COMPARATIVE STUDY ON QUALITY (REQUIRED BY THE COSMETIC INDUSTRY) OF SYNTHETIZED SOAPS THAT TAKE INTO THEIR COMPOSITION THE HALLOYSITIC KAOLIN OF THE REGION OF MAR DE ESPANHA (MG) IN COMPARISON OF COMPOSITIONS USING PERLITE, CAULINITE AND BENTONITE.

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INTRODUCTION

Halloysitic kaolin is a fine-grained, white-colored rock composed primarily of kaolinite and halloysite and secondarily muscovite, quartz and feldspar. Deposits derived from the *in situ* alteration of feldspar-rich rocks are called primary and, when of sedimentary origin, secondary. The deposits that are southeast of Minas Gerais, are part of the Eastern Pegmatitic Province (PPO), and fit into the Paraíba do Sul, Raposo and Andrelândia Groups. Halloysite is a mineral of tubular morphology which has distinct technological properties from kaolinite. Because of this, it can be used in special industry segments such as carrier mechanism and also in the control of release of active ingredients. Although Brazil has 4.5% of the world's currently marketed kaolin reserves, there are no records of industrial grade halloysitic kaolin deposits.

Considering that the soaps should protect the skin, allowing the preservation of its natural microbiota, it was proposed in this study to verify the physico-chemical quality parameters of the soaps that in its composition a percentage of halloysitic kaolin was used in its synthesis.

For this, quality tests (1) (required by the cosmetic industry) were performed such as: general characteristics, crack; mass absorption rate; weight loss; foam formation; measurement of mass compression strength, and color. In order to compare, bar soaps with five distinct compositions of clay minerals (halloysitic kaolin with and without quartz) were analyzed.

EXPERIMENTAL PART

The halloysitic kaolin extracted in the city of Mar de Espanha (MG) (Fig.1), was benefited (Fig.2) at the Mineral Technology Center (CETEM), where it passed the grading steps (# <44μm) followed by separation magnetic, chemical targeting and gravitational hydraulic classification (Fig.3). The resulting fractions were analyzed by X-ray diffraction, X-ray fluorescence and scanning electron microscopy, which identified the presence of halloysite crystals, as well as euhedral kaolinite, feldspar, muscovite, quartz and impurities. The synthesis and technological tests carried out on the soap samples were carried out at the Universidade Tecnológica Federal do Paraná (UTFPR - Campus Pato Branco).

The soaps were produced by the cold process (2) and the formulation (3) was composed of 20% palm oil (*Elaeis guineensis oil*), 34% sunflower oil (*Helianthus annuus oil*), 13% coconut oil (*Cocos nucifera oil*), 23% deionized water, 9% sodium hydroxide and 1.5% of the clay minerals (halloysitic kaolin with CH+Q quartz and without CH-Q quartz). In a beaker, it was added to the oil phases (coconut oil, sunflower oil and palm oil), in a second beaker was added water and sodium hydroxide and waited to cool. When the two beakers reached the temperature of about 40°C, the beaker containing the leach was added to the beaker having the oil phase. The clay was then added stirring the solution with the aid of a mixer, and the final result was placed in a refractory vessel, allowing to stand for 30 days.

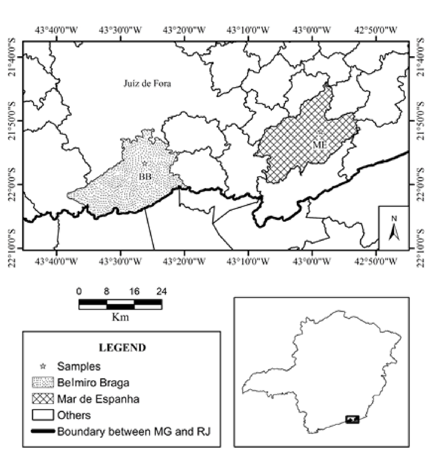

Figure 1: Kaolin extration site.

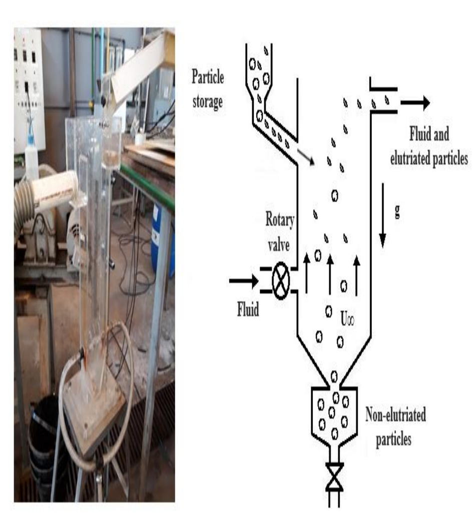

Figure 2: Gravitational Hydraulic Sorter (CHG)

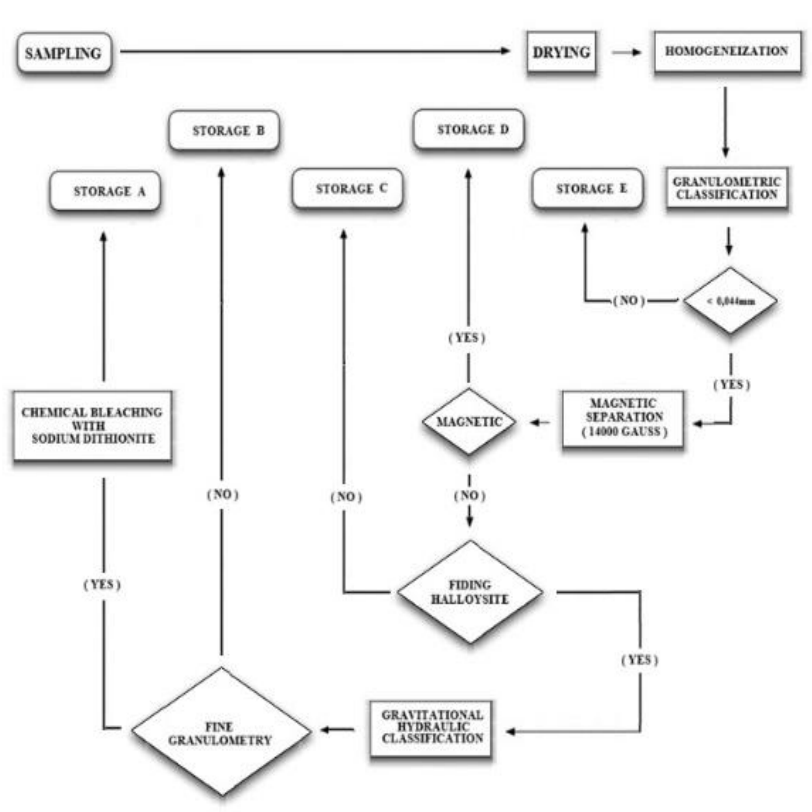

Figure 3: Processing of halloysitic kaolin.

Quality tests were carried out (4) under which the soaps produced must present industrial quality for the parameters: crack; mass absorption rate; weight loss; foam formation; texturometer and colorimeter.

To evaluate the generation of cracks, half the bar of bar soaps were immersed in distilled water for 24 hours (Fig.4-A). After this time, the bars were removed and allowed to dry for 30 hours at room temperature (Fig. 4-B).

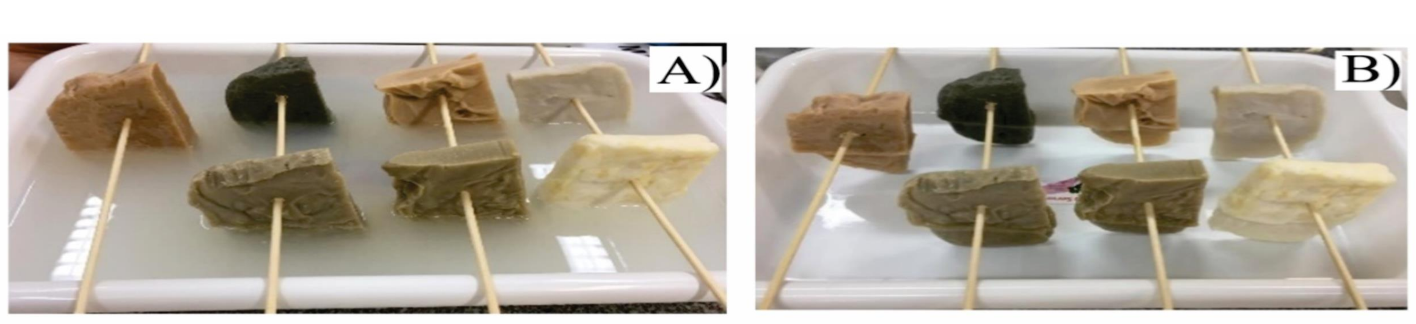

Figure 4: Bars of soap in water (A); bars of soap drying (B).

The determination of the rate of water absorption is made with samples of the clay-based soaps, which were weighed (m1) and immersed in distilled water and left for 24 hours at room temperature. Water was drained after this period, and the specimens were weighed again (m2). The difference between the masses of the percentage of water absorbed. For each sample, triplicates were performed, obtaining an average of the results (eq.1).

% water absorption = [(m2-m1) / m1] .100 (eq.1)

For the mass loss analysis, the samples were weighed (m1), then submerged in distilled water and allowed to stand for 24 hours at room temperature. Subsequently, the gelatinous mass is withdrawn and weighed again (m2). From the mass differences, the mass loss percentage (eq.2) is calculated. For each sample, triplicates were performed.

% water loss = [(m2-m1) / m1] .100 (eq.2)

For foaming analysis, 2% solutions of each soap were prepared using distilled water. After the preparation, 50 ml of each was poured into a 100 ml beaker, which was inverted 10 times, in synchronized motions. Immediately after this operation, the height of foam formed was determined, repeating these determinations after 5 minutes. Triplicates of each soap were made.

Using a texturometer (5), calibrated in order to measure the compressive force, penetration tests were performed on the soaps. The penetrations were 8mm at a speed of 1mm/s, at an ambient temperature of about 25°C. The maximum positive force is the force required to penetrate the soap to the distance of 8mm: the higher this value, the greater the hardness of the soap.

Colorimetric analyzes were obtained using a Meter CR-400 Colorimeter (Konica Minolta), where:

L * = Brightness (minimum value 0 indicates black and maximum value 100 indicates white)

a * = red / green coordinate (+a indicates red and -a indicates green)

b * = yellow / blue coordinate (+b indicates yellow and -b indicates blue)

RESULTS

The results of the quality cosmetic tests (1) performed on quartz kaolin (CH+Q) and quartz (CH-Q) kaolin soap were:

- in soaps produced with the sample CH+Q did not show cracks (level zero) and the formulation with the sample CH-Q presented crack (level II);
- in soaps produced with the sample CH+Q presented a higher foam formation average than the soap with the sample CH-Q;
- the CH+Q sample showed a greater mass loss compared to the CH-Q sample;
- measurements of the mass compression force (6) were made on the soaps in order to estimate their hardness. The diagrams (Fig.4) produced show that the measurements of the compressive force were 8mm at a speed of 1mm / s, at an ambient temperature of about 25°C. The maximum positive force (the force required to penetrate the soap) up to the 8mm distance was 4.63N for the soap produced with the CH-Q sample and 3.66N for the soap produced with the CH+Q sample.
- the same graphical diagrams (Fig.5) constructed from the compression force measurements allow the average force (force x time) to be calculated. This impulse value also corresponds to the value of the hardness of the soap. For the bar of soap constructed with the sample CH+Q presented the value of 19.40 N.sec and for the soap constructed with the sample CH-Q presented the value of 28.91 N.sec.
- the comparison between the CH-Q/CH+Q ratios calculated from the maximum positive penetration force and also the impulse, have the values: 1.26 for the mass compression force, and 1.49 for the impulse . The difference between these reasons is approximately 16%.

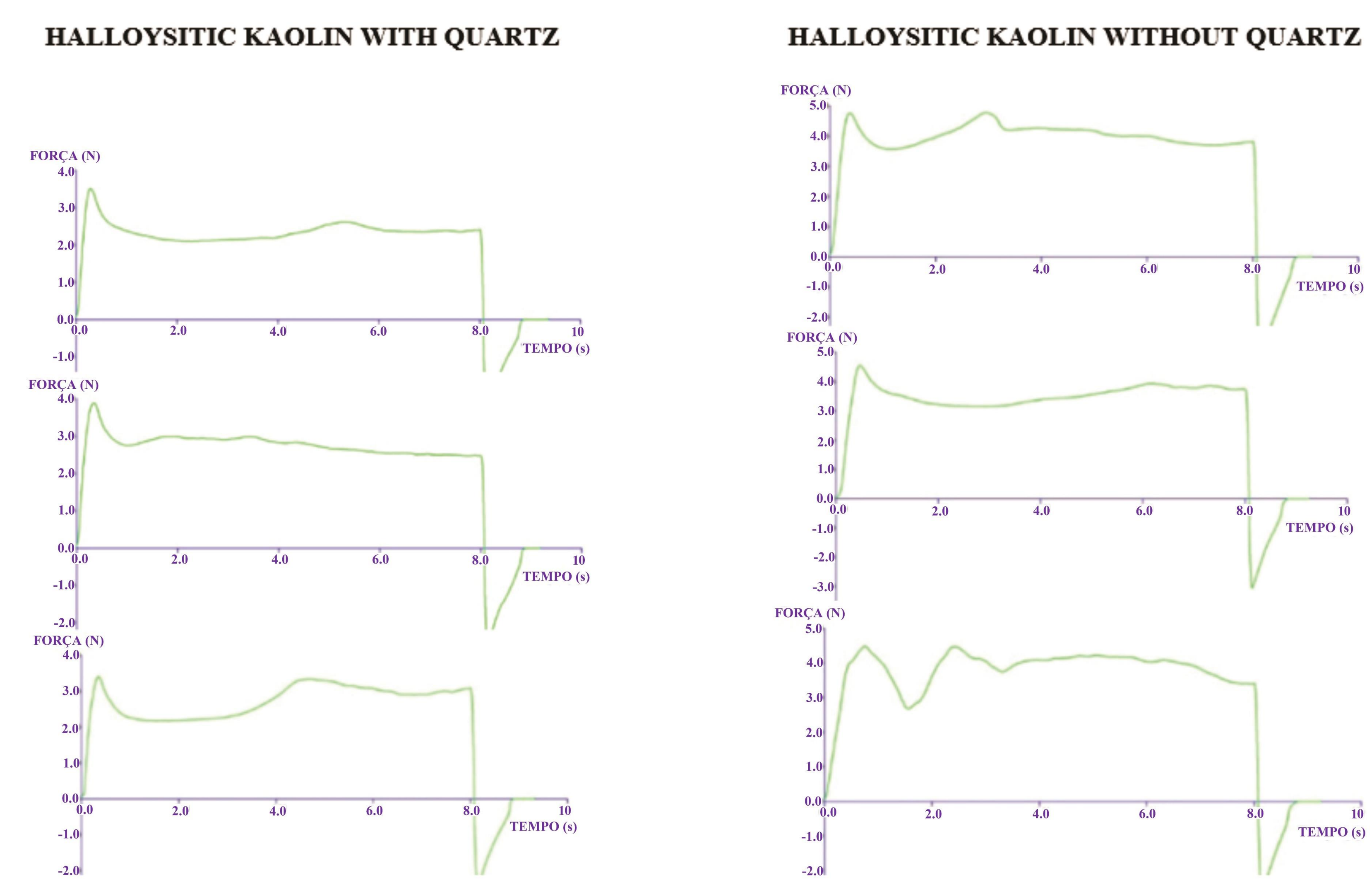

Figure 5: Tests of compression force measurements on soaps

CONCLUSION

Halloysitic kaolin with quartz (CH+Q) and without quartz (CH-Q) presented technological properties that demonstrated to be compatible as an input to be used as components in the synthesis of bar soaps. The soaps containing quartz halloysitic kaolin (CH-Q) showed a higher hardness, a lower mass loss when immersed in water, a lower formation of foam, and increased cracking (Type II). The difference between the ratios (CH-Q)/(CH+Q) in the calculation of the impulse compared to the calculation of the maximum penetration force was 16%, being similar and equal to the hardness of the soap.

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THANKS

V REUNIÃO ANUAL SOBRE ARGILAS APLICADAS