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RESEARCH ARTICLE

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PSEUDOBOEHMITE NANOCARRIERS IN COSMETIC FORMULATIONS

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ABSTRACT

The aim of this study is to contribute to the preparation and characterization of nanoemulsions for anti-aging cosmetic use. Palmarosa oil and Rosehip nanoemulsions were prepared with different active cosmetic ingredients such as mandelic acid and hyaluronic acid, in concentrations of 1%, 3% and 5% (wt%) of pseudoboehmite. After the nanoemulsions analysis, they were characterized in the following parameters: visual analysis, pH, density and optical microscopy. The results obtained show the possibility of using different compositions, the most suitable were: palmarosa oil nanoemulsion with 3 or 5wt% of mandellic acid/pseudoboehmite and palmarosa oil nanoemulsion with 1,3 or 5% of hyaluronic acid/pseudobohemite.

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INTRODUCTION

The development of controlled release systems imply a multidisciplinary scientific approach, currently representing one of the main areas of the Biological Sciences. These systems are developed to allow the drug to be released in a controlled and / or sustained manner and can reach therapeutic levels in the biological target, avoiding possible toxic reactions inherent to the active principle (FERREIRA, 2012). These systems have a phase with droplets dispersed in another, in which energy, mechanical and chemical, are used to reduce the surface tension (RAWLINGS, 2004) always containing an aqueous and an oily phase (BRASIL, 2010).
Usually, nano emulsification occurs by high energy processes with the mixture of the oil and water phases, resulting in a coarse emulsion with a large droplet size and, later, achieved by means of high-pressure homogenizers or ultrasound generators, to promote shear capable to deform the particle (PEREIRA, 2011 and CLEMENTS, 2011). The fine ceramic of the pseudoboehmite type, has a high surface area, being able to adsorb active ingredients, being a modifier of the physical environment activating the solubilization process and it can be classified in the non-toxic category (MUNHOZ, 2010 and SOUZA, 2013). One of the most studied techniques of its synthesis is the sol-gel process, as it has advantages such as low cost and easy manipulation, making it possible to obtain materials on a nanometric scale, adding different properties to it, in comparison to conventional materials (PAGANINI, 2012). Considering these affirmations, this study aims to contribute to the Cosmetic area by presenting nanoemulsions containing mass concentrations of 1%, 3% and 5% of pseudoboehmite, 7% of Palmarosa essential oil or Rosehip vegetable oil and 2, 1% mandelic or hyaluronic acid as active ingredient.

MATERIALS AND METHODS

In this study, pseudoboehmite was obtained by the sol-gel process using the following reagents: aluminum nitrate, ammonium hydroxide, acetone; and the nano emulsions were prepared with Rosehip vegetable oil, Palmarosa essential oil, mandelic acid, hyaluronic acid and the surfactant polysorbate (Tween 20). The main characteristic of the raw materials was the absence of impurities to enable its use in the cosmetic product.

Obtaining Pseudoboehmite by Sol-Gel Process: Aluminum nitrate aqueous solutions (98wt%) and polyvinyl alcohol (8wt%) were mixed in the proportions of 64.9wt% and 35.1wt%, for thirty minutes, using a magnetic mixer, as shown in the figure 1 (Preparation of pseudobohemite). The ammonium hydroxide solution was dripped into the obtained mixture. The initial temperature of the system was 26°C and the final -6°C. After total precipitation and aging for one week at a controlled temperature of 12° C, pseudoboehmite was obtained. The solution containing the pseudoboehmite was filtered through a Buchner funnel and washed with 1L of distilled water and later with 0.5 L of acetone. The precipitate was placed in a glass flask for later use in the nanoemulsion. The process of preparing and obtaining the nanoparticle is the most important part of the process of obtaining a nanostructure in the order of 1 to 100nm providing the reduction of the size, shape and porosity of the particles, and is crucial to the nanocomposites final properties (KOCH, 2002). The pseudoboehmite obtained was lyophilized in the Terronelyophilizer - model LS300, and later characterized by X-ray diffraction, scanning electron microscopy and thermal analysis.

Nanoelmulsions Preparations: The pseudoboehmite is solubilized in water, then the active ingredient (mandelic acid or hyaluronic acid) is added. Simultaneously, in another becker, the Tween 20 surfactant is solubilized in Palmarosa essential oil or Rosehip vegetable oil. Both solutions are kept under stirring for 30 minutes at 40°C. Then, the aqueous phase is poured over the oily phase under constant and continuous agitation at 600 rpm, in the magnetic stirrer with heating (SOLAB - model SL-91). The mixture remained under continuous stirring until cooling to room temperature at 25 ± 3°C. The nanoemulsion was obtained from the prepared emulsion, by centrifuging the samples for 15 minutes at speeds of 1000, 2500 and 3500 rpm, respectively, in an Eppendorf centrifuge, model 5804R (ALMEIDA and BAHIA, 2003). The nanoemulsions were obtained with different concentrations of pseudoboehmite nanoparticles and different active ingredients, mandelic acid and hyaluronic acid according to the compositions in Tables 1 and 2. The concentration of active ingredients in emulsions (2.1wt%) is in accordance with the literature (BORGES, 2006 and SCOTTI, 2006). The nanoemulsions obtained in the following parameters: visual analysis, pH, density and optical microscopy.

Table 1. Composition of nanoemulsions with mandelic acid

Nano Emulsion	Composition (% in weight)				
	Pseudo bohemite (PSB)	Palmarosa (PR)	Rosehip (RM)	Mandelica cid (AM)	Tween 20
A	1.0	7	-	2.1	8.8
B	1.0	-	7	2.1	8.8
C	3.0	7	-	2.1	8.8
D	3.0	-	7	2.1	8.8
E	5.0	7	-	2.1	8.8
F	5.0	-	7	2.1	8.8

Table 2. Composition of nanoemulsions with hyaluronic acid

Nanoemulsion	Composition (% in weight)				
	Pseudobohemite (PSB)	Palmarosa (PR)	Rosehip (RM)	Hyaluronic acid (AH)	Tween 20
G	1.0	7	-	2.1	8.8
H	1.0	-	7	2.1	8.8
I	3.0	7	-	2.1	8.8
J	3.0	-	7	2.1	8.8
L	5.0	7	-	2.1	8.8
M	5.0	-	7	2.1	8.8

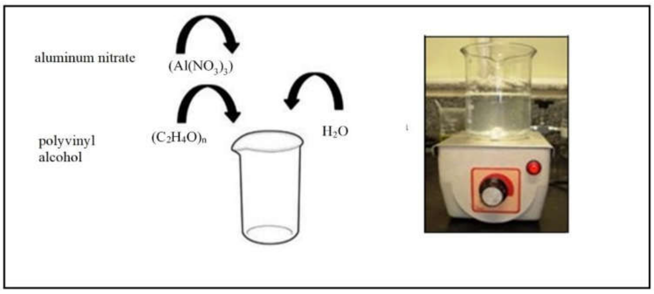

Figure 1. Preparation of pseudobohemite

RESULTS AND DISCUSSION

The results obtained from X-ray diffraction (Figure 2) show a typical pseudoboehmite diffractogram with non-intense peaks according to the observed peaks 2Θ = 13o (020) and 28º (021), in accordance with the International Center Diffraction Data (ICCD). The morphology of pseudoboehmite shows the presence large clusters of particles (Figure 3).

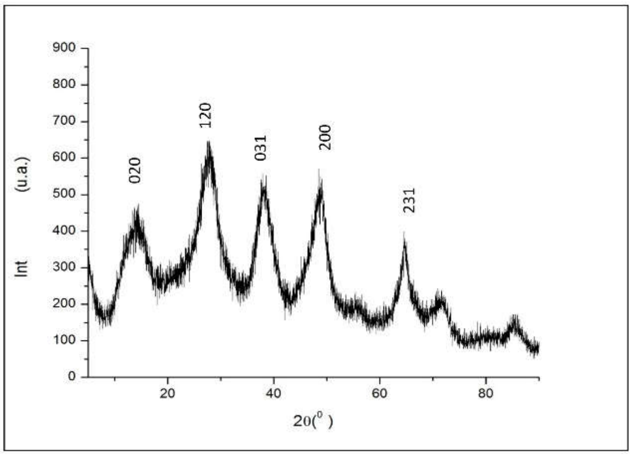

Figure 2. DRX obtained for pseudobohemite

These clusters are formed by weak interaction forces, which can include Van der Waals forces and forces of electrostatic attraction between particles of opposite charge (MUNHOZ, 2010).

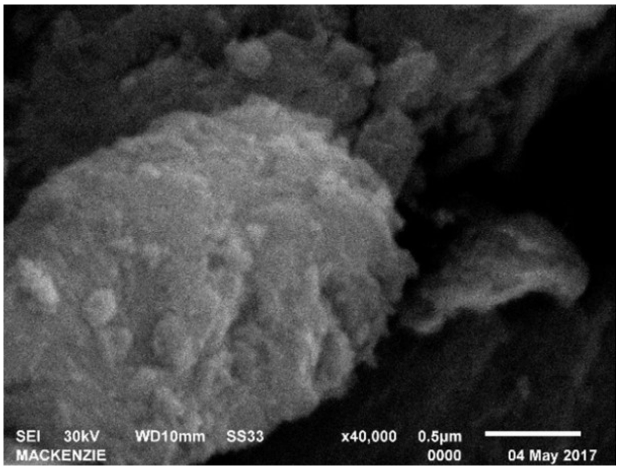

Figure 3. Electron microscopy of synthetized pseudobohemite scan

High porosity is also observed, providing a high surface area for a nanostructure in the order of 1 to 100nm. Thermal analyzes show the typical Differential Scanning Calorimetry (DSC) curve of the pseudoboehmite (Figure 4a) showing an endothermic peak close to the temperature of 100°C, due to water vaporization. A transformation of pseudoboehmite into γ-alumina and a decomposition of PVAL in the same temperature range as the DSC analysis shows a complex peak in the 200-400°C range. A thermogravimetric analysis (TG) shows the mass loss corresponding to these phenomena (Figure 4b). In DSC analysis at about 1200°C, a peak attributed to the transformation of the last metastable phase of alumina into α-alumina is observed. Visually, samples containing mandelic acid presented phase separation with all concentrations of pseudobohemite.

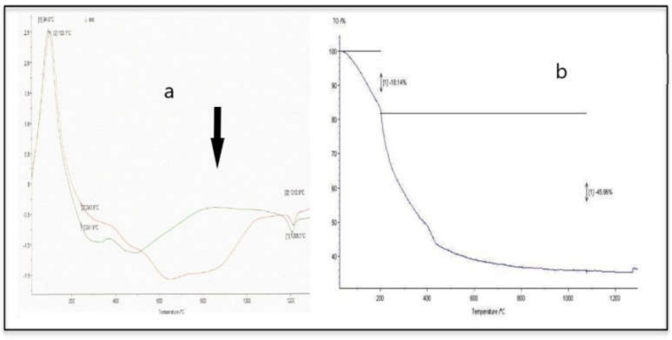

Figure 4. Thermal analysis of pseudobohemite: DSC (a) and TG (b)

Samples containing hyaluronic acid and Palmarosa essential oil showed similar behavior to samples containing mandelic acid. However, samples with hyaluronic acid and Rosehip vegetable oil did not show phase separation and, therefore, did not form nanoemulsions. The pH (Figure 5) is a parameter for monitoring the stability in nanoemulsions, since changes in its value indicate the occurrence of chemical reactions or bacterial growth (ANDRADE, 2008).

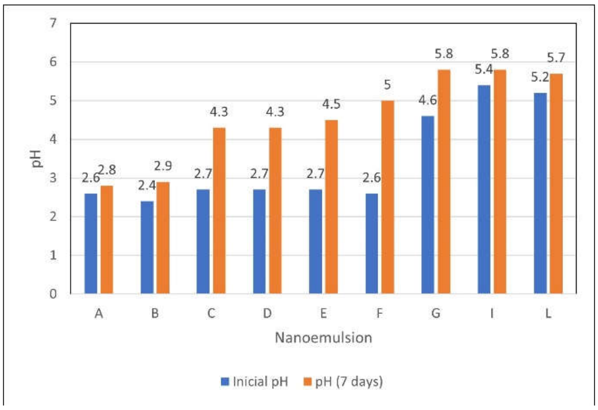

Figure 5. PH behavior of the nanoemulsions

Table 3. Particle Size of the nanoemulsions A – D

Nanoemulsion	A	B	C	D
Particle size	35.4	107.2		12.9
	41.6	114.5	1010.6	294.3

Table 4. Particle Size of the nanoemulsions E, F, G, I, L

Nanoemjulsion	E	F	G	I	L
Particle size	253	192.5	21.5	530.0	21.3
	285.2		29.2		32.3
			189.7		

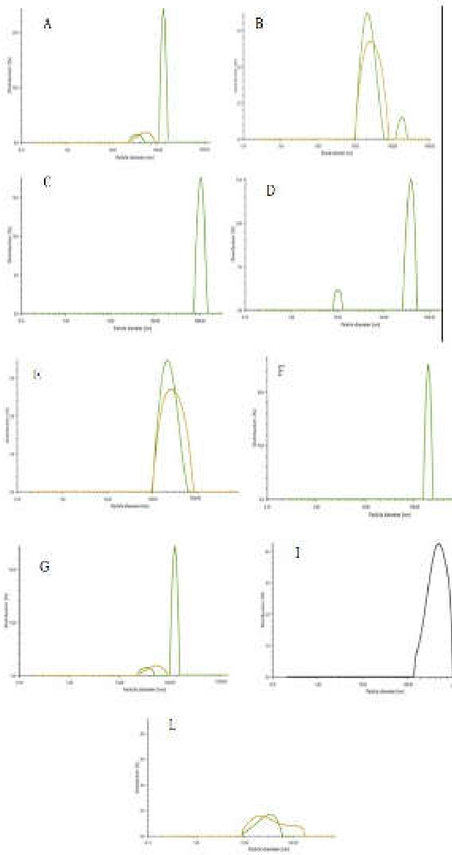

Figure 6. Particle size distribution of the nanoemulsions

The results obtained are in accordance with Silva Júnior *at al.* (2013), who state that nanoemulsions prepared with vegetable oils can exhibit a decrease in pH due to the hydrolysis of fatty acid esters. All formulations have an acidic pH, and formulations C, D, E, F, G, I, L have a pH similar to the skin´s pH (4.6 - 5.8), which is a very important fact from the cosmetic or dermatological perspective (LEONARDI, 2004). The density of mandelic acid and hyaluronic acid nanoemulsions as a function of time, measured at 25°C, presents values similar to water values 1.00 g.cm^{-3} (main component), the results are compatible with the literature (SILVA JUNIOR et al, 2013). The particle size distribution of the nanoemulsions showed the following results: sample B has a higher polydispersity index containing mandelic acid, rosehip vegetable oil and 1wt% of pseudoboehmite, and sample I, was the best polydispersity index of the nanoemulsions containing hyaluronic acid, Palmarosa essential oil and 3wt% of pseudoboehmite as can be seen in Table 3 and Figure 6.

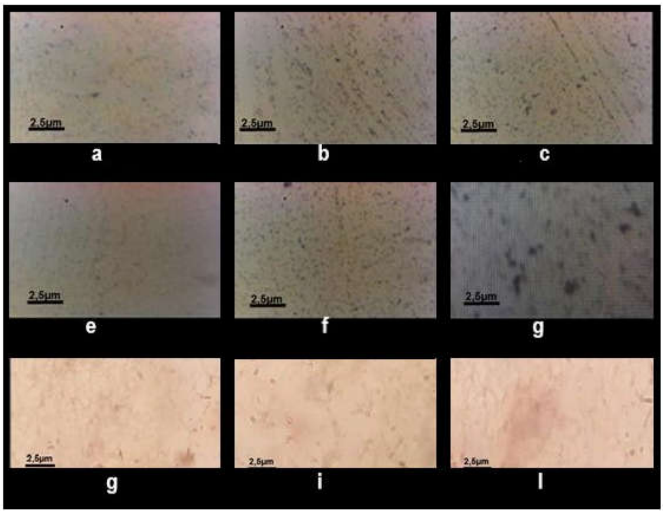

Figure 7. Micrographs of the nanoemulsions

The micrographs (Figure 7) were obtained in a Philips optical microscope, using a 400X magnification objective. The microphotographs were captured with the aid of an Opton digital camera. Samples A (1.0wt% PSB / PR), D (3.0wt PSB / RM) and E (5.0wt% PSB / PR) were the most uniform, with sample D (3.0wt % PSB / RM) was the one that presented, good particles dispersion as well as uniformity. The difference in dispersion is due to the concentration of the pseudoboehmite. Sample I (3.0wt% PSB / PR), containing hyaluronic acid, showed the greatest uniformity and good dispersion of the nanoparticles.

CONCLUSION

The results obtained allowed to conclude the possibility of obtaining nanoemulsions with pseudoboemite nanoparticles. The most suitable nanoemulsions with mandelic acid and Palmarosa essential oil or Rosehip vegetable oil are 3wt% and 5wt% of pseudoboehmite. Meanwhile, all pseudoboehmite concentrations in nanoemulsions of hyaluronic acid and Palmarosa essential oil are suitable for obtaining cosmetic products.

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