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Analysis of the Influence of Lipid Components on the Spreadability of Hair Heat Protectants by Central Composite Design

Análise da Influência de Componentes Lipídicos no Espalhamento de Protetores Térmicos do Cabelo por Delineamento Composto Central

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Abstract

Thermal procedures aiming hair dressing, such as hair brushes, hair dryers or hair straighteners, usually involve higher temperatures, which progressively damages the hair fiber structure. The search for heat protectants for reducing this thermal damage has been growing, especially containing *green* components from vegetable source. To be efficient, the heat protectants should adequately spread throughout the entire hair shaft, covering the entire surface to be protected. Thus, the influence of the lipids in the *in vitro* spreadability of heat protectants was determined in this study. This influence was determined in a central composite rotational design 2^3, and spreadability was the dependent variable. Cetostearyl alcohol, shea butter and coconut oil concentration ranged between 0.64 and 7.36% (w/w) and the emulsions were prepared by hot homogenization with mechanical stirring. *In vitro* spreadability was determined by using an apparatus containing a square glass plate positioned on a circular plate with a 1 cm diameter hole in the center, on which a fixed amount of sample was applied to be pressed onto the surface by a fixed weight (5 g). Changes in the concentration of the components directly influenced the product spreadability ($p < 0.05$), which was higher around the central point for shea butter and coconut oil concentrations (4%) and at low concentrations of cetostearyl alcohol (0.64%). The studied components directly influenced the spreadability of the final product, which can be optimized in a rational pathway to obtain an adequate coverage throughout the hair shaft and desired thermal protection.

Keywords: Cosmetics. Factorial Design. Hair Coverage. Hair Damage. Thermoprotective.

Resumo

Procedimentos térmicos para pentear os cabelos, como escovas, secadores ou chapinhas, geralmente envolvem temperaturas elevadas, o que danifica progressivamente a estrutura da fibra capilar. A busca por protetores térmicos para redução deste dano térmico vem crescendo, principalmente contendo componentes verdes de origem vegetal. Para serem eficientes, os protetores térmicos devem se espalhar adequadamente por toda a haste capilar, cobrindo toda a superfície a ser protegida. Assim, a influência dos lipídios na espalhabilidade in vitro de protetores térmicos foi determinada neste estudo. Essa influência foi determinada em um delineamento composto central rotacional 2^3 *e a espalhabilidade foi a variável dependente. As concentrações de álcool cetoestearílico, manteiga de karité e óleo de coco variaram entre 0,64 e 7,36% (p/p) e as emulsões foram preparadas por homogeneização a quente com agitação mecânica. A espalhabilidade in vitro foi determinada por meio de um aparato contendo uma placa de vidro quadrada posicionada sobre uma placa circular com um orifício central de 1 cm de diâmetro, na qual foi aplicada uma quantidade fixa de amostra para ser pressionada na superfície por um peso fixo (5 g). A variação na concentração dos componentes influenciou diretamente na espalhabilidade do produto* ($p < 0,05$)*, sendo maior em torno do ponto central para as concentrações de manteiga de karité e óleo de coco (4%) e em baixas concentrações de álcool cetoestearílico (0,64%). Os componentes estudados influenciaram diretamente na espalhabilidade do produto final, que pode ser otimizado de forma racional para se obter uma cobertura adequada em toda a haste capilar e a proteção térmica desejada.*

Palavras-chave: *Cosméticos. Planejamento Fatorial. Cobertura Capilar.. Danos Capilares. Termoprotetor.*

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1 Introduction

The hair is one of the main parts of the human body that is directly related to the self-image. The hair comb has a social function and an impact on self-esteem and are one of the few body characteristics that we can easily change, whether in length, color or shape (BOLDUC; SHAPIRO, 2001). For many people, these changes involve daily processes, so the demand for products that meet these needs has thrilled the cosmetic industry in the last years (BHUSHAN, 2010).

The hair strand has basically three layers: cuticle, cortex and medulla. The outermost region is the cuticle, consisting of layers of keratin scales that overlap each other, with protective function, and acting as a barrier. When undamaged, it has a smooth surface that reflects light, yielding a natural hair bright. The cortex is the intermediate region responsible for the hair tensile strength and elasticity. This is the largest part of the hair shaft, granting its shape and color. Finally, the medulla is the innermost portion and may not be present in all the hair shafts (BOLDUC; SHAPIRO, 2001; SERRÃO *et al.*, 2018; LIMA *et al.*, 2019; KOCH *et al.*, 2020). The hair is mainly composed of the protein α-keratin, whose interactions, such as the disulfide bonds, hydrogen bonds, Coulomb interactions and other weaker interactions define the hair shape, whether straight, wavy, curly, etc. Moreover, the hair color is due to the melanin present in the cortex, a pigment produced in the melanocytes located in the hair bulb (DRAELOS, 1991; PARK *et al.*, 2018; SERRÃO *et al.*, 2018; KOCH *et al.*, 2020).

Thus, as the cortex constitutes the main hair structure responsible for shape and color, the cuticle is the first barrier to protect the whole hair fiber from external aggression. For the chemical transformations during hair dressing or dyeing to occur in the cortex, it is necessary that the cuticle scales are opened so that the cortex can be reached by the active substances. As many of these transformations involve redox reactions, it is inevitable that there are harmful side effects to the structure of the hair shaft (JOHNSON, 1997; KOCH *et al.*, 2020; PARK *et al.*, 2018).

In the search for different hair shapes, people often undergo various physical procedures that usually involve higher temperatures, such as hair brushes, hair dryers or hair straighteners (e.g., flat and curling irons). Although they are very efficient for the desired effect, these procedures also cause cumulative damage to the hair fiber, with weakening, dehydration, pH reduction and loss of the natural elasticity of the hair (JOHNSON, 1997; LIMA *et al.*, 2019). The heating process removes the free and strongly bound water from the hair fiber, which can lead to vaporization, carbonization and even decomposition of the hair shaft, with thermal denaturation of the keratin, breaking chemical bonds and unfolding the α-helix structure (LEE *et al.*, 2011; LIMA *et al.*, 2019).

To circumvent this degradation, heat protective cosmetics can be applied to the hair, seeking to minimize the damage. Hereupon, many compounds, such as the silicones, have been widely used as heat protectants due to their high resistance to heat, water or oxidizing agents. In addition, lipids and silicones are good electrical insulators and film formers, preventing the loss of water and keeping the hair hydrated, while improving the sensory aspect, in terms of combing and disentangling (DAVIS *et al.*, 2011; RELE; MOHILE, 2003; ZHOU *et al.*, 2011).

Several hair conditioning agents are good thermal protectors, either due to the antistatic effect, with the cationic compounds, or the emollient effect, for the presence of waxes, butters and oils in the composition. Among the lipid components routinely used in hair heat protectants, there are cetostearyl alcohol, shea butter and coconut oil. These products are commercially available in the form of creams and leave-on sprays (rinse off) to be utilized before or post-thermal exposure as pre-treatments or hair restoratives (DAVIS *et al.*, 2011; RELE; MOHILE, 2003; ZHOU *et al.*, 2011).

Products that spread more easily promote greater coverage of the hair shaft, protecting them more effectively. However, excessively high spreadability can cause the product to be easily removed from hair. Thus, this work aims to analyze the influence of the concentration of three lipid components, cetostearyl alcohol (a wax), shea butter and coconut oil, on the spreadability of hair heat protectants, using a central rotational composite design 2^3 for this analysis.

2 Material and Methods

2.1 Material

The cosmetic product components were: cetostearyl alcohol, propylene glycol and EDTA, obtained from EMFAL Empresa Fornecedora de Álcool (Betim, Brazil); shea butter, cyclomethicone 245, polyquartenium 7 and castor oil, purchased from MIX das Essências (Belo Horizonte, Brazil); refined coconut oil and cetyltrimethylammonium chloride (50%), obtained from Indústria Química Anastácio (São Paulo, Brazil); butylhydroxytoluene (BHT), purchased from Voest-Alpine (Linz, Austria); and Phenochem®, acquired from All Chemistry (São Paulo, Brazil).

2.2 Experimental design

In the first step, the components present in the formulations of the heat protectants were determined from a market survey of the main products commercially available, and the three most frequent lipids (among waxes, butters and oils) that could potentially influence the spreadability were selected.

Thus, to determine the influence of the concentration of these three components on the spreadability of the resulting cosmetic product, a central composite rotational design (CCRD) 2^3 was performed, with five replicates at the central point, totaling 19 experiments, each one corresponding to one product. The independent variables were the concentration of cetostearyl alcohol (X_1), concentration of shea butter (X_2) and concentration of coconut oil (X_3), analyzed at three equidistant variation levels, coded with -1, 0 and +1, in addition to two axial points (-1.68 and +1.68), considering the spreadability (Y) as the dependent variable (response). The experimental design matrix and the coded and real values of the independent variables are included in Table 1. The concentration ranges were defined in preliminary tests.

All the other components remained constant. Castor oil (10 mg/g) was included as emollient; BHT (0.1 mg/g), as an antioxidant; cyclomethicone (5 mg/g), as a sensory agent; polyquartenium-7 (5 mg/g), as a conditioning cationic polymer; cetyltrimethylammonium chloride (10 mg/g), as a cationic surfactant with conditioning properties; propylene glycol (30 mg/g) as a humectant; EDTA (1 mg/g) and Phenochem® (5 mg/g), as stabilizing agents; and water, as the vehicle (batch 100 g).

A second-order model was adopted in order to fit the response variables:

$$= \beta_0 + \sum \beta_i X_i + \sum \beta_i^2 X_i^2 + \sum \beta_{ij} X_i$$

where Y is the dependent variable, X_i and X_j are the coded independent variables, β_0 is the constant, β_i is the linear coefficient, β_i^2 is the quadratic coefficient and β_{ij} is the interaction coefficient (GONZALEZ-MIRA *et al.*, 2011; PELISSARI *et al.*, 2013).

Statistical analysis of the experimental data and the response surface methodology was performed using Statistica 7.0 software (StatSoft Inc, Oklahoma, USA) (GONZALEZ-MIRA *et al.*, 2011; PELISSARI *et al.*, 2013).

2.3 Preparation of the heat protectants

The emulsified formulations were prepared by hot-melt homogenization. First, the components were separated into oily phase (OP): cetostearyl alcohol, shea butter, castor oil, coconut oil and BHT; and aqueous phase (AP): propylene glycol, cetyltrimethylammonium chloride, EDTA and distilled water. Both phases were heated separately to 65 °C in a water bath and then the AP was slowly poured onto the OP, and mixed in a propeller stirrer (Q235-1 model, Quimis) at 100 rpm until complete cooling. At temperature below 40 °C, the following components were incorporated: cyclomethicone, Phenochem® and polyquartenium-7. At the end of the process, the products were placed in covered plastic cream jars and stored at room temperature, protected from light and heat.

2.4 Determination of the *in vitro* spreadability

The *in vitro* spreadability test was carried out at room temperature, according to method previously reported (SILVA *et al.*, 2019). The apparatus consisted of a circular glass plate (diameter = 20 cm; thickness = 0.3 cm), with a central hole of 1.2 cm diameter, placed on a support square glass plate (20 cm x 20 cm), which was positioned over a sheet of graph paper. The samples were added to the hole and the excess was removed. The circular plate was then removed and a glass slide (5 g) was positioned over the sample.

After 1 minute, the sample diameter was registered on two perpendicular directions to calculate the average diameter (d). All determinations were performed in triplicate and the average spreadability (S) was then calculated using the equation:

$$S = \frac{d^2\,x\,\pi}{4}$$

3 Results and Discussion

3.1 Formulation of the Heat Protectants

The composition of the nine heat hair protectants found on the market was analyzed and the most frequent thermoprotective agents present in the formulations were selected for inclusion in the composition of the developed product. Among them, three lipid components were present in all the analyzed products and were selected as potential influencers on the product spreadability: cetostearyl alcohol, shea butter and coconut oil. Thus, the concentration of these compounds was varied according to the CCRD matrix (Table 1), and 19 formulations were prepared.

Table 1 – Central composite design matrix and spreadability of the obtained heat hair protectants

Test	Independent Variables*			Spreadability (mm^2)
	X_1 (% w/w)	X_2 (% w/w)	X_3 (% w/w)	
1	-1.00 (2)	-1.00 (2)	-1.00 (2)	340.99
2	-1.00 (2)	-1.00 (2)	1.00 (6)	499.12
3	-1.00 (2)	1.00 (6)	-1.00 (2)	491.40
4	-1.00 (2)	1.00 (6)	1.00 (6)	552.53
5	1.00 (6)	-1.00 (2)	-1.00 (2)	394.53
6	1.00 (6)	-1.00 (2)	1.00 (6)	392.44
7	1.00 (6)	1.00 (6)	-1.00 (2)	294.00
8	1.00 (6)	1.00 (6)	1.00 (6)	222.92
9	-1.68 (0.64)	0.00 (4)	0.00 (4)	4427.86
10	1.68 (7.36)	0.00 (4)	0.00 (4)	299.10
11	0.00 (4)	-1.68 (0.64)	0.00 (4)	336.41
12	0.00 (4)	1.68 (7.36)	0.00 (4)	397.80
13	0.00 (4)	0.00 (4)	-1.68 (0.64)	380.39
14	0.00 (4)	0.00 (4)	1.68 (7.36)	423.46
15	0.00 (4)	0.00 (4)	0.00 (4)	330.52
16	0.00 (4)	0.00 (4)	0.00 (4)	335.89
17	0.00 (4)	0.00 (4)	0.00 (4)	340.99
18	0.00 (4)	0.00 (4)	0.00 (4)	330.26
19	0.00 (4)	0.00 (4)	0.00 (4)	375.03

*X_1 = cetostearyl alcohol concentration (% w/w), X_2 = shea butter concentration (% w/w), X_3 = coconut oil concentration (% w/w).
Source: resource data.

All the heat protectant creams had a bright white color, a characteristic odor, and a viscous and creamy appearance, which facilitate the product application. None of the products were so fluid that they could not be used in the *in vitro* spreadability studies. Thus, all the products were considered to have a potential good spreadability and could be utilized the further tests. The concentration range of the compounds, as proposed for the CCRD study, was also considered suitable, since all the products could remain emulsified and no incompatibility was observed.

3.2 *In vitro* spreadability

The 19 products were prepared according to the composition described in the CCRD matrix and analyzed as their *in vitro* spreadability. The corresponding results are displayed in Table 1. The spreadability values varied in the range of 294 to 4427.86 mm², and the highest spreadability was obtained in one of the products (test 9), also observed as the most fluid formulation. Moreover, as expected, the three chosen components (shea butter, coconut oil and cetostearyl alcohol) clearly had direct influence on the product spreadability.

Spreadability is referred as the area occupied by the formulation on a flat surface after being applied a constant pressure, in this case, a constant weight (5 g). For a good acceptance by the consumer market, it is basilar that the product presents good spreadability, as it results in proper distribution of the product on the skin, constituting a strong point in the consumer decision process (ESTANQUEIRO *et al.*, 2016).

To this moment, there is some limitations in establishing a standard for this *in vitro* spreadability test, as the values can vary according to the temperature, time and weight added to the sample (BORGHETTI; KNORST, 2006; DEUSCHLE *et al.*, 2015). Thus, previously published works had considered products with varied spreadability as suitable, with values ranging between 1100 – 2300 mm^2 (DANELUZ *et al.*, 2020), or 6000 – 13000 mm^2, with variations in the weights applied to the sample (0 to 2000 g) (BORGHETTI; KNORST, 2006).

3.3 Statistical Analysis

Throughout the cosmetics development, it is important to verify the product behavior within a certain experimental space. Considering that spreadability is a critical attribute for the efficiency of the product, it was essential to identify the factors that could affect this property. Thus, in this study, a CCRD 2^3 was used, in order to analyze the influence of the lipid components on the spreadability of the heat protectant.

The obtained spreadability data were submitted to the statistical analysis according to the proposed model, followed by an analysis of variance (ANOVA) with a confidence level of 90%. Only the significant data (Table 2) were used to analyze the behavior of the adjusted mathematical models in the equation:

$$Y = 369{,}19 - 550{,}92X_1 + 16X_3 + 567{,}76X_1^2 - 138{,}06X_2^2 - 125{,}75X_3^2 - 59{,}23X_1X_2 - 36{,}55X_1X_3 - 20{,}75X_2X_3$$

where Y is the spreadability of the heat protectants and X is the coded variables, X_1 = concentration of cetostearyl alcohol, X_2 = concentration of shea butter, and X_3 = concentration of coconut oil.

Table 2 – Regression coefficients of the adjusted equation and analysis of variance (ANOVA) for spreadability (dependent variable) of the central composite design

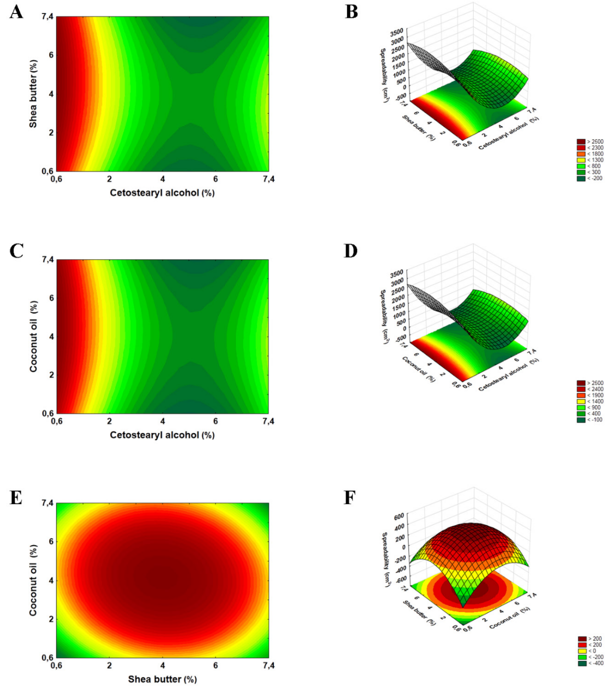
Parameter	Coefficient	Result	p-value
Linear	β_0	369.19	0.000002
	β_1	-550.92	0.000000
	β_2	-	-
	β_3	567.76	0.000000
Quadratic			
	β_1^2	-138.06	0.000011
	β_2^2	16.00	0.034067
	β_3^2	-125.75	0.000016
Interaction			
	β_{12}	-59.23	0.000857
	β_{13}	-36.55	0.005220
	β_{23}	-20.79	0.034857
R^2	-	0.62	-

*X_1 = cetostearyl alcohol concentration (% w/w), X_2 = shea butter concentration (% w/w), X_3 = coconut oil concentration (% w/w).

Source: resource data.

The coefficient of determination (R^2) found in the ANOVA was 0.62, with $p < 0.05$, showing that the change in the concentrations of the components significantly influences the product spreadability (Table 2). From the experimental design, the response surfaces involving the spreadability of the products as a function of the concentration of the components (Figure 1) were then possible to be obtained.

Figure 1 – Response surfaces of spreadability as a function of the concentrations of shea butter and cetostearyl alcohol, 2D (A) and 3D (B); concentrations of coconut oil and cetostearyl alcohol, 2D (C) and 3D (D); concentrations of shea butter and coconut oil, 2D (E) and 3D (F)



Source: resource data.

When observing the interaction profile between shea butter and cetostearyl alcohol (Figures 1A-B), there is greater spreadability in the concentration of shea butter around the central point (4%) and in the lower concentrations of cetostearyl alcohol (0.64%). Regarding the effect of the interactions between coconut oil and cetostearyl alcohol (Figures 1C-D), similarly, greater spreadability occurs in the concentration of coconut oil in the region of central point (4%) and in the lower concentrations of cetostearyl alcohol (0.64%). Finally, in the interaction between shea butter and coconut oil, a response surface with a different profile was obtained, with greater spreadability around the central point for both components (4%) (Figures 1E-F).

Thus, cetostearyl alcohol was a component with high influence on spreadability, since a product with lower viscosity was obtained with low concentrations of cetostearyl alcohol, but still keeping the consistency of a semi-solid cream, and with higher spreadability. In fact, cetostearyl alcohol is a lipid wax with the highest melting point (m.p. = 51-59 °C) (FUKUSHIMA *et al.*, 1976), when compared to shea butter (m.p. = 36.6 °C) (SAMUEL, 2017) and coconut oil (m.p. = 24.4 °C) (YOUNG, 1983).

Moreover, the other two components showed significant interactions with cetostearyl alcohol and between themselves in influencing the product spreadability, as could also be seen for the presence of the interaction coefficients in the adjusted equation of the model. This indicates that the components not only individually influenced the spreadability, but also their interactions influenced this critical attribute of the product (RODRIGUES; IEMMA, 2005).

Finally, the importance of carrying out a central composite design in the development of cosmetic products was demonstrated, since the presence of significant linear, quadratic and interaction coefficients in the adjusted equation of the model was observed. Therefore, the one-at-a-time approach, which varies only one component at each prepared formulation, should be less efficient as it does not deeply explore the entire space of solutions (RODRIGUES; IEMMA, 2005).

4 Conclusion

Formulations of hair heat protectants were developed and the influence of the lipid components cetostearyl alcohol, shea butter and coconut oil on the spreadability of the resulting product could be determined. All the three components directly influenced the spreadability of the final product, with greater influence of cetostearyl alcohol when analyzed alone, as higher spreadability was obtained in the product with lower concentrations of this wax (0.64%), and in the central values of shea butter and coconut oil (4%). Significant interactions were also observed between the influences of the three components, thus demonstrating the importance of carrying out a central composite design in the development of similar products, instead of varying one factor at a time. Therefore, optimized spreadability can be obtained by varying the composition of the hair protectants, aiming a proper distribution of the product throughout the hair shaft, with a complete coverage and greater protection of the hair against the degradation by the thermal agents.

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