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Physical and mechanical properties of the hair shaft: A review of measurement techniques to verify the efficacy of cosmetic products

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ABSTRACT
Hair is a natural fiber made up of keratin, a protein that contains a high concentration of sulfur from the amino acid cystine. The hair structure is made up of three main regions: the cuticle, the cortex and the medulla. The human hair strand has some unique characteristics, such as: mechanical strength, shine, and combability. Several measurement techniques are reported in the literature to measure the mechanical properties of hair with the main purpose of proving the efficacy of cosmetic products. Some of these methods are: optical and electron microscopy, measurement of mechanical resistance through dynamometers, compression tests to measure combability, evaluation of gloss by specular reflection, among others. In this sense, this work includes a literature review of the techniques for measuring the physical and mechanical properties of the hair fiber, which are extremely important methods in the development of new cosmetic products.

Keywords: Hair, Mechanical strength, Brilliance, Combability, Measurement.

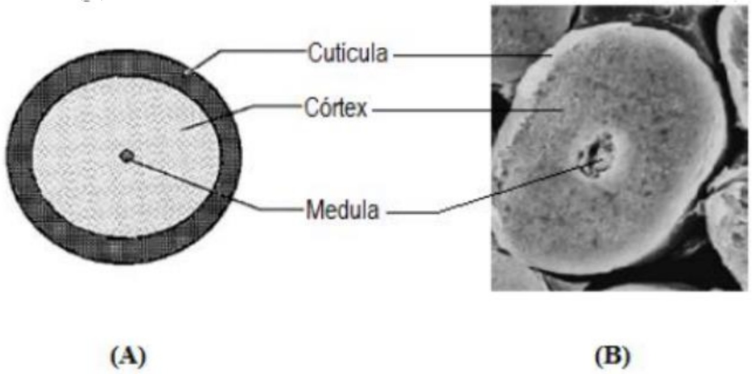
1 INTRODUCTION

1.1 MORPHOLOGY AND CAPILLARY STRUCTURE

Hair is made up of proteins called keratins, which are produced in the keratinocytes of the epithelial tissue. Small amounts of water-soluble substances are also present, such as: pentenes, phenols, uric acid, glycogen, glutamic acid, valine and leucine (WILKINSON; MOORE, 1990).

Hair is a natural polymer consisting mainly of fibrous structural proteins called keratins (NAKANO, 2006), which grow from cavities called hair follicles, where the development and keratinization of the hair occurs, that is, the transformation of hydrogen sulfide groups into disulfide bonds (ROBBINS, 2001). The hair shaft is mainly composed of 3 distinct regions: cuticle, cortex and medulla, as can be seen in Figure 1.

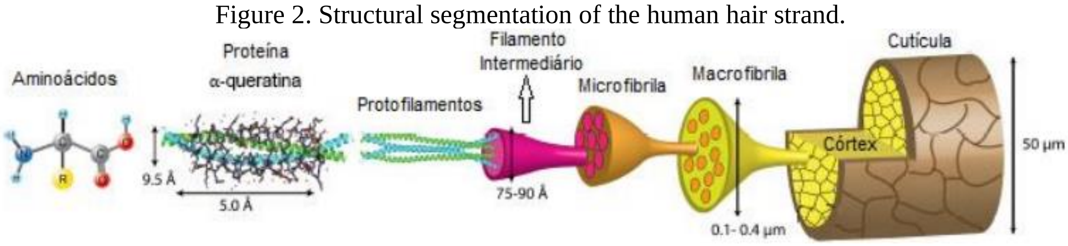
Figure 1. Schematic representation of the cross-section of a hair fiber (A) and electron microscope image of the cross-section of the human hair strand (B).



Source: (MAURICIO, 2019).

The association of the medulla, cortex, and cuticle form a hair fiber, but it is the amount of subfibrils that gives hair its unique physical properties. This segmentation of the capillary structure is shown in Figure 2. The cuticle is structurally amorphous (FEUGHELMAN, 2002), colorless and has cells in the form of "scales" that overlap (VELASCO, *et al*. 2009), and can measure from 350 to 450 nm in thickness (DRAELOS, 2000). The main function of the cuticular matrix is to protect against external aggressions, functioning as a barrier to larger molecules, in addition to promoting protection, they are transparent and responsible for brightness and sensory properties. Therefore, as it is the region of greatest contact with the external environment, this is the area that suffers the most damage to the hair fiber (FEUGHELMAN, 2002).

Figure 2. Structural segmentation of the human hair strand.



Font: (YANG, *et al.*, 2014).

The cuticle protects the cortex and medulla, which are the most delicate regions of the hair. The cortex makes up about 90% of the hair fiber (SAKAI; KIKUCHI; FUJII, 2013), with elongated structures parallel to the direction of the fiber (SANT'ANNA, 2000). This structure is composed of macrofibrils of 0.1 - 0.4 μm in diameter, which in turn are made up of smaller filaments called microfibrils. The cortex is what gives strength, flexibility, elasticity, responsible for the shape of the fiber and hair color (MAURICIO, 2019).

Composing a small percentage of capillary mass, but no less important, the medulla is the innermost part of the capillary axis and is located in the center of the fiber, which can be continuous, fragmented or completely absent, and still has a high lipid content when compared to the rest of the fiber (WAGNER, 2007). The shape of the medulla may vary, and it may be empty or filled with spongy keratin along the thread (ROBBINS; CRAWFORD, 1991). Its function is not yet completely elucidated in the literature, although its cells can become dehydrated and its spaces can be filled with air, which affects both hair color and shine (MAURICIO, 2019).

1.2 HAIR TYPES

Human hair presents a great variety in terms of its shape and color, this difference occurs due to genetic reasons that vary from individual to individual, so all this heterogeneity depends on the race or mixture of breeds inherited by the individual (HALAL, 2011). It is known that the shape of the hair determines the degree of shine and the efficiency of fat in covering the strands (oiliness). Straight hair has more shine than curly hair, allowing maximum light reflection and easier movement of fat through the fiber (DIAS, 2004). Regarding hair types, there are three different types: Asian, Caucasian and African as shown in Figure 3.

Figure 3. Classification of hair according to ethnic origin.

TIPO DE CABELO	MODO DE INSERÇÃO	SECÇÃO TRANSVERSAL	ASPECTO
Cabelo Caucasiano	Ângulo < 90ºC	Oval	Ondulados a retos
Cabelo Africano	Curvado	Elíptico	Bastante ondulados
Cabelo Asiático	Ângulo reto	Redondo	Lisos

Fonte: (PÉREZ, 2005).

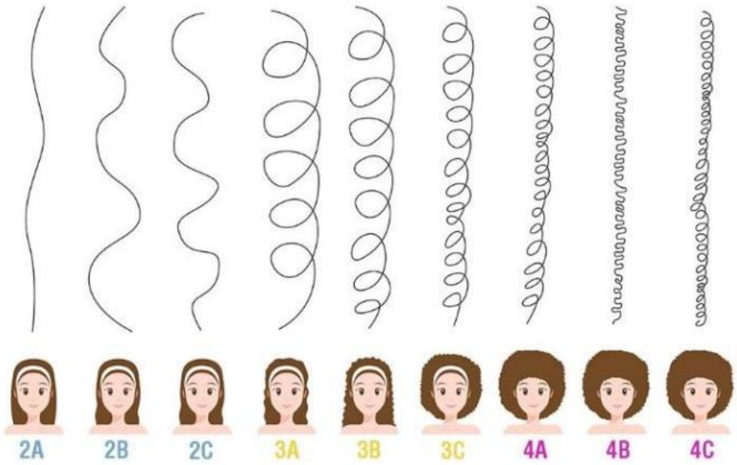
Caucasian hair has a greater variety of the three ethnic groups. The strands can have wavy or straight shapes and their fibers have a more oval shape, growing at an oblique angle to the scalp. This type of hair has a strand with a very varied diameter, but it has the smallest diameter among the three groups (DIAS, 2004).

Asian hair, on the other hand, is the hair that is born perpendicular to the scalp, thus resulting in completely straight hair. Its fibers are thicker and have a cylindrical shape, having the largest diameter among the three ethnicities. The growth rate of Asian hair is the fastest of the three groups (DIAS, 2004).

African hair is characterized by very wavy strands and, for this reason, it is drier hair, since the sebaceous glands are less active in this group. In addition, the distribution of oil throughout the fiber is uneven. This group is the one with the slowest rate of hair growth. Its fibers have an elliptical shape and also have the lowest water content (DIAS, 2004).

There is also a classification of hair in the literature that takes into account the level of curvature of the hair fiber, subdividing them into three groups: type 2 or wavy; type 3 or curly; and type 4 or curly. In addition, these classifications are subdivided into: (A) hair with looser curls, (B) more defined curls, and (C) hair with smaller, more defined curls. This classification of the wires is shown in Figure 4.

Figure 4. Classification according to the curvature of the wire from type 2A.



Fountain: (SANTOS, 2019).

1.3 PHYSICAL AND MECHANICAL PROPERTIES OF THE HAIR SHAFT

1.3.1 Mechanical resistance

Mechanical properties of hair, such as strength and elongation at breakage are used as primary indicators of hair integrity and provide information on the effect of a formulation on the hair fiber (ESTIBALITZ FERNÁNDEZ *et al.*, 2012). However, the methodologies used to measure the mechanical properties of hair are often time-consuming, require multiple measurements, and are prone to variation (TINOCO *et al.*, 2018).

The mechanical strength of the hair can be evaluated by appropriate equipment designed to measure the strength or elasticity properties of the hair fibers. For example, these characteristics can be measured using a dynamometer (RIBEIRO; MARTINS; TINOCO, 2021). The device exerts a tension force or load on the single fiber fixed to a support, breaking the fiber, which is recorded by the software, converting the information into an elongation load graph (RUETSCH; WEIGMANN, 1996).

1.3.2 Brilliance

Brightness is the ability of light to reflect through the contact of light with a given surface. The light rays falling on a surface, at an angle of incidence of 90º, promote its specular reflection. Other proportions of light radiation are scattered and detected at other angles; this reflection is called diffuse (EVANS, 2018). As the outermost surface of the hair is the cuticular region of the hair shaft, the integrity of the cuticle is decisive for the interaction of the hair with light and, therefore, directly affects the shine of the hair.

1.3.3 Combability

Hair combability can be defined as the subjective perception of the easy or difficult way to comb the hair. It is directly related to the forces opposing the action of combing the hair. This is an important attribute in the evaluation of hair conditioning (DIAS *et al*., 2005; DIAS *et al*., 2008). For the consumer, better combability reflects better hair conditioning.

Other factors related to combability involve malleability and mechanical damage that can occur when we comb our hair normally, and are aggravated when we detangle it (GARCIA, DIAZ, 1976; SYED *et al*., 1996; ISHII, 1997).

2 OBJECTIVES

Within this context, the present study aimed to carry out a literature review on the various non-invasive techniques used in order to analyze the hair fiber and the benefits that the cosmetic industry can prove from these analyses.

Thus, this work aims to bring an approach to measurement techniques and equipment used to evaluate the various physical and mechanical parameters of the hair fiber, in addition to understanding how these measures identify the various benefits communicated in hair cosmetic products for the consumer in relation to the hair shaft.

3 METHODOLOGY

In November and December 2023, scientific papers were selected through an electronic search in the following databases: Scholar Google, Scopus, Web of Science, ScienceDirect, MedLine, and PubMed. The scientific papers included in this review were: theses, dissertations, term papers, book chapters and scientific articles.

The job search process was performed using the following descriptors: "*cosmetics", "hair",* "hair care*", "mechanical resistance*", "shine*", "*gloss*", "*combability*" and "photoprotection*". These terms and their combinations were selected to perform the job search. Searches were also carried out with the same descriptors mentioned above, but in Portuguese. This search process aims to identify works specially developed in Brazil.

At the end of the search process, from the total number of papers initially tracked by the descriptors, duplicate papers were excluded by titles. Then, after reading the texts, the works that did not address the proposed theme were excluded. The reference lists of the pre-selected articles were also examined, in order to find studies with potential relevance to be added to the bibliographic reference of this work.

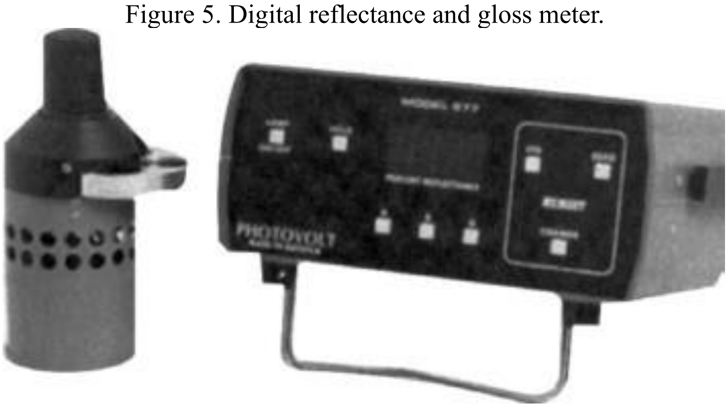
4 METHODS FOR EVALUATING THE EFFECTIVENESS OF HAIR PRODUCTS

Characterization of the structure and physical and mechanical properties of hair are essential to develop better cosmetic products and advance Biological and Cosmetic Science (BHUSHAN, CHEN, 2006).

4.1 GLOSS METERS

Glossmeters are equipment developed to measure the shine of hair. The regularity of the hair's surface helps to determine the reflection of light. When light follows a uniform surface, such as in a mirror, the angle of incidence is exactly the same as the angle of reflection. However, the hair is not completely uniform and at some points the light beam is reflected forming different angles (0 to 75º) and this type of reflectance is known as diffuse reflectance (REIMER *et al*., 1995; ISHII, 1997; SCHUELLER, ROMANOWSKI, 2001; DIAS, 2004). The measurement principle of this equipment is based on reading the reflection (whether specular or diffuse) of light by the surface of the wires. Figure 5 shows a commercially available digital reflectance and gloss meter.

Figure 5. Digital reflectance and gloss meter.



Source: (CAMPO, 2008).

The work of LEITE; CAMPOS (2018) provides a reference methodology on the use of gloss meters. The equipment used was the GL200 ® Skin Glossymeter (Courage-Khazaka), which measures the shine of the hair surface. The analyses were performed on hair samples of 5 g, one sample per group, at a temperature of 20 to 22 ºC, relative humidity (RH) of 50 to 60 % and in the absence of light to avoid interference. Nine measurements were taken, three in each region of the wire, which was divided into three regions, i.e., upper, middle and ends. The work of FARIAS; CORNÉLIO (2023) also makes use of the GL200 ® Skin Glossymeter (Courage-Khazaka) probe to evaluate the shine of hair strands treated with vegetable oils before and after chemical bleaching and straightening treatments.

4.2 MECHANICAL TESTS: TENSION AND COMPRESSION TESTS

To perform these tests, a dynamometer is used (illustrated in Figure 6) - equipment often used to evaluate: hair breakage tension, elasticity, combability and detangling. Different tests are used to measure different properties of the yarn. To assess the tensile stress of the strand, the device exerts a tensile force on the hair shaft and measures this applied force over time, until the strand breaks. The greater the force supported by the wire, the greater its mechanical resistance (RIBEIRO; MARTINS; TINOCO, 2021).

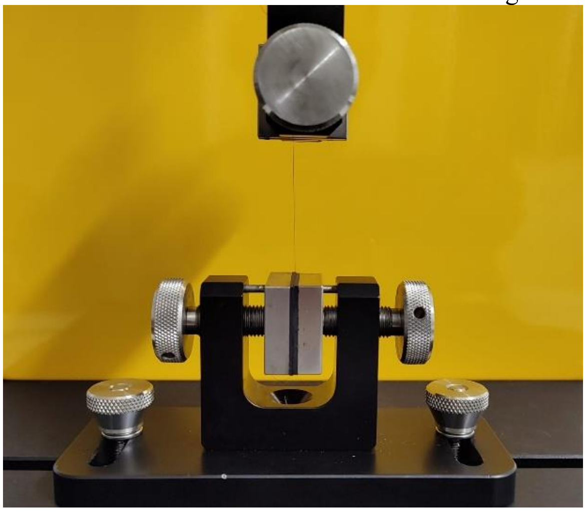
Figure 6. Dynamometer operating a breakdown stress test.

Fonte: (ERIK *et al*., 2008).

The works of RIBEIRO; MARTINS; TINOCO (2021) and TINOCO *et al*. (2018) bring mechanical testing methodologies with the use of dynamometers. A Hounsfield dynamometer was used in both studies. For these mechanical wear tests, 30 individual hair fibers with low diameter variability were selected. Elongation measurements were performed at a rate of 1.5 mm/min, with a preloaded force of 0.01 N, until fiber rupture.

Another equipment widely used to analyze mechanical parameters of tension and compression of hair strands is the Texturometer or Texture Analyzer. The work of FARIAS; CORNÉLIO (2023) studies the mechanical properties of hair fibers using Ametek-Brookfield's *Texture Analyzer CT3* equipment. In this work, the elongation measurements were performed at a speed of 2.5 mm/s, with a preload of 6.8 g. The analysis is performed until the capillary fiber ruptures (FARIAS; CORNELIUS, 2023). Figure 7 shows the Ametek-Brookfield Texturometer operating a tension test.

Figure 7. Ametek-Brookfield CT3 texturometer during tension test.



Source: (AUTHORED BY AUTHORS, 2023).

The shape and type of hair can strongly influence the behavior of hair fibers, particularly their resistance to mechanical stress (CRUZ *et al*., 2017). For example, the high curl of African hair combined with the lower resistance of its cortex to mechanical stress makes this type of hair more brittle and more sensitive to excessive handling, when compared to other hair types (WEI; BHUSHAN; TORGERSON, 2005; MCMICHAEL, 2003).

To study parameters of combability and detangling of the strands, the strand of hair is secured with a support, and then two combs pass through it. In this test, the equipment measures the compression force necessary for the comb to penetrate the strand of hair and, consequently, detangle the strands. In this case, the lower the force applied by the equipment, the easier it is for the comb to penetrate the strand and, consequently, the better the combability of the hair.

The works of GARCIA; DIAZ (1976), ISHII (1997) and DIAS *et al*. (2008) provide methodological information on the test of measuring the combability of the locks. In both studies, a dynamometer (Instron model 4464) was used with two non-metallic combs hanging on a support. The tested wick was placed on the top clip of the equipment (5.0 kg load cell). When turned on, the combs that hang from side supports comb the analyzed strand. The clip speed during the measurement procedure was 500,000 mm/min. The force required for the combs to pass through the wick was measured by the equipment and the software, Series IX Software, of the equipment itself. Texturometers can also be used in the combability analysis of the locks. The work of (LEITE; CAMPOS, 2018) uses the TXA XT Plus® Texturometer to analyze the combability of locks treated with cosmetic formulations containing vegetable oils and silicone. Figure 8 shows the Ametek-Brookfield Texturometer operating a compression test.

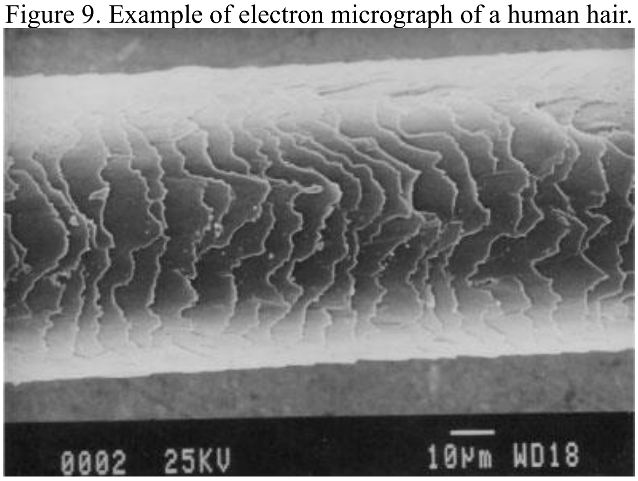
Figure 8. Ametek-Brookfield CT3 texturometer during compression test.

Source: (AUTHORED BY AUTHORS, 2023).

4.3 MICROSCOPY TECHNIQUES

Scanning electron microscopy (SEM) is widely used for hair analysis of hair strands. This technique allows observations of thick, non-transparent samples under an electron beam. It also makes it possible to determine the shape of a material, the size of the particles that compose it and their arrangement (DIAS, 2004). Figure 9 presents an example of an electron micrograph of an Afro-ethnic hair.

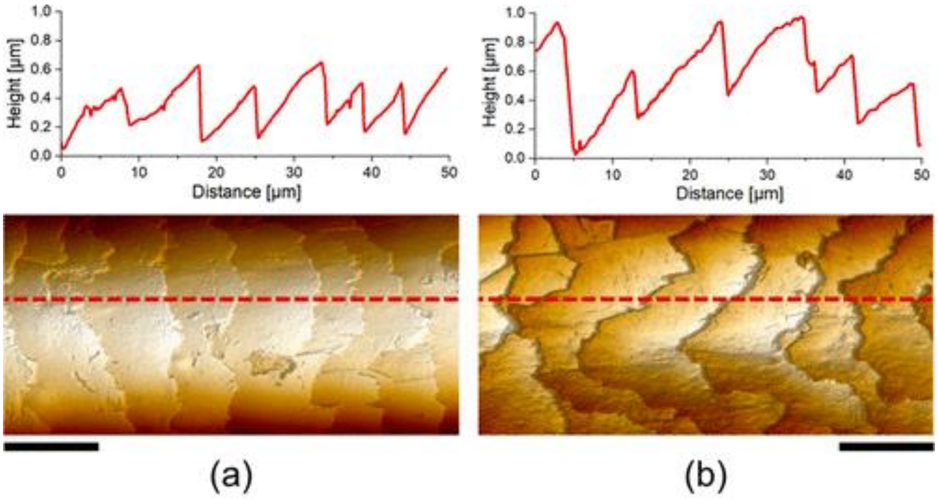
Figure 9. Example of electron micrograph of a human hair.



Source: (VELASCO *et al*., 2009).

Another widely used microscopy technique is Atomic Force Microscopy (AFM) or tunneling microscopy. This equipment allows you to observe images of samples through the microscope. The image is captured by a probe that has physical contact with the sample and follows a plane parallel to the surface while acquiring each point of the topographic component. The deflection of the probe is then measured by a computer program (software) that generates the image. Through this equipment, it is also possible to obtain quantitative data on the distribution of electrical charges and the strength of the sensor to go through the sample (SMITH, 1997; DIAS, 2004). Images of atomic force micrographs performed on strands of hair are shown in Figure 10.

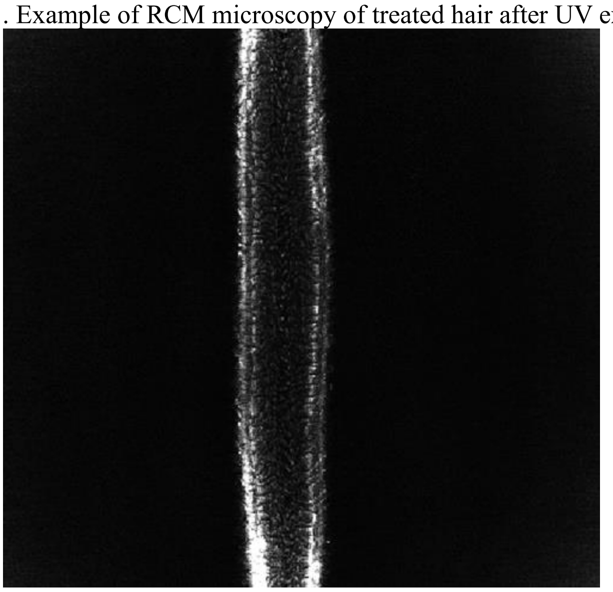
Figure 10. Atomic force micrograph images of (a) virgin hair and (b) bleached hair.



Cast iron: (LIU *et al*., 2022).

Confocal Reflectance Microscopy (RCM) is also a microscopic technique that can provide valuable information about hair fibers. The work of LEITE; CAMPOS (2018) used RCM techniques to visualize hair fibers before and after exposure to ultraviolet (UV) radiation. The microscope used was the Vivascope 1500 ®. Two hairs of each hair were evaluated for the integrity of the hair cuticle before and after irradiation with UV light. This made it possible to analyze the effect of the formulation on the surface of the hair, as well as its preventive action against UV damage caused by sun exposure. Figure 11 presents an example of an MRI micrograph of a hair fiber.

Figure 11. Example of RCM microscopy of treated hair after UV exposure.



Source: (MILK; CAMPOS, 2018).

4.4 PIEZOELECTRIC SENSORS

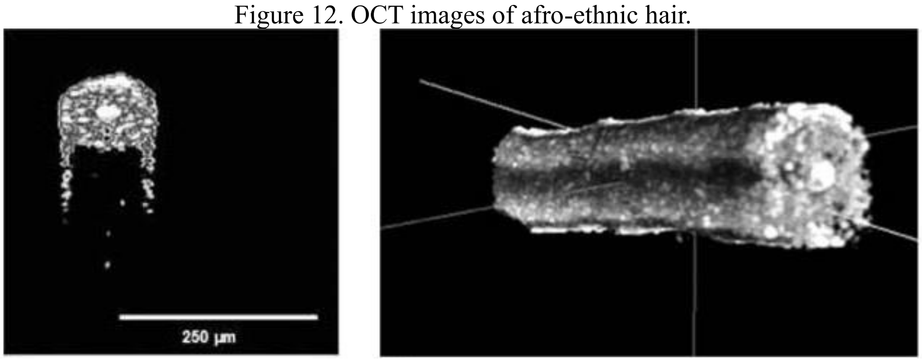
The piezoelectric principle is based on the deformation of a crystal by mechanical action. When this occurs, a load shift is induced, thus creating a voltage signal. On hair, it is possible to apply this technique to tactile perceptions of hair properties, such as: conditioning, cleanliness and surface roughness. During the evaluation, the sensor is placed on a mechanical arm that touches the section of hair and is then released. This is repeated several times. The results are expressed as arbitrary stress values (REIMER *et al*., 1995; ISHII, 1997; SCHUELLER, ROMANOWSKI, 2001).

4.5 OPTICAL COHERENCE TOMOGRAPHY

Optical coherence tomography (OCT) is a diagnostic imaging technology based on low-length coherence interferometry, in which the coherence characteristics of photons are exploited, leading to an imaging technology capable of producing non-contact and non-destructive images (FREITAS *et al*., 2008), high-resolution cross-sectional images of the internal microstructure of living tissues such as: retina, skin (HUANG *et al., 1991) and teeth (FREITAS* et al., 2006). OCT imaging is primarily based on an optical property of the sample, the backscatter coefficient. The false color in the image represents the backscatter coefficient, where the white color represents high dispersion and the black color represents low dispersion.

Figure 12 shows images of Afro-ethnic hair, where it is possible to identify the main hair structures: medulla, cortex and cuticle (FREITAS *et al*., 2008). The mean diameter of the spinal cord was 29 ± 7 µm and the diameter of the hair was 122 ± 16 µm in these samples studied in the study by FREITAS *et al*. (2008).

Figure 12. OCT images of afro-ethnic hair.



Fonte: (FREITAS *et al*., 2008).

4.6 THERMAL ANALYSIS

The study of the effect of hair products on the mechanical and thermal properties of hair is essential in the development of new formulations. In the work of TINOCO *et al.* (2018), Differential Scanning Calorimetry (DSC) measurements were performed to assess whether keratin-based particles penetrate the capillary cortex and protect the hair from thermal denaturation.

Thermal studies of the hair samples were conducted using a power-compensated DSC instrument (DSC 6000, Perkin Elmer) and aluminum pans (maximum pressure: 1 bar), in a temperature range of 50 to 250 °C (heating rate: 5 °C min−1, sample weight: approximately 2 mg). The DSC device was calibrated using high-purity indium and zinc. Each test group was measured in triplicate and mean values are reported (TINOCO *et al.* 2018).

4.7 PHOTOPROTECTIVE ACTION

Developing hair care products that are effective in protecting against ultraviolet (UV) damage has been a challenge, as some parameters for evaluating efficacy are not yet well defined (DARIO; BABY; VELASCO, 2015). The work of LEITE; CAMPOS (2018) evaluated the photoprotective effects of a multifunctional hair care formulation containing botanical extracts, vitamins, and UV filters.

Hair samples with and without application of the formulations (control) under study were subjected to UV radiation in the range of 280–400 nm emitted by a solar simulator with a 150W 96000 xenon arc lamp (Oriel Corporation, Stratford, CT) coupled to a source connected to an Oriel 81 045 dichroic mirror and an Oriel WG 305 59450 filter (LEITE; CAMPOS, 2018).

The formulations were irradiated for a period of 25 min each (with an irradiance of 12 mW cm−2), being turned in half the time to ensure the homogeneity of the irradiation process. Subsequently, comparative evaluations were carried out to verify possible damage caused to the hair by solar radiation and to evaluate whether the proposed formulation had a protective effect against this damage (LEITE; CAMPOS, 2018).

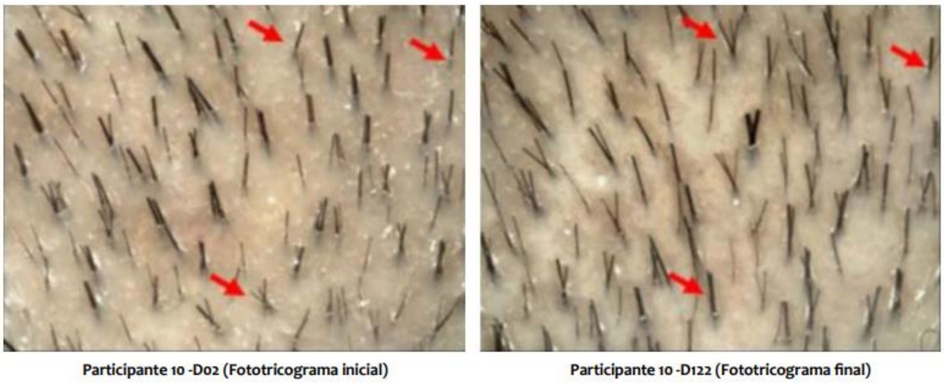
4.8 PHOTOTRICHOGRAM AND MACRO PHOTOGRAPHY

The phototrichogram corresponds to a non-invasive method in which the hair of the experimental region is shaved and an image is captured after two to three days to determine the amount of hairs in the anagen and telogen phases (SINCLAIR et al., 2011). Macrophotography, on the other hand, corresponds to the capture and comparison of paired images before and after treatment to verify the improvement in hair growth (BLOCH; SQUIRE; SARRUF, 2018).

The work of BLOCH; ESCUDEIRO (2020) provides methodological information on the use of phototrichogram and macrophotography techniques applied in hair loss studies. In BLOCH's work; ESCUDEIRO (2020), for the phototrichogram analysis, the site in the frontoparietal region was selected for standardized scraping of the hairs, with an area of 2 cm2. Then, the participants attended again after two days to capture images of the wires with the Dermoscope Dynamic® equipment (FotoFinder Systems, Inc., Maryland, USA), using 20-fold magnification. This procedure was performed prior to treatment (baseline visit) and was repeated after 120 days of use of the investigational product.

In both visits, the analysis took place in the same region. The captured images were analyzed using the Image Pro Premier® program (Media Cybernetics, Rockville, USA) to compare the total area with hair loss between the experimental times. An example of an image from the phototrichogram analysis developed in this work is illustrated in Figure 13. In this study, we evaluated the efficacy of a capillary ampoule containing human hair follicle stem cells in reducing hair loss in women affected by androgenetic alopecia (BLOCH; ESQUIRE, 2020).

Figure 13. Image of the phototrichogram analysis at different times for patients in the treated group (participant reference: 10). The arrows indicate the increase in the number of wires.



Fonte: (BLOCH; ESCUDEIRO 2020).

4.9 EVALUATION OF THE EFFECTIVENESS OF COSMETIC HAIR CARE PRODUCTS

Evaluating the effectiveness of hair products may involve the use of devices that often have high sensitivity. These tests are specific and provide information only about one attribute of the assay. Usually, equipment is used to obtain the image, which is evaluated subjectively (VELASCO *et al.*, 2009).

The analyses should be performed in pre-defined regions of the strands to standardize the method and obtain more reliable results. The advantages of these techniques, when compared to merely subjective evaluations, are:

- There is no need for a panel of volunteers;
- Some assessments can be performed quickly;
- Use of specific wicks;
- Standardized test condition;
- They can be used for complex studies.

5 FINAL THOUGHTS

The results obtained in this review show that several analytical techniques can be applied to verify the efficacy of hair cosmetic products. This work also addresses several methodologies that can be used to analyze product performance, such as: increase in hair shine, increase in mechanical resistance, improvement in combability, reduction of hair loss, photoprotective action, among others.

The development of new cosmetic products depends on these analysis techniques in order to properly prove the efficacy of the products and components present in the formulation. This work is of paramount importance for companies and industries and also for Universities and Research Centers that work in the areas of Research, Development and Innovation (RD&I) in the cosmetics sector.

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