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Full Length Research Paper

Formulation and characterization of a green tea extract containing emulsion prepared with Abil®EM90 as a lipophilic surfactant

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The purpose of this study was to formulate and develop a stable water-in-oil (W/O) emulsions containing the extract of the green tea leaves. Abil®EM90 was used as an emulsifier and effects of emulsifier ratios were optimized to obtain a stable emulsion without adding any preservative. For this purpose, 25 formulations were formulated initially and were kept at 25℃ to choose the desired formulations for further stability study. 4 formulations were selected to keep them at various storage conditions for 21 days. Finally, a formulation containing 5% Abil®EM90, 3% green tea extract and 16% liquid paraffin oil was selected as final formulation to be subjected to the stability studies of 28 days. Formulation was observed for any type of phase separation, color change and liquefaction at defined intervals. It was found that a formulation containing 5% Abil®EM90, 3% green tea extract and 16% liquid paraffin oil was the most desirable, stable formulation regarding its physical characterization, including viscosity of the emulsion. Hence, it could be imperative choice as skin product in the form of stable topical emulsion.

Key words: Green tea, Abil®EM90, formulation, emulsion, stability.

INTRODUCTION

Botanical extracts are playing an increasingly important role in cosmetics because botanical secrets have been passed down through generations of herbal folklore and nowadays. For cosmetic industry, isolation and purification of the active ingredient within the crude extract are sometimes not needed because such isolation and purification may lead to a loss in the biological activity. An incorporation of the extract into the cosmetic formula, however, may cause several problems, since the botanical extracts usually contain hundreds of chemical structures. An incompatibility between ingredient used in the formula for examples and component of the extract lead to decrease in product stability and safety. Therefore, it is the duty of formulator to solve these problems and to produce the products related to the consumer needs by using various formulation techniques and knowledge. Several cosmetic formulas are available in market such as emulsions, suspensions and gels. Emulsion system including lotions and creams is an area of potential for cosmetic products and the various techniques of emulsion preparation have been widely studied. The emulsion is versatile in application, depending on the bioactive incorporated. Typically, emulsion system consists of an immiscible liquid (internal) phase finely dispersed in another liquid (external) phase that is, oil dispersed in water (o/w emulsion) or water dispersed in oil (w/o emulsion) type (Viyoch et al., 2003).

Plant derived polyphenols are antioxidants that have photoprotective, anti-inflammatory and anticarcinogenic properties. Flavonoids are a subgroup of polyphenols that are admired antioxidants in cosmeceuticals (Choi and Berson, 2006). Major flavonoids found in green tea are

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catechins. Epigallocatechin -3- gallate (EGCG) is approximately 59% of total catechins found in green tea. Green tea also contains gallic acid, kaempferol, myricetin and quercetin (Cabrera et al., 2006). Major limitation with oral use of catechins is their strong affinities for certain elements and proteins, which hinder digestibility and absorption of these substances (Sumpio et al., 2006). Moreover extensive biotransformation and poor bioavailability makes it impossible to show effects at lower doses (Khan and Mukhtar, 2007). Topical emulsions have been used as dermatological formulations for a long time (Marti-Mestres and Nielloud, 2002). Water in oil type emulsions are frequently used because of their ease of application, restriction of water evaporation from skin surface and by their emollient action (Carter, 2007).

Because of their thermodynamic instability, emulsions have always represented a significant challenge to the formulator and manufacturer of cosmetic products. Through years of experience and with the availability of many emulsifying agents and other types of excipients, the formulator generally has had little difficulty in coming up with a formulation, which provides the physical and chemical characteristics required of any cosmetic product. However, because of the often complex nature of these cosmetic formulations and the relatively uncertain conditions to which they may be exposed during manufacture and storage, the assessment of any changes, which may take place over an extended period of storage and use is not as readily carried out, particularly the prediction of long-term stability (Zografi, 1982).

Abil®EM90 is a non-proprietary name for dimethyl copolyol / modified polyether-polysiloxane. It is miscible with ethyl acetate, methyl ethyl ketone, mineral oil and toluene. It is soluble in isopropylmyristate and very slightly soluble in ethanol. Practically, it is insoluble in glycerin, propylene glycol and water. Generally it is non-irritant and non-toxic with slight irritation to the eye (Rowe et al., 2003).

Therefore, the purpose of this study was to formulate and develop a stable water-in-oil (W/O) emulsions containing the extract of the green tea leaves. Abil®EM90 was used as an emulsifier and effects of type of the emulsifier, oil, as well as concentrations of the extract on physical characteristics and stability of the prepared emulsions were focused in this study. Furthermore, the viscosity of the prepared product and stability studies were performed for 28 days without adding any preservative and added substances.

EXPERIMENTAL

Chemicals

The following substances were used for the preparation of w/o emulsions: The oil used was liquid paraffin (η: 110 to 230 mPa.s, Merck). Abil®EM90 (cetyl dimethicone copolyol, Goldschmidt, France) was used as a lipophilic surfactant. Concentrated green tea extract was prepared in Lab No. 42 in Department of Pharmacy, the Islamia University of Bahawalpur, Pakistan. Lemon oil was purchased from a local market.

All materials were used without further purification.

Preparation and characterization of emulsions

Emulsions were prepared by homogenization and ingredients used for the preparation of emulsions with varying concentration of oil and surfactant are enlisted in Table 1. For preparing emulsion as a general oily phase that consisted of paraffin oil and surfactant, Abil®EM90 was heated up to 75 ± 1℃. At the same time, aqueous phase consisting of water was heated to the same temperature and then the green tea extract was added to it after heating. After that, aqueous phase was added to the oil phase drop by drop. Stirring was continued at 2000 rpm by a homogenizer (Euro-Star, IKA D 230, Germany) for about 15 min until complete aqueous phase was added, 2 to 3 drops of lemon oil were added during this stirring time to give good fragrance to the formulation. After the complete addition of the aqueous phase, the speed of the mixer was reduced to 1000 rpm for homogenization, for a period of 5 min and then the speed of the mixer was further reduced to 500 rpm for 5 min for complete homogenization; until the emulsion cooled to room temperature.

Emulsions formed were divided into five categories based on Abil®EM90 concentration and oil concentration respectively shown in Table 1. A total of 25 formulations from category 1, 2, 3, 4 and 5 were formed according to the afo0rementioned procedure and were kept at 25℃ in incubator (Sanyo MIR-153, Japan) for a p eriod of 21 days. All formulations were observed for any change in physical appearance for example color, liquefaction and phase separation. After 21 days formulation number 6, 11, 16 and 21 were selected to keep them further in different storage conditions (8 ± 0.1℃ in refrigerator, 25 ± 0.1℃, Cold Incubator, Sanyo MIR-15 3, Japan, 40 ± 0.1℃, Hot Incubator, Sanyo MIR-162, Japan and 40 ± 0.1℃, Hot incubator, Sanyo MIR-162, Japan with 75% relative humidity). 4 formulations were kept in these conditions for a period of 21 days. Formulation number 21 from category 5 was found the most stable with respect to physical appearance at various storage conditions. Then 4 samples of formulation number 21 from category 5 were subjected to stability studies for a period of 28 days subjecting them to varying storage conditions of temperature and humidity.

Viscosity of prepared emulsions

The viscosity of the prepared emulsion number 21 from category 5 was determined by heat-cool cycling between refrigerator temperature and 40℃ + 75% relative humidity with st orage at each temperature of 24 h for a period of 4 weeks. Viscosity test was performed at different conditions provided to emulsions to note the effect of these conditions on the storage of emulsion. These tests were performed on samples kept at 8 ± 0.1℃ (in refriger ator), 25 ± 0.1℃ (Cold Incubator, Sanyo MIR-153, Japan), 40 ± 0. 1℃ (Hot Incubator, Sanyo MIR-162, Japan) and 40 ± 0.1℃ (Hot I ncubator, Sanyo MIR-162, Japan) with 75% relative humidity (RH). The viscous properties of the emulsion were measured by using Brookfield digital rheometer (Model DV-III, Brokkfield, USA).

Physical characteristics of emulsions

The physical characteristics or appearances of the prepared emulsions were determined by visualization of a single person. These characteristics included color, liquefaction and phase separation. The phase separation of the prepared emulsions was

Table 1. Category wise composition of 25 formulations.

Formulation No	Abil®EM90	Liquid paraffin (%)	Distil water (%)	Green tea extract (%)	Fragrance (%)
Category 1					
1	3.0	16	79	3	1
2	3.0	18	77	3	1
3	3.0	20	75	3	1
4	3.0	22	73	3	1
5	3.0	24	71	3	1
Category 2					
6	3.5	16	78.5	3	1
7	3.5	18	76.5	3	1
8	3.5	20	74.5	3	1
9	3.5	22	72.5	3	1
10	3.5	24	70.5	3	1
Category 3					
11	4.0	16	78	3	1
12	4.0	18	76	3	1
13	4.0	20	74	3	1
14	4.0	22	72	3	1
15	4.0	24	70	3	1
Category 4					
16	4.5	16	77.5	3	1
17	4.5	18	75.5	3	1
18	4.5	20	73.5	3	1
19	4.5	22	71.5	3	1
20	4.5	24	69.5	3	1
Category 5					
21	5.0	16	77	3	1
22	5.0	18	75	3	1
23	5.0	20	73	3	1
24	5.0	22	71	3	1
25	5.0	24	69	3	1

observed by using centrifuge machine (Hettich EBA 20, Germany). Centrifugation of all samples kept at various storage conditions was performed at 5000 rpm and for 10 min for each sample. Samples were subjected to centrifugation at 0 time (fresh), 12, 24, 36, 48 and 72 h for 7, 14, 21 and 28th day to observe any phase separation.

RESULTS AND DISCUSSION

Physical characteristics

No color change and phase separation was observed after visualization throughout the study period at all storage conditions, however, slight liquefaction was observed in sample kept at 40℃ in the last week of observation. Results are shown in Table 2.

Due to the complexity of the nature of emulsions, it is difficult to generalize the results beyond the scope of the experiments. The optimum initial surfactant location for a given system would be dependent on the end results desired (Lin, 1968). We were interested to attain the desired stability of emulsion by changing the surfactant and oil concentration. It has been previously reported that, optimum concentration for Abil®EM90 is 2 to 4%. Cetyl dimethicone copolyol (Abil®EM90) used as a lipophilic surfactant is the siliconic polymeric surfactant and shows good trapping capacity, prolongs the release of active molecules and produces a w/o emulsion with strong interfacial film (Tirnaksiz and Kalsin, 2005).

Table 2. Physical charactristics of formulation number 21 of Category 5 kept at 8, 25, 4040℃ and 40℃ + 75% RH.

Parameter		Fresh	12 h	24 h	36 h	48 h	72 h	7 Days	14 Days	21 Days	28 Days
Colour	A	LG	LG	LG	LG	LG	LG	LG	LG	LG	LG
	B	LG	LG	LG	LG	LG	LG	LG	LG	LG	LG
	C	LG	LG	LG	LG	LG	LG	LG	LG	LG	LG
	D	LG	LG	LG	LG	LG	LG	LG	LG	LG	LG
Liquefaction	A	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve
	B	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve
	C	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	+ve
	D	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve
Phase separation	A	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve
	B	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve
	C	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve
	D	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve

LG = Light green, -ve = no change, +ve = little change; A = at 8℃; B = at 25℃; C = at 40℃; D = at 4 0℃ + 75% RH (relative humidity).

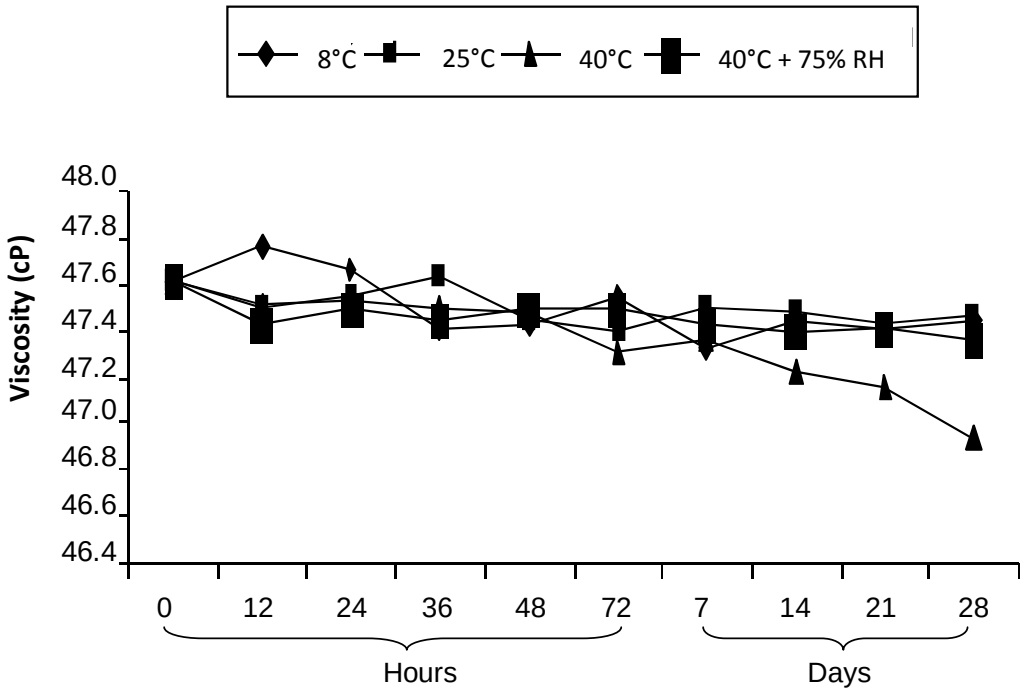

Figure 1. Viscosity (cP) of formulation number 21 of category 5.

Viscosity

Thus, according to the rheological study, the formulations remained stable throughout the test period of 28 days, especially at the lower temperatures. However samples kept at 40°C shown decline in viscosity due to effect of high temperature. These results are shown in Figure 1.

Regarding the physical stability of an emulsion, it is known that its degradation by heating is a consequence of changes in the solubility of the components of the emulsion or facilitation of the coalescence phenomenon. Hence, study of the rheological behavior during the development of cosmetic formulations is fundamental to assessing their stability. Such studies also enable the

behavior of the product during use to be predicted, since rheological properties may be correlated with the effectiveness of the product. The storage temperature can alter the viscosity of the product, and higher temperatures lead to precocious instability, by reducing the continuous phase viscosity and favoring an increase in particle motility and the interactions between the phases (Gonçalves and Maia Campos, 2009).

Conclusions

The investigations presented lead us to conclude that stable water in oil emulsion containing green tea extract can be formulated to explore its potential for desired topical effects. The physical characteristics and stability of the prepared products were the major criteria in this study, to determine the suitable formula for possible future application to the skin. It was found that a formulation containing 5% Abil®EM90 and 16% liquid paraffin oil was the stable formulation regarding its physical characterization, including viscosity of the emulsion. Up to our knowledge, it is the first time to predict the stability by varying emulsifier and oil concentration, without adding any stabilizers. Hence it could be a new way to formulate stable topical emulsions.

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