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Stability studies of cosmetic emulsions prepared from natural products such as wine, grape seed oil and mastic resin

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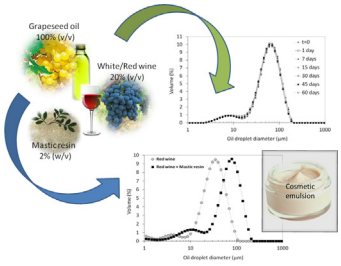
HIGHLIGHTS

- Still wine and grapeseed oil can contribute natural components to cosmetic emulsions.
- Low wine concentrations yield rheologically stable O/W emulsions.
- The optimum wine percentage in the aqueous phase was found to be 20% (v/v).
- Mastic resin at 2% (w/v) is optimum for preparing O/W emulsions with grape seed oil.
- Combining wine and mastic resin results in bigger droplets and lower viscosity.

GRAPHICAL ABSTRACT



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ABSTRACT

An attempt was made in this study to use diluted wine as the aqueous phase and grapeseed oil as the oil phase for the preparation of oil-in-water cosmetic emulsions. Two monovarietal wines of Hellenic origin were used in this study; a red one from *Sangiovese* grapes and a white one from *Muscat of Samos* grapes. The oil-to-water ratio in the emulsions was 20:80 (v/v) and the wine concentrations in the aqueous phase were in the range of 5–100% (v/v). Glycerol monostearate was used as emulsifying agent. The only extra additive was mastic resin from *Pistacia lentiscus* var. *Chia*, which is reported to have healing and antibacterial properties. The study of the emulsion stability involved droplet size determinations and viscosity measurements for a period of sixty days. It was found that the optimum percentage of wine (red and white) in the aqueous phase of such emulsions is 20% (v/v) and of the mastic resin 2% (w/v).

1. Introduction

During the last decade consumers have widely expressed their interest in natural products forcing the industry and the scientific community to search for alternative sources and raw materials. Cosmetology is a highly affected area by this phenomenon and the use of natural components from olive oil to jojoba extracts forms the latest trend. One of the most significant reasons for this change is the appearance of allergies and skin irritations due to synthetic preservatives (e.g. parabens), colourants, stabilizers etc. that have not yet been fully tested in the long run for their consequences on consumers' health.

Wine and grapeseed oil are both natural products. Red wines especially have a high content in natural antioxidants such as

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flavanoids, natural colourants such as anthocyanins and natural aroma compounds such as terpenes and esters [1,2]. Grape seed oil on the other hand is considered to be a dietary oil of high quality with a high concentration of unsaturated linoleic acid, vitamin E and phytosterols [3,4]. In this study, an attempt was made to prepare oil-in-water cosmetic emulsions by adding white or red wine in the aqueous phase and using grape seed oil as the oil phase in order to examine their stability with time and exploit their beneficial constituents. The study was complemented by using the natural resin from the mastic tree of *Pistacia lentiscus* var. *Chia* as additive to examine its impact on the organoleptic and rheological characteristics of the emulsions. The mastic resin was chosen as a natural additive of Hellenic origin with healing and antibacterial properties [5–9].

2. Materials and methods

The oil-to-water ratio for the preparation of the emulsions was 20:80 (v/v) and commercial glycerol monostearate (GMS) was used as emulsifier at 6%, 8% and 10% (w/v). Two Hellenic wines, the white 'Samena Golden' from the grape variety 'Muscat of Samos' with 12.4% (v/v) ethanol and the red 'Sangiovese Karipidis 2002' from the grape variety 'Sangiovese' with 12.7% (v/v) ethanol were added at concentrations 5%, 10%, 20%, 50% and 100% (v/v) in deionised water to form the aqueous phase. Grapeseed oil (Henry Lamotte, Germany) was the oil phase in all cases. Mastic resin (Chios Mastiha Growers Association, Hellas) was added at concentrations 1%, 2% and 3% (w/v).

Emulsification was performed using an impeller with sawtooth edges placed for rotation in the centre of a 600-ml borosilicate-glass beaker and at approximately 1 cm above the bottom of the beaker. Initially, grapeseed oil was transferred to the beaker and heated at 70 °C. GMS was then slowly dissolved in the hot grapeseed oil. The aqueous phase was also heated separately in a conical flask at the same temperature and was subsequently added gradually to the oil phase under intense agitation at 400 rpm for 2 min. At this point, the heating was turned off and the emulsion formed was kept agitated until temperature dropped to 25 °C.

Applying this procedure, attempts were made to prepare emulsions with various combinations of the constituents described above. Out of these attempts, the following three series of 20:80 (v/v) O/W emulsions were successfully prepared:

(a) with grapeseed oil, 5–20% (v/v) white wine in the aqueous phase and 6% (w/v) GMS as emulsifier (series code: **W**); emulsions were not successfully prepared with 50% and 100% (v/v) white wine in the aqueous phase even when 10% (w/v) GMS was added.
(b) with grapeseed oil, 5–20% (v/v) red wine in the aqueous phase and 6% (w/v) GMS as emulsifier (series code: **R**); emulsions were successfully prepared also with 50% and 100% (v/v) red wine in the aqueous phase but only when 10% (w/v) GMS was added.
(c) with grapeseed oil, deionised water (i.e. 0% wine in the aqueous phase) and 1–3% (w/v) mastic resin as additive (series code: **M**).

In all cases, an O/W emulsion with 20:80 (v/v) grapeseed oil and deionised water was prepared as control sample (code: **C**).

The study of the emulsion stability involved the following procedures:

(a) Surface tension measurements of the aqueous phase using a Sigma 70 tensiometer (KSV Instruments Ltd., Finland) at 25 °C. The Wilhelmy plate technique was applied in the Wilhelmy Constant Run programme mode. A platinum plate was partially immersed into the surface layer of an aqueous phase and the monitored surface tension decreased with time while the plate remained into position. The analysis ceased when the surface tension value was stabilized. Prior to each analysis, the surface tension of deionised water was measured as control.
(b) Interfacial tension measurements of the two-phase system using a K6 tensiometer (Krüss, Germany) at 25 °C. The Du Nouy ring method was employed. A platinum ring was used first to zero the indicator with grapeseed oil and then to measure the interfacial tension between grapeseed oil and each aqueous phase. Deionised water was used first as the aqueous phase for the reference measurement. Then followed the other aqueous phases which contained wine, measured from the most diluted one (5%, v/v wine) to the least diluted (100%, v/v wine). The interfacial tension of each system was measured at the point where the ring broke away from the interfacial layer of the two phases.
(c) Determination of the mean surface droplet diameter $D_{[3,2]}$ and droplet size distribution with the use of a Mastersizer 2000 with a HYDRO 2000MU unit (Malvern Instruments Ltd., UK). $D_{[3,2]}$ is the so called Sauter mean diameter and it is defined as the diameter of a sphere that has the same volume-to-surface area ratio as a particle or droplet of interest.
(d) Viscosity measurements with a LVTDV-II digital viscometer (Brookfield, USA) attached to a temperature controller (PolyScience, USA) and using 25/13R accessories at 1.32 min^{-1} and 25 °C. All measurements and macroscopic observations were performed at 0, 7, 15, 30, 45 and 60 days.

3. Results and discussion

As explained also in paragraph 2 (experimental part of Section 2), with the use of 6% (w/v) GMS as emulsifier, emulsions were successfully prepared with 5%, 10% and 20% (v/v) of red or white wine in the aqueous phase. In the case of higher wine concentrations, i.e. 50% and 100% (v/v), emulsification of grapeseed oil was achieved neither with the red nor with the white wine. However, by increasing the amount of the emulsifier up to 10% (w/v), emulsions were obtained only when using the red wine. For reasons of comparison among the various emulsions, the results discussed below refer only to the samples with 0–20% (v/v) wine (white or red) in the aqueous phase, and do not include the final two emulsions achieved with higher red wine and GMS concentrations.

3.1. Surface and interfacial tension measurements

Surface tension values of the aqueous phase of an emulsion are indicatory data of the emulsion stability with time. Table 1 shows the corresponding values for each emulsion of the study. An alcoholic solution with 12% or 13% (v/v) ethanol has surface tension values γ = 49.5 mN/m and 48.1 mN/m, respectively [10,11]. In Table 1 it is shown that the surface tension values for 100% (v/v) wine with alcoholic grade around 12.5%, which is the average alcoholic grade of the two wines used in this study, are considerably lower than the above reference values. Even more surprising is the

Table 1
Surface tension values of the aqueous phases of the emulsions under study at 25 °C.

Aqueous phase	Surface tension γ (mN/m)	
Wine (%, v/v)	White wine	Red wine
100	42.0 ± 0.6	45.9 ± 0.8
50	48.6 ± 0.6	50.1 ± 0.8
20	51.0 ± 0.5	57.3 ± 0.7
10	54.0 ± 0.4	60.9 ± 0.4
5	62.9 ± 0.7	65.3 ± 0.9
0	71.6 ± 0.5	71.7 ± 0.4

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