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STABILITY STUDY OF GALACTOMANAN BIOHYDROGEL ASSOCIATED WITH SHA BUTTER[1]

ESTUDO DE ESTABILIDADE DE BIOHIDROGEL DE GALACTOMANANA ASSOCIADO A MANTEIGA DE KARITÉ

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ABSTRACT

This study shows the development of a biohydrogel from galactomannan from the species Delonix regia associated with emulsified shea butter. Due to characteristics such as atoxity, biocompatibility and mainly its use as a vehicle of active principle, biohydrogels have aroused the interest of large industries for natural hydrogels. Shea butter was chosen in the composition of the biohydrogel, because it is used as an emollient, with moisturizing power and has a protective action, has antioxidant properties capable of protecting against free radicals and

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UV rays. The biohydrogels obtained were submitted to organoleptic and physical-chemical evaluation, followed by preliminary stability tests. In view of the analyzes, it was observed that samples remained stable in the organoleptic evaluation and also showed physical-chemical characteristics that can serve the biohydrogel industry, however, when subjected to higher temperatures, they lost water, showing that hydrogels are quite susceptible to changes due to heat exposure. However, even with the loss of water, these formulations have a great potential to become a product for hair and body use.

KEYWORDS: Karite butter. Galactomannan. Biohydrogel.

RESUMO

Este estudo mostra o desenvolvimento de um biohidrogel a partir da galactomanana advinda da espécie Delonix regia associada à manteiga de karité emulsionada. Devido características como atoxidade, biocompatibilidade e principalmente sua utilização como veículo de princípio ativo, biohidrogéis têm despertado o interesse das grandes indústrias por hidrogéis naturais. A manteiga de karité foi escolhida na composição do biohidrogel, por ser utilizada como emoliente, com poder hidratante e possuir uma ação protetora, apresenta propriedades antioxidantes capazes de proteger contra os radicais livres e raios UV. Os biohidrogéis obtidos foram submetidos a avaliação organoléptica e físico-química, seguidos de testes de estabilidade preliminares. Diante das análises, observou-se que as amostras se mantiveram estáveis na avaliação organoléptica e apresentaram também características físico-químicas que podem atender a indústria de biohidrogéis, porém, quando submetidas a temperaturas mais elevadas perderam água, evidenciando que os hidrogéis são bastante suscetíveis a alterações devido a exposição ao calor. Entretanto, mesmo com a perda da água, estas formulações apresentam um grande potencial de se tornarem um produto para uso capilar e corporal.

PALAVRAS-CHAVE: *Manteiga de Karité. Galactomanana. Biohidrogel.*

1. INTRODUCTION

The cosmetics sector, in particular the hair and body products market, has followed a new trend according to the demands of its consumers, betting on products free of petrolatum , sulfates, parabens and silicones, replacing these products with ingredients from from nature.

In this sense, biohydrogels have aroused the interest of large industries for presenting characteristics such as biocompatibility, and the ability to become gels due to their high degree of swelling, resembling living tissue. In addition, they act as a controlled release system of active principles, being a good candidate to replace synthetic polymers (AOUADA, 2009; MOURA, 2005; SABADINI, 2015). Therefore, the use of natural polymers, obtained from renewable sources, such as plants, algae and microbial cultures, especially yeasts and fungi, has a great industrial application because they present low-cost products, are biocompatible , non-toxic, biodegradable and are abundant. in nature (SABADINI, 2015).

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Shea butter is made up of fatty acids and vitamins, having a great importance in the cosmetics area (BAREL; PAYE; MAIBACH, 2009; MARANZ; WIESMAN, 2004; MAANIKUU; PEKER, 2017; SEMMLER, 2011).

In this context, this work presents the development of a biohydrogel from a natural polymer associated with emulsified shea butter. The formulations obtained were evaluated in order to ensure the reliability of the product for possible studies and later applications.

2. MATERIALS AND METHODS

Galactomannan was provided by the Chemical Technology Laboratory of the State University of Ceará. Shea butter was supplied by the company Mapric ® Products Pharmaceuticals .

2.1 *Preparation of the emulsion*

The emulsion was prepared using distilled water, shea butter and fatty acid diethanolamide (DEA) as a surfactant. Initially, distilled water was placed in a 100 mL beaker , followed by the addition of diethanolamide , until the mixture was homogenized; finally, the shea butter was added , heating it for 10 minutes at a temperature of around 50°C until complete homogenization. The emulsions were left to rest for 48 hours and after this period, the emulsion's pH, appearance, color and visual stability were evaluated.

2.2 *Incorporation of emulsified shea butter in galactomannan biohydrogel*

After obtaining the shea butter emulsions and forming the galactomannan biohydrogel , 5% of the shea butter emulsions were reserved for 5 grams of the biohydrogel , in which the emulsion proportions varied by 6.8615g (Formulation A) and 11 .9037g (Formulation B). Then, the formulations were placed in an ultrasound for agitation promoted by ultrasonic energy, for 10 minutes. Then, the samples were placed at rest in the refrigerator for 48 hours to later be analyzed for their organoleptic characteristics and physicochemical properties.

2.3 *Organoleptic and physicochemical parameters*

2.3.1 pH Assessment

The determination of the pH of the prepared samples was carried out at room temperature for 4 weeks using a MACHEREY-NAGEL pH indicator strip.

2.3.2 Density

To analyze the density of the samples, a 25 mL pycnometer was used . Initially, the empty pycnometer was weighed, then it was weighed with water that was used as a standard, and finally the pycnometer containing the ready-made formulations was weighed.

2.3 .3 Organoleptic Properties

The analyzes involved macroscopic aspects such as odor, color and homogeneity, in order to verify some type of instability.

2.4 *Preliminary stability tests*

From the physical-chemical and organoleptic parameters, the stable samples were submitted to preliminary stability tests (ANVISA, 2004).

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2.4.1 Centrifugation

falcon centrifuge test tube , the samples were subjected to cycles of 1000, 2000 and 3500 rpm for 15 minutes each cycle. And then the samples were analyzed macroscopically in relation to their appearance, color, odor, phase separations and pH determination.

2.4.2 Thermal Stress

The samples were submitted to heating in a water bath, in the temperature range of 40 to 80°C. Increasing the temperature by 5°C and remaining for 30 minutes at each temperature. These samples were then subjected to organoleptic tests and pH determination.

2.4.3 Freeze-thaw cycle

The formulations were subjected to different temperature conditions. First, the samples were conditioned in the refrigerator for 24 hours with a temperature range of around 4°C, then the samples were removed and placed in the oven for another 24 hours with a temperature range of 45°C, ending the first cycle. Finally, a total of 6 cycles were carried out, that is, in a period of 12 days with alternation between the refrigerator and the oven. Then, they were analyzed in relation to organoleptic aspects and pH determination.

3. RESULTS AND DISCUSSION

3.1 *Physico - chemical and organoleptic parameters*

3.1.1 pH Assessment

According to Gomes (1999), the pH of the hydrolipidic layer that protects the hair should be slightly acidic, between 4 and 6 on the pH scale. When using products that are too acidic or too alkaline, hair damages, presenting an opaque and dry appearance. The formulations had a pH around 7 and this pH was maintained, as it would not damage the skin or hair.

3.1.2 Density

Analyzing the formulations, it can be seen that there was no significant change since the formulations have different amounts of emulsion.

Table 2 - Determination of the relative densities of formulations A and B

Determination of relative density (25°C)			
SAMPLE **(A)** (g)	mH_2O (g)	ρH_2O (g/mL)	SAMPLE **(A)** (g/mL)
25.1196	26.3246	≅ 1.00	≅ 0.9542
SAMPLE **(B)** (g)	mH_2O (g)	ρH_2O (g/mL)	SAMPLE **(B)** (g/mL)
24.6968	26.7392	≅ 1.00	≅ 0.9269

Source: Prepared fur author .
Where ρ= density .

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3.1.3 Organoleptic Properties

For the formulations obtained, no changes were observed in relation to their organoleptic properties as shown in Table 3. These could then proceed to the stability test.

Table 3 - Organoleptic parameters of the formulations

Formulations	Coloring	Odor	Aspect	pH
THE	whitish	characteristic	Gel	7.0
B	whitish	characteristic	Gel	7.0

Source: Prepared fur author .

3.2 Evaluation of preliminary stability tests

The stability study is very important for these biohydrogel samples , as it will provide information about their behavior under different environmental conditions, such as humidity and temperature, evaluating the extent to which these formulations can remain unchanged (ANVISA, 2004).

3.2.1 Centrifugation

According to Anvisa (2004), the first test that must be performed is the centrifugation test, which is related to the gravitational force in order to verify the increase in the movement of the particles, thus causing a stress in the sample and anticipating possible processes of instability. When centrifuged, the biohydrogels remained stable, maintaining the appearance of a gel, without changes in color or odor, without the formation of phases and changes in pH.

3.2.1 Thermal Stress

In the temperature range of 40-65 ºC , the formulations behaved similarly, even with differences in the amount of water and surfactant. From 65 ºC , there were changes in its characteristics. Regarding their color, they became yellowish, and in their aspects, they became slightly dry due to loss of mass, that is, which is possibly due to the loss of water during the cycle.

3.2 .3 Freeze-Defrost Cycle

During the first, second and third cycles, the samples did not undergo any changes in characteristics such as: gelatinous appearance and whitish color, in addition to maintaining neutrality in the pH value. During the fourth cycle, samples A and B began to undergo changes, showing yellowish colors and the gelatinous aspect was dry, due to the evaporation of water. On the other hand, the odor and the pH value remained the same. Similar results were found by Moura (2005), who, when performing tests with hydrogels , involving temperature, observed a decrease in their volume with increasing temperature, evidencing that they are quite susceptible to changes when exposed to heat. Biohydrogels are also quite susceptible to changes in their appearance due to temperature (DRESSLER , 2008),

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a fact demonstrated in this work, where the samples had the appearance of a plastic film.

4. CONCLUSIONS

The association of emulsified shea butter with galactomannan biohydrogel provided a stable formulation. In the results obtained by the preliminary stability tests, it was observed that after a certain temperature and a repetition of cycles, mass loss occurred, probably due to water evaporation, since the biohydrogel is composed of a high amount of water due to swelling. Regarding the parameters adopted by ANVISA, regarding storage for long periods of time on the shelf, the samples behaved well, maintaining their organoleptic properties and pH, which is an advantage for the product for commercialization purposes. Thus, the formulations need further studies in future evaluations in order to obtain more detailed information about this biohydrogel .

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