Designing sesame oil emulsions for sunscreen actives

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INTRODUCTION

The term sunscreen is widely used to describe a cosmetic consumer product that bears a clear message of "protection against solar radiation." The chemicals contained in these sunscreens are called UV filters or UV reflectors. Today's sun products contain additional specific skin or body care active ingredients accompanied by a corresponding claim. These products have primary skin care claims (such as moisturizing, wrinkle diminishing, and firming) and offer the sun protection as an additional benefit [1, 7, 8].

Emulsions are a technologically simple, yet elegant, skin delivery system. Over the years, formulating chemists have used different types of ingredients to design different types of emulsions. In most cases, the characteristics of emulsions depend on how the ingredients interact and how the emulsions are formulated and manufactured. The texture, stability, and delivery of an emulsion are determined by the combination of emollients and emulsifiers. Understanding the mechanisms of emulsion formation is of great importance in the development of sunscreen products. The difficulties vary depending on emulsion types. Such difficulties can be managed if all of the necessary steps of ingredient selection and investigation are carefully done before drawing up a final formula [2-4].

Sesame (*Sesamum indicum* L., Pedaliaceae) is a very old crop first cultivated in Africa. The oil extracted from this plant consists of glycerides with about 43% of oleic and linoleic fatty acids, and 9% of palmitic and 4% of stearic fatty acids. Refined sesame oil is a special oil because of its antioxidant properties allowing for greater shelf life. Also such property improves its flavor and taste, which allows its use in the food industry. Since ancient times, people have recognized the sesame seed property to prolong youth and beauty. In present days, sesame oil is used in the production of a wide variety of cosmetic products for this outstanding same reason. Sesame oil is rich in antioxidants, which are molecules that prevent the oxidation of other molecules. By slowing down the oxidation in skin cells, clinical studies have shown that sesame oil's antioxidants can slow down the aging process of the skin[5,6].

The aim of this work was to formulate sunscreen w/o emulsions using sesame oil and different types of surfactant systems and assess the effect of surfactant systems on the formulation stability.

MATERIALS AND METHODS

Four emulsions were analyzed. They differed by the concentration of surfactants (Table 1). All the emulsions were made of 60% (w/w) of sesame oil (VitalAtman - Brasil) as the oily phase, 10% of surfactants, and 40% (w/w) of purified water. Emulsions were prepared at 65 ◦C: Hot water was slowly added to the heated oily phase containing the melted surfactants. This technique, called the phase inversion method, is widely used in batch processing since it has been found that it can yield

small droplet sizes. The emulsions were stirred by Ultra-Turrax for 10 minutes. Two sunscreen emulsions groups were prepared: Group A with 2% of Avobenzone and 2,5% of Octildimetil PABA, and group B with 2% of Avobenzone and 4% of octyl methoxycinnamate, (w/w). All the emulsions were prepared in triplicate and analyzed immediately after their production.

<u>1 - Intrinsic stability:</u> For each emulsion, sample vials were filled with 15ml of the emulsion and hermetically closed. They were stored vertically at room temperature (25 ˚C) and were evaluated for four months. Changes such as phase separation or creaming rate were recorded.

<u>2 - Stability under storage:</u> Other sample vials were filled as described in Section 1. Twelve vials containing samples were used. Three were stored vertically at room temperature (25 ˚C), three in a hot air oven at 45 ˚C ±2, and three at 4 ˚C ± 2. Observations were made each week for four months.

<u>3 - Stability under centrifugation:</u> Four centrifugation vials, filled with 15ml of emulsion, underwent a centrifugal acceleration of 3,500 rpm for 30min (BioEng, Model BE 5100). To avoid modifications induced by possible heating, the temperature was measured in one tube at the end of the experiment. It should not exceed 30 ˚C.

<u>4 - pH:</u> The P 2000 Gehaka pHmeter was used for the determination of the pH value of the emulsions at room temperature (25 ˚C).

<u>5 - Freeze/thaw cycles</u>: Three sample vials filled with the emulsion and hermetically closed were vertically stored for 24 h in a freezer at −5 ˚C and then for 24 h at room temperature (25 ˚C). The emulsion was observed and any change was recorded. This cycle was repeated six times.

<u>6 – SPF (Sun Protection Factor) Evaluation by the Mansur technique:</u> Mansur *et al.* (1986) developed a very simple mathematical theorem (Equation 1) which replaces the in vitro method, using the UV spectrophotometry:

$$SPF_{spectrophotometer} = CC \times \Sigma\, EE(\lambda) \times I(\lambda) \times Abs(\lambda) \qquad \text{(Eq. 1)}$$

Where;
CC= Correction constant (equal to 10)
EE (λ) = Erithematogênic Effect of the radiation at a wavelength
I (λ) = Intensity of sunlight at a wavelength
Abs (λ) = Absorption value of the solution at a wavelength

RESULTS AND DISCUSSION

Stability is reflected by the value of the creaming percentage previously used to evaluate emulsion formulations. No change could be macroscopically observed in emulsions over the 24 h. All the systems showed a light tendency to creaming, except Formulation 3 (3A and 3B). No excessive heating was observed after centrifugation.

All the emulsions that underwent freeze/thaw cycles presented damage, which was dependent on the formulation characteristics. However, not a single one could stand five stable cycles. On formulations 1, 1A, and 1B, rupture occurred after one cycle; formulations 2, 2A, and 2B creamed after one cycle and rupture occurred after two cycles. A tendency to rupture (visible droplets of oil dispersed in the emulsion) was observed in formulations 3 and 4 after four cycles.

Measurements of pH are useful data especially in quality assurance and skin compatibility. Their variation may explain some unexpected differences observed between emulsions. The pH values of the formulations remained in the range of 5 and 6, which is quite compatible to the skin. The value of SPF was around 8, confirming the incorporation of UV filters in the formulation and adequacy of the method of analysis.

Emulsion formation is influenced by secondary emulsifiers such as the ethoxylates and propoxylates. The degree of ethoxylation or propoxylation will help to shift the hydrophilic and lipophilic balance of the entire emulsion. Surfactants with a high degree of ethoxylation with high-HLB value will favor the formation of an o/w emulsion. On the other hand, compounds with a high degree of propoxylation with low HLB values will favor the formation of a w/o emulsion. There are situations where a combination of ethoxylated and propoxylated surfactants will provide unique characteristics of emulsion behavior, especially with active ingredients.

The disadvantage of monitoring the stability under storage is the lengthy period required before any distinction can be made in the case of emulsions with small stability differences, such as emulsions 3A and 3B. However, if the developed formulation is intended to be stored or exposed at high temperatures (storage in tropical countries, transport, etc.), this test is very critical. The whole analysis should be carried out again after storage of the emulsion in the representative conditions (temperature, length of time). Storage at 4 $^{\circ}$C is interesting in the case of formulations containing active substances likely to crystallize, such as sunscreen filters. The stability study under centrifugation not only reveals that formulation 3 was the most stable one, but also gives excellent information about the stability of the system compared with the creaming volume percentages.

CONCLUSION

Emulsions will continue to grow in importance in the personal care industry. As consumers become more and more sophisticated in their demands, the formulations will become ever more complex. A number of tools and rules can help to make the selection of ingredients for the preparation of emulsions. However, the preparation of cosmetically elegant emulsions will always require an element of art. Consequently, it is the consumer, not the computer, who will ultimately determine what is acceptable. From these results we can conclude that the sesame oil emulsions are suitable for use as a vehicle for sunscreen products since they will have stability and compatibility with the skin. However, among the formulations, only type 3 showed better stability properties. Finally, a good understanding of the lipophilic and the hydrophilic portion of surfactants will allow researchers to design emulsions that will improve the base for sunscreen actives.

FIGURES AND TABLES

Table 1. Surfactant systems of emulsion formulations

Formulation	HLB	Surfactant System (%)		
		Tween 80	Span 80	Tween 20
1	8	35	65	-
2	11,3	65	35	-
3	15	-	13	87
4	15	100	-	-

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