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Review Article

Application of design of experiments in the development of cosmetic formulation based on natural ingredients

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Abstract

Development of cosmetic formulations based on natural ingredients presents a challenge in order to instability and sensory limitations of raw ingredients. Organogel which is a cold emulsifier and offers refined sensory properties, physical-chemical characteristics and stability could be solved this limitation. The aim of this study is to develop cosmetic formulations by cold process containing natural ingredients and assess through design of experiment (DOE) the influencing factors on the physical-mechanical and sensory properties of these formulations. Physico-mechanical and sensory properties of cosmetic formulations containing natural ingredients obtained by cold process were evaluated. The DOE was performed for the texture analyses. In addition, the influence of natural actives substances in the developed formulations was assessed. The different concentrations of polymers, organogel and active substances influenced the texture profile of the formulations, being the concentration of organogel the most influential factor. The formulation containing sunflower oil, a polymer from a natural source and the highest concentration of organogel influenced the firmness, consistency, cohesiveness and viscosity. The work of shear decreased in the formulation added of the polymer and increased when the formulation was supplemented with active substances. The sensory analysis showed that the formulation based on 5% of organogel, sunflower oil and polymer it was preference for the selected formulation. In conclusion, the application of DOE for the rational development of cosmetic formulations based on natural ingredients led to formulations with physico-mechanical and sensory properties suitable for the application on the skin.

Keywords: DOE, Organogel, Texture Profile, Sensory Properties, Cosmetics

Introduction

Products for Personal Hygiene, Perfumery and Cosmetics containing natural ingredients are expected to lead global movements for the next few years.[1] By the year 2025 the global market for natural products are estimated to reach approximately 25.11 billion dollars.[2] In addition, according to Mintel's report, 41% of Brazilians are interested in cosmetic products with natural ingredients.[1]

Another global movement is sustainability, which aims to reduce impacts to the environment.[3] One of the strategies for sustainability used in the cosmetic industry is cold emulsification, which eliminates the phase of heating and cooling the emulsions, making the process faster and more productive with reduced energy consumption and cost. However, it is challenging to find natural raw materials to make this process possible.[4]

The cold emulsification process is one of the advantages of using organogel, which consists of a semi-solid system, in which the oil phase is immobilized by a three-dimensional network of the structuring agent. The oil phase may consist of vegetable or mineral oils, or organic polar solvents. The structuring agent may consist of waxes, monoglycerides, polymers or polyol ethers. The organogel acts as an emulsifier and offers refined sensory properties, physical-chemical characteristics and stability.[5]

In this context, according to global market trends, the development of cosmetic formulations based on natural ingredients presents a challenge because the raw materials can often present instability and sensory limitations.[4]

The development of cosmetic formulations can be optimized using design of experiment (DOE), which is one type of experimental design. DOE is a statistical tool widely used to identify which factors influence the studied parameter, leading to the desired result. Therefore, the costs and the number of experiments are reduced during the development phase of the product.[6,7]

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The texture of emulsions during application is known to be of great importance regarding the consumers' preference. The texture of a product is a complex and multidimensional notion that has therefore to be carefully considered.[8]

It is important to employ methods that evaluate the physico-mechanical properties of the formulation during the R&D stage to define the formulation and achieve a stable product with sensory characteristics in accordance with the formulation purpose.

The TA texturometer XT/Plus® 50 (Extralab) determines the texture profile of formulations and predicts the sensory properties through the shear work data, which results in a more practical and integrative development of a cosmetic formulation.[9-11]

In summary, the application DOE contributes to optimize the development of natural cosmetics through statistical analysis of the physical-mechanical evaluation, as well as the sensorial evaluation by the final consumer.

In this context, the aim of this study was to develop cosmetic formulations by cold process containing natural ingredients and assess through DOE the influencing factors on the physical-mechanical and sensory properties.

Materials and Methods

Formulations Development

The developed formulations were based on Glycerin; Helianthus Annuus (Sunflower) Seed Oil (and) Polyacrylic Acid (and) Xylityl Sesquicaprylate (and) Glyceryl Stearate (and) Euphorbia Cerifera (Candelilla) Wax (and) Sodium Hydroxide; Propylene Glycol; Hydroxypropyl Starch Phosphate; Aqua; Disodium EDTA; Helianthus Annuus (Sunflower) Seed Oil; BHT; C13-15 Alkane; 2-Phenoxyethanol (And) Methylisothiazolinone (And) Aqua/Water. In addition, the formulations were supplemented or not with active substances Olive fruit extract and *Spirulina* sp.

Design of Experiment

The DOE was performed considering as variation factors the type of emollient, presence and absence of polymer and concentration of organogel with two levels each (2^3), (Table 1). The response was evaluated according to the texture profile of the formulations.[6,7]

Physico-mechanical Properties

The TA.XT/Plus® 50 (Extralab) Texture Analyzer (Stable Microsystems, United Kingdom) evaluates the physico-mechanical properties of cosmetic formulations. The parameters assessed are related to the texture profile and spreadability.[9-11] The texture profile was evaluated to determine the influence of the DOE factors. The texture parameters evaluated were: firmness, cohesiveness, consistency and viscosity index. The Back Extrusion rig A/BE 35 mm was used at room temperature. The return distance used was 100 mm, the return speed was 20 mm s^{-1} and the contact force was 30 g. The pre-test speed was 2 m/s, the test speed was 2 mm/s, the post-test speed was 1 mm/s, the distance was 10 mm and the force of recognition was 15 g.

Table 1. Factors Studied in Factorial Experimental Planning and Their Levels

Factors	Level	
Emollient	**Sunflower Oil**	**Hemisqualane**
Polymer	0%	2%
Organogel	3%	5%

The spreadability test assessed the work of shear of the formulations. The probe TTC Spreadability rig HDP/SR used. And the program was: return distance of 25 mm, return speed of 20 mm/s, contact force of 30 g, pre-test speed of 2 mm/s, test speed of 2 mm/s, post-test speed of 1 mm/s, distance of 10 mm and force of recognition of 15g.

Sensory Analysis

Sensory analysis was performed on 10-trained subjects after written consent. The opinions regarding the formulations selected by the DOE were evaluated. A standard amount of formulation was applied on the anterior region of the forearm. The assessed parameters were: touch sensation, spreadability, cohesiveness, consistency and firmness/viscosity. The scale was: 1-bad, 2-regular, 3-good, 4-very good and 5-excellent. The preference towards the formulations was also evaluated.

Statistical Analysis

The DOE analysis was carried out in the Minitab® 18 software. The independence, homocesticity and normality of the data were analyzed. Box-cox transformation was performed for non-normal results following the analysis of variance (ANOVA). The results were presented as Pareto and main effects graphs. The work of shear was evaluated in the GraphPad® Prism software. ANOVA followed by the Tukey test was performed for the data with normal distribution and Kruskal-Wallis test, followed by the Dunns test was performed for non-normal distributions. The sensory analysis was expressed as relative frequency of the parameters. All analyzes were performed in triplicate and with 95% confidence level.

Results

Design of Experiment

The firmness, consistency, cohesiveness and viscosity index followed a non-normal distribution, being necessary to carry out the Box-cox analysis to perform the analysis of variance. According to the results of the analysis of variance, it was possible to observe that the variation factors had a positive influence on the parameters evaluated. Figure 1 shows the impact of the variation factors on the tested parameters obtained by DOE. Among the variation factors, the variation in the organogel concentration was

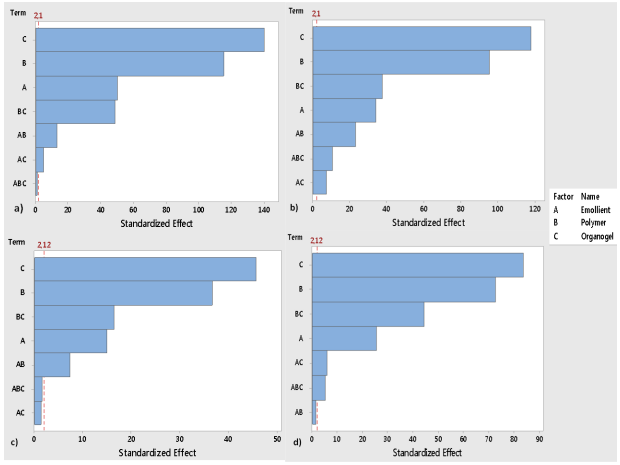

Figure 1. Pareto chart to evaluate the influence of firmness (a), consistency (b), cohesiveness (c) and viscosity index (d) parameters in relation to organogel, polymer and emollient factors obtained through design of experimental.

the one with the greatest influence on the texture profile, followed by the polymer presence and emollient type. The main effects of variation factors on the texture profile were evaluated and the highest effect was observed for 5% of organogel, followed by the presence of polymer and use of sunflower oil as emollient (Figure 2).

Physico-mechanical Properties

The formulation containing 5% of organogel presented the higher firmness, consistency, cohesiveness and viscosity index. Thus, new formulations were developed containing 5% of organogel and the variation factors were the presence or absence of the emollients and polymer. The evaluated formulations were: FO: formulation containing sunflower oil, FOP: formulation containing sunflower oil and polymer, FH: formulation containing hemisqualane, and FHP: formulation containing hemisqualane and polymer. The formulations containing the polymer had a work of shear lower than the other formulations (Figure 3). However, the addition of the active substances (hydroxytyrosol and *Spirulina* sp.) in FOP resulted in a significant increase in the work of shear (Figure 4).

Sensorial Analysis

The results from the sensorial analysis indicated that all the formulations, except the formulation with hemisqualane (FH), were considered very good. The formulation with sunflower oil and polymer (FOP) was the favourite of most of the participants and it presented a better sensation to the touch with 80% of the answers being very good or excellent (Figure 5). The low work of shear of this formulation can be correlated with its better sensory characteristics.

Discussion

The texture profile may be influenced by different components of a cosmetic formulation, such as the presence and concentration of waxes, polymers, surfactants, etc.[9] Organogel was used as a cold emulsifying agent in the formulation developed in this study. The emulsifying agents may directly influence the texture profile of the formulations, as was corroborated by the DOE. In addition, the polymer used also presented features to increase the texture profile of the formulation, working as a rheology modifier.

It has been suggested that mostly fatty alcohol-based emollients are able to improve the texture profile of the formulation.[12] However, in the present study, it was

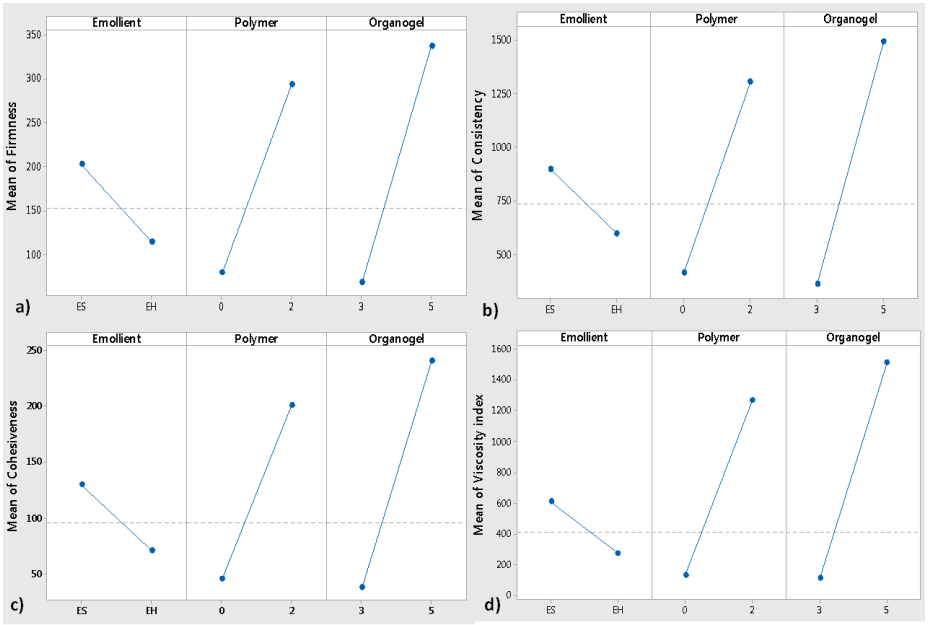

Figure 2. Main effects plots to evaluate the influence of the parameters of firmness (a), consistency (b), cohesiveness (c) and viscosity index (d) in relation to organogel, polymer and emollient levels obtained through design of experimental.

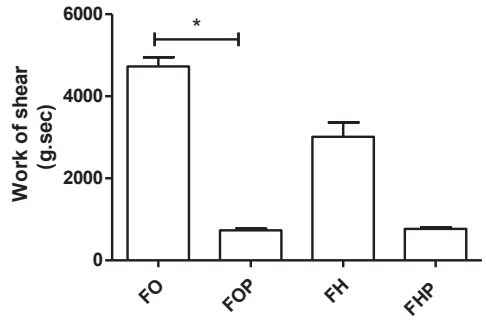

Figure 3. Work of shear of formulations with sunflower oil (FO), sunflower oil and polymer (FOP), hemiesqualane (FH) and hemiesqualane and polymer (FHP). * *P* value <0.05.

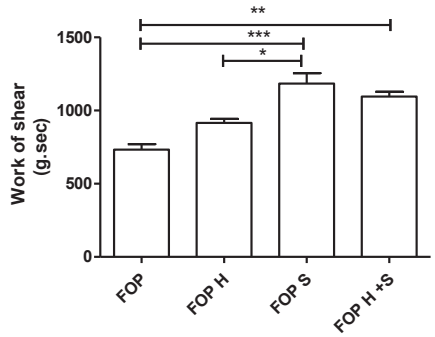

Figure 4. Work of shear of formulations with sunflower oil and polymer (FOP) with the active substances of olive extract standardized in 20% of hydroxytyrosol (FOP H), *Spirulina* sp. (FOP S) and olive extract standardized in 20% of hydroxytyrosol and *Spirulina* sp. combined (FOP H + S). *) *P* value <0.05, **0.01<*P*<0.05 ***$P<0.001$.

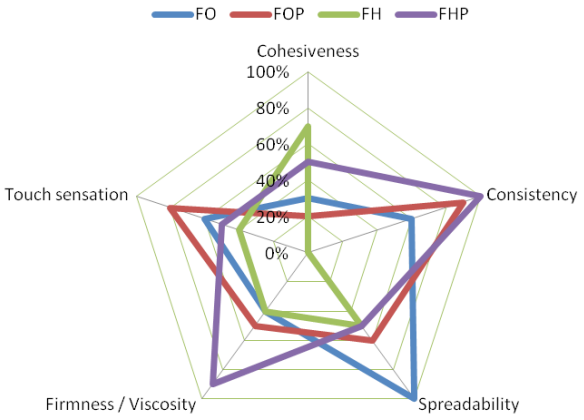

Figure 5. Sensory analysis of formulations with sunflower oil (FO), sunflower oil and polymer (FOP), hemiesqualane (FH) and hemiesqualane and polymer (FHP) against the best evaluations regarding touch sensation, spreadability, cohesiveness, consistency and firmness/viscosity.

observed that the sunflower oil improved the texture profile of the formulations, with the advantage of also improving the sensory characteristics. The organogel has sunflower oil in its composition; therefore, the improvement in the texture profile related to the increase in the concentration of organogel may be related to the indirect increase in the sunflower oil concentration.

The increased work of shear for the formulations in the presence of the polymer is corroborated by the literature, since generally polymers improve the spreadability of cosmetic formulations.[11] The addition of *Spirulina* sp. to FOP significantly increased the work of shear, which may be related to the rich composition of proteins and polysaccharides of *Spirulina* sp.[13] In addition, it can be used as a texture agent aiming to decrease the concentration of other ingredients in the formulation, such as rheology modifiers.[10]

Considering that, Spirulina sp., The microalgae - Blue Green Algae, according to its composition provides multiple benefits to the skin when added to cosmetics,[13-15] an excellent ingredient to obtain multifunctional cosmetic formulations but can also improve the texture profile of the formulations.

The formulation containing sunflower oil and polymer presented low work of shear, which can be related to the better sensory characteristic of the formulation.

The work of shear is related to the spreadability of the formulation. Therefore, through the work of shear it is possible to correlate the texture profile with the sensory properties, which are usually inversely proportional. For example, a formulation with a low work of shear can lead to better sensory characteristics.[10,11,16] In this study, although the FOP formulation presented lower work of shear compared with the other formulations, it did not present the best spreadability in the sensorial analysis. However, this formulation was rated the as the best in other sensory parameters, such as touch sensation.

Conclusion

The design of experiment optimized the development of cosmetic formulations containing natural ingredients by assessing the influence of different parameters related to the texture profile, which resulted in a rational development of the cosmetic formulations. Finally, a cosmetic formulations based on natural ingredients with sensory and physico-mechanical properties suitable for topical application was obtained.

Competing Interests

None.

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