Development of hair care formulations based on natural ingredients

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Abstract

Introduction: The development of hair care cosmetic formulations with ingredients obtained from natural sources is a challenge since it can compromise the stability and performance of formulations. The search for innovative ingredients obtained from natural sources is important to overcome this challenge. In addition, the evaluation of the physicomechanical and sensorial proprieties, stability and effectiveness of hair formulations are fundamental to obtain formulations with good acceptance by consumers. In this context, the aim of this study was to develop and evaluate the stability, physical-mechanical, and sensorial properties, as well as the efficacy of hair care formulations based on natural ingredients.

Methods: Shampoo and conditioner formulations containing natural ingredients such as hydrolyzed rice peptides and polysaccharides obtained from plants and biotechnological processes were developed. Stability, spreadability, hardness, texture profile, rheological behaviour and sensory properties of these formulations were evaluated. In addition, the efficacy of hair care formulations was assessed by break force, combability and brightness of hair samples.

Results: Shampoo formulation containing hydrolyzed rice peptides had better results in the texture, hardness and spreadability parameters. Conditioner formulations with polysaccharides showed better performance in the tests. Regarding the hair effectiveness tests, all treatments increased the mechanical strength of the hair fiber as well as the strength in the combing test, but only shampoo formulation containing hydrolysed rice peptides increased the brightness of the hair. Moreover, the sensory analysis showed a correlation with the efficacy test by instrumental measurements.

Conclusion: This study has a significant contribution since it showed that it is possible to develop stable and efficacy formulations with ingredients obtained from a natural source and the importance of the correlation between instrumental measurement data and sensory analysis.

Keywords: Hair care, Cosmetics, Efficacy, Sensorial properties, texture profile, polysaccharides

Introduction

Hair promotes well-being in people and also protects the scalp from solar radiation and mechanical abrasion. The development of hair care formulations is important for the treat and protect the hair fiber from daily external hair aggressions. The primary functions of hair care formulations improve the physical properties of the hair fiber, such as texture, strength, and combability. The secondary functions are the improve of sensory proprieties, such as brightness, softness, frizz reduction and hair film formation.\(^1,2\)

Shampoos are liquid formulations composed mainly of anionic surfactants that remove dirt from the hair and scalp. Conditioners are emulsion-type formulations composed of cationic surfactants, which neutralize the hair fiber and provide conditioning effect. Actives substances can be added to give a treatment function to the hair fibers.\(^1,2\)

The search for suitable raw materials is one of the most important stages of the protocol for research and development of cosmetic products.\(^3\) Nowadays, there is a global trend towards the use of natural based ingredient formulations. In addition, the improvement of technologies and techniques of extraction, isolation, characterization, and production are increased market demand for natural ingredients.\(^1\)

Some ingredients of natural origin have potential for application in the development of hair care formulations. Hydrolyzed rice peptides could have potential for repair and fortify the damaged hair fiber. In addition, it could provide a conditioning and film effect, which protects the hair from external aggressions.\(^4\) A blend of vegetable polysaccharides, green tea, panthenol, and xylitol is an innovative raw material with antioxidant and film forming properties. It has the potential of anti-pollution effect, protection against photodegradation and improves

brightness, malleability and strength. A biotechnological polysaccharide, derived from bacterial fermentation, is another innovative raw material which promotes film forming on hair fiber. This active substance could reduce the effects of external aggressions, especially pollution, solar radiation, heavy metals and surfactants. Moreover, it improves the texture and touch sensation of formulations. Hemisqualane is a sugarcane-derived saturated hydrocarbon with a light, non-volatile, dry-touch emollient action that gives the formulation an appearance and sensory property equivalent to synthetic silicones. It is a natural alternative to this type of raw material. The hair application provides a soft and silky hair fiber, besides good spreadability and texture of formulation.

The research and development process requires some analyzes to ensure that formulations are pleasing and have quality and safety. Stability assessments determine the shelf life of the product against external factors and storage conditions. The analysis of sensory properties, rheology behavior, and texture profile are important to understand the behavior of ingredients in the formulation and its acceptability by the consumer. In addition, studies of effectiveness in hair fiber are essential to evaluate the performance of hair care formulations and consumer acceptability. Tensile and combability tests can be performed using the TA XT Plus® Texturometer (Stable Micro Systems) on the hair fibers. Noninvasive image analysis using Skin Glossymer GL 200 determines hair brightness through the principle of reflectance. In this context, the aim of this study was to develop and evaluate the stability, physical-mechanical and sensorial properties of hair care formulations based on natural ingredients as well as the efficacy of these formulations in the improvement of mechanical properties and gloss of hair.

Materials and Methods

Formulations Development

The developed formulations of shampoos were based on disodium laureth sulfosuccinate, sodium laureth sulfate, lauryl glucoside, quar hydroxypropyltrimonium chloride, xylitol sesquicaprylate, disodium EDTA, propylene glycol, sodium chloride, citric acid, and aqua. The active substance used was 2% of hydrolyzed rice protein.

The developed formulations of conditioners were based on cetyl alcohol, C13-C15 alkane, Hydroxyethylcellulose, behentrimonium chloride, polyquaternium-10, xylitol sesquicaprylate, disodium EDTA, propylene glycol, and aqua. The actives substances used were 2% of a biotechnological polysaccharide (biosaccharide gum-4) in one of the studied formulation and 2% of a blend of vegetable polysaccharides, green tea, panthenol and xylitol (Water (and) galactoarabinan (and) Salvia hispanica seed extract (and) glycerin (and) trehalose (and) xylitol (and) panthenol (and) Camellia sinensis leaf Extract (and) sodium phosphate) in another formulation.

Stability Tests

Determination of pH and Evaluation of Organoleptic Characteristics

The formulations were stored at different temperatures (25, 37, 45, and 4°C) for 28 days. Every seven days were evaluated the organoleptic characteristics (color, odor, aspect) and determinate the pH in triplicate.

Centrifugation Test

The formulations were submitted to three cycles of 30 minutes at 3000 rpm in the centrifuge (CENTRIBIO®) to see possible phase separation or changes in the composition of the formulation.

Determination of Viscosity

Ford Cup viscometer determines the viscosity of a solution through a graph of flow time versus thickness. Ford cup viscometer was filled with the shampoo formulation until it overflowed, and then the orifice was released to start counting time until the first flow has stopped. Measurements were made in triplicate and mean times were used to determine centipoise viscosity (cP) on the graph.

Rheological Behavior

The Brookfield digital rheometer model RVDV3T, equipped with the CP-CPA-52Z spindle and connected to the Rheocalc® software, was used to evaluate the apparent viscosity of emulsions through deformation and flow properties in function of an applied shear rate. The outward curve was formed with a progressive increase in rotational speed, while the return curve with a gradual reduction of this speed. Analyzes were performed in triplicate at the initial time, 14 and 28 days for conditioners stored at 25, 37, 45, and 4°C. The parameters used for evaluated 0.5 g of formulation were initial rotational speed of 0 rpm, a final rotational speed of 20 rpm with 6 points and time between each speed of 10 s.

Analysis of Texture Profile, Spreadability, and Hardness

Measurements were made in triplicate on TA XT Plus® Texturometer (Stable Micro Systems). The texture profile determination consists of the insertion, with predefined speed and depth, of the back extrusion probe into the formulation, generating a graph of Strength (g) x Time (t) that provides work of shear values, viscosity index, consistency, firmness, and cohesiveness. Shampoos formulations analysis was used the probe A/BE d45 with diameter 45 mm, return distance 100 mm, return speed 20 mm/s and contact force 10 gF. Conditioners formulations analysis was used the probe A/BE d40 with diameter 40 mm, return distance 100 mm, return speed 20 mm/s and contact force 30 gF. The spreadability test consists of insert an analytical probe (HDP/SR) into the formulation which provides the “Work of Shear” parameter. A smaller value of this parameter indicates good spreadability.
Shampoo formulations parameters were returned distance 25 mm, return speed 20 mm/s and contact force 15 gF. Conditioners formulations parameters used were return distance 25 mm, return speed 20 mm/s and contact force 30 gF. Hardness test consists of the insertion of a cylindrical probe into the formulation provides the parameters of hardness, elasticity, cohesiveness, adhesiveness, stickiness and compressibility.\textsuperscript{11} Shampoo formulations parameters used in quintuplicate were P/50 aluminum cylindrical probe, pre-test speed 2 mm/s, distance 17 mm, trigger force 15 gF, return distance 80 mm, post-test speed 1 mm/s.

**Efficacy Tests**

**Hair Sample Preparation**
The hair sample was brown without chemical treatments or dyes. The washing protocol consisted of 3 cycles per hair samples of 4 minutes each. The first step of the cycle was to wash the hair samples for 1 minute with shampoo and rinse. Then, conditioner formulation was applied for a minute and rinsed. All hair samples were dried with a 1200 W Taiff\textsuperscript{®} hair dryer 15 cm apart for 5 minutes.

**Hair Sample Treatments**
Seven hair samples were submitted for the treatment with the developed formulations. Hair sample H1 was treated with shampoo and conditioner without active substances. Hair sample H2 was treated with shampoo without active substances and conditioner with a blend of vegetable polysaccharides. Hair sample H3 was treated with shampoo without active substances and conditioner with the biotechnological polysaccharide. Hair sample H4 was treated with shampoo with rice peptides and conditioner without active substance. Hair sample H5 was treated with shampoo with rice peptides and conditioner with a blend of vegetable polysaccharides. Hair sample H6 was treated with shampoo with rice peptides and conditioner with the biotechnological polysaccharide. Hair sample C was not treated.

**Hair Combing Analysis**
The combing test measures the force required to comb hair samples. The higher the force required to comb the hair sample, the more damaged is the fiber.\textsuperscript{1,2,13} Two non-metallic combs were mounted on the TA XT Plus\textsuperscript{®} Texturometer (Stable Micro Systems). Twenty-five hair fiber from each hair sample was separated and attached to the machine. The parameters used were return distance 55 mm, return speed 20 mm/s, and contact force 50 gF.

**Tensile Test**
The tensile test gives the average value of the maximum force required to break the hair fiber. The increase of maximum force needed to break the hair fiber after treatment suggests action on the internal structure of the hair fiber.\textsuperscript{1,2,13}

It was performed on the equipment TA XT Plus\textsuperscript{®} Texturometer (Stable Micro Systems). Twenty-five hair fiber from each hair sample was separated and attached to the machine. The parameters used were return distance 55 mm, return speed 20 mm/s, and contact force 50 gF.

**Hair Gloss Analysis**
Brightness analysis was evaluated with the equipment Skin Glossymeter\textsuperscript{®} GL 200 (Courage & Khazaka). This equipment measures the surface gloss of the hair sample. The principle of the method is about reflectance. High values indicate a hair sample with a regular surface. Measurements were made in triplicate in three regions of each hair samples.\textsuperscript{14}

**Hair Sensory Analysis**
The hair sensory analysis was applied to 10 subjects. The parameters analyzed were softness, brightness, hydration, combability, and appearance. After look and touch the hair samples, the items rated these parameters as poor, regular, good or very good. The scores obtained were transformed into frequencies.

**Statistical Analysis**
The results obtained were statistically analyzed using GraphPad Prism 6\textsuperscript{®} and Origin 8\textsuperscript{®} software. The statistical tests used varied according to the sample distribution obtained. All data were considered nonparametric and compared by analysis of variance (one-way ANOVA) followed by the post-tests Kruskal Wallis, Mann-Whitney, or Student’s t test (P<0.05).

**Results and Discussion**

**Stability Tests**

**Determination of pH Values and Evaluation of Organoleptic Characteristics**
Shampoo and conditioner formulations did not change the color, odor, and appearance during 28 days at different temperatures (25, 37, 45 and 4°C). The pH of shampoos remained between 5.5 and 6.0 (Figure 1). This pH is ideal for not damaging the hair fiber and also is compatible with the pH stability of the hydrolyzed rice protein (5.0-6.0).\textsuperscript{4} The conditioners had a pH between 4.5 and 5.0 (Figure 1). The ideal pH of conditioner formulations should be around 4.0-4.5, due to the neutralization of hair ions is close to the keratin isoelectric point (3.7).\textsuperscript{15} However, a blend of vegetable polysaccharides has a pH of stability between 4.5 and 7.05, requiring the adjust of this value. After this improve, pH 5.0 was obtained for the vehicle, and this could justify the range in the stability test.

**Centrifugation Test**
The conditioner formulations did not show phase separation after the centrifugation test. Shampoos formulations have single phase, so they were not submitted
Development of hair care formulations

Determination of Viscosity

The viscosity of shampoo formulations obtained on the Ford Cup No. 6 viscometer was 3300 cP to shampoo formulation without active substance (Sv) and 1700 cP to shampoo formulation with hydrolyzed rice protein (Srp). Despite the large difference between Sv and Srp, both were prepared in the same way and considered with suitable viscosity for this type of formulation.

Rheological Behavior

Conditioner formulations showed a non-Newtonian pseudoplastic flow behavior with thixotropy due to a decrease in apparent viscosity as a function of increased shear rate and the recovery of initial viscosity after removal of shear rate. In characterization (T0), the conditioner formulation without active substances (Cv) at room temperature showed the flow rate of 0.411. The flow rate of conditioner formulation with a blend of vegetable polysaccharides (Cvp) was 0.402, and biotechnological polysaccharide (Cbp) was 0.401. Thus, at characterization, the formulation with the lowest flow rate was the conditioner formulation with biotechnological polysaccharide, but it was not statistically different from other formulations to assume better spreadability. Moreover, the flow rate was close among the three formulations during the 28 days of study (Figure 2). This parameter may be related to the spreadability of the formulations. The apparent viscosity of the Cv was higher when compared to Cvp and Cbp at time zero. This means that the Cv flows with more difficult since higher viscosity values make it difficult to flow. The apparent viscosity of the Cv was higher when compared to Cvp and Cbp at time zero. This means that the Cv flows with more difficult since higher viscosity values make it difficult to flow. In 14 days, the formulations Cv and Cvp had their apparent viscosity considerably increased at 45°C and, at the other temperatures, all formulations had very close apparent viscosities. In 28 days (T28) was showed the viscosity decrease of Cv and Cvp at 37°C. However, the Cbp viscosity increased at 45°C. The apparent viscosity at 4 and 25°C in 28 days had no significant difference between T0 (Table 1). Despite these slight variations, the formulations maintained their behavior during the study period.

Analysis of Texture Profile, Spreadability, and Hardness

The evaluation of the physical-mechanical properties of a formulation through its texture, spreadability, and hardness profile provides important parameters. These parameters can be correlated with the sensory characteristics of the formulations and consumer acceptance.

Shampoo formulation with hydrolyzed rice protein (Srp) presented lower values of cohesiveness, consistency, firmness, viscosity index, and work of shear in the analysis of texture profile, spreadability and hardness when compared to the vehicle formulation (Sv) (Figure 4). The cohesiveness is the union of particles in the formulation, which form a polymer network. The
higher the cohesiveness, the stickier is the formulation. The viscosity index is directly related to consistency. These parameters were higher for vehicle formulation (Figure 4). The spreadability may be related to the firmness and hardness parameters of the formulations. It is characterized by the work required to spread the formulation on a surface and is defined by the work of shear value. Lower work of shear values, as well as firmness and hardness, indicate that the formulation is more comfortable to spread. Srp formulation had a decrease of work of shear when compared with Sv formulation which indicates spreadability improvement with the addition of the active substance. However, lower adhesion values and higher elasticity values are desired, and this was observed for the Sv formulation. In the statistical analysis of the obtained data, the parameters evaluated in the texture and spreadability profile showed a significant statistical difference ($P<0.05$) between both formulations and a low coefficient of variation.

In texture profile analysis of the conditioner formulations, all formulation had close parameters. Nevertheless, the vehicle presented higher firmness, consistency, cohesiveness, and viscosity index values which corroborate with the higher viscosity and flow index values obtained in the rheological behavior of this formulation. Higher firmness, consistency, viscosity and

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**Table 1.** Apparent viscosity of conditioner without active substances (Cv), conditioner with a blend of vegetable polysaccharides (Cvp) and conditioner with biotechnological polysaccharide (Cbp) at times T0 (initial), T14 and T28 (final) and temperatures (25, 37, 45 and 4°C)

<table>
<thead>
<tr>
<th>Conditioner Formulations</th>
<th>Temperature (ºC )</th>
<th>Viscosity (cP)</th>
<th>Time ( Days )</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>25</td>
<td>37</td>
<td>45</td>
</tr>
<tr>
<td>Cv</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>T0</td>
<td>2307 ± 3.64</td>
<td>2307 ± 3.64</td>
<td>2307 ± 3.64</td>
</tr>
<tr>
<td>T14</td>
<td>2309 ± 3.18</td>
<td>2294 ± 6.28</td>
<td>2740 ± 4.31</td>
</tr>
<tr>
<td>T28</td>
<td>2332 ± 1.79</td>
<td>2009 ± 3.59</td>
<td>2418 ± 3.75</td>
</tr>
<tr>
<td>Cvp</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>T0</td>
<td>2135 ± 3.30</td>
<td>2135 ± 3.30</td>
<td>2135 ± 3.30</td>
</tr>
<tr>
<td>T14</td>
<td>2289 ± 2.51</td>
<td>2204 ± 1.44</td>
<td>2507 ± 2.61</td>
</tr>
<tr>
<td>T28</td>
<td>2094 ± 1.17</td>
<td>1955 ± 3.32</td>
<td>2567 ± 3.47</td>
</tr>
<tr>
<td>Cbp</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>T0</td>
<td>2206 ± 1.12</td>
<td>2206 ± 1.12</td>
<td>2206 ± 1.12</td>
</tr>
<tr>
<td>T14</td>
<td>2322 ± 5.76</td>
<td>2322 ± 2.62</td>
<td>2322 ± 2.62</td>
</tr>
<tr>
<td>T28</td>
<td>2262 ± 1.25</td>
<td>2123 ± 0.67</td>
<td>2123 ± 0.67</td>
</tr>
</tbody>
</table>

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**Figure 3.** Rheograms of conditioner without active substances (Cv) [a], conditioner with a blend of vegetable polysaccharides (Cvp) [b] and conditioner with biotechnological polysaccharide (Cbp) [c] at times T0 (initial), T14 and T28 (final) and temperatures (25, 37, 45 and 4°C).
flow rate values are related to the work required to spread the formulation. However, the work of shear had no statistically significant difference ($P < 0.05$) between the three formulations (Figure 5). According to the analyses of texture profile, spreadability and rheological behavior, it was observed that the addition of active substances to the vehicle made the formulation less thick and more fluid. Therefore, it has improved spreadability and contributed to better formulation performance.

Efficacy Tests

**Hair Combing Analysis**

Hair treatments with conditioner formulations increase the softness and smoothness of the hair. Also, it reduces the frizz and the force necessary to comb. Antistatic agents are adsorbed on the hair fiber, which reduces the hair friction. The obtained results in the combability test showed an increase in the required force to comb for the treated hair samples when compared to the control. Figure 6 shows that there was a significant difference in the force required to comb between C and the other treatments. Also, there was a considerable difference among H1 and others treatments, except for H6. It can be noticed that in the treatments with the addition of active substances, the combing force was higher than compared to the treatment with vehicles. This could have been avoided by...
adding a higher concentration of antistatic agents to the formulation because it decreases the combing force.\textsuperscript{9,15}

\textbf{Tensile Test}

The results showed that different treatments were able to increase the hair fiber resistance when compared to the control since the maximum force required to break the hair fiber was higher (Figure 7). Hair samples treated with shampoo added with hydrolyzed rice peptides, regardless of the conditioner used, had a significant difference ($P<0.05$) in the force required to break the hair fiber when compared to the control. This is interesting because peptides are added to hair formulations with the intention of increase hydration, brightness, smoothness and improve mechanical properties of the hair fiber, such as strength and resistance.\textsuperscript{19} However, with this result, it was possible to observe the action of this active substance on the internal structure of the capillary fiber (cortex).\textsuperscript{9}

\textbf{Hair Gloss Analysis}

Figure 8 showed the results of hair gloss analysis. There was a considerable increase in brightness in the H4. This brightness increase probably occurred due to the action of the hydrolyzed rice peptides. However, the coefficient of variation for hair samples was high, which caused no significant difference compared to the control. There was a reduction in brightness in the H2, H3, and H6 hair samples. The H5 and H1 hair samples were similar to the control. It would be expected that in the conditioner treatments added with the film-forming active substances would have an increase in brightness. The brightness increases it is due to the regular hair fiber surface and light reflectance.\textsuperscript{9}

\textbf{Hair Sensory Analysis}

The results of hair sensory analysis were demonstrated in Figure 9. H6 was the one with the highest scores (very good) in all evaluated parameters except combing, which corroborates the results obtained in the combing and brightness test. H5 and H3 also had a good evaluation of hair sensory analysis.

\textbf{Conclusion}

According to the obtained results, the ingredients obtained from natural sources influenced the stability, physical-mechanical, and rheological properties of hair care formulations. In addition, the ingredients obtained from

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\textbf{Figure 6.} Required force to comb the hair samples treated with shampoo and conditioner without active substances (H1), shampoo without active substances and conditioner with a blend of vegetable polysaccharides (H2), shampoo without active substances and conditioner with biotechnological polysaccharide (H3), shampoo with hydrolyzed rice peptides and conditioner without active substance (H4), shampoo with hydrolyzed rice peptides and conditioner with a blend of vegetable polysaccharides (H5), shampoo with hydrolyzed rice peptides and conditioner with biotechnological polysaccharide (H6) and without any treatment (C). The treated hair samples showed a significant difference between C ($P<0.0001$) and H1 ($P<0.006$).

\textbf{Figure 7.} Maximum required force to break the hair fiber treated with shampoo and conditioner without active substances (H1), shampoo without active substances and conditioner with a blend of vegetable polysaccharides (H2), shampoo without active substances and conditioner with biotechnological polysaccharide (H3), shampoo with hydrolyzed rice peptides and conditioner without active substance (H4), shampoo with hydrolyzed rice peptides and conditioner with a blend of vegetable polysaccharides (H5), shampoo with hydrolyzed rice peptides and conditioner with biotechnological polysaccharide (H6) and without any treatment (C). The results showed statistical differences between C and H4 ($P<0.008$), H5 ($P<0.0001$) and H6 ($P<0.0001$).

\textbf{Figure 8.} Hair gloss analysis of the hair samples treated with shampoo and conditioner without active substances (H1), shampoo without active substances and conditioner with a blend of vegetable polysaccharides (H2), shampoo without active substances and conditioner with biotechnological polysaccharide (H3), shampoo with hydrolyzed rice peptides and conditioner without active substance (H4), shampoo with hydrolyzed rice peptides and conditioner with a blend of vegetable polysaccharides (H5), shampoo with hydrolyzed rice peptides and conditioner with biotechnological polysaccharide (H6) and without any treatment (C). The results showed statistical differences between C and H4 ($P<0.009$), H5 ($P<0.02$) and H6 ($P<0.04$).
the natural sources were effective in the improvement of mechanical properties, texture and gloss of hair fiber evaluated by instrumental measurements and sensory analysis. Finally, this study has an important contribution since it showed that it is possible to develop stable and efficacy formulations with ingredients obtained from a natural source and the importance of the correlation between instrumental measurement data and sensory analysis.

Competing Interests
The authors declare that there is no conflict of interest.

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Figure 9. Hair sensory analysis of the hair samples treated with shampoo and conditioner without active substances (H1), shampoo without active substances and conditioner with a blend of vegetable polysaccharides (H2), shampoo without active substances and conditioner with biotechnological polysaccharide (H3), shampoo with hydrolyzed rice peptides and conditioner without active substance (H4), shampoo with hydrolyzed rice peptides and conditioner with a blend of vegetable polysaccharides (H5), shampoo with hydrolyzed rice peptides and conditioner with biotechnological polysaccharide (H6) and without any treatment (H7).


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