Mimicking the Surface Properties of Human Skin: Tribo-mechanical & Adhesive Properties of a Synthetic Stratum Corneum

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Abstract: A Synthetic Stratum Corneum (SSC) was produced from a mixture of PVA (poly vinyl alcohol) hydrogel and SDS (sodium dodecyl sulphate) surfactant, later mixed with rapeseed oil and, finally crosslinked with glutaraldehyde. The thermal properties of the material were studied via TGA (thermogravimetric analysis) and DSC (differential scanning calorimetry) measurements and the composition was analysed by FTIR (fourier transform infrared). The hydration degree of the SSC measured at 30% of relative humidity and 25°C was 23% whilst at wet conditions it grew up to the 80%. Moreover, the surface adhesion of the SSC, evaluated under dry, normal and wet conditions indicated substantial variations depending on the skin conditions with values for the work of adhesion of -16, -122.4 and -233.4 mN/m, respectively. The viscoelastic properties of the material were also evaluated by tensile tests and stress relaxation measurements. The elastic modulus of the samples was found between 8.8 - 1.9 and 3.3 - 1.2 for dry and wet conditions, respectively. Further, stress relaxation times of 64.4 sec and 83.2 sec were calculated from the SLS (standard linear solid) model at dry and wet conditions, respectively. Lastly, the SSC presented a frictional performance in close agreement to that of human skin under normal (36% relative humidity and 25°C) and wet conditions. The friction coefficient, measured between 1 and 50 mN, was found between 0.8 to 0.45 at dry conditions whereas, at wet conditions, it ranged between 2 and 0.45. At higher applied forces between 0.5-3.5 N, the friction coefficient was nearly steady with values of 0.4 and 0.55 for dry and wet conditions, respectively.

Key Words: Synthetic stratum corneum, Tribo-mechanical properties, Adhesion, PVA hydrogel, Rapeseed oil
unsaturated lipids was coated on top of Lorica to analyse the surface properties of the model in relation with its frictional performance\(^{28}\). In vitro models of SC from plastic surgery have been used to study the mechanical performance of the SC and determine its contribution to the overall skin response\(^{41}\). However, the availability of ex vivo samples from cadaveric or surgical source is limited and the ethical and sanitary arrangements required to manipulate biological tissue imply a limitation at industrial scale. Besides, although isolated human SC could serve as a model, the isolation process alter the natural state and properties of the SC due to the use of chemical agents. Furthermore, in vivo studies present inter-individual variability and testing-device limitations.

Different polymers and, particularly hydrogels have been research in the last decades for biomedical uses thanks to its inherent biocompatibility and hydration properties\(^{32, 33}\). Hydrogels have been used to manufacture contact lenses\(^{40}\) or wound dressings\(^{35}\), in cartilage replacement applications\(^{36}\), drug delivery systems\(^{37}\) or artificial organs\(^{38}\). Within the existent hydrogels, Poly vinyl alcohol (PVA) represents a relatively economical choice with ease manufacturing. PVA has been used to develop wound dressings\(^{39, 40}\) due to its ability to maintain hydration and prevent dryness, its elastic and non-toxic properties and the wide number of crosslinking methods already known \(^{42}\). In our previous research\(^{42}\), it was suggested a modification of the shearing properties of PVA blocks to approach the frictional performance of human skin by simply modifying the crosslinking approach. Additionally, the properties and uses of rapeseed oil (Ro) are broadly researched currently for bio diesel applications, in the biotechnology field (antibiotics, antioxidants, vitamins) and as a source of vegetable oil with positive health properties\(^{43}\). Rapeseed oil is relatively cheap and facile to acquire. It is a rich source of natural components with antioxidant properties such as vitamin E, phenolic compounds, phytosterols or phospholipids. Moreover, some of these compounds protect the skin against lipid peroxidation due to UV exposure and ionizing radiation\(^{44}\). Rapeseed oil is rich in phospholipids which are fundamental constituents of the cells membranes with an essential role their physicochemical properties such as, the homeostasis or the signal transduction. Further, the main fatty acids in the rapeseed oil are saturated fatty acids in an amount of 7.46 wt.%, 64.06 wt.% of mono un-saturated fatty acid and 28.48 wt.% of total poly-unsaturated fatty acid. These fatty acids are mainly Palmitic, Palmitoleic, Stearic, Oleic, Linoleic and α-Linoleic acids some of which are widely present in the SSL\(^{45}\).

Therefore, in this paper the development of an alternative Synthetic Stratum Corneum (SSC) is described and the evaluation of its tribo-mechanical behaviour is presented. The SSC was produced from a mixture of PVA hydrogel and SDS surfactant, later mixed with rapeseed oil and, finally crosslinked with glutaraldehyde. The thermal properties of the films were determined via DSC and TGA measurements. FTIR measurements were performed to confirm the presence of the rapeseed oil and evaluate the changes in the functional groups after wetting the samples. The SSC was texturized with the features of the thigh of an individual and the roughness parameters were determined. Further, the contact angle and hydration degree at different relative humidities were obtained. The mechanical properties of the films were evaluated by tensile tests and stress relaxation measurements from which the viscoelastic parameters were calculated based on the SLS model. Further, the adhesive properties were determined based on pull-off measurements and the frictional behaviour of the SSC was evaluated in two different range of forces between 1 - 50 mN and 0.5 - 3.5 N.

2. Tribo - mechanical properties of the human stratum corneum

2.1 Elastic modulus

The elastic modulus of the stratum corneum is a key parameter to describe the frictional performance of human skin. In brief, the SC has been assessed by indentation tests at different conditions which results showed an elastic modulus scattered in a wide range of values (MPa – GPa)\(^{46, 50}\). Results from nano-indentation on porcine isolated stratum corneum were analysed by Yuan and Verma\(^{51}\) who found an elastic modulus of 120 and 26 MPa for the stratum corneum at dry and wet conditions respectively. Isolated stratum corneum measured by indentation indicated an elastic modulus of 2.6 ± 0.6 MPa according to Geerlings\(^{52}\). Further, Pailler-mattei et al.\(^{60}\) showed large differences between the results from in vivo skin and isolated stratum corneum. The latest indicated a mean elastic modulus around 1 GPa which decreased with the indentation depth. Moreover, Crichton et al.\(^{52}\) analysed the mechanical properties of the individual layers of mouse skin. Their results indicated a larger elastic modulus for dermis (7.33 MPa) than for the stratum corneum (at 0.75 - 1.62 MPa) under similar conditions. Van Kuilenburg et al.\(^{53}\) presented a collection of the elastic modulus of the human skin from indentation as a function of the length scale. These results indicate a decrease of the elastic modulus of several orders of magnitude with the increase of indentation due to the layered structure of the skin. Thus, at indentation depths no larger than 10 mm, the elastic properties of the skin can be described only by the stratum corneum which elastic modulus vary between GPa and MPa.
depending on the indentation and the skin conditions (dry or wet)\(^{48-50}\).

2.2 Viscoelastic properties: stress relaxation measurements

The viscoelastic properties of the human skin are mostly due to the nature of dermis yet, the stratum corneum also contributes especially, at wet conditions\(^ {41, 52}\). According to the literature the SC exhibits a short period of viscoelasticity which is mainly associated to the gradient of hydration from the top to the bottom of this thin layer\(^ {22}\). In this paper, the viscoelastic properties of the SSC were analysed based on the Standard Linear Solid model (SLS):

\[
E(t) = 
\frac{E_1}{E_1 + E_2} \left[ 1 + (\frac{E_2}{E_1}) t \right]^{-1} 
\]

(1)

The elastic moduli, \(E_1\) and \(E_2\), and the viscosity, \(\eta\), were calculated by fitting the stress relaxation results in Eq.1. Later, the relaxation time, \(\tau\), was calculated as:

\[
\tau = \frac{\eta}{E_1 + E_2} 
\]

(2)

2.3 Frictional performance

The frictional behaviour of the skin is significantly affected by the properties of the stratum corneum, especially at low forces\(^ {46-60}\). When a smooth hard material contacts the skin, already at very low forces the true area of contact reaches the nominal one and the effect of the surface properties of the skin are mainly commanded by the Skin Surface Lipid Film (SSLF) on top of the stratum corneum. Thus, the friction coefficient is mainly determined by means of the adhesive properties of the skin with little effects of its roughness. Previous work of several authors gives a wide overview of the most relevant factors influencing skin friction\(^ {44-60}\). Derler et al.\(^ {59}\) analysed the contact situation of in vivo skin against rough and smooth surfaces at dry and wet conditions. Their results suggested the adhesive component of friction as the main friction mechanism whilst the contribution of the hysteresis due to the viscoelastic properties of the skin was minor\(^ {47, 61}\). Additionally, hydration increases the coefficient of friction of skin as it has been extensively probed in other research\(^ {45, 60-64}\). The effect of water, creams and moisturizers affects mainly the elastic properties of the stratum corneum with a subsequent effect in its adhesive properties and frictional performance\(^ {12}\). Thus, under dry conditions the skin shows a friction coefficient typically between 0.25 and 0.5 depending on the body location, the contacting material, age of the individual or indenter size among other. Conversely, in the hydrated case, the friction coefficient increases as consequence of the softening effect of the stratum corneum and the increase of the adhesion forces at the contact. A range of friction coefficient from 0.7 to 2.6 have been found for the coefficient of friction at wet conditions for a range of applied forces between 0.7 and 6.4 N\(^ {59}\). The mechanism behind the differences between dry and wet conditions is not clearly known yet. Persson\(^ {63, 64}\) found that the effect of capillarity depends on the elastic modulus of the soft material, so that a decrease on the elasticity addresses to an increase of the capillary force that, likely enlarges the friction force.

3. Material and methods

3.1 Synthesis

The Synthetic Stratum Corneum (SSC) was created from a mixture of poly vinyl alcohol (PVA) (Mw 31,000-50,000) purchased from Sigma Aldrich (USA) and rapeseed oil (Ro) provided by Grease Factory of Lanzhou (China) with no further treatment. Besides, a 0.5% wt. of Sodium Dodecyl Sulphate (SDS) 99% from Sigma Aldrich (US) was combined with the PVA to enhance the solubility of the rapeseed oil in the PVA aqueous solution. Glutaraldehyde (GA) 50% solution reagent (AMRESCO (USA) was used as a cross linker agent. To start with the crosslinking reaction, sulphuric acid (H\(_2\)SO\(_4\)) 96% provided by Acros Organics (USA), was used as initiator. Further, a flat square Teflon mould \(150 \times 80\) mm was used to create the films. To resemble the roughness of the human skin on the surface of the SSC films, a copy of the thigh surface of an individual between 25 - 30 years was obtained after applying the Panasil Initial Contact from Kettenbach (DE). The rubber copy was scanned by confocal microscopy and from this image an acrylate mould was obtained by laser engraving with a Trotec Speedy 300, manufactured by Trotec Laser (USA).

The specific procedure to synthesize the films is explained as follow: first, a solution of 20 wt. % (w/v) of PVA and 0.5 wt. % of SDS in deionized water (DW) was prepared by introducing the components in a round bottom flask with a reflux and stirring over night at 90°C. Then, 2 wt.% of Ro, respect to the PVA amount, was added to the solution and it was vigorously stirred during 3 minutes. Second, certain amount of PVA-oil solution was taken and 5 wt.% of H\(_2\)SO\(_4\) (aq) (pH = 1), with respect to the taken amount, was added as a catalyst for the formation of the acetyl groups between PVA and GA. The mixture was stirred for one minute and afterwards, GA cross linker was added in a ratio 10:1 of PVA to GA, respectively. The mixture was stirred for one minute and it was subjected to vacuum to avoid the presence of bubbles in the formed films. Finally, the acrylate mould of the thigh roughness was placed
on the bottom of the Teflon mould with the texture looking up. Then, the solution labelled as PVA-oil-GA, was poured in the mould and introduced in the oven at 40°C for 16 hours.

3.2 Characterization

3.2.1 Thermal & chemical properties: TGA, DSC, FTIR

The thermal behaviour of the SSC was analysed by TGA and DSC measurements. The weight loss, water content and degradation of the samples was evaluated from TGA measurements on a TGA 7 from Perkin Elmer (US). The samples were subjected to a heat program from room temperature to 350°C at 10°C/min under a N2 flow of 25 ml/min. Moreover, thermal transitions were determined on a DSC from Mettler Toledo with temperature range from room temperature to 300°C at 2°C/min under a N2 flow of 25 ml/min. The glass transition and melting points of the samples were evaluated. Additionally, a Fourier Transform Infrared (FTIR) spectroscope with a resolution of 0.5 cm⁻¹ from Perkin Elmer (US) was used to collect the infrared spectra of the SSC samples. The spectroscopy of SSC samples was measured within the range of 7800 – 370 cm⁻¹ at normal (36% relative humidity and 25°C) and wet (immersed in deionized water for 2 mins) conditions.

3.3 Surface & hydration properties

The surface properties of the SSC, such as roughness and contact angle were determined in the texturized samples. A laser confocal microscope VK 9700 from Keyence with z-axis resolution of 1 nm was used to determine the arithmetic mean (Ra), root mean square (Rq) and peak-to-valley (Rp) of the samples at a magnification of 10x. Additionally, the contact angle formed by water drops on top of the SSC was measured by a contact angle device from Dataphysics (Germany) model OCA 20. Further, the hydration degree of the SSC was determined with a corneometer MP5 from Courage+Khazaka Electronic (Germany) at 25°C and 30%, 70% relative humidity and also in fully hydrated samples. The latest were immersed in water 10 seconds and then, placed on a tissue paper. Later the measurements were performed.

3.4 Mechanical properties

3.4.1 Tensile & stress relaxation tests

The mechanical properties of the SSC were determined with an extensometer Zwick Roell model Z1.0 (Germany). Tensile tests were performed in 4 SSC samples at both, normal (25°C and 36% relative humidity) and wet conditions. The dimensions of the tested samples are specified in the ISO 37, ASTM D412 Tensile Testing of Rubber and Elastomers. The samples, with a grip to grip separation of 45 mm, were subjected to a preload of 0.05 N and the tests were performed at a velocity of 10 mm/min. From the tensile tests, the elastic moduli of the samples were obtained as the slope of the stress-strain curve at strains of 0 - 0.1, 0.1 - 0.2 and 0.2 - 0.3 for the normal conditions. In the case of the wet samples, the elastic modulus was also calculated as previously at strains of 0 - 0.05, 0.05 - 0.1 and 0.1 - 0.15.

Additionally, stress relaxation tests at both, normal and wet conditions were carried out at strains of 2% and 10% during 150 and 300 seconds, respectively. The wet conditions were achieved after immersing the SSC samples in deionized water during one minute. The results of these tests were correlated to the Standard Linear Solid (SLS) model and the characteristic parameters of each model were calculated by fitting the experimental results.

3.5 Adhesive properties from pull-off measurements

Series of pull–off measurements were performed by using a Vacuum Adhesive and Friction Tester (VAFT) designed at the University of Twente with a spherical indenter of 6 mm diameter made of Chrome Steel AISI-52100. The snap-off forces obtained from these measurements were used to calculate the work of adhesion, W₁₂, between the 2 materials based on the JKR model. An unloading speed of 50 µm/s was used during the pulling-off. The work of adhesion, W₁₂, was extracted from Equation (3):

\[ F_{aJKR} = 1.5\piRW_{12} \]

With \( F_{aJKR} \) the snap-off force, R the radius of the indenter and \( W_{12} \) the work of adhesion when \( z = z_0 \).

3.6 Frictional performance at two different range of forces

Friction tests were performed in a range of forces between 1 to 50 mN with the VAFT mentioned in the previous section. The measurements were carried out at a velocity 10 µm/s during a sliding distance of 400 to 2000 µm. The SSC samples were glued to a bottom layer of PVA hydrogel obtained by freezing/thawing cycles as it was described in our previous research. Additionally, friction test at loads between 0.5 and 3.5 N were conducted on a HC 4057 pin-on-disk machine (CSM, Switzerland). In this case, the friction measurements were conducted with cylindrical pins made of Chrome Steel AISI-52100 of 10 mm length and 30 mm radius. The measurements were performed at normal conditions of 36% relative humidity and 25°C and wet conditions. It has been shown in earlier research that, the friction coefficient presents its maximum reaction to the water effect during the first 2 min.
of exposure\textsuperscript{27, 54}. Thus, several drops of deionized water were dropped on the SSC during 2 min and later the excess of water was removed with a tissue. Then, the friction measurements at wet conditions were performed.

4. Results and discussion

4.1 Thermal properties & Fourier Transform Infrared (FTIR) spectroscopy

4.1.1 Thermogravimetric Analysis (TGA)

The normalized loss weight as a function of the temperature is presented in Figure 1 for the SSC, PVA crosslinked with GA (PVA-GA) and PVA physically crosslinked via freezing/thawing cycles (PVA f/t). Moreover, an example of the onset of the degradation, calculated as the intersection of the two tangent lines of a drop, is also illustrated in Figure 1.

The results extracted from the TGA curves above, such as water percentage, the onset of the degradation temperature and the residue for each sample are collected in Table 1.

Although the boiling point of water is 100°C, some water molecules remained attached to the polymer chains at higher temperatures as indicate the DSC results presented in the following section, particularly in the case of PVA-GA and SSC. Thus, the water content of the samples was calculated as the loss weight between room temperature and 120°C which corresponded mainly to free water clustered in the hydrogel whereas another small percentage remained until higher temperatures linked to the polymer chains. Based on these results, SSC samples contained a 25% of water as indicated in Table 1, which is considerably lower than the 90% of the PVA crosslinked via freezing/thawing and similar to the 15% obtained for the PVA-GA samples. The onset of the degradation temperature for the SSC corresponded to 288°C, lower than the 340°C of the PVA-GA likely due to the presence of oil with smoke point around 200°C. The degradation of the PVA crosslinked via freezing/thawing cycles started around 230°C. Furthermore, PVA-GA and SSC samples show a less evident change of the curve around 380°C which seems to be related to the decomposition of main chain of PVA while, the previous step corresponded to the elimination of side-groups between 200 and 330°C approximately. The residue of each sample is also given in Table 1. The maximum value was obtained for the SSC and it is of 5% which might corresponds to not decomposed traces of the main chain on PVA\textsuperscript{67, 68}.

4.1.2 Differential Scanning Calorimetry (DSC)

The results from the DSC measurements are shown in Figure 2. Images a), b) and c) from Figure 2 correspond to the individual tests for each analysed sample: PVA-GA, PVA f/t cycles and SSC, respectively; image d) collects the information of the three samples.

In Table 2 the temperatures correlated to each transition,
which were calculated as the intersection of the two tangent lines of an specific step, are presented (see image a) from Figure 2. The first transition in a DSC curve used to be correlated to the glass transition temperature of the polymer. However, in these cases the large peak representing the first transition shows mainly the evaporation of water, overlapped with the glass transition. Thus, it was not possible to determine the exactly glass transition temperature. It is being indicated a glass transitions temperature for the PVA polymer around 80°C (49-73), yet, for crosslinked samples the glass transition shifts. Moreover, the increase of the water content in the hydrogel causes also a change of the glass transition towards lower temperatures (72).

The SSC here presented was composed of PVA chemically crosslinked with GA. In its structure were also contained around 20%wt. of water and rapeseed oil. Thus, the effects of water and oil likely shifted the glass transition yet, the exactly temperature could not be determined probably hidden by the large peak of water. Furthermore, the second relaxation in the DSC graphs of PVA-GA and PVA f/t cycles was attributed to the melting point of the polymer. The peaks in these cases appeared at 338 and 223.5°C, respectively. In the SSC samples a second peak emerged at 168°C which can be attributed to the crystalline relaxations of the PVA (72) while the last peak corresponded to the melting point of the PVA at around 218°C as indicates Table 2.

### 4.1.3 Fourier Transformed Infrared (FTIR) spectroscopy

The fatty acids presented in the rapeseed oil were determined by comparing SSC samples at normal and hydrated conditions. The results of the spectroscopy are shown in Figure 3 and the main peaks related to the PVA, GA and fatty acids of the rapeseed oil are indicated. Strong resonance absorption appeared around 3300 cm⁻¹ due to stretching of intermolecular and intramolecular hydrogen bonds in the PVA polymer chains and the water molecules (73, 74). As it can be seen from Figure 3, the intensity of the peak at hydrated conditions was higher due to the increase of -OH groups in the sample linked to the water. The small peak appearing around 2950 cm⁻¹ was related to the CH2 stretching of the in the acetal bound between PVA and GA (73, 74).

Between 2850 and 2930 cm⁻¹ two sharp peaks appeared which correspond also to CH2 stretching bands related to fatty acids chain of the rapeseed oil (75). Another peak related to the presence of rapeseed oil appeared at 1746 cm⁻¹ and it is most likely related to the C = O (ester/acid) stretching of the glycerides present in the oil (75). This peak appears only in the SSC samples at normal conditions which indicates that after immersion in water an unknown amount of oil is removed that is, in wet conditions this peak is not visible. On the contrary, the peak appearing around 1690 cm⁻¹, related to hydroxyl groups (-OH), is larger in the case of wet samples due to the higher amount of hydroxyl groups introduced by the water molecules. Further, -CO stretching bands around 1100 cm⁻¹ can be attributed to the acetal ring and also to other-CO stretching groups in the fatty acids of the rapeseed oil (75).

### 4.2 Surface & hydration properties

The SSC samples were stamped with an acrylate mold previously texturized with the features of the human skin of the thigh of an individual. The roughness parameters $R_a$, $R_s$, $R_m$ and $R_d$ corresponding to the average of 4 SSC samples are presented in Table 3. These values have been correlated to human skin roughness from the literature which indicates values of $19.6 \pm 4$ for $R_v$ (76) and $30 \pm 7$ for $R_m$, the latest obtained as an average of different body places (77). Other researches showed similar values as well in the range of the roughness measured on the SSC (78, 79).

Additionally, the average and standard deviation of the contact angle of the samples are presented as well as the contact angle of the samples.

| Sample | $R_a$ (µm) | $R_s$ (µm) | $R_m$ (µm) | $R_d$ (µm) | CA (deg) |
|--------|------------|------------|------------|------------|----------|
| SSC 1  | 21.4 ± 0.1 | 26.8 ± 0.2 | 42.8 ± 0.1 | 0.19 ± 0.2 | 74.3 ± 3 |
| SSC 2  | 22.0 ± 0.3 | 27.5 ± 0.2 | 43.2 ± 0.3 | 0.21 ± 0.3 | 74.5 ± 2 |
| SSC 3  | 21.7 ± 0.2 | 26.9 ± 0.3 | 42.9 ± 0.2 | 0.19 ± 0.2 | 74.2 ± 2 |
| SSC 4  | 21.9 ± 0.1 | 27.2 ± 0.1 | 43.1 ± 0.1 | 0.20 ± 0.1 | 74.4 ± 2 |

Figure 3. FTIR curves for SSC at normal conditions of 25°C and 30% relative humidity (grey line), SSC at wet conditions compared to PVA crosslinked via freezing/thawing cycles (red line).

Figure 4. Confocal microscope images of the SSC after stamping with the features of the thigh. On the left side, a 3D colour image is presented with a height scale on its left; on the right side, a grey scale image of the stamped texture is given.
contact angle of the samples is indicated. The contact angle of the SSC was 74.3 deg which is within the range of values given by Ginn et al.\(^8\) in previous research between 58 - 104 deg for different skin conditions. This result is also in agreement with other previous research\(^6, 8\). Furthermore, a 3D image and a 2D grey scale image of a SSC sample is shown in Figure 4.

The hydration of the SSC samples was evaluated after equilibration at 30% and 70% relative humidity. Further, it has been indicated that the friction coefficient of human skin at wet conditions presents its maximum during the first 2 min after applying water\(^27, 55\). Thus, other 4 SSC samples were immersed for 2 minutes in water to wet them and the hydration was measured afterwards. These results are displayed in Figure 5.

The average hydration measured at 30% was 23.4 (A.U.) and 49.7 (A.U.) at 70% relative humidity whilst the fully hydrated sample presented a hydration value of 80.3 (A.U.). Hydration of the skin changes considerably from person to person and depends also on the temperature and humidity of the environment. However, the SSC samples exhibited a hydration level in the range of the values obtained at different body sites for the human skin\(^83, 84\).

4. 3  Tensile & stress relaxation tests

The mechanical properties of the SSC were analysed by tensile tests and stress relaxation measurements in 4 SSC samples under both, normal and wet conditions. A discussion of the obtained results is discussed in the following sections.

4. 3. 1  Tensile tests

The elastic modulus of the SSC was evaluated as the slope of the curve ‘stress vs strain’. The initial elastic modulus, \(E_0\), calculated at \(t = 0\) sec, was considerably higher than the elastic moduli, \(E_i\), calculated between 0 - 0.1, 0.1 - 0.2 and 0.2 - 0.3 strains at normal conditions. At wet conditions the samples were breaking at lower elongations so that, the \(E_i\), was calculated between 0 - 0.05, 0.05 - 0.1, 0.1 - 0.15 strains. The average values for each case are presented in Table 4.

The initial elastic modulus, \(E_0\), was about 2.5 times higher at normal conditions compared to the hydrated case. Further, a considerable decrease of the modulus was observed at different strains for instance, in the normal case, where \(E_i\) presented a decrease of 4.5 times the initial value \(E_0\). Figure 6 presents the evolution of the elastic modulus at normal and wet conditions as a function of the strain.

There is a clear decrease of the modulus at wet conditions respect to normal with a faster stabilization of the modulus at larger strains in the wet case. Moreover, the fracture of the wet samples occurred at lower elongations, specifically at 15% strain whereas the normal samples were stretched until 30% of their elongation as indicates in Figure 6. From the previous figure it can also be observed that the elongation at fracture of the SSC depends also on the water content of the samples.

4. 3. 2  Stress relaxation tests

Stress relaxation measurements were performed at a strain of 10%. The results were fitted in the Standard Linear Solid (SLS) model.
model and the elastic moduli, $E_1$ and $E_2$, and the viscosity, $\eta$, of the model were calculated. Further, the relaxation time, $\tau$, as the quotient of the viscosity of the material and the sum of the two elasticities $E_1$ and $E_2$, was also obtained. Table 5 collects the data of the 4 studied samples and the average values for the normal and wet conditions.

As it is expected because of the plasticizer effect of water in hydrogels\(^{(85)}\), the elastic moduli of the two springs, $E_1$ and $E_2$ presented lower values in the hydrated case. Especially notable were the differences in viscosity between normal and wet conditions. In normal conditions, the viscosity was considerably higher than the value found at wet conditions yet, the relaxation time is higher in the wet case than the normal. This can be explained due to a poorer resistance of the material to be deformed in wet conditions as a consequence of the presence of water. Water modifies the thermodynamics of the material and it leads to a higher mobility of the PVA polymer chains within the hydrogel. Hence, the material becomes more easily deformable with a consequent decrease of the resistance to be deformed under stress so that, the viscosity, $\eta$, decreases. However, the softening effect of water in the hydrogel causes a decrease of the $E_1$ and $E_2$ and a subsequent, lower relaxation time than under normal conditions.

### 4.4 Adhesive properties from pull-off measurements

The adhesive properties of the SSC obtained from pull-off measurements at forces between 1 and 50 mN are presented in Figure 7 for very dry (20% relative humidity; 25°C), normal (36% relative humidity; 25°C) and wet conditions. The wet conditions were considered after 2 mins of soaking the sample in accordance with frictional results on human skin which show a maximum variation of the COF (coefficient of friction) during the first 60 sec after soaking\(^{(27, 55)}\). Then, the excess of water was removed with a paper tissue. The results presented in Figure 7 indicate a clear increase of the snap-off force with the humidity.

Moreover, the adhesive forces presented a slight increase with the normal force which can be explain due to the increase of the area of contact at higher loads.

From 10 mN on the adhesive force presented more steady values that vary depending on the environmental condition. Figure 8 presents the average adhesive forces of 4 samples at 50 mN for each condition. Although the scattering is considerable, the differences between conditions are clear, especially when comparing the case of 20% relative humidity with 36% or wet conditions. At wet conditions, snap-off force presents the higher value due likely to the modification of the surface properties of the skin under the presence of water so that, changes in capillarity and adhesion appeared due to Van der Waals interactions.

Additionally, the work of adhesion for the skin samples was calculated from Equation (3) at different conditions and the results are presented in Table 6. According to these results, at very dry conditions of 20% relative humidity, the work of adhesion was considerably low at 80 mN. As the humidity increased, the work of adhesion increased almost ten-fold as it can be seen for 36% relative humidity. At wet conditions, the adhesive force indicated a highest value of -233.4 ± 7 mN/m which evidence the influence of water in the adhesive properties of the skin which can aid to explain the differences on its frictional performance. Moreover, the results obtained for the work of adhesion at the studied conditions are in the range of values obtained in previous research\(^{(36, 86)}\). The differences in the adhesive force previously presented cannot be explained in terms of JKR contact area so that, they might be a result of the role of capillary forces in the adhesion of human skin as it has been previously suggested.
4.5 Frictional performance of the SSC at two different range of forces

The frictional behaviour of the SSC samples was evaluated between 1 and 50 mN, so-called the meso scale, and from 0.5 to 3.5 N, the macro scale, for both, dry (36% relative humidity and 25°C) and wet conditions (after 2 mins of wetting the samples). These results are displayed in Figure 9 as a function of the logarithm of the normal force. At dry conditions, the COF at the meso scale showed a power law decreasing from 0.8 to 0.4 as the normal force increased.

The influence of water in the adhesive properties of the skin increased the COF to values around 2 under wet conditions. As the force increased, the COF decreased to values around 0.5. At the macro scale, the friction coefficient showed a more steady behaviour with values between 0.4 and 0.3 at dry conditions. The effect of water at the macro scale was not that relevant as for the meso scale and with a slight variation with respect to the dry case with values between 0.5 and 0.45. The results obtained for the SSC at both scales were in good agreement with the frictional results of ex vivo skin obtained in our previous research\(^{35}\). Additionally, these values were compared to in vivo skin results from the literature which indicate a COF for dry skin between 0.26 - 0.85; for wet skin the literature presents values ranging from 2.62 - 0.92 depending on the force and other boundary conditions\(^{25, 28, 59, 62}\).

5. Conclusions

The present work describes the development of a Synthetic Stratum Corneum (SSC) and its tribo-mechanical performance. To better understand the material’s performance and determine its water content and melting temperature, TGA and DSC measurements at dry and wet conditions were accomplished. The surface features of the thigh of an individual copied in the SSC were analysed under confocal microscope and the roughness parameters determined. Moreover, the hydration of the SSC samples was evaluated at 20% and 36% relative humidity and for wet samples with a good correlation with results of in vivo skin at different body places from the literature. The mechanical properties of the SSC were analysed under tensile and stress relaxation tests. The elastic modulus presented similar values to in vivo skin from the literature which supports the suitability of the SSC in terms of mechanical properties. The adhesive response of the SSC under different conditions showed the important influence of water on the surface properties of the material. Further, these values SSC were in accordance with human skin results from previous research. Lastly, the frictional performance of the SSC evaluated at the meso and the macro scale indicated a proper correlation ship with the tribological behaviour of the skin in the same range of forces. The friction coefficient at the meso scale showed was described as a power law function of force, with values of COF at 1 mN around 2 and 5 for normal and wet conditions, respectively. At the macro scale, the results were steadier as a function of the normal force with only slight differences from dry to wet conditions and a COF around 0.4 at normal conditions and 0.5 at wet conditions.

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