



# Development of shampoo formulations with guarana extract: Influence of thickening agents in the texture profile

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Received 5 Oct. 2019

Revised 19 Oct. 2019

Accepted 19 Nov. 2019

ePublished 15 Jan. 2020

## Abstract

**Background:** The texture analysis represents an excellent indicator for the physical stability evaluation of cosmetic formulations. This analysis can also be useful to evaluate the effects of the addition of ingredients that can modify the viscosity, such as botanical extracts, emollients and thickening ingredients. This way, it is possible to choose the formulation with greater acceptability, once the texture analysis can be correlated with the sensorial analysis. Thus, the aim of the present study was to develop a shampoo formulation added with guarana (*Paullinia cupana*) extract and to evaluate the texture profile, in order to choose the one with greater acceptability.

**Materials and Methods:** To this end, eight shampoo formulations were developed. In four formulations was used acrylates/C10-30 alkyl acrylate crosspolymer as thickening agent in the concentrations of 1% and 2%, and in the other four, salt (NaCl) was utilized in the same concentrations. Half of the formulations were stirred manually, and the other half was stirred during 30 minutes at 300 rpm using a mechanical stirrer (Fisatom 713D). The texture profile evaluation was performed using a TA.XT Plus Texture Analyzer (Stable Microsystems, Surrey UK). The formulations were evaluated in terms of hardness and spreadability and the parameters calculated were hardness, elasticity, adhesiveness, cohesiveness, compressibility, firmness and work of shear.

**Results:** In the obtained results, it was observed that the formulations with NaCl presented smaller values of hardness, firmness and work of shear, which are not desirable parameters in a shampoo formulation. The formulation with acrylates polymers at 2% and mechanical stirrer presented less hardness, adhesiveness, firmness and work of shear than the manual stirrer showing the influence of the process in the final result. Thus, the formulations with acrylates polymers at 2% concentration and mechanical stirrer showed the best texture profile for a shampoo formulation.

**Conclusions:** The developed formulations with guarana extract presented suitable texture profile for the aimed purposes and, the agitation process and thickening agents under study had influence in the texture profile. In addition, the evaluation of texture profile was important during the formulation development for the choice of a formulation with suitable parameters, which can optimize the research & development protocol and predict sensory properties such as formulation spreadability.

**Keywords:** Texture profile, Viscosity determination, Shampoo formulations, Guarana extract.



## Background

The use of hair care cosmetics and its influence in the well-being have been reported in previous studies of our research group, once alterations in the hair fiber and scalp can influence in the quality of life.<sup>1</sup> Previous studies showed that the hair care is essential to the women's quality of life and well-being, considering that the hair has a significant role in the self-image.<sup>1,2</sup>

Considering the rich composition of the plant extracts, they have great application in products for the skin and hair.<sup>3</sup> Among these extracts, we highlight the guarana plant extract which has bioactive compounds, such as caffeine and catechins<sup>4</sup> with potential for application in

cosmetic formulations for skin and hair care.

Guarana (*Paullinia cupana*) is a plant found in Amazonia discovered in the year 1895 and known for its stimulating activity. Its composition is rich in compounds such as caffeine, theophylline and theobromine, which confer the stimulant activity by which it is known. In addition, guarana is rich in compounds such as saponins and tannins, which confer detergent activity, astringent and is still rich in phenolic compounds such as catechins and epicatechins, which presents potent antioxidant activity.<sup>4-6</sup>

Due to its rich composition, guarana extract presents great potential to be applied in cosmetic products, specially shampoo formulations. Considering the presence of



saponins in its composition, the detergent activity can improve the cleaning properties of the shampoo.<sup>4</sup> Also, the tannins and phenolic compounds can be benefic to the hair fiber, once the antioxidant activity can protect the hair fiber from external aggressions.<sup>3</sup> This way, the application of guarana extract in hair care cosmetic products can result in innovative products for the treatment of this condition.

Although the guarana has great application in the food industry, it also presents a varied therapeutic use. According to Schimpl et al, guarana extract has several therapeutic applications, such as: antidepressant, anxiolytic, in improving cognitive functions and fat burning.<sup>7</sup> Due to its potent antioxidant activity, it is used as a protector of DNA damage, by reducing the production of reactive oxygen species (ROS) and consequently reducing the cancer-triggering factors. Among other pharmacological activities, due to the astringent and antioxidant activity of guarana,<sup>8,9</sup> it could act in the control of oiliness of the skin and hair. However, in the aforementioned review study, the use of guarana extract in topical formulations is not reported. In this context, the application of guarana in cosmetic products can result in innovative products.

The application of botanical extracts in cosmetic formulations it is very common in the Research and Development field.<sup>3</sup> At the same time, it is known that glycolic extracts can reduce the viscosity of the formulation, causing alterations in its stability and sensorial properties. In this context, the choice and application of the correct ingredients in the cosmetic formulations, such as thickening agents in shampoo formulations, it is very important for the final result.

In this context, the texture analysis represents an excellent indicator for the evaluation of the physical stability of cosmetic formulations. With the rapid and precise evaluation of the texture properties, it is possible to evaluate instability signs related to pH alteration, temperature variation, alteration of organoleptic characteristics and rheological characteristics.<sup>10</sup>

Such analysis may also be of great utility in assessing the effects of the addition of active principles on the stability of formulations, since actives such as plant extracts, salts and others can significantly alter the consistency of the product and consequently stability, texture and sensory of the same compromising the quality and reproducibility of the formulations.<sup>11,12</sup>

In addition, the sensorial suitable for the purposes is of fundamental importance for adherence to the use of cosmetic products and dermatological treatments.

There are studies that combine texture analysis with sensory perception, but most of them focus on the food industry.<sup>13-15</sup> A study by Savary et al, showed the application of texture analysis in the evaluation of the spreadability of different emollients in cosmetic formulations. The results showed significant correlations between in vivo sensory analysis and instrumental analysis of the evaluation of oils and emulsions.<sup>16</sup> These results are important to guide the

research and development of cosmetics, justify the choice of raw materials and improve the sensory of formulations.

Thus, the use of the texture profile evaluation of shampoo formulations can help the development of suitable formulations with the adequate sensorial and consequently attend the needs of the consumer.

### Objective

The aim of the present study was to develop a shampoo formulation added with guarana botanical extract and to evaluate the influence of the agitation process and the use of different thickening agents in the texture profile, in order to choose the one with greater acceptability.

### Materials and Methods

#### Development of the Formulations

To this end, eight shampoo formulations were developed. The formulations were developed based on TEA lauryl sulfate (40%), disodium laureth-sulfosuccinate (27%), cocamide DEA, cocamidopropyl betaine, glicerol, EDTA, water and 2% guarana extract. In order to evaluate the performance of the thickening agents, four formulations with acrylates/C10-30 alkyl acrylate crosspolymer in the concentrations of 1 and 2% were developed, and in the other four, salt (NaCl) was utilized in the same concentrations. Half of the formulations were stirred manually (F1, F2, F5, F6) and the other half was stirred during 30 minutes at 300 rpm using a mechanical stirrer, Fisatom 713D, (F2, F4, F7, F8). The ingredients and the concentration in the developed formulations are represented in Table 1.

#### Texture Profile Evaluation

The texture profile evaluation was performed using a TA.XT Plus Texture Analyzer (Stable Microsystems, Surrey UK). The formulations were evaluated in terms of hardness and spreadability and the parameters calculated were hardness, elasticity, adhesiveness, cohesiveness, compressibility, firmness and work of shear. The accessory for the hardness analysis was a cone shaped acrylic probe with a 45° angle and for the spreadability test the TTC Spreadability rig HDP/SR was used. The measures were made five times and in triplicate respectively and were performed at room temperature (24°C).

#### Viscosity Determination

The viscosity of the shampoo formulation was determined using a Ford cup size 6. The Ford cup is a viscosimeter easy to handle, in which the viscosity is related to the emptying time of the cup with a known volume, which presents a calibrated opening in the base. Openings with the numbers 2, 3 and 4 are commonly used for lower viscosity liquids and openings equal or higher than 5 are used to measure superior viscosity liquids (310cst). The analysis was done in triplicate and the viscosity calculus is made using the curve viscosity versus time of emptiness.<sup>17</sup>

**Table 1.** Shampoo formulations developed in the study

Ingredients (INCI)	F1 (% p/p)	F2 (% p/p)	F3 (% p/p)	F4 (% p/p)	F5 (% p/p)	F6 (% p/p)	F7 (% p/p)	F8 (% p/p)
TEA lauryl sulfate (40%)	18	18	18	18	18	18	18	18
Disodium laureth-sulfosuccinate (27%)	10	10	10	10	10	10	10	10
Cocamide DEA	3	3	3	3	3	3	3	3
Cocamidopropyl betaine	4	4	4	4	4	4	4	4
Acrylates/C10-30 Alkyl Acrylate Crosspolymer	1	2	1	2	-	-	-	-
NaCl	-	-	-	-	1	2	1	2
Glycerin	5	5	5	5	5	5	5	5
Panthenol	1	1	1	1	1	1	1	1
Guarana ( <i>Paullinia cupana</i> ) extract	3	3	3	3	3	3	3	3
NaOH qsp pH 6.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
EDTA	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Aqua qsp 100	54.5	53.5	54.5	53.5	54.5	53.5	54.5	53.5

\* INCI, International Nomenclature of Cosmetic Ingredients.

### Statistical Analysis

The experimental data obtained in the texture profile evaluation were submitted to a statistical analysis using the software GraphPad Prism 5. The data obtained presented a normal distribution, thus test used was the analysis of variance (ANOVA) with multiple comparison.

## Results

### Development of the Formulations

All developed formulations did not present instability signs thus, allowing us to proceed to the texture and visual analysis of the thickening agents.

It was possible to observe that the agitation process influenced the visual aspect of the formulations, and the formulations prepared with the addition of NaCl using the mechanical stirring process were more consistent than the formulations prepared by manual stirring.

At the same time, formulations containing acrylate polymer were less consistent when made by mechanical

agitation, showing the influence of the preparation process on the texture profile of the formulations under study.

### Texture Profile Evaluation

The results of the parameters obtained in the evaluation of the texture profile – hardness test – are presented in Table 2.

The results of the calculated parameters in the evaluation of the texture profile in the spreadability test are shown in Table 3.

The statistical analysis was performed using ANOVA with multiple comparison test; it was possible to observe statistical difference among almost all formulations. The formulations with NaCl at 1% and 2% developed using manual stirring (F5 and F6) didn't present statistical difference in the hardness parameter (Figure 1). This was also observed in the Firmness and Work of Shear results (Figure 2 and Figure 3).

**Table 2.** Results of the parameters evaluated in the Hardness test

Ingredients (INCI)	F1 (% p/p)	F2 (% p/p)	F3 (% p/p)	F4 (% p/p)	F5 (% p/p)	F6 (% p/p)	F7 (% p/p)	F8 (% p/p)
TEA Lauryl Sulfate (40%)	18	18	18	18	18	18	18	18
Disodium Laureth-Sulfosuccinate (27%)	10	10	10	10	10	10	10	10
Cocamide DEA	3	3	3	3	3	3	3	3
Cocamidopropyl Betaine	4	4	4	4	4	4	4	4
Acrylates/C10-30 Alkyl Acrylate Crosspol	1	2	1	2	-	-	-	-
NaCl	-	-	-	-	1	2	1	2
Glycerin	5	5	5	5	5	5	5	5
Panthenol	1	1	1	1	1	1	1	1
Guarana ( <i>Paullinia cupana</i> ) Extract	3	3	3	3	3	3	3	3
NaOH qsp pH 6.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
EDTA	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Aqua qsp 100	54.5	53.5	54.5	53.5	54.5	53.5	54.5	53.5

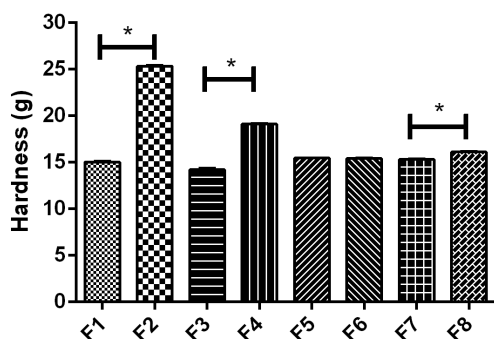
**Table 3.** Calculated parameters in the Spreadability test

	F1	F2	F3	F4	F5	F6	F7	F8
Hardness	15.04	25.4	14.22	19.12	15.46	15.42	15.3	16.12
Adhesiveness	473.9020	265.9686	310.3464	313.2940	551.6010	557.3122	640.9650	319.6686
Cohesiveness	-0.0333	-0.3707	-0.0602	-0.4217	-0.0264	-0.0204	-0.0147	-0.1039
Elasticity	-5.4427	-1.8596	-3.3421	-5.9040	-5.5149	-5.3304	-5.9439	-41.5258
Gumminess	-0.5021	-9.4181	-0.8572	-8.0629	-0.4092	-0.3148	-0.2172	-1.5903
Compressibility	458.5894	194.0254	292.7008	220.3654	537.3772	546.1618	631.6674	289.5698

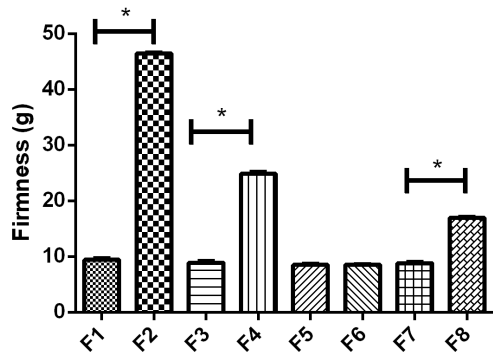
In the obtained results, it was observed that the formulations with NaCl presented smaller values of hardness, firmness and work of shear, which are not desirable parameters. The formulation with acrylates polymers at 2% and mechanical stirrer presented less hardness, adhesiveness, firmness and work of shear than the manual stirrer.

### Viscosity Determination

The result obtained for the formulation with NaCl at 2% with the addition of guarana extract was 1100 cP, while the formulation with acrylate polymer obtained a viscosity of 1900cP. It was also made the comparison with the vehicle formulations and it was observed a decrease of 300cP in both formulations with the addition of the botanical extract in the formulation.



**Figure 1.** Hardness values of the shampoo formulations developed (\* means statistical difference  $P < 0.05$ ).



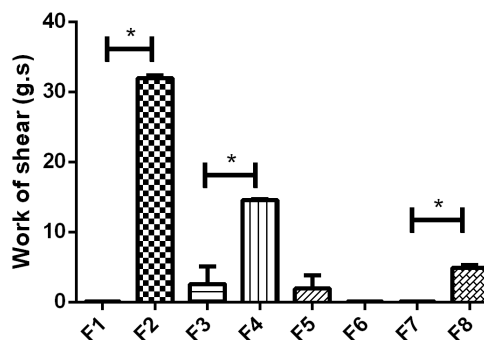
**Figure 2.** Firmness values of the shampoo formulations developed (\* means statistical difference  $P < 0.05$ ).

### Discussion

Though the standardization is something that is always a concern when developing an emulsion, in the development of shampoo formulations, the standardization of the process is very important as well. The application of salt as a thickening agent in shampoo formulations has been well established in the research and development field. It is also well known that NaCl promote the increase viscosity when added in shampoo formulations.

In this context Abu-Jdayil et al evaluated the influence of salt ingredients in different concentrations, temperatures and the influence of the time of agitation process in the rheological properties of shampoo formulations.<sup>18</sup> It this study was observed that there is an optimum concentration of salt that was 1.5% and, after 2% concentration the salt promotes the reverse effect, promoting a reduction in the viscosity. It was also observed an optimum time of mixing which was 30 minutes.

In our study 30 minutes of mixing was standardized in both mechanical and manual stirring and only concentrations equal or under 2% were evaluated. It was observed that the process presents a great influence in the viscosity, being this difference visually observed as well as observed in the texture analysis. The formulation with NaCl at 2% presented higher hardness and firmness after a mechanical stirring when compared to the formulation with manual stirring. In the statistical analysis was observed that without the mechanical stirring, there was no difference detected in the equipment among 1% and 2% of NaCl in the formulation. This shows the influence



**Figure 3.** Work of shear values of the shampoo formulations developed (\* means statistical difference  $P < 0.05$ ).

of the process in the dispersion of the thickening agent in the formulation.

It is known that surfactants present a micellar critical concentration, in which above presents the formation of micelles and, with the increase of surfactant concentration presents the formation of a network structure. The salt addition promotes an interaction with this network and consequently a viscosity increase.<sup>19</sup> Thus, this shows that the mechanical stirring promoted a better dispersibility of the salt in the formulation. At the same time, for the acrylate polymer the inverse behavior was observed, being the formulation with manual stirring more viscous than the formulation with mechanical stirring. This was also a sign of better dispersibility of the polymer in the formulation, which promoted a better interaction with the network structure.

According to Cornwell, the shampoo formulation needs to present good cleansing and foaming properties, as well as needs to present good rheological properties to present a good acceptability in the cosmetic market. The shampoo needs to present the right appearance and consistence in the hands and, need to spread easily in the hair.<sup>20</sup>

In the viscosity determination was observed that the addition of the glycolic guarana extract promoted a reduction of 300cP in both formulations, with salt and acrylate polymer. In this context, a better viscosity donor must be chosen in order to control this viscosity reduction, in order to achieve the "ideal" parameters.<sup>20</sup>

In our study, we observed that the formulations with NaCl as 1% and 2% concentration and manual stirring presented low values of hardness and firmness, meaning that they presented low viscosity, being considered formulations with undesirable parameters. This is also due to the addition of the glycolic extract that reduces the viscosity of the formulation. Analyzing the formulations with acrylate polymer, the manual stirring presented higher values of firmness and hardness when compared to the formulation with mechanical stirring.

Though it seems that higher the hardness and firmness of the formulation, better the acceptability, studies showed that, the higher the hardness, smaller is the spreadability of the formulation and this parameter is inversible to the elasticity.<sup>21-23</sup> This means that the formulation does not present a soft profile. Also, smaller adhesiveness is a desirable parameter in cosmetic formulations. Thus, the formulation with acrylate polymer at 2% concentration and mechanical stirrer showed the best texture profile.

Comparing the NaCl with the acrylate polymer in the concentration of 2% and mechanical stirring, the Acrylate presented higher values of work of shear, which is the work needed to spread the formulation. At the same time, in the viscosity determination, the formulation with acrylate polymer presented viscosity of 1900cP after the addition of the extract while the formulation with NaCl presented only 1100cP.

Considering that the viscosity is the measure of the resistance of a liquid to flow, the higher the viscosity, higher the resistance, which is also consistent to the results obtained in the texture profile evaluation. At the same time, low viscosity liquids overflow, which is not desirable for a shampoo formulation, hence it can flow through the hands before the application in the hair.<sup>20</sup> Thus, the formulation with 2% acrylate and mechanical presented better results among all the formulations developed and the ones with greater acceptability.

The texture profile evaluation has been widely explored in the food research and development field and recently has been explored by the cosmetic industry, but mostly focused in emulsions and formulations for skin care.<sup>13-16</sup> In our study we observed that the data obtained in the texture profile evaluation were consistent to the obtained in the viscosity determination. In this context, we can highlight that this test was of great importance during the development of the shampoo formulation and can be used during the research and development of hair care formulations.

It has also been reported that the texture profile can be correlated to the sensorial evaluation,<sup>16</sup> in this context, the texture profile evaluation can help the development of a shampoo with great acceptability.

The formulation with guarana extract and acrylate polymer at 2% concentration prepared with mechanical stirring presented the suitable parameters, as described by Cornwell, with a good appearance and consistence in the hands and, easy to spread in the hair.<sup>20</sup> This was observed with the previously described values of hardness, adhesiveness and work of shear.

In conclusion the developed formulations with guarana extract presented suitable texture profile for the aimed purposes and the agitation process and thickening agents under study had influence in the texture profile. In addition, the evaluation of texture profile was important during the formulation development for the choice of a formulation with suitable parameters, which can optimize the research & development protocol and predict sensory properties such as formulation spreadability.

Finally, the findings of this research showed the importance of the standardization of the process during the prepare of hair care formulations. Also, we evaluated the benefits of using the texture analysis for the choice of a formulation with desirable parameters, once it optimizes the research and development protocol before the sensorial analysis.

#### Acknowledgements

We would like to thank the São Paulo Research Foundation – FAPESP (grant numbers: 2017/04149-0 and 2017/19272-0) and Coordination of Superior Level Staff Improvement - CAPES (grant number 001) for financial support of our research.

## Competing Interests

None

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