, and B immediately after application, probably due to the large standard deviations associated with the measurements performed on 6 different participants for the lower speed. However, in Chapter 3 the yield stress for C was found to be considerably higher than for the other products, which indicates that its viscosity will increase with increasing stress for longer. Therefore in this case the increase in sliding speed may have caused an increase in viscosity which would explain the increased DCOF for C.

Although there are too many variables which could be associated with the differences observed relating to the probe and speed to form any conclusions, it remains of interest to assess the shift in trends. In both cases where the friction was measured against a finger-pad, A appeared to have the largest DCOF after 5 minutes. For the friction measured against a solid probe, this was found for B. The DCOF for C was also almost as high as A, but both immediately and after 5 minutes. These trends also become clear in Figure 5.17. The DCOF for C appears to be almost independent of the test-bed which demonstrates that the finger-pad could be being “carried” by the product rendering the DCOF dependent on the properties of the product. The lower DCOF observed against the smooth test-bed after 5 minutes also highlights that the test-bed texture likely enables the product to anchor itself onto the surface.

Furthermore, in contrast with the results found for the original method, the comparison in mean DCOF values between all four test-beds highlighted the lack in significant differences for different mechanical properties. Another difference with previous results was the reduced friction immediately after product application for the smooth test-bed. Lastly, the addition of glycerin into the test-bed did also incur a significant increase in friction after 5 minutes for B and A, which is in accordance with previous results, but a decrease in friction immediately after application for B.

There are a few probable explanations for these observations. Firstly, the finger-pad is characterised by concentric ridges [13]. In the case where the BPC product is still present on the test-bed surface, the ridges could be contributing to an increase in

adhesion due to a larger contact area with the BPC product. As highlighted in Figure 5.18, C is more adhesive and therefore a larger contact area with the finger-pad will lead to higher friction which is also observed in Figure 5.15. The significant correlation of adhesive measurements with the panel ratings for the attribute “Greasy”, which refers to the feeling of a thick, fatty layer of product remaining on the skin, further validates this theory.

The finger-pad being more compliant than the probe will also likely increase the contact area, as illustrated in Figures 5.22 and 5.23. As mentioned above, this will have an effect on the contact pressure. As the finger-pad deforms both laterally and normally and is dragged across the surface, the centre of pressure will move from the centre of the finger to the side, as shown in Figure 5.23, therefore leaving part of the skin to sit on top of the product. The increase in friction with decreasing contact pressure for a finger-pad was also found by Derler et al. [110].

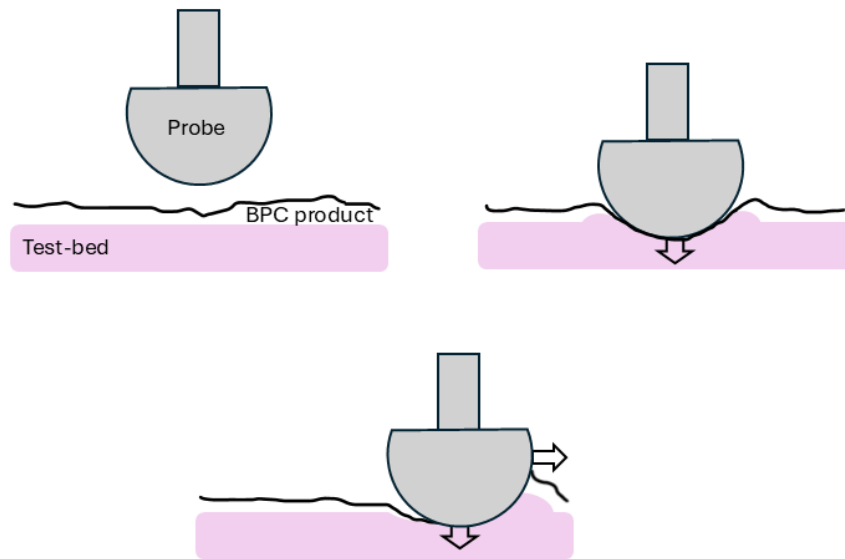


Figure 5.22: Diagram of the probe - BPC product interaction during movement.

As the contact pressure is reduced for the finger-pad, it will likely not penetrate as deep into the test-beds. Hence in the case of the bi-layered test-bed, the effect of a stiffer upper layer is observed rather than the effect of the bulk properties. In contrast, on a more compliant test-bed the probe will likely cause more deformation of the test-bed as highlighted in Figure 5.22. As the probe starts to move, the accumulation of test-bed material in front of the probe will cause an increase in friction. This

could explain why for B, which is likely to render the skin more compliant due to its hydrating properties, the friction was higher when measured against the solid probe. It can therefore be reaffirmed, that the finger pad friction may be more affected by the BPC product characteristics, specifically adhesion, whilst the probe friction will be influenced in a majority by the mechanical properties of the test-bed.

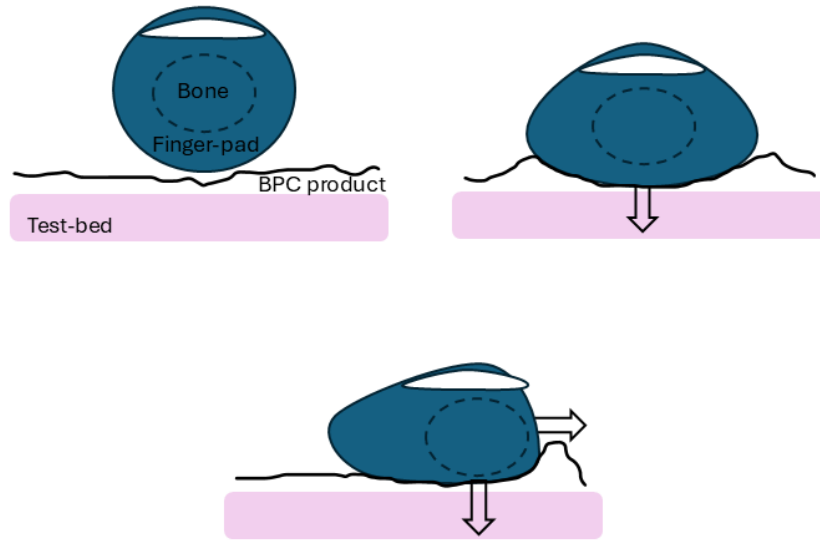


Figure 5.23: Diagram of the front facing view of a finger - BPC product interaction during movement, with exaggeration of the deformation to highlight the differences with a solid probe.

Finally, from the linear regression analysis conducted on the adhesion results, significant positive correlations were found for the bi-layered test-bed (TB4) and the attribute scores *Greasy* and *Sticky Tacky*. The increase in perception of *greasiness* with increasing adhesion was also found in a study by Tang et al. [41], which the authors attributed to the increase shear resistance of the BPC products. Additionally, positive correlations with *Greasy* and *Sticky Tacky* were found for the human finger-pads at 10 mm/s against TB4 and for the probe against TB4, respectively, for the DCOF immediately after product application. Lastly, although not significant, a linear relationship was found for the Coefficient of Variation (COV) measured against human volar forearms and the scores for *Sticky Tacky* 5 minutes after product application in Chapter 3. From these results it can be said that the scores for *Sticky Tacky* are likely to be attributed to the deformation of the volar forearm skin caused by the adhesion of the product. Conversely, the perception of the attribute *Greasy*

is more likely to be influenced by the product characteristics and the adhesion of the product on the finger-pad.

The linear regression analysis also revealed a significant negative correlation between the adhesive force and the sensory panel scores for the attribute *Drag*. This seemed counter-intuitive, as the attribute is defined as “the difficulty of moving fingers across the skin” (from Table 3.2 in Chapter 3). However, additional linear regression analyses revealed a significant positive correlation between the viscosity measured at a shear rate of 1 s^{-1} and the adhesive force after 5 minutes, as well as a significant negative correlation between the viscosity measured at a shear rate of 1 s^{-1} and the attribute scores for *Drag* after 5 minutes. Hence these correlations could be explained by the fact that a reduced viscosity will allow for the layer of product to be pushed by the finger-pad and accumulate in front of it as it slides across the surface, thus increasing the perceived *drag*. In contrast, an increased viscosity and adhesion will cause the product to remain in place whilst the finger slides across the top of the layer.

5.5 Conclusions

The aim for this chapter was to utilise the learnings from the preliminary testing outlined in Chapter 5 to develop a synthetic skin test-bed which replicated the frictional behaviour of human volar forearm skin in the presence of different BPC products. The results highlighted in this chapter are summarised below.

- The addition of deadener into the test-beds at a weight percentage of 40 led to a increase in compliance and viscoelasticity which matched the mechanical properties of human volar forearm skin for compressive loads in the range of 0.5 to 1N.
- The manufacture of a bi-layered test-bed with added glycerin in the most superficial layer and deadener in the deeper layer successfully replicated the frictional behaviour of human volar forearm for all products and conditions except for B immediately after application. This was associated to potential disparities in the water absorption rates. Although there was a significant difference for B immediately after application, the test-bed was able to replicate the trends in DCOF for all three products.

- The calculation of the deformation component of friction confirmed that the contact was already no longer fully lubricated for the measurements immediately after product application, and that adhesion was contributing to the DCOF.
- The measurement of the DCOF of a finger-pad against the bi-layered test-bed with glycerin (TB4) revealed significant differences for the products A and C when compared with measurements against a solid probe.
- Simple linear regression analysis identified significant correlations between the DCOF immediately after application and attribute scores for *Sticky Tacky* (positive), and *Greasy* (positive), for the solid probe and finger-pads against TB4 respectively. However, their accuracy remains limited due to the size of the data set.
- Some statistically significant differences were found between the results from the DCOF measurements against TB4 in an industry setting and the ones obtained from the original method, for both the human finger-pad and the probe. However, due to the number of confounding factors the significance of these differences remains limited.
- Similar to the finger-pad data obtained at the University, the DCOF was increased compared to the values found for a solid probe, for both of the occlusive products A and C with the latter also yielding the highest adhesive force values.
- Additionally, it was found that the influence of the test-bed mechanical properties were greater for the DCOF measured against the probe than for the human finger-pad.
- These findings confirmed the greater impact of product characteristics on the friction when measured against a human finger-pad.
- Combining the results from the linear regression analysis for all chapters revealed that the perception of *Sticky tacky* was most likely associated with the deformation of the volar forearm skin whereas the perception of *Greasy* may be more impacted by the product characteristics.
- The significant negative correlation found for the adhesive force and sensory panel scores for *Drag* were found to be related to the product viscosities at a shear rate of 1 s^{-1} , which were measured in Chapter 3. Thus it was hypothesized that a lower viscosity would cause the accumulation of product in front of the

finger-pad, whilst an increased viscosity would cause the finger-pad to slide on top of the layer of product thus decreasing the perceived *drag*.

These findings demonstrate the potential of the developed test-bed to provide meaningful insights into the complex interactions between product properties, human skin behaviour, and sensory perception. Future work could be used to further refine the test-bed and enhance its predictive capabilities in consumer product evaluation.

Chapter 6

Conclusions, limitations, recommendations and future work

6.1 Conclusions

For this thesis, sensory panel scores for attributes rated during application, as well as immediately after and 5 minutes after application, were provided by Unilever. Several experiments were undertaken in order to find measurable variables linked to the skin's frictional behaviour and BPC product characteristics, which could be used to predict these scores. Additionally, the results were utilised to inform on the design of a synthetic skin test-bed, to replace human participants in the developed protocol. Findings from the different chapters are summarised below.

- Rheological measurements were performed for five products in order to relate BPC product characteristics to the sensory attributes evaluated while the products were first applied to the skin. The results from a simple linear regression analysis revealed that the Log of the instantaneous maximum viscosity (Log(IVM)) was significantly correlated to the attribute scores for *Wetness* ($p < 0.05$). A multiple regression analysis showed that the Log(IVM) and the log of the product's yield stress were significantly negatively correlated to the attribute scores for *Spreadability* ($p < 0.05$). Lastly, a multiple regression analysis showed that the measured thixotropy of the products was significantly positively correlated to the attribute scores for *Creaminess*.
- From the friction measurements performed on human volar forearms for 12 participants with three of the five previously tested products, a strong, but not significant, positive linear relationship was found between the coefficient of variation (COV), which can be used to measure “stick-slip”, after 5 minutes and the

attribute scores for *Sticky tacky*. The results also revealed potential correlations between attributes *Greasy* and *Oily* and the DCOF after 5 minutes however, more data points are needed in order to confirm any significant correlations.

- As it is likely that immediately after application both the BPC product characteristics and the frictional behaviour will influence the perception of the product, a larger data set would allow for a multiple regression analysis to be performed for more accurate results.
- In Chapter 4, the influence of key design elements on the frictional behaviour of test-beds in the presence of BPC products was evaluated. It was found that the addition of glycerin into a silicone mixture enabled the absorption of water into the test-bed, which caused the DCOF to increase. Additionally, cross-linking the silicone mixture with silicone deadener allowed for an increase in compliance and visco-elasticity of the test-beds which also increased the DCOF for the measurements done 5 minutes after product application. Lastly, the addition of human volar forearm texture reduced the DCOF both immediately and 5 minutes after product application. However, this effect was not observed for test-beds with close to the maximum amount of deadener, as the amount of deformation of the test-beds did not allow for any sliding to occur. Lastly, adding a stiffer upper layer of less than 1mm in thickness to a 5mm layer cross-linked with deadener caused an increase in stiffness of the test-beds which became more prominent above a normal load of 2N. The bi-layered structure also caused the a wrinkling effect which was reflected in the raw friction data.
- Following on from Chapter 4, the decision was made to increase the length of the test-beds, to allow for full sliding to occur for the most compliant and visco-elastic test-beds. In Chapter 5 it was found that the bulk mechanical properties of a bi-layered test-bed with added deadener at a weight percentage of 40 in its deeper layer (TB4) matched the properties of human volar forearms under compressive loads of 0.5 to 1N when measured with a solid probe of 16 mm in diameter.
- The frictional behaviour of the human volar forearm in the presence of three different BPC products was also replicated by the textured bi-layered test-bed with glycerin in the most superficial layer at a weight percentage of 40 and deadener in the second layer at a weight percentage of 40 (TB4), both immediately and 5 minutes after product application.

- The friction was then assessed for the bi-layered test-bed (TB4) against human finger-pads, which showed some significant differences in the DCOF for two of the BPC products compared to the friction against a solid probe as well as differences in trends. Additionally, the DCOF of the finger-pad against TB4 was found to be significantly positively correlated with the attribute scores for *Greasy* immediately after product application.
- Results from the indentation tests for test-bed TB4 allowed for the calculation of the deformation component of friction, which revealed the influence of adhesion on the DCOF immediately after application. This finding reaffirmed the transition from a fully lubricated contact during application to a mixed lubrication regime immediately after application.
- The frictional behaviour of the synthetic skin test-bed was further assessed in Chapter 5, by comparing the frictional behaviour of several test-beds against a human finger-pad whilst also measuring the adhesion in the presence of the three BPC products. The results showed that the DCOF was independent of the test-bed's mechanical properties, in contrast with the friction measured against a solid probe. Some differences arose from changes in speed, however the number of confounding factors limited their significance.
- The adhesion measurements yielded significant positive correlations with attribute scores for *Sticky tacky* and *Greasy* immediately and 5 minutes after product application. Combined with the previous correlations found, this helped strengthen the theory that the perception of the attribute *Sticky tacky* is in a majority influenced by the deformation of the volar forearm skin, which occurs due to the adhesion of the product. In contrast the attribute *Greasy* is more likely influenced by the characteristics of the products and their interactions with the finger-pad.
- The adhesion was also found to be significantly negatively correlated to the attribute scores for *Drag*, which was explained by the significant positive correlation found between the viscosity at a shear rate of 1 s^{-1} and the adhesive force measured 5 minutes after product application, which also yielded a significant negative correlation with the scores for *Drag*. Hence it was hypothesized that the increased viscosity would prevent the accumulation of product in front of the finger-pad during sliding, and therefore would create a layer for the finger-pad to slide on top of thus reducing the perceived *drag*.

The methodology used for the assessment of the “in-use” and “after-use” attributes yielded promising results, as highlighted by the strong linear relationships found. Replicating these tests with more products should strengthen the understanding of the mechanisms contributing to the sensory perception of BPC products.

6.2 Limitations

The results presented in this thesis highlighted that the perception of various attributes is more than likely the result of several mechanisms acting in synchronicity. Although the experimental results outlined did lead to some significant correlations and strong linear relationships with sensory attribute scores, the number of confounding factors limits the ability to validate the theories on the mechanisms involved. For example, in the method used for the measurements of friction in Chapters 4 and 6 the normal load and speed were applied manually. Although participants had direct feedback of the normal load on a screen, they were given a window of 0.5 to 1N for which to aim as maintaining a specific load was challenging. The verbal guidance provided in order to maintain a constant sliding speed did enable some control, however variations were inevitable, especially due to lack of experience and the added difficulty of maintaining the normal load. These discrepancies may have contributed to the lack of significance for the correlation found between the attribute *Sticky Tacky* and the coefficient of variation (COV) after 5 minutes. Repeating these tests with the forearm held in a passive position whilst a probe slides across it may improve the chances of obtaining a significant positive correlation between the two variables.

Another limiting factor was the relatively small sample size. As most of the measurements were done for three BPC products, the ability to perform multiple regression analyses was restricted. As highlighted in the literature review, it is likely that the friction occurring immediately after the application of the BPC products will be in the mixed lubrication regime [91, 97], meaning that both the specific rheological characteristics of the products and changes in frictional behaviour are likely to influence the sensory perception.

Additionally, while the Product C has a simple composition, the other two products cover a wide variety of ingredients which each carry a specific function. The

ingredients focussed on were limited to the 5 highest in concentrations but it is unclear whether ingredients not mentioned, at lower concentrations, may have a greater impact on the frictional behaviour of skin which could further explain the trends.

Moreover, while the development of a synthetic skin test-bed to replicate the behaviour of volar forearm skin in the presence of three BPC products yielded encouraging results, there was a noticeable shift in trends when the solid probe was replaced by a human finger-pad. More experiments with a higher degree of control are needed to fully explain these trends. For example, the angle at which the finger was held could cause a significant change in contact area which could affect the resulting friction [96]. It could also be of interest to measure the friction induced vibrations, as these have been found to play a major role in tactile perception [94]. Moreover, the friction was only measured for one sliding direction (laterally, rather than aligned with the longest finger dimension). As sliding direction has been hypothesized to affect the tactile perception due to the uneven distribution of receptors in the finger-pad [15], this could have impacted the significance of correlations found with attribute scores.

Lastly, the use of linear regression analysis could be a limiting factor in finding correlations between measured variables and attribute scores, as it is likely that the skin's response to different stimuli is non-linear [101].

6.3 Revisiting the Aims and Objectives

The overarching aim of this PhD was to develop a methodology for assessing attributes related to the sensory perception of beauty and personal care (BPC) products. This was successfully addressed by establishing relationships between measurable product and skin/test-bed parameters and sensory attributes, and by designing a synthetic skin test-bed that could act as a prototype screening tool.

Aim 1: Understand the differences between BPC products and their effect on the frictional behaviour of human skin

This objective was achieved through a series of rheological and friction experiments:

- Rheological measurements (Chapter 3) identified key parameters such as Log(IVM), yield stress, and thixotropy that were significantly correlated with sensory attributes like *Wetness*, *Spreadability*, and *Creaminess*.

- Friction measurements on human skin revealed a potential linear relationship between the coefficient of variation and the attribute *Sticky tacky*, reinforcing the link between physical product properties, skin interaction, and perceived sensory experience.
- The findings provided an insight into the predictive capability of sensory attributes based on quantifiable variables.

Aim 2: Understand the effect of synthetic skin test-bed design elements on its frictional behaviour

This objective was addressed in Chapters 4 and 5:

- Various design elements (texture, glycerin content, cross-linking, bi-layered structures) were evaluated, with their impact on dynamic coefficient of friction (DCOF) systematically studied.
- The optimised test-bed (TB4) was shown to replicate the mechanical properties and frictional response of human volar forearm under specific conditions.
- These findings directly informed the design of a synthetic skin substitute with human-like frictional characteristics.

Aim 3: Develop a methodology for assessing BPC products on a synthetic skin test-bed

Chapter 5 demonstrated that:

- The TB4 test-bed successfully replicated human frictional behaviour in the presence of BPC products and exhibited significant correlations between measured friction/adhesion values and sensory attributes.
- Correlations with *Sticky tacky*, *Greasy*, and *Drag* demonstrated the test-bed's potential for predicting sensory perception.
- The methodology has potential as an industry screening tool, though replication with a broader range of products is needed to validate its generalisability.

6.3.1 Summary

This research successfully achieved its aim by:

- Establishing potential quantitative relationships between product parameters and sensory perceptions.
- Designing and validating a synthetic test-bed capable of replicating human skin frictional responses.
- Laying the groundwork for a non-human based screening method suitable for early-stage product evaluation.

6.4 Recommendations for the manufacture of a synthetic skin test-bed.

The following process was determined to be the most accurate for replicating the frictional behaviour of human volar forearm skin against a solid probe, following the application of BPC products.

The mould used measured 150×50 mm, with a 1 mm deep groove of the same dimensions CNC-milled into the base, as shown in Figure 4.1 in Section 4.2. To replicate the skin texture on the test-bed, a negative of human volar forearm skin was created using SILSKIN silicone, onto which the test-bed mixture was cast.

However, this method proved time-consuming and resulted in partial loss of texture due to the need to separate the two silicones with petroleum jelly, which prevented them from curing together. An alternative approach using additive manufacturing to directly print the texture into the mould may offer a more efficient and precise solution.

Test-bed Preparation

1. Mix addition-cure silicone (Silskin 10 Special Effects, Fibreglass, MB) at a 1:1 ratio of base to catalyst.
2. For the upper layer, add glycerin at 40 wt% of the combined silicone base and catalyst.

3. Mix thoroughly using a plastic mixing stick and degas the mixture until no air bubbles appear at the surface.
4. Pour the mixture into the 1 mm-deep groove of the mould and scrape off any excess using a flat mixing stick.
5. Secure the frame onto the mould.
6. Leave to cure for a minimum of 30 minutes.
7. For the second layer, again mix the addition-cure silicone at a 1:1 ratio, this time incorporating silicone deadener at 40 wt% of the total silicone weight.
8. Mix thoroughly and degas until the surface is free of bubbles.
9. Pour the second mixture on top of the cured first layer until the mould is full.

6.5 Future work

Based on these findings and the limitations outlined above, the priority should be the repeat of experiments with a greater range of BPC products. It is likely that the measurement of the DCOF alone will not be enough to accurately predict attribute scores and therefore larger data sets would allow for a multiple regression analysis. This would enable the combination of rheology variables with DCOF and adhesion measurements, to establish significant correlations with attribute scores for the “after use skin feel”.

Although this thesis focused on the mean DCOF, additional understanding could be achieved from the raw friction data. One example of this was the calculation of the coefficient of variation which is used to determine whether stick-slip is occurring. A further option to consider is measuring changes in the static coefficient of friction.

Furthermore, the methodology currently used at Unilever could be adapted to use lower speeds and fewer strokes. Lower speeds, which been used in similar studies [75, 41], may allow for an increased understanding of the friction behaviour of finger-pads and synthetic skin test-beds in the presence of various BPC products. The large number of strokes could also be contributing to an increase in sweat secretion which may be adding a layer of complexity to the system.

Integrating temperature into the methodology is also worth considering, as the temperature of skin may be contributing to the evaporation rate of ingredients from the BPC products, especially as it is rubbed with a finger against the skin. This could be done by means of a heated pad underneath the test-bed. A pilot study using this method was carried out at the University of Sheffield for a final year project, which yielded encouraging results. Similarly, tailoring humidity levels to specific environmental conditions may allow for a greater understanding of consumer trends. Likewise, the adaptation of the test-bed mechanical properties and deformation behaviour, by tailoring deadener percentage and layer thicknesses may be of interest for adapting the test-bed to a specific demographic.

Lastly, although introducing a finger-pad probe into the system may allow for a more accurate representation of the consumer experience, studying the deformation of the synthetic skin test-bed using a solid probe may still be of value. Understanding the effect of various BPC products on the deformation of skin could be essential in understanding trends. Additionally, methods such as Digital Image Correlation and Optical Coherence Tomography may be used to evaluate localised skin strain and deformation in more detail.

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Appendices

Table 1: Resulting mean DCOF extracted from the raw data of the solid probe against human volar forearm skin in the presence of BPC products A,B and C as well as dry on the dominant forearm (D).

Participant number	Moisture	SD	Mean DCOF						
			Dry (D)	A Imm	A 5m	B Imm	B 5m	C Imm	C 5m
1	53	0.940	0.907	0.314	0.390	0.287	0.667	0.506	0.587
2	44	0.47	0.602	0.211	0.305	0.267	1.10	0.266	0.239
3	54	2.16	0.837	0.465	0.335	0.505	0.860	0.245	0.2572
4	51	2.05	0.666	0.605	0.491	0.422	1.35	0.317	0.379
5	53	0.82	0.773	0.361	0.405	0.213	0.904	0.356	0.298
6	56	2.45	0.999	0.374	0.461	0.456	0.858	0.225	0.500
7	41	1.70	0.711	0.327	0.515	0.520	1.53	0.325	0.258
8	43	3.68	0.908	0.257	0.406	0.253	0.677	0.357	0.506
9	45	1.25	0.783	0.180	0.294	0.262	0.804	0.493	0.558
10	43	2.16	0.450	0.457	0.525	0.4721	0.719	0.437	0.234
11	51	2.05	0.761	0.4759	0.618	0.5465	0.737	0.319	0.339
12	47	0.94	0.794	0.3212	0.372	0.257	0.854	0.209	0.287
Average			0.753	0.362	0.426	0.372	0.921	0.338	0.370
Standard deviation			0.141	0.117	0.094	0.120	0.259	0.094	0.126

Table 2: Resulting mean DCOF extracted from the raw data of the finger-pads of 6 human participants against test-bed TB4 in the presence of BPC products A,B and C.

Participant number	Mean dynamic COF					
	A Imm	A 5 min	B Imm	B 5 min	C Imm	C 5 min
1	0.655	1.019	0.360	0.604	0.570	0.663
5	0.566	0.988	0.253	0.532	0.455	0.564
6	0.455	0.879	0.366	0.774	0.331	0.469
8	0.282	0.683	0.269	0.722	0.452	0.348
9	0.310	0.670	0.408	0.951	0.573	0.614
10	0.323	0.713	0.323	0.560	0.466	0.405
Mean	0.432	0.825	0.330	0.690	0.474	0.510
Standard Deviation	0.140	0.143	0.055	0.145	0.082	0.113

Table 3: Resulting mean DCOF extracted from the raw data for each repeat of the solid probe against synthetic test-beds.

Synthetic test-bed	Mean DCOF					
	A Imm	A 5 min	B Imm	B 5 min	C Imm	C 5 min
TB1	0.102	0.116	0.112	0.096	0.111	0.093
	0.085	0.111	0.105	0.095	0.098	0.108
	0.112	0.124	0.092	0.106	0.113	0.108
TB2	0.113	0.134	0.080	0.140	0.137	0.130
	0.098	0.159	0.098	0.122	0.143	0.145
	0.142	0.165	0.086	0.183	0.156	0.182
TB3	0.240	0.323	0.270	0.352	0.327	0.354
	0.240	0.417	0.274	0.378	0.355	0.358
	0.204	0.205	0.231	0.624	0.352	0.359
TB4	0.351	0.365	0.288	0.893	0.369	0.356
	0.246	0.424	0.233	0.659	0.341	0.353
	0.255	0.417	0.264	0.798	0.357	0.321
TB4	0.439	0.632	0.390	1.458	0.573	0.523
	0.252	0.439	0.305	1.310	0.419	0.383
	0.279	0.487	0.214	0.593	0.371	0.400

Table 4: Resulting mean DCOF extracted from the raw data for each repeat of the human finger-pad against synthetic test-beds in an industry setting.

	Test-bed							
	TB1		TB2		TB4		TB5	
	Imm	5 minutes	Imm	5 minutes	Imm	5 minutes	Imm	5 minutes
A	0.419	0.662	0.308	1.025	0.479	1.021	0.319	0.928
	0.364	0.720	0.512	1.021	0.471	0.913	0.261	0.940
			0.466	0.853	0.449	0.945		
			0.337	0.957				
B	0.527	0.645	0.320	0.698	0.570	0.807	0.225	1.026
	0.360	0.608	0.452	0.750	0.452	0.682	0.198	0.852
			0.322	0.681	0.525	0.751		
			0.392	0.734				
C	1.090	1.073	0.874	0.996	0.966	0.965	0.950	0.641
	0.549	0.629	0.781	0.752	0.784	0.811	0.721	0.620
			0.657	0.609	0.866	0.869		
			0.751	0.678				



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Approved: 26/10/2023

Phoebe-Claire Bramley
Registration number: 200236788
School of Mechanical, Aerospace and Civil Engineering
Programme: Mechanical Engineering PhD

Dear Phoebe-Claire

PROJECT TITLE: Measuring the effect of skin cream on the friction coefficient of skin.

APPLICATION: Reference Number 056989

On behalf of the University ethics reviewers who reviewed your project, I am pleased to inform you that on 26/10/2023 the above-named project was **approved** on ethics grounds, on the basis that you will adhere to the following documentation that you submitted for ethics review:

- University research ethics application form 056989 (form submission date: 25/10/2023); (expected project end date: 01/03/2024).
- Participant information sheet 1128419 version 2 (25/10/2023).
- Participant consent form 1128420 version 1 (09/10/2023).

If during the course of the project you need to [deviate significantly from the above-approved documentation](#) please inform me since written approval will be required.

Your responsibilities in delivering this research project are set out at the end of this letter.

Yours sincerely

Jennifer Rowson
Ethics Admin
School of Mechanical, Aerospace and Civil Engineering

Please note the following responsibilities of the researcher in delivering the research project:

- The project must abide by the University's Research Ethics Policy: <https://www.sheffield.ac.uk/research-services/ethics-integrity/policy>
- The project must abide by the University's Good Research & Innovation Practices Policy: https://www.sheffield.ac.uk/polopoly_fs/1.6710661/file/GRIPPpolicy.pdf
- The researcher must inform their supervisor (in the case of a student) or Ethics Admin (in the case of a member of staff) of any significant changes to the project or the approved documentation.
- The researcher must comply with the requirements of the law and relevant guidelines relating to security and confidentiality of personal data.
- The researcher is responsible for effectively managing the data collected both during and after the end of the project in line with best practice, and any relevant legislative, regulatory or contractual requirements.



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Approved: 30/01/2023

Phoebe-Claire Bramley
Registration number: 200236788
School of Mechanical, Aerospace and Civil Engineering
Programme: Mechanical Engineering PhD

Dear Phoebe-Claire

PROJECT TITLE: A proposed methodology for the validation of the surface structure and tribological properties of an artificial skin model.

APPLICATION: Reference Number 050484

On behalf of the University ethics reviewers who reviewed your project, I am pleased to inform you that on 30/01/2023 the above-named project was **approved** on ethics grounds, on the basis that you will adhere to the following documentation that you submitted for ethics review:

- University research ethics application form 050484 (form submission date: 26/01/2023); (expected project end date: 30/03/2023).
- Participant information sheet 1113566 version 7 (26/01/2023).
- Participant consent form 1113567 version 3 (26/01/2023).

If during the course of the project you need to [deviate significantly from the above-approved documentation](#) please inform me since written approval will be required.

Your responsibilities in delivering this research project are set out at the end of this letter.

Yours sincerely

Jennifer Rowson
Ethics Admin
School of Mechanical, Aerospace and Civil Engineering

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Downloaded: 15/04/2025

Approved: 26/04/2022

Phoebe-Claire Bramley
Registration number: 200236788
School of Mechanical, Aerospace and Civil Engineering
Programme: Mechanical Engineering PhD

Dear Phoebe-Claire

PROJECT TITLE: A proposed methodology for the validation of the surface structure and tribological properties of an artificial skin model.

APPLICATION: Reference Number 046093

On behalf of the University ethics reviewers who reviewed your project, I am pleased to inform you that on 26/04/2022 the above-named project was **approved** on ethics grounds, on the basis that you will adhere to the following documentation that you submitted for ethics review:

- University research ethics application form 046093 (form submission date: 25/04/2022); (expected project end date: 26/04/2022).
- Participant information sheet 1104615 version 3 (25/04/2022).
- Participant consent form 1104616 version 2 (25/04/2022).

If during the course of the project you need to [deviate significantly from the above-approved documentation](#) please inform me since written approval will be required.

Your responsibilities in delivering this research project are set out at the end of this letter.

Yours sincerely

Jennifer Rowson
Ethics Admin
School of Mechanical, Aerospace and Civil Engineering

Please note the following responsibilities of the researcher in delivering the research project:

- The project must abide by the University's Research Ethics Policy: <https://www.sheffield.ac.uk/research-services/ethics-integrity/policy>
- The project must abide by the University's Good Research & Innovation Practices Policy: https://www.sheffield.ac.uk/polopoly_fs/1.6710661/file/GRIPPpolicy.pdf
- The researcher must inform their supervisor (in the case of a student) or Ethics Admin (in the case of a member of staff) of any significant changes to the project or the approved documentation.
- The researcher must comply with the requirements of the law and relevant guidelines relating to security and confidentiality of personal data.
- The researcher is responsible for effectively managing the data collected both during and after the end of the project in line with best practice, and any relevant legislative, regulatory or contractual requirements.