

Formulation, Preparation and Evaluation of a Natural Cosmetic Product

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Abstract

Background: Nowadays the demand for natural products is increasing. By definition natural products are considered all those products which have at least one natural ingredient. It is recommended to use as few ingredients as possible in order to avoid incompatibilities.

Aim: This study intends to formulate, develop and prepare natural cleansing milk for the skin that is prone to acne. This preparation is meant to give a deep cleansing effect and lessen day by day acne lesions.

Study Design: This is an original article. It is an experimental study on formulation and

characterization of cosmeceutical products for dermal use.

Methods and materials: Formulations were prepared under the Good Laboratory Practice guidelines, using the inverse method of preparation for emulsions. Their control was based on methods described by the European Pharmacopoeia (Ph.EUR).

Materials used are sunflower oil, rosemary essential oil, chosen randomly from the Albanian market, stearine, triethanolamine, boric acid, citric acid, distilled water. Necessary equipments are water bath (Model HH-S6 with 6 places); pH-meter, (Model

InnoLab ph 7110); viscometer (Fungilab, series Alpha, ALPH 141057); balance (Model CL501, Capacity 500g X 0.1g, OHAUS CORPORATION, USA), etc.

Results: The formulation containing 5 drops of rosemary oil, 25% sun flower oil, 5% stearine, 10% triethanolamine and distilled water up to 100%. It results stable cleansing milk, with a nice milky color and consistency. It has the characteristic smell of the ingredients especially rosemary oil. Samples showed a pH value between 4.5 and 8.5 for the whole period of control study, four weeks. As far as it concerns the viscosity remains stable at 1.9 Pa.S. from the first measurement until the end of study.

Conclusion: This formulation of cleansing milk is natural, can be prepared even in a galenic laboratory in pharmacy. It preserves the required characteristics during 4 weeks such as pH within range 4.5-8.5, color, viscosity and aroma. It is recommended to use within 30 days after opening. It should be stored in a dark, well closed bottle away from light and heat.

Keywords: natural, essential oil, cleansing milk, acne.

INTRODUCTION

Nowadays the demand for natural products is increasing rapidly. By definition natural products are considered all those products which have at least one natural ingredient. It is recommended to use as few ingredients as possible in order to avoid incompatibilities. Up to date studies on dermatologic pathologies shows that acne vulgaris is one of eight most common illnesses of the skin with a prevalence of 9.38%, all ages included. Throughout different countries, in different ethnical groups the prevalence goes from 35% up to approximately 100% in adolescents, who have had acne at least once in their life (1).

A few years ago, hygiene was considered as the main factor for the appearance of acne. Although today it is shown that lack of hygiene is not the only one, but a hard cleaning of the skin or cleaning the skin with severe soaps and certain chemicals can irritate the skin and worsen acne. So the use of delicate products as skin cleansers or even better the use of natural products would be of benefit for skin prone to acne.

Essential oils are known for their anti acne effectiveness. Essence is a mixture of natural aromatic substances produced from plant species. It has oily consistency, lipophile character, evaporates easily from the plant through distillation process into the water. Essential oils can be distilled from leaves, radices, bark and other parts of aromatic plants.

The most effective essential oils against acne are considered that of thyme, rosemary, cinnamon, tea tree oil, green tea etc (2,3,4,5).

Great Britain studies show that thyme essential oil is very effective against acne especially those of the face used as concentrated alcoholic solution. Data showed that the tincture of thyme is more effective than the local treatment with benzyl peroxide to calm the acne situation (6,7,8,9).

From laboratory tests, it is proven that the essential oil of rosemary destroys bacteria *P. acnes* which leads to the appearance of acne. Initially it attacks cellular walls and at the end kills the bacterial cells. Apart from antibacterial effect rosemary can be used also to reduce redness and swelling which is very beneficiary in the treatment of cystic acne. It has been studied thoroughly also the essential oil from bark of cinnamon tree. It resulted very effective against *P. acnes*. Studies report that it reduces the menstrual pain and cholesterol levels. Cinnamon is a safe tool against staphylococcus bacteria and *E. coli* (10).

Tea tree oil has antimicrobial, antiseptic and anti inflammatory properties, that's why it is very popular and successful against acne. Investigations shows that tea tree oil is also effective in wound healing which means it can be used for the treatment of acne or just calms them down.

These are some data which made the research

group decide to formulate a cleansing emulsion with rosemary essential oil for a double effect.

The aim of this study is to formulate, prepare, quality control of a natural cleansing emulsion, in order to clean, fade the acne lesions on the skin or even, in some cases, prevent day by day the appearance of acne.

This study intends to formulate, develop and prepare natural cleansing milk for the skin that is prone to acne. This preparation is meant to give a deep cleansing effect and lessen day by day acne lesions.

METHODS AND MATERIALS

The method of formulation is based on the guidelines of Good Laboratory Practices, it is known as inverse method of emulsion preparation.

Quality control tests are described in the European Pharmacopoeia (Ph.EUR), VIth edition.

Materials used for the formulation phase are sunflower oil (from the market in Albania), Stearin, Triethanolamine, Boric acid, Citric acid, distilled water, rosemary essential oil; laboratory equipment such as chemical glasses (250ml and 500ml), porcelain mortar, water bath (Model HH-S6 with 6 places); pH-meter, (Model InnoLab pH 7110); viscometer (Fungilab, series Alpha, ALPH 141057); balance (Model CL501, Capacity 500g X 0.1g, OHAUS CORPORATION, USA), etc

Technological process of preparation

There have been prepared different formulations by inverse method of preparation of emulsions and at the end it has been chosen the most stable one. Below are explained different formulas.

Base formulation I consists of sunflower oil at 25%, stearine at 5 %, triethanolamine at 10 %, distilled water up to 100 % and rosemary essential oil 5 drops.

The measured pH was 9.2 which exceed the permitted range of values 4.5-8.5 for this kind of product. This means that it could be a suitable environment for the development of bacteria and as a consequence it could worsen the situation of acne. That's why it was modified the base formulation in order to have a suitable pH value. First it was added boric acid which is a well known ingredient with cleaning, antiseptic, antibacterial effect, etc. The test of pH showed that it was not enough to get the pH within 4.5-8.5. At this point it is not advisable to increase its percentage because it could damage the homogeneity of the emulsion and irritate the skin. So it was used a system of ingredients to get the appropriate pH, boric acid in combination with citric acid. It was chosen the citric acid, because it is part in many preparations for topical use. First it is prepared a solution of both acids with 3% boric acid and 1% citric acid (11,12).

Here below is explained the method of preparation. This solution will replace the distilled water used in formulation I.

Boric acid has a lower rate of solubility and as such it is the first to be dissolved in hot distilled water. After cooling down, it is added the quantity of citric acid, stirred and brought to 100 g with some more distilled water.

So base formulation II contains sunflower oil at 25%, stearine at 5%, triethanolamine at 10%, citric acid at 1%, boric acid at 3%, distilled water up to 100% and rosemary essential oil 5 drops.

In a mortar of 250 mL it is added the quantity of sunflower oil and stearine and placed in water bath at 70-75°C as it is shown in Figure 1.

In a glassware is added the quantity of triethanolamine (TEA) and then other small quantities of the prepared acidic solution stirring continuously up to the necessary level of it.

After the complete melting of oily phase, the mortar is placed in the bench to cool down a little bit. In the mean time the watery phase has become a transparent solution without any solid particle of acid. At this point when both phases are at the same temperature the watery phase is poured part by part, stirring slowly and continuously, into the oily phase until it becomes a homogenous emulsion (Figure 2). It is stirred slowly and continuously to prevent the foam formation, if it happen it should be wiped away with a paper filter. At the end of the process is added the necessary quantity of rosemary essential oil triturating softly until the created mass becomes cool (at room temperature).



Figure 1. Fatty Phase melting in water bath at 70-75°C.



Figure 2. Mixing two phases in a porcelain mortar.

For the packaging are used dark glass bottles of pharmaceutical quality.



Figure 3. Dividing samples to use for stability testing.

Samples are divided in different groups in order to check their quality and stability via accelerated aging method. The accelerated aging test

simulates real-time aging and shelf life by using elevated temperatures to artificially speed up the aging process. Accelerated aging is an optional requirement for testing, but real-time aging is required when establishing an expiration date for a product or product packaging (13). So Group 1 of samples was kept at room temperature and humidity, Group 2 in the oven at 40°C and Group 3 in the fridge at 2-4°C Samples were controlled on regular basis for a period of five weeks.

RESULTS

First the pH-meter (Figure 4) was calibrated using 3 buffer solutions such as: Acid Buffer acid, Neutral buffer and basic buffer and after that it is performed the measurement of pH per each sample.

Viscosity was measured using a viscometer Fungilab (Figure 5) with 100 rotations per minute (100 RPM) and spindler R2. 24 hours after preparation, viscosity of samples kept at room temperature was 1.5 Pa.s., whilst those kept in the fridge showed a value of 0.8 Pa.s.

The organoleptic control showed that samples had milky white, slightly misty and with the scent of rosemary essence. Regardless the storage conditions such as room temperature or the 2-4°C, samples kept the color, aroma and appearance unchanged. Samples that were placed in the thermostat at 40°C after week II changed color and smell and therefore were excluded from further control tests.

The samples observed with the naked eye and with a 4.5 times magnifying lens present a homogeneous structure. In terms of pH control, the following results:

Samples of formulation I after 1 week kept in different conditions of temperature and humidity gave the following results:

- Base pH (without essential oil) = 9.32
- pH at room temperature = 9.37
- pH at 2- 4°C = 9.5
- pH at 40°C = 9.4

Samples of formulation II obtained after the addition of acids as above, stored in different conditions of temperature and humidity, gave the results summarized in table 1.

Table 1. pH control values.

Time Parameter	24 hours after preparation	Week I	Week II	Week III	Week IV	Week V
pH at 20°C	7.4	7.27	7.305	7.224	7.28	7.18
pH at 4°C	7.4	7.7	7.691	7.403	7.54	7.61
pH at 40°C*	7.4	7.357	7.209	-	-	-

*Samples changed organoleptic characteristics after week II and no further control tests were conducted on them

Since we are dealing with an emulsion, the rheological control of the preparation is focused on determining the viscosity. The results of determinations over time are summarized in Table 2.

Table 2. Viscosity control values.

Time Parameter	24 hours after preparation	Week I	Week II	Week III	Week IV	Week V
Viscosity at 20°C	1.4 Pa.s	2.1 Pa.s	2.1 Pa.s	1.5 Pa.s	2.3 Pa.s	2.5 Pa s
Viscosity at 4°C	1.4 Pa.s	3.1 Pa.s	2.3 Pa.s	1.7 Pa.s	1.8 Pa.s	1.6 Pa.s
* Viscosity at 40°C	1.4 Pa.s	1.2 Pa.s	1.1 Pa.s	-	-	-

*Samples changed organoleptic characteristics after week II and no further control tests were conducted on them

DISCUSSION

Acne is one of the biggest problems among many other health problems all over the world. A very large number of people have these problems and hope to find solutions as quickly and efficiently as possible. Patients are tired of long and expensive treatments and want immediate solutions.

There are many products on the market that are used to treat acne. They can be synthetic or natural, plant products. Medicines used in these cases are thought to remove acne faster, but are not always recommended. In many patients, herbal products are effective and help somewhat in their elimination. It is worth noting that essential oils have also been proven to help treat acne. Among them, rosemary oil has turned out

to be one of the most effective in the treatment of acne due to its antimicrobial and antioxidant properties. There is a growing interest in the use of natural extracts, also as alternatives to synthetic additives, due to (a) their synergy with

other preservation methods (b) their safety profile and (c) their specific properties as antioxidant, antidiabetic, antimutagenic, antitoxic and antibacterial.

Rosemary oils are obtained by steam distillation of fresh branches and leaves. Rosemary extract is used in the treatment of various diseases, due to its hepatoprotective potential, therapeutic potential for Alzheimer's disease and its antiangiogenic effect. In turn, they prevent oxidation and microbial contamination. Therefore, rosemary extract can be useful for replacing or even reducing the level of synthetic antioxidants in foods and drugs. Cyclic diterpene diphenols, carnosolic acid and carnosol have been identified among the most effective antioxidant components of rosemary. In addition,

its extract contains carnosic acid, epirosmanol, rosmanol, methylcarnosate and isorosmanol. As a preservative, rosemary extracts offer several advantages and technological benefits to consumers (14).

The natural cleansing product proposed in this paper is an innovative preparation which can be prepared in the pharmacy "ex tempore". It is simple, safe and cost-effective. Although preparatory personnel should have in mind some steps. The cleaning emulsion can be prepared either by adding the oil phase to the water phase or vice versa. The inverse method was chosen for the reason that during the addition of the aqueous phase to the oily one, phase inversion occurs and a more stable emulsion is created. A stable O/W emulsion was thus obtained.

Care must be taken during the preparation: add the rosemary essential oil after the temperature of the preparation has decreased. This is because the essential oil evaporates at high temperature.

Another aspect where care must be taken is the way the emulsion phases are mixed. It should be performed evenly, counter-clockwise and at a constant rate. This is because, due to irregular mixing, the preparation can absorb air. This phenomenon can cause degradation of the fatty phase of the emulsion and result in an inhomogeneous preparation that expires very quickly.

If we observe the obtained results, we can recommend packaging the emulsion in dark glass containers and keep them at a temperature between 4°C - 25°C.

From the pH values obtained for the samples stored at room temperature, during the 5 weeks of control, it is observed that they fluctuate from 7.4 to 7.18 in the last week, remaining within the range of 4.5-8.5.

Regarding viscosity at room temperature of 20 °C, the lowest value was at the initial moment with 1.4 Pa.s. and the highest 2.5 Pa.s. the last week. At a temperature of 4°C, the lowest value resulted in the last week of control and the highest in the first week. It should be emphasized that the emulsion needs 24 hours to stabilize the internal structure and reach the final mistletoe. At room temperature viscosity increases, while at low temperature, viscosity decreases over time. In both cases, from the rheological point of view, the emulsion behaves as a pseudoplastic fluid, whose viscosity is directly proportional to the temperature or tangential stress values. In conclusion, we can confirm that as the temperature decreases over time, the viscosity decreases.

CONCLUSIONS

From the above data and results, it can be concluded that a cleansing emulsion with essential oil was formulated and prepared. It will act as a synergistic product with the drugs for the treatment of acne. It was observed that both at room temperature and at 4°C the samples showed stable appearance elements such as color, odor and homogeneity; viscosity and pH values are also rather stable within the accepted range of values. After carrying out all the necessary tests,

it is estimated that this natural emulsion can be prepared "ex tempore" in the pharmacy to help treat acne and remove the lesions afterwards. This is a preparation that accelerates healing and reduces the intensity of post-acne marks.

It is recommended to use within 30 days of opening. Store in dark glass bottles or if possible, in dark places away from light and heat.

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