Hydrolates as water phase in cosmetic creams: physicochemical and in vivo assessment

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Introduction

Hydrolates are considered a highly diluted water-soluble fraction of essential oils and as such do not have many of the undesirable skin reactions. According to the Cosmetic Ingredient Database, they have already found their way into the cosmetics industry (European Commission, 2009). Hydrolates could also be a promising way to replace the water phase in cosmetic products, reducing wastewater disposal costs and environmental impact. Considering the proven antimicrobial activity of many hydrolates, they can potentially act as preservatives in cosmetic formulations (Aćimović et al., 2020). However, there is limited research on cosmetic emulsions with hydrolates as an aqueous phase. The main objective of the present study was to evaluate the physical stability and preliminary topical safety profile of creams formulated with some of the most commonly used hydrolates as water phase.

Materials and methods

Preparation of cream samples: For preparation of the O/W emulsion-type cream Brij TM S2 and Brij TM S721 emulsifiers were used. Cetaryl alcohol, coconut oil decyl oleate, dimethicone, petrolatum, isopropyl myristate, caprylic/capric triglycerides, butyrospermum parkii were used as an oil phase. Panthenol, carbomer, glycerol, sodium benzoate were added in the water phase which also consisted of purified water (in placebo cream) or specific hydrolate. All chemicals used were of cosmetic grade. Commercially available Anthemis nobilis L., Daucus carota L., Thymus vulgaris L., Melissa officinalis L., and Lavandula angustifolia Mill. hydrolates, produced by Promontis (Vilandrica, Serbia), were used. Emulsifier was mixed with other lipophilic components and then heated to 70°C on the thermostatic heating plate of the magnetic stirrer IKA-MAG (IKA Werke, Staufen, Germany). The oil phase was slowly added to the already heated water phase with constant stirring until reaching room temperature. For stirring, a RW16 basic (IKAWerke) propeller-rotary laboratory stirrer was used. The placebo cream sample (P) was prepared with purified water. The investigated creams were made using one of the hydrolate to replace purified water: (A) cream with hydrolate of Daucus carota, (B) cream with hydrolate of Lavandula angustifolia, (C) cream with hydrolate of Melissa officinalis, (D) cream with hydrolate of Thymus vulgaris, and (E) cream with hydrolate of Anthemis nobilis.

Physico-chemical analysis and stability study: The organoleptic characteristics (color, texture, shine, odor, homogeneity) of the creams were examined. The electrical conductivity and pH values of the samples were measured by directly immersing a conductometer electrode (CDM 230, Radiometer, Denmark), and the pH meter electrode (pH 211 Microprocessor pH Meter) into the sample. The measurements were performed initially and after 30 days of storage at room temperature. To observe the alterations of phase separation and changes in appearance under stress conditions, international organizations for cosmetics, such as IFSCC have published a number of recommended guidelines (IFSCC Monograph, Number 2). The cream samples were exposed to a centrifuge test (3000 rpm for 30 min) and freeze-thaw cycling. The creams were stored in a refrigerator at 4°C, and then thawed in an oven set at 45°C for 24 hours to complete a cycle.

In vivo skin irritation potential: In order to assess topical safety (skin irritation potential) of investigated creams, the in vivo effects were assessed by measuring the appropriate biophysical parameters before and after the
application of the cream samples under occlusion on human skin (Tasic-Kostov et al., 2019). The Multi Probe Adapter System MPA®️9 (Courage & Khazaka, Germany) was used with the appropriate probes to measure electrical capacitance (EC), transepidermal water loss (TEWL), skin surface pH levels, and erythema index (EI). The study was performed in accordance with the Declaration of Helsinki and the experimental protocols after being approved by the local Ethical Committee on Human Research of the Faculty of Medicine in Niš (No. 12-2691/2-4). After signing informed consent, 20 healthy volunteers were included in the study.

**Results and discussion**

All prepared creams were white, with a different odour depending on the used hydrolate, a slight gloss, a semi-solid consistency, and homogeneous. After 30 days of storage at room temperature, the color and consistency of the samples remained unchanged; the odour became milder. The homogeneity was satisfactory for all samples, without any signs of phase separation. The pH of the external water phase of the emulsions decreased after 30 days of storage, being higher in sample C. Electrical conductivity increased slightly in sample C, while it decreased slightly in the other samples compared to baseline values. However, these changes were not statistically significant. Preliminary stability tests were carried out utilizing two separate techniques: centrifugation and freeze-thaw cycles. The investigated cream formulations showed satisfying physical stability. Regarding in vivo investigation, after 24 hours, EI, a parameter which could indicate an unfavorable skin irritation profile of the preparation showed a statistically significant decrease compared to baseline values. A certain increase in TEWL was registered after the application of samples A, B and P. These results were consistent with earlier studies showing that occlusion per se could lead to decrease in SC barrier function, and therefore an increase in TEWL (Zhai and Maibach, 2002). Additionally, there were no significant variations in TEWL, EI and pH relative changes between creams containing hydrolate and placebo, suggesting that the hydrolate did not cause any skin irritation.

**Conclusion**

Considering the demonstrated antimicrobial activity of many hydrolates, they can potentially be used as preservatives in cosmetic formulations. Our results show that creams formulated with certain hydrolates as water phase exhibited satisfactory physical stability during a 30-day study. In addition, the samples exhibited acceptable skin irritation profiles. Further studies, such as a cosmetics challenge test, should be conducted to assess hydrolates in cosmetics as potential alternatives to commonly used preservatives for cosmetics.

Figure 1. The influence of the cream samples on in vivo measured skin parameters; the results are shown as relative changes of mean values on the second vs. First day and standard error of means. *p < 0.05

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**References**


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