



## Study of the shape and angularity of abrasive particles, vegetable and mineral, to be incorporated into solid soap with exfoliating action: Kaolin, bentonite, rice microspheres, bamboo and apricot seed

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### **Jumara Aparecida Batista Gonçalves**

MSc student in metallurgical and materials engineering  
Polytechnic School (Department of Metallurgical and  
Materials Engineering) - University of São  
Paulo, Sao Paulo, Brazil

### **Gilmar de Oliveira Pinheiro**

Ph.D student in metallurgical and materials engineering  
Polytechnic School (Department of Metallurgical and  
Materials Engineering) - University of São  
Paulo, Sao Paulo, Brazil

### **Francisco Rolando Valenzuela Diaz**

Ph.D chemical engineering  
Polytechnic School (Department of Metallurgical and  
Materials Engineering) - University of São  
Paulo, Sao Paulo, Brazil

### **ABSTRACT**

This research work presents a contribution to the areas of cosmetology and materials, focused on the use of natural products as exfoliating agents in solid soap.

It consists of a literature review on the use of different exfoliating agents in cosmetics, a field survey to demonstrate the availability of these

products in commercial stores, and tests to characterize their abrasive particles.

The characterization in terms of shape and angularity of the abrasive particles of the selected exfoliating agents, was obtained through the Scanning Electron Microscopy-SEM tests, using the Scanning Electron Microscope - FEI Inspect 50™, of Particle Size and Shape Distribution.

This research work consists of presenting the analysis of images of exfoliating agents, obtained through different methods, with the objective of classifying them according to their shape and angularity, which is one of the characteristics to be considered in an abrasive particle.

The results obtained were in agreement with the literature in terms of shape and angularity, normally found in abrasive particles, as well as satisfactorily demonstrating the hypothesis of the use of kaolin, bentonite, rice microspheres, bamboo and apricot seed as exfoliating agents to be incorporated into solid soaps, motivating the expansion of research regarding the observation of their performance, but in this case with a tribological look on the subject.

**Keywords:** Solid Soap, Abrasive Particles, Tribology.

## **1 INTRODUCTION**

Soap is found in several solid and liquid forms, its production occurs by the chemical reaction between a fat and an alkali, resulting in soap and glycerol.

A careful selection and choice of materials for soap production has a significant impact on the finished product. Caustic potash will produce softer potassium soaps, and caustic soda will produce a harder sodium soap. Attention should also be paid to oils and fats as well as to additives that have the function of assigning color, odor or some other specific function, as well as exfoliating the skin. In addition to the care with the selection, mentioned above, it is worth mentioning that the pre-preparation of the raw materials will also have an influence on the color and odor of the finished soap. Although production methods and techniques may have changed over time, the basic chemistry of soap remains largely unchanged.<sup>(1-3)</sup>.

In a more comprehensive language, the term "soap" is attributed to products for general cleaning, while the term "soap" has assumed a more functional definition, used to refer to personal hygiene articles in general.<sup>(2)</sup>

It is observed that cosmetic products with exfoliating function has been gaining space in the market, which can be evidenced by the offer of numerous options of these articles. Exfoliating products can be found in the form of creams, lotions, gels, liquid soaps, body or facial oils and bar soaps, they offer the benefit of exfoliating the skin bringing the feeling of freshness, repair and firmness through the removal of more aged skin, traditionally known as dead skin.

Facial exfoliation can be performed through mechanical, chemical or enzymatic actions, can be classified according to its action and details of operation, such as: Gommage, Scrub, Exfoliation and peeling. The mechanical action of exfoliation is more related to a form of gentle abrasion. For this method, abrasives considered light are used, and several products can be used, such as: ground fruit pits, coffee powder, andiroba bagasse, clays, apricot seed microspheres, rice and bamboo, among others.<sup>(4-6)</sup>

In order to observe the use of abrasive vegetable and mineral particles as exfoliating agents, it was necessary to resort to the knowledge available in the area of science known as tribology. In general terms, tribology can be defined as the study of friction, lubrication, and wear.<sup>(7)</sup> Wear is one of the main focuses of study of tribology, and four modes of wear are traditionally accepted: adhesive wear, fatigue wear, corrosive wear and abrasive wear.<sup>(8,9)</sup>

In abrasive wear, the removal of material from the surface occurs by the action of abrasive particles, which theoretically corresponds to the case of the facial exfoliation process.

The shape of the abrasive particle is an important property to consider when it comes to abrasive wear. Therefore, an effort to explore the shape of the particles to be used as exfoliating agents is of considerable importance. Thus, this study aims to present the form of the exfoliating particles most commonly used in cosmetic products and compare with the recommendation found in the literature. The natural products selected for the study were: Kaolin, Bentonite, Rice Microspheres, bamboo and apricot seed.

A vast search of the literature on the subject was necessary to understand the development, approach and definitions applied in this work. Thus, the themes explored address soap production, skin cell structure and renewal, use of exfoliating products, exfoliating agents and tribology.

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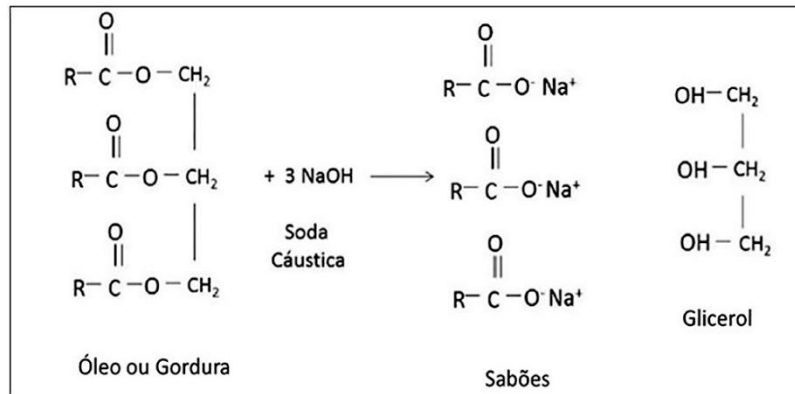
#### **A look at development**

*Study of the shape and angularity of abrasive particles, vegetable and mineral, to be incorporated into solid soap with exfoliating action: Kaolin, bentonite, rice microspheres, bamboo and apricot seed*

## 1.1 SOAP PRODUCTION

The production of soap occurs by the saponification reaction that occurs between oils or fats, of animal or vegetable origin, and an alkali such as caustic soda. The result of this reaction is the formation of soap and glycerol, as illustrated in Figure 1.

Figure 1. Illustration of the saponification reaction for soap production.



Source: Souza<sup>(10)</sup>. - Adapted by the author.

The production of soap can be carried out from three manufacturing methods, which can be applied on a small or large scale. These methods include the semi-boiling process, the complete boiling process, continuous process and cold process. Among the processes mentioned, the best known is the *Cold Process*, which is the most common batch process. This process consists of adding to the fats an amount of caustic soda solution sufficient to ensure complete saponification. The mixture is kept under stirring until it reaches the "trace" point. At this point the dough acquires a gelatinous aspect, at which time dyes, perfumes and additives are added, as illustrated in Figure 2. The trace point is the moment when the mixture of oils, fats and caustic soda solution emulsify and begin to thicken. In the cold process is not included the step of separation of the glycerin that is produced, being thus maintained in the product. After addition of the additives, the mixture is poured into molds, as illustrated in Figure 3, and kept in a dry and cool place, away from light for drying for approximately thirty days. During this period, the pH of the soaps should be monitored and can be measured with a pH indicator tape, as illustrated in Figure 4.

Figure 2. Illustration of the *Trace* point of the mixture of oils, fats and caustic soda, during the saponification reaction.



Source: The author (2023).

Figure 3. Illustration of the mixture poured into molds.



Source: Soap Queen©. Available in: <https://www.soapqueen.com/bath-and-body-tutorials/tips-and-tricks/adding-honey-to-cold-process-soap-tips-tricks-recipe/>

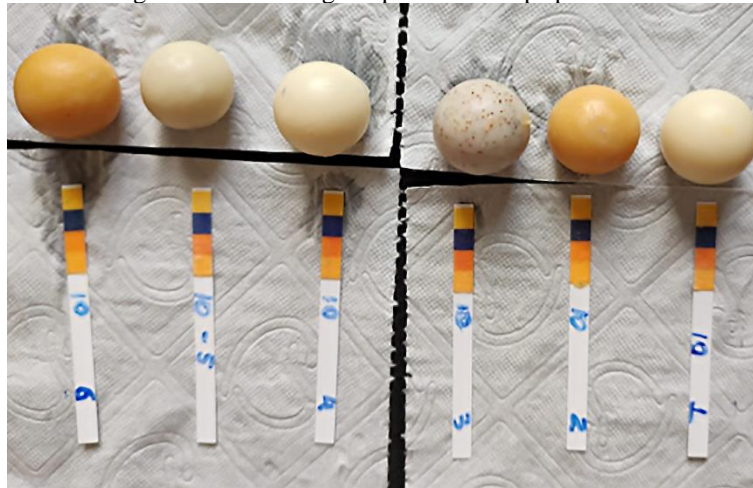
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#### A look at development

*Study of the shape and angularity of abrasive particles, vegetable and mineral, to be incorporated into solid soap with exfoliating action: Kaolin, bentonite, rice microspheres, bamboo and apricot seed*



Figure 4. Monitoring the pH of the soaps produced.

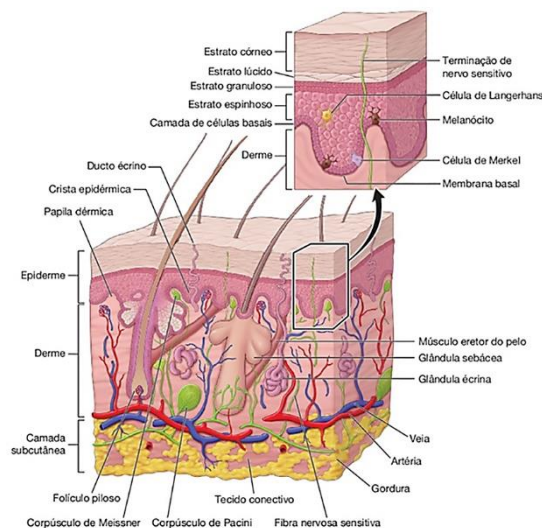


Source: The author (2022).

## 1.2 SKIN, CELL RENEWAL AND THE USE OF EXFOLIATING PRODUCTS

As defined by the authors Soutor and Hordinsky<sup>(11)</sup>, Cestari<sup>(12)</sup> and Li<sup>(13)</sup> The human skin is an organ of complex composition of its own structure, consisting of components with well-defined structure and function and is divided into three distinct layers: epidermis, dermis and hypodermis. The stratum corneum is located in the epidermis and performs a semipermeable barrier function. The stratum corneum presents a structure similar to a construction of the type "bricks" (hardened stacked cells) and "mortar" (Ceramides, cholesterol and fatty acids), as illustrated in the Figure 5.

Figure 5. Representation of the structure of human skin.

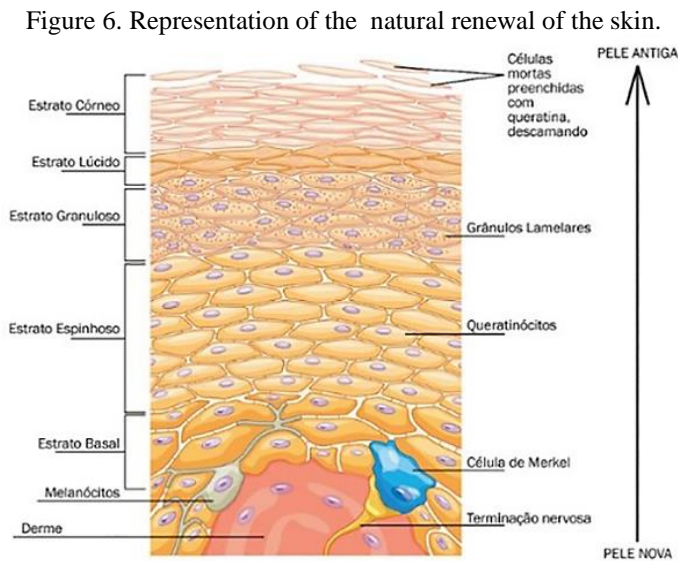


Source: Soutor and Hordinsky.<sup>(11)</sup>

### A look at development

*Study of the shape and angularity of abrasive particles, vegetable and mineral, to be incorporated into solid soap with exfoliating action: Kaolin, bentonite, rice microspheres, bamboo and apricot seed*

The formation of new skin and the scaling of old skin naturally tend to remain in a state of balance as the seasons and environment change. This replacement can slow down and the stratum corneum become thicker, which makes it difficult for cosmetics to penetrate and results in thick, rough skin. This effect can be minimized by treating the skin with the use of an exfoliating product, which has the function of removing dead cells from its surface, accelerating the renewal of new cells.<sup>(14)</sup>. Figure 6 represents the skin's natural renewal mechanism.



Source: Souza.<sup>(15)</sup> - Adapted by the author.

### 1.3 EXFOLIATING AGENTS

In general, exfoliating products when composed of solid particles are classified as mechanical exfoliants. In the production of mechanical exfoliants there are several natural alternatives for variation of exfoliating agents and can be found products that use ground fruit pits, coffee powder, andiroba bagasse, clays, apricot seed microspheres, rice and bamboo, among others.<sup>(4-6)</sup>.

Some authors have published studies that present studies on the exfoliating action provided by some of these products. In these works the authors made some relevant considerations on the subject, such as:

- Barel, Paye and Maibach<sup>(16)</sup>, state that exfoliation by mechanical abrasive action results in a fresh and smooth skin and suggest that formulators opt for clays that have softer particles, as this will provide a smoother exfoliation.
- Baran and Maibach<sup>(5)</sup>, state that ground fruit pits provide a more abrasive exfoliation because of their rough-edged particles, while more spherical and smooth particles produce a smoother exfoliation.

#### A look at development

*Study of the shape and angularity of abrasive particles, vegetable and mineral, to be incorporated into solid soap with exfoliating action: Kaolin, bentonite, rice microspheres, bamboo and apricot seed*

- c) Sun and Parr<sup>(17)</sup> share the concern of the authors Barel, Paye and Maibach<sup>(16)</sup> e Baran e Maibach<sup>(5)</sup> as for the hardness and shape of the particles. In their work "Formulation of Body Exfoliants" they demonstrate the same concern when they state that abrasives should be evaluated based on three factors: hardness, particle dimensions and particle shape. The authors warn of effects such as rough sensation on the skin, which can cause irritation and sometimes injury, when exfoliants formulated with larger, hard, irregularly shaped abrasives are used.
- d) Rocha and Oliveira<sup>(4)</sup>, published an article with the aim of exploring the development of an exfoliating cream formulated with coffee powder. Due to the tests carried out regarding the sensory evaluation and physicochemical evaluation of their formulations, they concluded that Coffee powder can be used in body scrub formulas aiming at the production of cosmetics in a more sustainable way.
- e) Feather<sup>(6)</sup>, with the article titled "*Exfoliating Agents for Skincare Soaps Obtained from the Crabwood Waste Bagasse, a Natural Abrasive from Amazonia*", present the bagasse of andiroba as an exfoliating agent. The author concluded that this raw material in question has the potential to be added to soaps as an exfoliating agent because it presents a high heterogeneous particle size distribution, with dimensions compatible with the exfoliating agents added to commercial soaps, as well as the surface of the particles presented characteristics similar to those of other exfoliating agents of plant origin.
- f) Martin<sup>(18)</sup>, in his title article "*Clay minerals: Properties and applications to dermocosmetic products and perspectives of natural raw materials for therapeutic purposes—A review*", bring as justification for the use of clays in cosmetics the peculiarity of their physical and chemical properties. The authors state that clay minerals, taking into account certain specifications, may have different applications in pharmaceuticals and dermocosmetics, including use as an exfoliating agent.
- g) Zague<sup>(19)</sup>, Shigueyama<sup>(20)</sup> Baby<sup>(20)</sup> Give<sup>(21)</sup>, Funck<sup>(22)</sup> and Saints<sup>(23,24)</sup> Claim In his works that: i) clays are natural actives and are among the numerous possible ingredient options for face mask formulas. ii) Clay face masks are formulations that can contain more than twenty-five percent solid particles dispersed in a liquid. iii) Clays are one of the most used natural actives in facial mask formulations with exfoliating action. iv) The sensation of physical exfoliation, observed after its use, is due to the hardening and contraction of the clay mask and v) because they have the requirements required for the formulation of face masks, Kaolin and Bentonite are commonly used for this purpose.

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#### A look at development

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- h) Li, Zhai, Pang, Kong e Zhou<sup>(13)</sup>, stated, in their work on the influence of the exfoliating facial cleanser on the biotribological properties of human skin, that the properties of the exfoliating particles used in these products play important roles in the tribological behavior of the skin, by removing dead skin from its surface, facilitating the penetration of cosmetic products and improving their appearance.

#### 1.4 EXFOLIANTS AND TRIBOLOGY

In order to observe the use of some natural products as exfoliating agents, it was necessary to resort to the knowledge available in the area of science known as tribology. The term tribology comes from the Greek, where Tribe means to rub and Logos means study. In general terms, tribology can be defined as the study of friction, lubrication, and wear. <sup>[7,25]</sup>.

According to Kato and Adachi <sup>(8)</sup>, Peterson <sup>(9)</sup> and Suguio<sup>(25)</sup>, wear is considered as one of the main focuses of study of tribology, being traditionally accepted four modes of wear: adhesive wear, fatigue wear, corrosive wear and abrasive wear. In the case of abrasive wear, the removal of material from the surface occurs by the action of hard particles, which theoretically corresponds to the case of the facial exfoliation process, where the removal of dead skin takes place. Also according to the authors cited, abrasive wear occurs, among other aspects, due to the shape and angularity of the abrasive particle.

The shape of a particle can be described, by a numerical factor that scales the deviation of its geometry with respect to a regular solid.<sup>(26,27)</sup> The shape of a particle is based on the measurement of its perimeter and the area of its two-dimensional projection, commonly obtained by optical microscopy.<sup>(7)</sup> Due to the evolution of image analysis techniques and software, new methods of particle shape characterization have been developed, one of these methodologies is the dynamic image analysis CAMSIZER™. This method allows the dynamic analysis of images during the processing of particles, allowing the rapid determination of their shape. Thus, through the analysis of the images it is possible to determine the aspects related to the morphology of the particles, the most common being the sphericity and the relation of aspects. Sphericity represents the irregularity of the projected perimeter of a particle, which is represented by values that the closer to 1 (one) the more uniform its surface will be and the closer to 0 (zero), the more irregular. For the interpretation of the aspect ratio (b/l) of a particle, the lower the values of b/l, the more elongated the particle will be.<sup>(28)</sup>

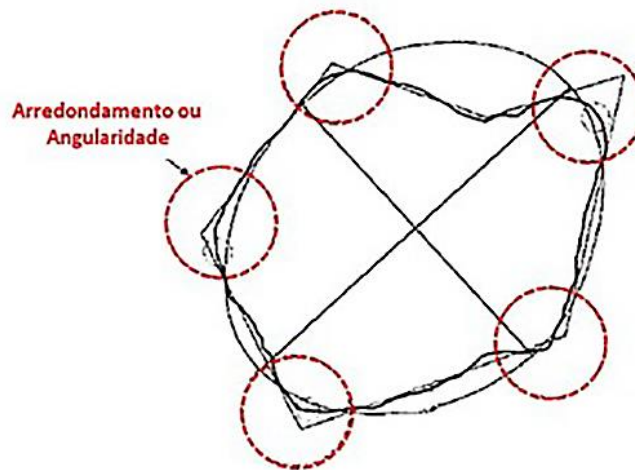
The angularity, or roundness, reflects variations in the corners of the particle, as illustrated in Figure 7.<sup>(29)</sup> The degree of angularity of a particle can be determined by the method of visual comparison, which consists of examining the particles and comparing them with images of particles organized into classes according to different degrees of rounding. The images of the particles to be



analyzed should be obtained in stereoscopic magnifying glass and then compared with the matrix of images illustrated in Figure 8.<sup>(30)</sup>

Saguio<sup>(25)</sup>, warns that in the case of the method of visual comparison, in order to avoid reading errors for classification of particles, attention should be directed to observation of their corners checking if they have angular aspect.

Figure 7. Particle contour (solid line) with its rounding component element (dashed circles).



Source: Barrete<sup>(26)</sup>. –Adapted.

Figure 8. Rounding degrees. (according to Russell, Taylor and Pettijohn. In: German Müller, 1967).



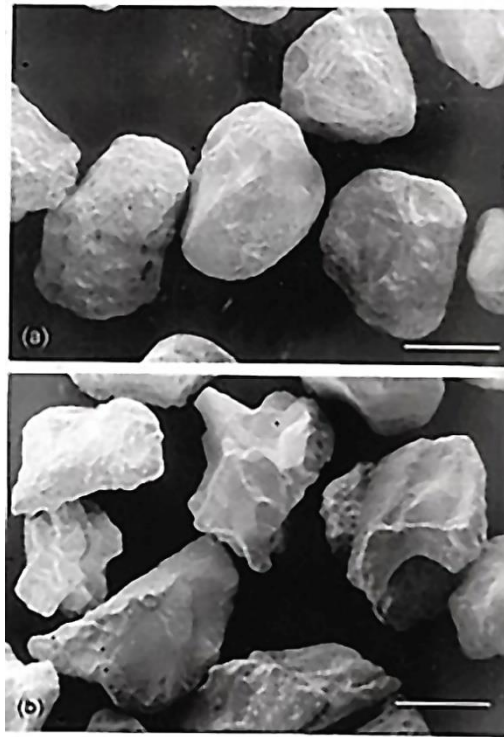
Source: SAGUIO<sup>(25)</sup> -Adapted

According to Saguio<sup>(25)</sup>, with the use of the image matrix, in a qualitative way, it is possible to characterize the degree of individual rounding of each particle considering the properties presented by Pettijohn<sup>(28)</sup>.

- (a) Particle with angular aspect: It presents sharp corners and large strongly defined concavities and small concavities smoother and less numerous.
- (b) Particle with subangular aspect: It presents a principle of wear of the corners, the larger concavities are well defined, but the small concavities are smoother and in smaller numbers.
- (c) Subrounded particle: It has well-rounded corners, large weakly defined concavities, small concavities in smaller numbers and gently rounded.
- (d) Rounded particle: It has gently rounded corners, large concavities only suggested and small concavities are absent.
- (e) Particle with Well Rounded aspect: It presents constructed surface of widely open curves, it is possible to recognize traces of the original shape of the particle and its contour is uniform.

Most of the particles responsible for abrasive wear are approximately equiaxial, but there can be significant variation in their angularity, which causes greater wear when compared to rounded ones. However the angularity is not simple to define, in general it is not measured quantitatively because of the difficulty of identifying and quantifying the characteristics of a complex three-dimensional shape, which are responsible for its abrasiveness, as shown in Figure 9 that illustrate two forms of particles.<sup>(7)</sup>

Figure 9. Scanning Electron Microscopy: (a) Rounded (b) Angular. (Courtesy of A.J.Sparks. Scale bar 100  $\mu$ m).



Source: HUTCHINGS<sup>(7)</sup>.

#### A look at development

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## 2 MATERIALS AND METHODS

For the research was carried out field survey, to search for cosmetic products that have exfoliating action as well as the agent used for this purpose.

We selected 5 types of exfoliating agents, two of the mineral type and three of the vegetable type. In the selection of exfoliating agents, the frequency of their use in commercial products and the ease of acquisition were considered.

Image assays to analyze the shape of the exfoliating particles were performed in order to compare the results with the requirements mentioned in the literature review.

### 2.1 FIELD SURVEY

The field survey was carried out in several stores of cosmetic products, in the region of Osasco-SP, in order to search for commercial products with exfoliating action and identify the abrasive agents used in their composition, according to their labels.

### 2.2 SCANNING ELECTRON MICROSCOPY ASSAY

#### 2.2.1 Materials and Equipment for Scanning Electron Microscopy Assay

##### *Materials*

- 1 gram of Kaolin Horri
- 1 gram of Bamboo Microspheres
- 1 gram of Microspheres Apricot seed
- 1 gram of Rice Microspheres
- 1 gram of Bentonite

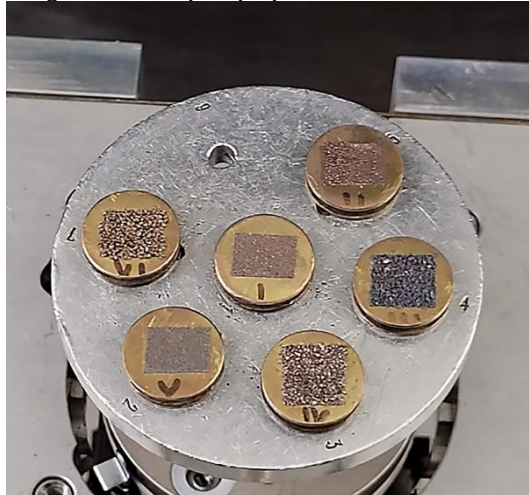
##### *Equipment*

Scanning Electron Microscope - FEI Inspect 50™

#### 2.2.2 Method for SEM-Scanning Electron Microscopy Assay

Through the SEM assay-Scanning Electron Microscopy it was possible to analyze the microstructure of all samples. For materials that are opaque to visible light, as is the case with the samples under analysis, only the surface can be observed, so it needs to be carefully prepared in order to reveal the details of the microstructure. The samples were prepared by covering them with a thin layer of gold, as shown in Figure 10.

Figure 10. Samples prepared for the SEM test.



Source: The author (2023).

The test was carried out at Labmicro in the Laboratory of Electron Microscopy and Atomic Force of the Department of Metallurgical and Materials Engineering, Polytechnic School of the University of São Paulo — PMT/EPUSP. The images of the surfaces of the samples were obtained using the Scanning Electron Microscope with Field Effect Cannon - FEG - Inspect 50, with secondary and backscattered electron detectors, EDS X-ray spectrometer and EDAX backscattered electron diffraction chamber EBSD - TEAM.

## 2.3 PARTICLE DISTRIBUTION AND FORM TEST

### 2.3.1 Materials and Equipment for Particle Distribution and Form Testing

#### *Materials*

- 5 grams of Kaolin Horii
- 3 grams of Bamboo Microspheres
- 5 grams of Apricot Seed Microspheres
- 5 grams of Rice Microbeads
- 5 grams of Bentonite

#### *Equipment*

CAMSIZER XT®- in X-Jet accessory

### 2.3.2 Method for Particle Distribution and Form Test

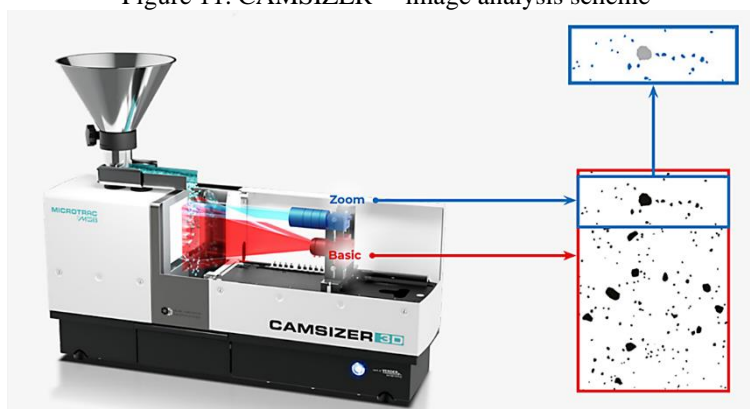
The test was carried out at the Laboratory of Technological Characterization – LCT of the Department of Mining and Petroleum Engineering of the Polytechnic School of the University of São Paulo — PMI/EPUSP.



The determination of the size and shape distribution of particles was performed by dynamic image analysis in the Retsch equipment, CamsizerXT model in X-Jet accessory with pressure of 100kPa; according to the procedure described by ISO 13322-2/2006.

The Camsizer XT® equipment consists of optical-electronic instruments for measuring the size and shape of particles by means of image analysis; the particles are analyzed with high resolution and individually, as illustrated in Figure 11.

Figure 11. CAMSIZER™ image analysis scheme



Source: Microtrac Retsch GmbH. Available at: <https://www.microtrac.com/products/particle-size-shape-analysis/dynamic-image-analysis/>

### 3 RESULTS AND DISCUSSIONS

#### 3.1 FIELD SURVEY

The research consisted of locating the products that had exfoliating action, consulting their labels in order to identify the type of agent used for this purpose. Thus, only products with exfoliating agents of mineral or vegetable origin were selected. The data were plotted in a spreadsheet and analyzed to identify the most common, and easily acquired, agent so that a solid soap could be manufactured with this incorporated agent. The result is shown in Table 1.

Table 1-Description and type of exfoliating agent in commercial product according to the label.

Product type	Exfoliating agent
Bar soap	Green clay (kaolin)
Bar soap	Bentonite clay
Face mask	Clay (kaolin)
Face and body mask	Clay (kaolin) + guarana seed powder + açai pulp powder
Face mask	Green clay (in nature) + Colágeno (in nature)
Face mask	Copper-rich green clay
Face mask	Kaolin +Carbon Black Cosmetic Pigments
Facial Exfoliating Cream	Apricot Seed Powder
Bar Soap	Apricot seed microspheres

#### A look at development

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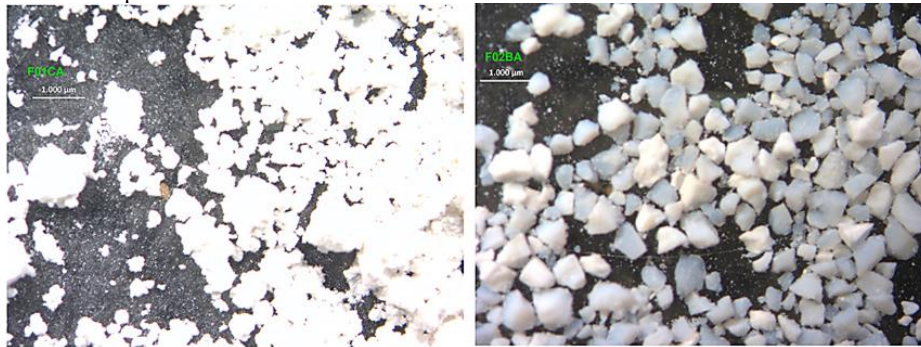
Bar Soap	Crushed passion fruit seed
Bar Soap	Bamboo charcoal
Bar Soap	Quartz Crystals
Bar Soap	Coffee powder
Bar Soap	Ground rice
Bar Soap	Ground rice
Detox Mask	Kaolin + Bentonite + Macorrina Clay

After analyzing the exfoliating agents found in commercial products and taking into account the cost and ease of acquisition, it was decided to analyze the kaolin and bentonite clays of mineral origin and the microspheres of bamboo, rice and apricot seed of vegetable origin.

### 3.1.1 Image Analysis – Optical Microscopy

Using the equipment Carl Zeiss <sup>TM</sup> Stemi 2000-C Stereo Microscope of the Clay Laboratory – LARG of the Department of Metallurgical and Materials Engineering of the Polytechnic School of the University of São Paulo – PMT-EPUSP, the images shown in Figure 13 were obtained.

Figure 13. Image of the samples of the exfoliating agents to be analyzed. 1000  $\mu\text{m}$  scale bar for F01Kaolin, F02Bamboo, F03Apricot samples and 500  $\mu\text{m}$  scale for F04Rice and F05Bentonite.



F01Caulim-Microscópio Estéreo, ampliação Total: 2,5x.

F02Bambu-Microscópio Estéreo, ampliação Total: 2,5x.



F03Apricot-Microscópio Estéreo, ampliação Total: 2,5x.



F04Arroz-Microscópio Estéreo, ampliação Total: 2,5x.



F05Bentonita-Microscópio Estéreo, ampliação Total: 2,5x.

Source: the author.

- F01Kaolin: the image shows clusters, typical of this type of clay. Despite the attempt to improve the resolution, in this image it was not clear the shape of the particles, making evident the need to use another method to analyze their shape.
- F02Bamboo: the image shows particles of well-defined shape. The particles are loose, which facilitated the observation of their shape, it was also possible to observe a sharp angularity, with corners and tips in evidence.
- F03Apricot: the image shows particles of well-defined shape. The particles are loose, which facilitated the observation of their shape, it was also possible to observe the presence

#### A look at development

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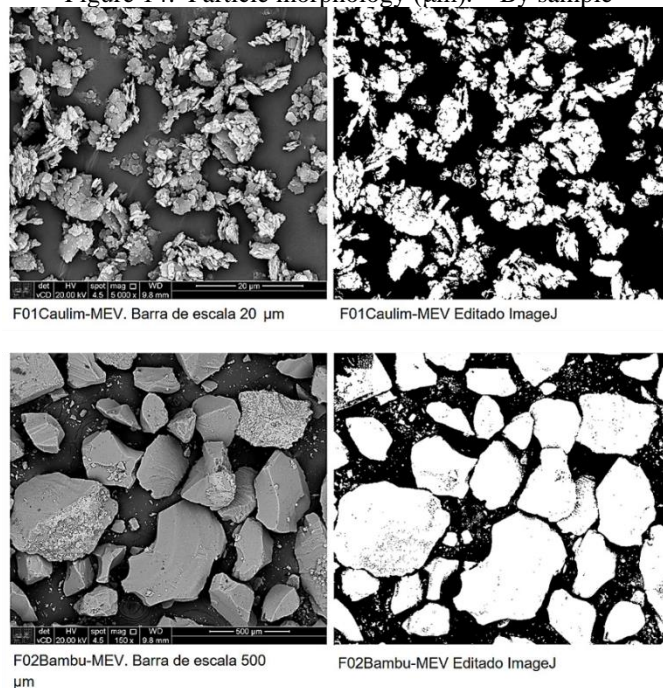
of a certain angularity, but their corners and tips do not appear to be as sharp or sharp as those of the sample F02Bamboo. The tips and corners have a rounded appearance.

- d) F04Rice: the image presents particles of shape that suggests the presence of tips and corners. The particles are loose and translucent in appearance. The fact that they are loose facilitated the observation of their shape, but the fact that they are translucent made it difficult to control the light and consequently the observation of their corners and tips, and it is not possible to say how angular they can be. Thus, it is evident the need to apply another method to analyze its angularity.
- e) F05Bentonite: the image presents small clusters, with an aspect that refers to a fine, loose and soft powder, typical for this type of clay. The image did not provide sufficient clarity to analyze the shape of the particles, requiring the use of another method to perform this analysis.

### 3.2 SCANNING ELECTRON MICROSCOPY ASSAY - SEM

In order to observe the morphology of the particles and thus identify the parameters of shape and angularity, the SEM - Scanning Electron Microscopy assay was performed, according to Figures 14 and 15. For better visualization the images were analyzed and treated in the public domain ImageJ 1.54d software developed at the *National Institute of Health* of the United States, based on the Java® programming language.

Figure 14. Particle morphology ( $\mu\text{m}$ ). – By sample



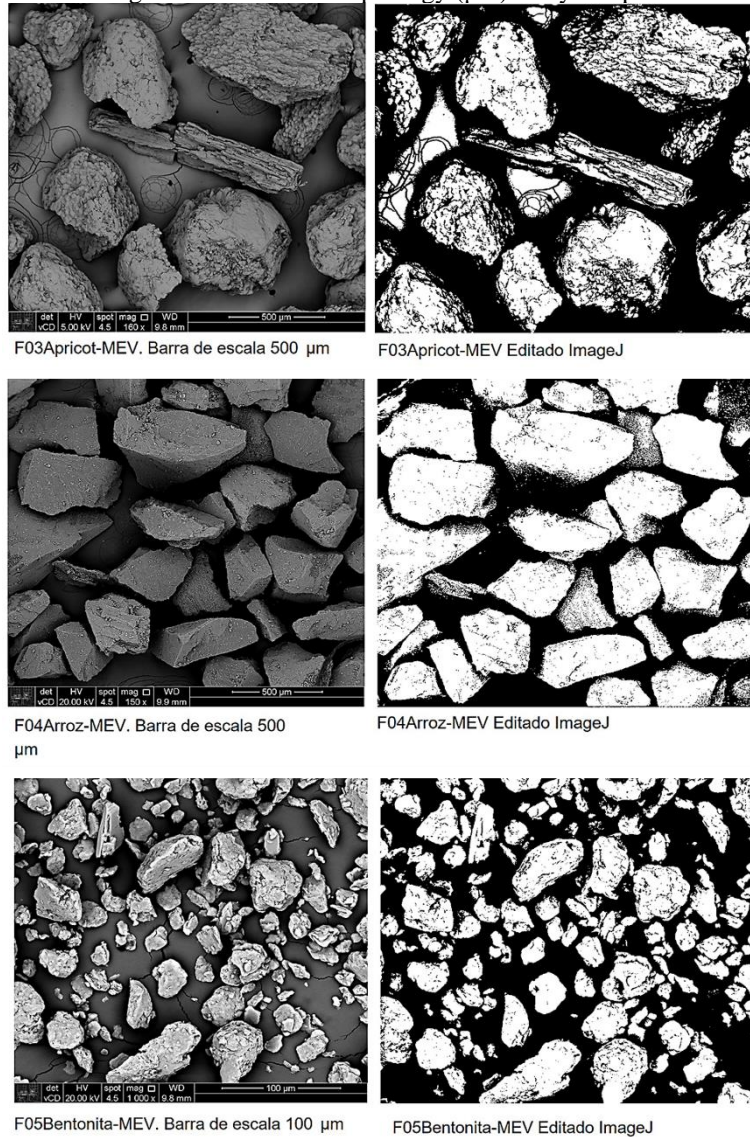
Source: the author.

#### A look at development

*Study of the shape and angularity of abrasive particles, vegetable and mineral, to be incorporated into solid soap with exfoliating action: Kaolin, bentonite, rice microspheres, bamboo and apricot seed*



Figure 15. Particle morphology ( $\mu\text{m}$ ). – By sample



Source: the author.

- a) Kaolin: its particles have the appearance of clusters, but it is possible, qualitatively, to verify that they have some angularity, based on their degree of rounding, thus being able to be classified as a subangular particle, according to criteria presented by Russell and Taylor<sup>(31)</sup> and Pettijohn<sup>(28)</sup> and illustrated in Figure 8.
- b) Microspheres of Bamboo: its particles have a more defined shape, presenting expressive corners. Qualitatively it is possible to classify them as an angular particle, according to criteria presented by Russell and Taylor<sup>(31)</sup> e Pettijohn<sup>(28)</sup> and illustrated in Figure 8.
- c) Apricot seed microspheres: its particles do not present expressive corners. In a qualitative way it is possible to classify them as a rounded particle, according to criteria presented by Russell and Taylor<sup>(31)</sup> e Pettijohn<sup>(28)</sup> and illustrated in Figure 8. Microspheres of Rice: its particles have a more defined shape, presenting expressive corners. Qualitatively it is

#### A look at development

*Study of the shape and angularity of abrasive particles, vegetable and mineral, to be incorporated into solid soap with exfoliating action: Kaolin, bentonite, rice microspheres, bamboo and apricot seed*

possible to classify them as an angular particle, according to criteria presented by Russell and Taylor<sup>(31)</sup> e Pettijohn<sup>(28)</sup> and illustrated in Figure 8.

- d) Bentonite: its particles have the appearance of clusters, but more dispersed than the particles of the sample F01Kaolin, do not present expressive corners. In a qualitative way it is possible to classify them as a rounded particle, according to criteria presented by Russell and Taylor<sup>(31)</sup> e Pettijohn<sup>(28)</sup> and illustrated in Figure 8.

Table 2. Angularity of Abrasive Particles

Sample	Angularidade
F01Caulim	Subangular
F02Bambu	Angular
F03Apricot	Rounded
F04Rice	Angular
F05Bentonite	Rounded

### 3.3 PARTICLE DISTRIBUTION AND FORM TEST

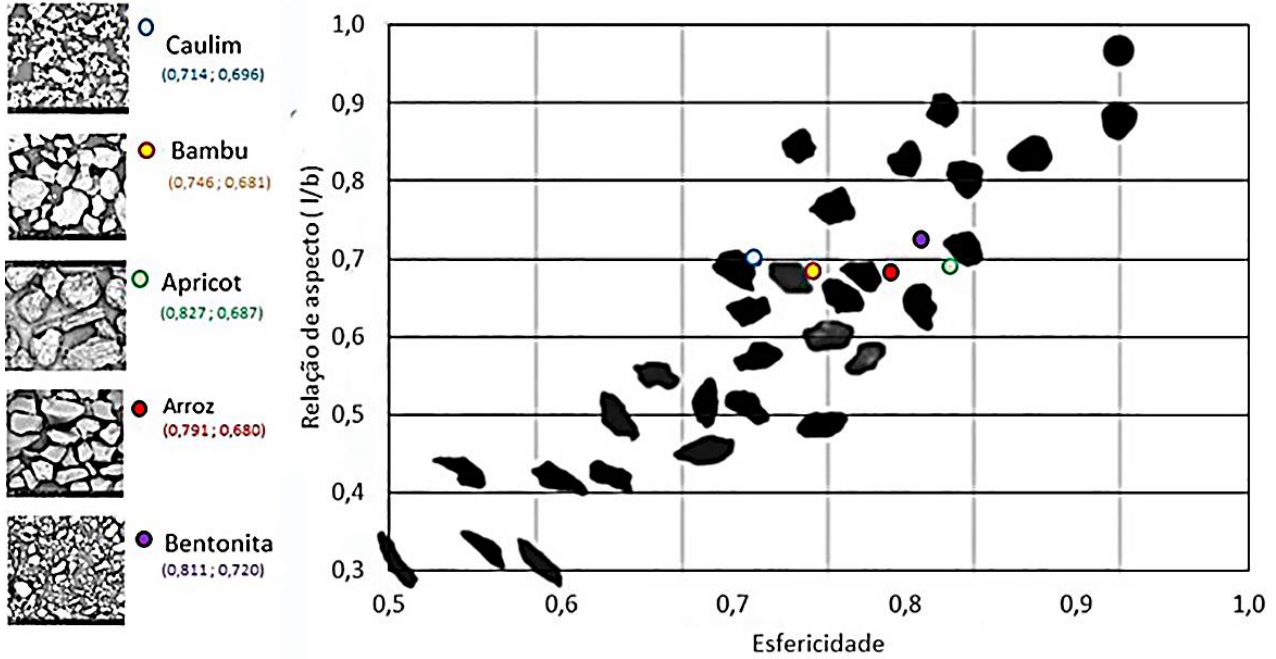
In the Particle Distribution and Form Assay, the values obtained for the particle form of the exfoliating agents used in the production of soaps showed some variation, as shown in Table 3. Figure 16 shows the representation of the distribution of particles by shape interval, which was obtained using the results shown in Figure 17. Figure 16 shows the location of the shapes of the particles, taking into account an average of the values obtained in the test, it was found that the particles occupy practically the same region in the graph, presenting a sphericity between 0.7 and 0.8, remembering that the particle will present a more sphere shape the closer to the value 1 for the ordered pair plotted in the graph. As for the aspect relation, which indicates the elongation of the particles, they are all within the same value interval, that is, of +/- 0.7.

Table 3 - Summary of particle size distribution and morphology ( $\mu\text{m}$ ).

Parameter	Sample	D[medium]	Average SPHT	b/l media
xc-min	F01Caulim	18,10	0,714	0,696
xc-min	F02Bambu	289,20	0,746	0,681
xc-min	F03Apricot	371,30	0,827	0,687
xc-min	F04Arroz	323,40	0,791	0,680
xc-min	F05Bentonita	48,10	0,811	0,720

Source: Adapted - Based on the analysis reports of the Laboratory of Technological Characterization – Polytechnic School of the University of São Paulo – Department of Mining and Petroleum Engineering, numbers: ID.LCT: 1493-22, 1494-22, 1495-22, 1496-22 and 1497-22, 15 Mar/2022. Where: D [mean] = mean diameter of the particles, mean SPHT (Mean sphericity) = relationship between the area of the particle and its perimeter, defined by  $(4\pi \cdot \text{Area})^{1/2} / (\text{Perimeter})^2$  and b/l (Mean aspect ratio) = ratio between the largest and smallest particle dimension

Figure 16. Representation of particle distribution by shape interval (SPHT and b/l) ( $\mu\text{m}$ )

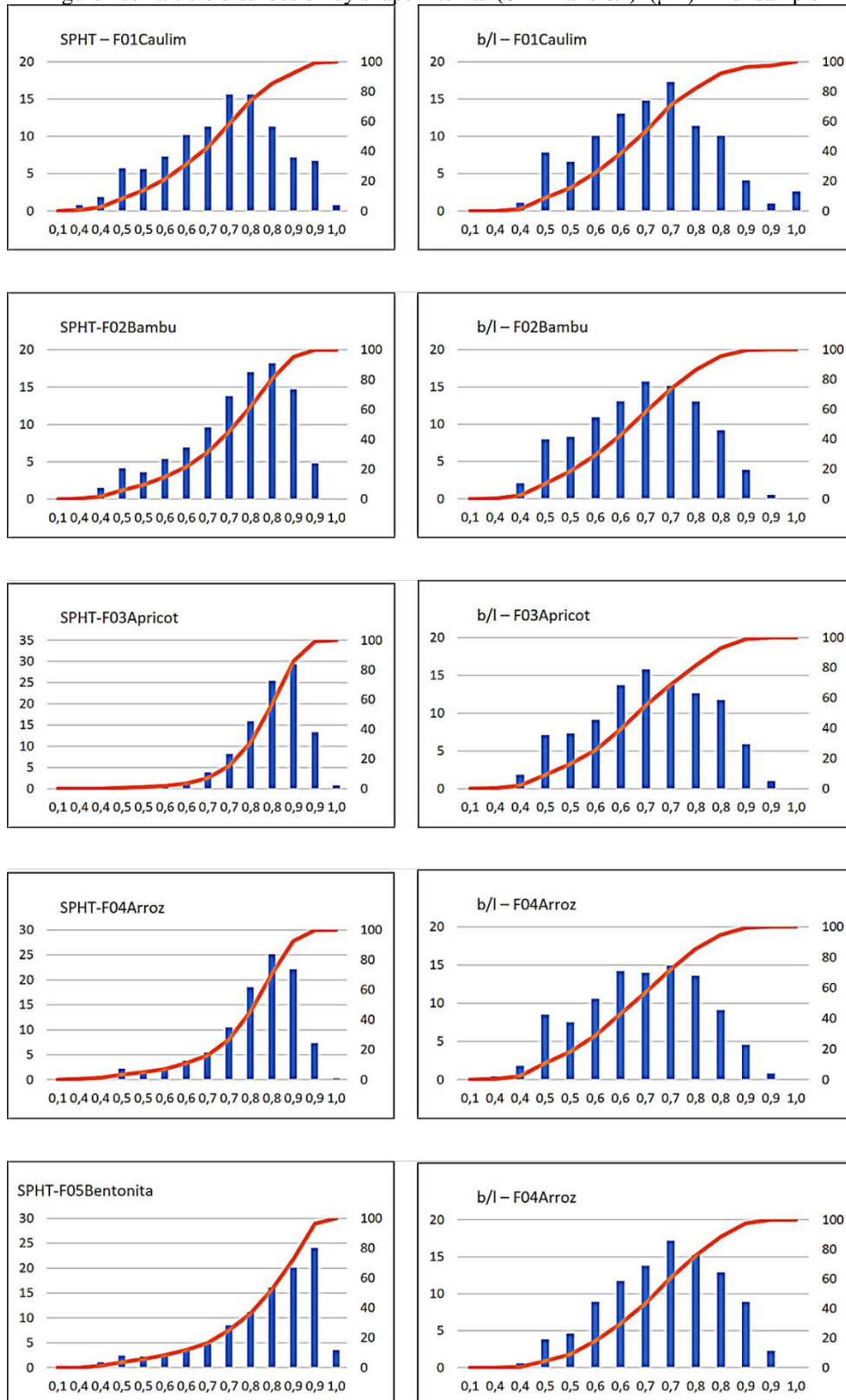


Source: Analysis report of the Laboratory of Technological Characterization – Polytechnic School of the University of São Paulo – Department of Mining and Petroleum Engineering, 15 Mar/2022. – Principle of operation of the automated particle size and morphology analysis system CAMSIZER®. Adapted.

Figure 18 illustrates a Representative Map of the Morphology of the particles where it is possible to observe the gradation of the morphology between the particles, of the exfoliating agents under study and their morphologies, in relation to the Aspect Ratio and the Sphericity. Table 4 describes the mean values of size, shape and angularity of the abrasive particles analyzed.



Figure 17. Particle distribution by shape interval (SPHT and b/l) ( $\mu\text{m}$ ) – Per sample



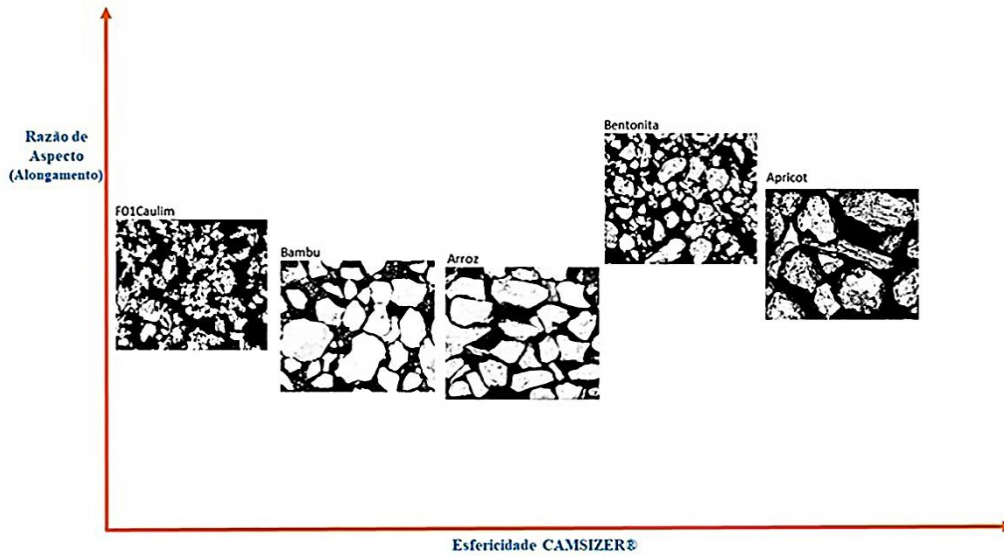
Source: The author. Based on the analysis reports of the Laboratory of Technological Characterization – Polytechnic School of the University of São Paulo – Department of Mining and Petroleum Engineering – Adapted.

**A look at development**

*Study of the shape and angularity of abrasive particles, vegetable and mineral, to be incorporated into solid soap with exfoliating action: Kaolin, bentonite, rice microspheres, bamboo and apricot seed*



Figure 18. Representative Map of Particle Morphology – General



Source: The author

Table 4 - Size, shape and angularity of abrasive particles

Sample	Average SPHT	average b/l	Angularidade
F01Caulim	0,714	0,696	Subangular
F02Bambu	0,746	0,681	Angular
F03Aprikot	0,827	0,687	Rounded
F04Arroz	0,791	0,680	Angular
F05Bentonita	0,811	0,720	Rounded

Source: The author

## 4 CONCLUSION

Considering the results obtained, it is possible to affirm that:

- As for the Sphericity parameter, the particles presented values between 0.71 and 0.82. Thus, it is possible to affirm that all the abrasive particles analyzed are similar in terms of sphericity.
- As for the Elongation parameter, the particles presented values between 0.68 and 0.72. Thus, it is possible to affirm that all the abrasive particles analyzed are similar in terms of their elongation.
- Regarding the Angularity parameter, the bamboo and rice microspheres were classified as angular, kaolin as subangular and the apricot seed microspheres and bentonite as rounded.

As the shape parameter varies little between the particles, the analysis of the abrasive potential of the exfoliating agents can be performed as a function of the angularity parameter.

According to Hutchings<sup>(7)</sup>, angular particles cause greater wear when compared to rounded particles. Thus, the bamboo and rice microspheres that presented higher angularity would have a

greater potential for use in cosmetics as exfoliating agents, compared to kaolin, bentonite and apricot seed microspheres.

These two parameters, in isolation, are not enough to affirm which exfoliating agent has the most intense abrasive action, but it may bring clues. In this line, and in a preliminary way, it is possible to affirm that bamboo and rice microspheres can present better performance as an exfoliating agent in cosmetic formulations, thus being able to be incorporated into solid soaps with exfoliating action.

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## REFERENCES

- Hilda Butler. Poucher's Perfumes, Cosmetics and Soaps. In: Butler H, editor. Poucher's Perfumes, Cosmetics and Soaps. Tenth Edition. Netherlands: Kluwer Academic Publishers; 2000. p. 453–65.
- Friedman M, Wolf R. Chemistry of soaps and detergents: Various types of commercial products and their ingredients. Clin Dermatol. 1996 Jan 1;14(1):7–13.
- Marcelo Shiniti Uchimura. Dossiê Técnico Sabão. São Paulo; 2007 May.
- Rocha MFL, Oliveira NP, Tescarollo ILT. Esfoliante formulado com pó de café como alternativa ao uso de microesferas de plástico . InterfaceHS - Rev Saúde, Meio Ambient e Sustentabilidade. 2020 Jun 1;15(1):81–93.
- Baran RB, Maibach HIM. Textbook of Cosmetic Dermatology. 5th Edition. Baran R, Maibach HI, editors. Vol. 1. Boca Raton: CRC Press; 2017.
- Pena DWP, Tonoli GHD, de Paula Protásio T, de Souza TM, Ferreira GC, do Vale I, et al. Exfoliating Agents for Skincare Soaps Obtained from the Crabwood Waste Bagasse, a Natural Abrasive from Amazonia. Waste and Biomass Valorization. 2021 Aug 5;12(8):4441–61.
- Hutchings I, Shipway P. Wear by hard particles. In: Gifford C, editor. Tribology Friction and Wear of Engineering Materials. 2nd ed. United Kingdom: Matthew Deans; 2017. p. 173–96.
- Kato K, Adachi K. Wear mechanisms. In: Kato K, Adachi K, editors. Modern Tribology Handbook. 1st ed. New York: CRC Press LLC ; 2000. p. 273–300.
- Peterson M., Winer W. Wear Control Handbook. 1st ed. Peterson M., Winer W., editors. Vol. 1. New York: American Society of Mechanical Engineers; 1980. 9–15 p.
- Souza AL de, Volpati D, Caseli L, Ceridório LF, Miranda PB, Nobre TM, et al. Filmes de Langmuir e Langmuir-Blodgett. In: Nanotecnologia Aplicada a Polímeros. Editora Blucher; 2022. p. 373–422.
- Soutor C, Hordinsky M. Dermatologia Clínica. 1st ed. Vol. 1. Porto Alegre: AMGH EDITORA LTDA; 2014. 1–5 p.
- Cestari S da CP. Noções de Anatomia e Histologia da Pele. In: Cestari S da CP, editor. Dermatologia Pediátrica. 1st ed. São Paulo: Editora dos Editores; 2018. p. 9–16.
- Li W, Zhai ZH, Pang Q, Kong L, Zhou ZR. Influence of exfoliating facial cleanser on the biotribological properties of human skin. Wear. 2013 Apr;301(1–2):353–61.
- Taliana L. Facial Skin Health: Antioxidant Facial Scrub From Red Dragon Fruit Extract. J Asian Multicult Res Med Heal Sci Study. 2020 Sep 29;1(2):1–5.
- Souza I, Spagolla RSNT, Bravo M da GL, Gaspar LR. Biologia, Histologia e Fisiologia da Pele. Cosmetics & Toiletries Brasil. Cosmet Toilet Bras. 2020 May 1;32(3):14–21.
- Barel AOB, Paye MP, Maibach HIM. Skin Feel Agents. In: Barel AOB, Paye MP, Maibach HIM, editors. Handbook of Cosmetic Science and Technology. 3rd ed. New York: CRC Press; 2009. p. 357–70.

Sun JZ, Parr JWP. Formulação de Esfoliantes Corporais. *Cosmet Toilet*. 2004 Feb 1;16(1):70–5.

Moraes JDD, Bertolino SRA, Cuffini SL, Ducart DF, Bretzke PE, Leonardi GR. Clay minerals: Properties and applications to dermocosmetic products and perspectives of natural raw materials for therapeutic purposes—A review. *Int J Pharm*. 2017 Dec 20;534(1–2):213–9.

Zague V, Santos D de A, Baby AR, Velasco MVR. Argilas: Natureza nas Máscaras Faciais. *Cosmet Toilet*. 2007 Jul 1;19(1):63–6.

Shigueyama JCD, Barros TAB. Compostos Naturais e Sintéticos em Máscaras Faciais. *Cosmet Toilet*. 2021 Nov 25;33(6):10–3.

Daré RG, Estanqueiro M, Amara MH dos AR, Truiti M da CT. Significância dos argilominerais em produtos cosméticos. *Rev Ciencias Farm Basica e Apl*. 2015 Sep 1;36(1):59–67.

Funck APG, Samy ME, Santos RDS. Argilas Minerais em Cosméticos. *Cosmet Toilet*. 2021 Jun 9;33(3):1–9.

Santos P de S, Hanna RAH, Coelho ACVC, Santos H de SS. Argilas e Argilominerais em Produtos Cosméticos: Caulim. *Cosmet Toilet Bras*. 2009 Dec 5;21(6):45–52.

Santos P de S, Santos H de S, Coelho ACV. Argilas e argilominerais em produtos cosméticos: Bentonita. *Cosmet Toilet Bras*. 2010 May 12;21(6):46–52.

Saguio K. Determinação das propriedades das rochas sedimentares em laboratório. In: Saguio K, editor. *Introdução a Sedimentologia*. 1st ed. São Paulo: Editora Edgard Blucher Ltda; 1973. p. 91–113.

BARRETT PJ. The shape of rock particles, a critical review. *Sedimentology*. 1980 Jun;27(3):291–303.

Jin ZM, Dowson D, Fisher J. Wear and friction of medical grade polyurethane sliding on smooth metal counterfaces. *Wear*. 1993 Apr 13;162–164(PART A):627–30.

Pettijohn FJ. *Sedimentary Rocks*. *Geol Mag*. 1957 Dec 1;94(6):516–516.

Hawlitsek G. Caracterização das propriedades de agregados miúdos reciclados e a influência no comportamento reológico de argamassas. [São Paulo]: Universidade de São Paulo; 2014.

Müller GM, Engelhardt W, Füchtbauer H. *Methods in Sedimentary Petrography (Part I)*. 2nd ed. Vol. 1. Michigan: John Wiley; 1967. 1–284 p.

Russell RD, Taylor RE. Roundness and Shape of Mississippi River Sands. *J Geol*. 1937 Apr 5;45(3):225–67.