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Development, physicochemical and functional analysis of anti-frizz leave-on emulsion with coconut oil

Flavia Scigliano Dabbur[1,2]*, Gisele Hédla da Silva Lima[1], Rosana Maia Costa[1], Clindia Lourenço Costa[1], Laís Estephane Silva Santos[1], Valesca Barreto Luz[3], Camila Calado de Vasconcelos[1,3], Josefa Renalva de Macêdo Costa[1]

[1]Curso de Farmácia, Centro Universitário Cesmac, Rua Cônego Machado, 984, 57051-160, Farol, Maceió – AL, Brazil
[2]Faculdade de Farmácia, Laboratório de Cosméticos, Universidade Federal do Rio Grande do Norte, Rua General Gustavo Cordeiro de Faria, s/n, 59012-570, Petrópolis, Natal – RN, Brazil
[3]Mestrado Profissional em Biotecnologia em Saúde Humana e Animal. Centro Universitário Cesmac, Rua Angelo Neto, 51, 57151-530, Farol, Maceió – AL, Brazil

*Correspondence to
Flavia Scigliano Dabbur
Email:
fladabbur@gmail.com

Abstract
Introduction: The cosmetics industry increasingly offers a greater variety of products to consumers, as people have a constant need to change their appearance. For the past years, women have followed a tendency to have their hair back to its natural color, since it was altered by chemical straightening for a long time. The purpose of this paper was to produce and assess the physicochemical stability of a leave-on emulsion formulation containing coconut oil for anti-frizz action.
Methods: It was developed a cationic emulsion formulation, with coconut oil as the active agent in 0.5% and 1.0% concentrations. It was performed functional tests in damaged hair strands. A preliminary stability test was conducted, and the samples were packaged in polyethylene containers, and subjected to different conditions (temperatures of 5°C, 25 °C and 45°C and direct exposure to sunlight). They were analyzed on day 0 and on day 15. The physicochemical analyses were: organoleptic characteristics (color, odor and appearance), pH, spreadability, apparent density and centrifugation. The functional tests (combability test and test on strands of hair) were conducted in strands of damaged hair. For the combability test, which measures the easiness of combing, the strands were previously washed with neutral shampoo. The samples, after being applied, were manipulated for 1 minute in the strands. After the application, a fine-tooth comb was slid through the hair length for at least 5 times. Then we measured the slid length. For the test on strands, they were placed in room temperature (23ºC) and relative humidity (33%) for 24 hours. After such period, we considered half the length of strands for width. After spreading, we examined the anti-frizz action. The results were photographed. The statistical test t was applied: sample data, with the BioEstat 5.3 program.
Results: We found that the color and odor were slightly altered only in samples exposed to sunlight. As for appearance and centrifugation, the samples remained homogeneous under all conditions. The pH of the samples ranged 4.0 – 4.6. In the spreadability test, we found variations of 7.93 –14.72. In the combability test, there was no significant difference in slid between the formulation with coconut oil 0.5% and the formulation with coconut oil 1.0%. Nevertheless, there was difference when they were compared to the sample without the active agent.
Conclusion: In the test on strands of hair, the sample with 1.0% of the active agent (coconut oil) had the best volume result.
Keywords: Hair. Quality control, Cosmetics, Coconut oil, Leave on emulsion.

Introduction

Hair care was a significant feature in Ancient Egypt, and archeological excavations found a great number of brushes, treatment lotions and other products for hair use.[1]

Human beings have a constant need to change their appearance. Such tendency may be connected to social issues and to people's ability to provide personal wellbeing. Hair is considered adornment.[1,2] Hair contributes to a person's image – either classical or alternative. It is associated with many concepts of boldness, youth, freedom, seduction and power. Hair is the most remarkable and variable feature of human beings, and it becomes a fundamental ingredient in one's identity.[3]

In the past years there has been an increasingly structured tendency in that women no longer accept having their hair altered by straightening. Many women, who once chose chemical alteration, want to restore their natural hair. In Brazil, this is known as "Hair Transition". It consists of letting hair grow and gradually have the

chemical remains cut off, until hair is totally natural. This process has been the focus of interest, having generated a new market.[4]

Hair can be generically classified into three big groups: Caucasian, Oriental (or Mongolian) and African-American (curly). The distinction is mainly originated by the difference in ellipticity. The difference in the format of hair strands affects physical characteristics, mechanical resistance, surface properties and reactivity to chemical agents.[5]

Hair growth happens in rhythms and cycles, having three phases which are repeated during one's lifespan. The phases are called anagen, catagen, and telogen. The anagen phase is the growth cycle; the catagen phase is the transition stage, signaling the end of the previous phase; and the telogen phase is the resting stage.[6]

Conditioners are substances which detangle hair, facilitate hairstyle, and reduce aggression from physical and chemical effects to which hair is subjected on a daily basis, such as the mere act of combing. They maintain the cosmetic appearance and softness of hair and reduce the frizzy aspect (anti-frizz effect). Conditioners are made of vegetal and mineral oils, waxes, long-chain alcohols, cationic substances, triglycerides, esters, silicones e fatty acids.[7]

Leave-on creams are emulsions that decreases the formation of static electricity on hair fibers. By reducing the charges on the keratin surface, they reduce the static aspect of hair. Furthermore, they cover the hair strands with lipophilic molecules, which incorporate the greasy material contained in the product that overgreases hair again. Such actions restore shine, softness and combability to hair.[8]

Emulsions are cosmetic preparations which may be unstable and undergo alterations, depending on their formulation and on packaging and storage conditions. Such conditions may be external, such as high temperature, direct light, direct contact with oxygen, high relative humidity, packaging material and microbial contamination. They may also be internal, related to the formulation composition and the interaction among ingredients, or interaction with the packaging material, physical and chemical incompatibility, such as pH, oxidation-reduction reactions, and hydrolase reactions.[9]

The formulation should remain stable. Any sign of instability indicates the need for reformulation. In order to provoke a possible instability, the pre-stability and accelerated stability tests are conducted. Such tests should be carried out under conditions that allow information on product stability in as short a time as possible. In order to achieve such purpose, samples should be stored under conditions that accelerate changes which are likely to occur during the validity period, i.e., before the expiration date.[10]

This paper is relevant, as there is a high rate of hair transition, in which women tend to abandon straightening procedures, in favor of natural hair. Therefore, the demand for anti-frizz products is likely to increase, in order to make the best of hair and ensure its shine, softness, conditioning and alignment, which brings about satisfaction and wellbeing.

The purpose of this paper was to develop and analyze the physicochemical and functional aspects (in strands of damaged hair), of a leave-on emulsion formulation with coconut oil (*Cocos nucifera* oil).

Materials and Methods

The anti-frizz formulation of the leave-on emulsion was adapted from Correa[11] Lauric oils are obtained from coconut trees, native to tropical countries. They stand out, when compared to other kinds of oils, for their high concentration of lauric acid. The use of such oils in the cosmetics industry, as vehicles for massage products or in cream formulations, is an excellent alternative, with the advantage of not becoming rancid easily, even in contact with water in cream formulations. They also have high durability. Table 1 shows the description of the anti-frizz formulation, which was developed and tested, along with concentration percentage and critical study of components.

Preliminary Stability Test

The preliminary pre-stability tests were performed according to ANVISA Guide for Cosmetics.[10] The samples were fractionated and packaged in polyethylene containers, and then subjected to three different temperatures (5°C, 25°C and 45°C) and to direct exposure to sun. They were evaluated on day 0 and on day 15.[10]

Physicochemical Analyses

The following tests were conducted: organoleptic characteristics – color, odor and aspect; pH direct reading at 10% solution; and centrifugation 300 rpm for 30 minutes.[9,12]

Spreadability is defined as the expansion of a semi-solid formulation on a surface, after a certain period of time.[13]

Functional Tests

The tests were adapted from protocols of companies that carry out security and efficiency tests in cosmetics.[14]

We acquired strands of damaged hair, ideal for final tests of products. The choice for damaged hair was due to the fact that its strands are more appropriate for applying the active anti-frizz agents.

Tests on Strands of Hair

The tests were adapted from protocols of companies that carry out security and efficiency tests in cosmetics. The hair strands were weighed (approximately 16 g each) and divided into 5 equal parts.

All strands were shampooed with a standardized amount of 2 mL neutral shampoo for 2 minutes and rinsed for 1

Table 1. Antifrizz Leave on Emulsion Formulation

Ingredients (INCI Name)	%	Function
Quaternium-18	1.00	Conditioning agent
Phenoxyethanol, Methylparaben, Ethylparaben, Propylparaben, Butylparaben, Isobutylparaben	1.00	Preservant
Butylated hydroxytoluene	0.05	Antioxidant
Cetearyl Alcohol	5.00	Emulsifier
Dissodium EDTA	0.20	Chelating agent
Propylene Glycol	3.00	Wetting agent
Aqua	qsp	Vehicle
Cocos nucifera oil	0.50/ 1.00	Active Antifrizz

Source: Adapted from Correa11.

minute. For removing excess water, the strands were dried with 2 paper towels.

One strand, only shampooed, was considered as a control, the other four were added 0.50 g of each sample, being sample A leave-on base formulation, sample B leave-on base formulation added 0.5% of coconut oil, sample C leave-on base formulation added 1.0% of coconut oil, sample D leave-on commercial product containing coconut oil.

Combability Test

After the samples were applied to hair, a fine-tooth comb was slid through hair length in order to observe the slip point. The distance covered by the comb was measured in centimeters, after 10 combing strokes.

The procedure was conducted by the same person, so that the final result was not altered. One characteristic of the combability test is that it has the same strength and the same rhythm when the strands are tested.

Test on Strands of Hair

After the combability test, the strands were detangled and placed in controlled temperature and relative humidity, 23°C and 33%, respectively, for 24 hours. After that the visual analysis of hair was conducted. The results were photographed.

Results and Discussion

Hair is part of male and female beauty and it should be treated well. Some strands are more fragile and require special treatment during beauty procedures.

The preliminary pre-stability tests consist of conducting tests in the initial phase of product development, by using different formulations with reduced durations (15 days).

Extreme temperature conditions were applied, in order to accelerate possible reactions among the components and the appearance of signs, which should be observed and analyzed, according to the characteristics of each type of product. This study does not aim at estimating product duration, but to aid with the screening of formulations.

Tables 2, 3 and 4 present the results of the physicochemical analyses of the leave-on emulsion, of the formulation with 0.5 % active agent and of the formulation with 1.0% active agent.

In the three samples, in the organoleptic analyses, we found that the color of the emulsion base formulation, when exposed to thermal stress – when compared to Day 0 – was not altered in white light nor in natural light. Only when the sample was exposed to direct sunlight, the color changed to yellowish, which indicated that one of the lipid components was oxidized.

The same performance was found as for odor. The sample turned rancid, which demonstrates that the incidence of direct light caused alterations in the formulations.

The change to color and odor may be related to the excessive microbial growth, resulting from the degradation of the preserving system, due to light.[15]

A suggestion for improving the leave-on emulsion formulation would be the addition of another antioxidant agent, such as oily vitamin E. This would provide a better antioxidant system, to avoid change to color and odor, and thus preserve the lipid components. An emulsion has a lipophilic part, which may undergo oxidation reactions, intensified by light.[15]

The pH underwent acidification in all samples of day 15, when placed under thermal stress, and upon incidence of direct sunlight. This result is below the ideal condition for an anti-frizz conditioning formulation. The pH is a very significant parameter, as alteration in its value may suggest chemical modifications to the formulation components. As emulsions are thermodynamically unstable systems, the sources of instability are common.[11,15]

However, in a stability study of an emulsion containing urea, Prestes et al,[16] reported that some samples showed higher pH in the formulations after thermal stress (45ºC), which differs from the findings presented here in.

As for appearance, the sample remained homogeneous and shiny throughout the period of the pre-stability test, even after the physical stress of centrifugation. This demonstrates physical stability of the sample.

The study conducted by Prestes et al[16] is in accordance with this study, in that the samples were stable when subjected to centrifugation throughout the stability test.

According to Borghetti and Knorts,[13] in a stability test for emulsions containing sunscreen, the formulations did not show alterations in appearance, color, odor and pH, after 6 months. Differently, this study showed alteration in color, odor and pH after only 15 days.

Table 2. Physicochemical Analysis of a Sample A (Base Leave on Cream)

	T0	T15			
		5ºC	25ºC	45ºC	Sunlight
Color (NL)	NC	NC	NC	NC	Changed
Color (AL)	NC	NC	NC	NC	Changed
Aspect	Homo	Homo	Homo	Homo	Homo
Smell	NC	NC	NC	NC	Changed
pH	4.63	3.79	3.98	4.04	4.10
Ei	14.72	8.45	8.29	8.52	7.93
Centrifugation	NC	NC	NC	NC	NC
Density	1.06 g/mL	1.02 g/mL	1.05 g/mL	1.01 g/mL	1.20 g/mL

Color: Natural Light (NL); Artificial Light (AL); No Change (NC), Homogeneous (Homo); Heterogeneous (Hetero).

Table 3. Physicochemical Analysis of a Sample B (Leave on with Coconut oil a 0.5%)

	T0	T15			
		5ºC	25ºC	45ºC	Sunlight
Color (NL)	NC	NC	NC	NC	Changed
Color (AL)	NC	NC	NC	NC	Changed
Aspect	Homo	Homo	Homo	Homo	Homo
Smell	NC	NC	NC	NC	Changed
pH	4.47	4.00	4.00	4.10	4.00
Ei	14.32	7.66	7.74	7.97	7.93
Centrifugation	NC	NC	NC	NC	NC
Density	0.95 g/mL	1.03 g/mL	1.17 g/mL	0.99 g/mL	1.13 g/mL

Color: Natural Light (NL); Artificial Light (AL); No Change (NC), Homogeneous (Homo); Heterogeneous (Hetero).

Table 4. Physicochemical Analysis of a Sample C (Leave on with Coconut oil a 1.0%)

	T0	T15			
		5ºC	25ºC	45ºC	Sunlight
Color (NL)	NC	NC	NC	NC	Changed
Color (AL)	NC	NC	NC	NC	Changed
Aspect	Homo	Homo	Homo	Homo	Homo
Smell	NC	NC	NC	NC	Changed
pH	4.47	4.14	4.00	4.10	3.94
Ei	10.52	8.05	6.44	7.66	8.16
Centrifugation	NC	NC	NC	NC	NC
Density	1.04 g/mL	0.99 g/mL	0.99 g/mL	0.99 g/mL	1.17 g/mL

Color: Natural Light (NL); Artificial Light (AL); No Change (NC), Homogeneous (Homo); Heterogeneous (Hetero).

The spreadability test is a physical test which has direct implications on distribution, uniformity and applicability of the emulsion on the strands. Between Day 0 and Day 15, the values presented little variation, which showed physical stability of the formulation.

Table 5 shows the sliding results, measured in centimeters, i.e., how much the comb slid on the strands after the product was applied.

In this test, we analyzed which formulation provided better sliding of strands when they were combed. The formulation containing 0.5 % and 1.0 % coconut oil had very similar results. When compared, the difference in sliding between them was small.

In comparison with the base formulation, we found that there was greater sliding, as the base formulation without the active agent did not slide as much on the strands.

Table 5. Results of Sliding Comb in Strands, in Centimeters, After 5 Combs with Samples

Maximum Slip Point (cm)	Sample
5.5	Control
4.0	A
9.5	B
10.0	C
8.0	D

When the active agent was added, the strands were more moisturized. Hence, the sliding was better it has been shown on Figure 1.

After the sliding procedure, the strands of hair were placed, for 24 hours, in a controlled environment, with 22°C temperature and 33 % relative air humidity, Figure 2.

The strands were measured at half length, to evaluate their width, after sample application and controlled packaging. It was visible that sample D showed more aligned strands.

Conclusion

The leave-on emulsion formulation showed oxidation. A suggestion for improving the formulation would be the addition of another antioxidant agent, such as oily vitamin E, in order to improve the antioxidant system. In the functional tests, the sample with the best results for

volume and combability was the sample D with 1.0 % coconut oil.

It is important to note that, in order for the formulation to have viable acceptance, it should be reformulated and, afterwards, undergo the accelerated stability test and functionality test again. It is possible, however, with prior laboratory tests, to conduct many preliminary tests with different formulations and active agents.

Competing Interests

None.

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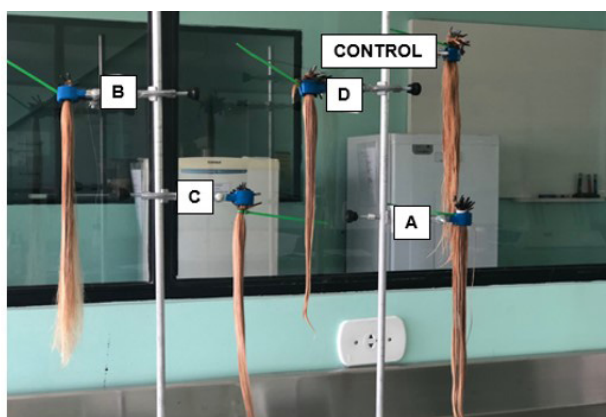
Figure 1. Hair strands after shampooing and applying the samples. Control: shampoo; A: leave on base; B: leave on with coconut oil 0.5%; C: leave on with coconut oil 1.0%; D: commercial leave on.
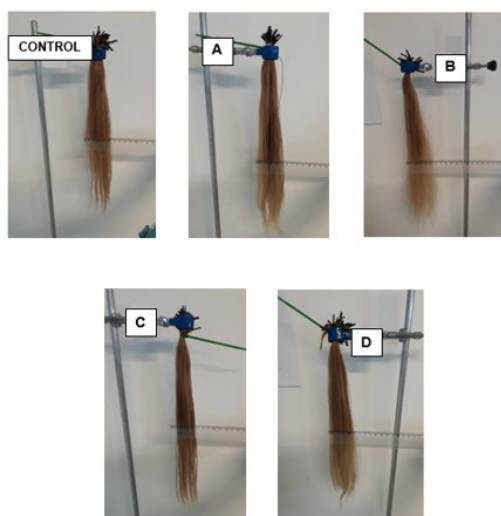
Figure 2. Hair damaged strands, with 0.50 g of each sample stored at 22°C and 33% relative humidity for 24 hours. Control: shampoo; A: leave on base; B: leave on with coconut oil 0.5%; C: leave on with coconut oil 1.0%; D: commercial coconut oil leave on.

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