Assessment of oil in water emulsion based on rose oil

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Rose oil and rose water are components that can be used in the prevention and care of vascular skin. Formulations containing the above-mentioned ingredients strengthen capillaries and improve the condition of vascular skin. The aim of this study was to form a stable emulsion directed to people with face vascular problems.
Compositions of six emulsions with different amounts of emulsifiers and thickeners were developed according to the specifications and requirements of KT-Skor software (based on the Kleeman's method).
The average particle size, dispersity index, sensory and skin moisturising properties of emulsions were evaluated.
The results obtained indicate emulsion VI (with 10 g of lecithin and 0.2 g of carboxmethcellulose) revealed the highest stability. A significant increase of skin capacitance after application of all emulsions was observed, which was maintained for about 120 minutes. The highest level was reached for areas of the skin after application of emulsions II and III. By analysing the sensory properties, it can be concluded that all the emulsions were homogenous and penetrated the skin well. However, all the examined emulsions had a low greasiness score. The respondents assessed emulsion VI as the weakest.
It was found that the addition of the thickener in the range 0.5-0.6 g and reducing the amount of the emulsifier at the same time, can change emulsion properties and improve the results of the sensory evaluation performed by respondents.

Keywords: rose oil, emulsions, particle size, vascular skin, sensory and hydrating properties

1. INTRODUCTION

Roses were used in ancient Greek, Indian, Sumerian, Chinese and Egyptian medicine. Rose gardening started in antiquity, and since the Middle Ages, roses have been cultivated for their beauty, but primarily for perfume, medicinal or culinary usage. Nowadays, rose preparations, such as oils, seed oil and fruit extract or hydrolate, are important commercial products, especially in the fragrance and flavour industries [1, 11, 19].
The genus *Rosa* includes 200 species and 18000 cultivars [8]. Despite the large number of rose varieties, there are only four species used for rose oil production: *Rosa damascena* Mill., *Rosa gallica* L., *Rosa moschata* Herrm. and *Rosa centifolia* L. [1, 22]. Roses are well known for their positive influence on skin, among other anti-aging properties, ability to promote skin tonicity; they are used to fight stretch marks, skin necrosis, dermatosis, irritations, wounds, and dehydration [11, 19]. Moreover, rose preparations are

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used for gastrointestinal disorders and have therapeutic properties – antiseptic, antiplasmodic, antiviral and antibacterial [3]. Rose oil has a great role in cosmetology, it is used for sensitive skin prone to broken capillaries. According to Authors [9] all organs of *Rosa Canina L.* (leaves, flowers, and fruits) contain favourable essential fatty acids (linolenic and linoleic acids) and also phenolic compounds therefore roses have such a wide range of applications. Besides rose oil contained beneficial compounds such as natural antioxidants which in the opinion of authors [21] are responsible for high stability. Roses are used in different forms. An extract preparation method has an impact on the composition, content of active compounds and pharmacological properties [11, 18].

Vascular skin is one of the most prevalent defects that cosmetologists and dermatologists have to fight [13]. It concerns a growing number of people, both women and men, however, women more often see it as a problem. People with this skin type are especially vulnerable to rosacea. It is a common disease, diagnosed in fair-skinned people of Celtic and northern European heritage, whereas it is rare in American and African blacks [2]. The characteristic symptoms are: presence of initially reversible, then consistent erythema, telangiectasia, papules, and pustules located within the central part of the face, chronic and recurrent dermatitis of the face skin [2, 13, 10]. Factors affecting the appearance of first symptoms are: inappropriate use of cosmetics, unsuitable face care, improper diet, lifestyle, and external factors such as UV radiation, wind, temperature fluctuations [13].

The aim of the study was to determine parameters of an emulsion that would be stable and acceptable to consumers, containing rose oil, which is rarely used in cosmetics. Application of this oil was dictated by its valuable properties for the skin. Contraction of blood vessels is an especially relevant property distinguishing rose oil from others. Therefore, an attempt was made to form an emulsion containing this oil and rose water which is addressed to people with vascular skin.

The novelty in this study was the application of rose oil in cosmetic emulsions, as before this fat had been mainly used by the culinary and food industry.

2. MATERIALS AND METHODS

2.1 MATERIALS

Emulsions were prepared using the following components: rose water (Filomed Sp. z o.o.) (INCI: Aqua, Rosa damascena Flower Oil, Rosa Canina (Rosehip) Fruit Extract, Glycerin, Panthenol, Allantoin, PEG-40, Hydrogenated Oil, Aniba Rosaeodora, Dmdm Hydantoin, Methylchloroisothiazolinone (and) Methylisothia - zolinone, d-limonene, citral, linalol, eugenol, citronelol, C.I. 16255), carboxymethylcellulose (Pronicel Sp. z o.o.), Rosa Canina (Rosehip) Fruit Oil unrefined (Greenaction), sunflower lecithin (RF Solutions), sodium benzoate (POCH), Vitamin Complex (CRODA) (INCI: Carthamus Tinctorius (Safflower) Seed Oil (and) Prunus Amygdalus Dulcis (Sweet Almond) Oil (and) Tocopheryl Acetate (and) Ascorbyl Palmitate (and) Linoleic Acid), Ginkgo biloba extract (FITOCO), citric acid (Jungbunzlauer).

2.2 PREPARATION OF EMULSIONS

The procedure of emulsions preparation was based on our own experience and optimising software based on Kleeman's method [12]. In this project, the amounts of emulsifier and thickener were variable. The third variable (amount of water) was dictated by the amounts of other ingredients and the need to produce an equal mass of all the emulsions – 100g.

All the emulsions were composed of two phases: water phase and oil phase. Appropriate amounts of lecithin (given in Table I) and vitamin complex (0.2 g) were added to 30 g of rosa canina (rosehip) fruit oil. To prepare the water phase, carboxymethyl cellulose solution was prepared separately by dissolving carboxymethyl cellulose (the amounts are given in Table I) in rose water. Ginkgo biloba extract (0.3 g) was added to rose water too. Oil and aqueous phases were heated in a water bath at 40°C. Fine emulsification (i.e. small droplet size between 1 μm and 10 μm with a narrow particle size distribution) was achieved by subjecting the pre-emulsions to pre-homogenization using a high shear blender for 1 min and then homogenization with a high shear mixer at 12000 rpm for 3.5 min. Finally, a preservative – sodium benzoate (0.2 g) - was added and pH was adjusted to 5.5 by

Table I - Changing amounts of components

Component [%w/w]	Emulsion					
	I	II	III	IV	V	VI
Lecithin	2.5	2.5	5.0	7.5	10.0	10.0
Carboxymethylcellulose	0.2	1.0	0.6	0.6	1.0	0.2
Rose water	Up to 100.0					

addition of 2 M citric acid. The volume of each mixture was 100 g.

2.3 METHODS

Determination of average particle size of emulsion and dispersion of emulsion

The laser diffraction method was used to measure the average particle size of the emulsion. It is a commonly used method for particle size measurements in the range of 0.1 to 1000 microns. The particle size is determined using the relationship between the angle of light scattering (laser) and the size of the particles, which caused the deflection. The sample to be analysed is diluted to an appropriate concentration and then placed in a glass measurement cell. A laser beam is generated and directed through the measurement cell, where the emulsion particles scatter it. The intensity of the scattered light is measured as a function of scattering angle using an array of detectors located around the sample [17].

The average particle sizes of the emulsions were measured and given as average particle size of 18 weeks of storage. Determination was carried out in the range 0.12 – 704.0 μm using a Microtrac Particle Size Analyzer (Leeds and Northrup, Philadelphia, U.S.A.).

Given average particle size in the manuscript was presented as a mean diameter (microns) of the volume distribution which represents the centre of gravity of the distribution. The average particle size = MV (according Microtrac software) value was weighted by the presence of coarse particles.

$$MV = \frac{\sum V_i d_i}{\sum V_i}$$

Where:
V – volume percent in a channel size
d – channel diameter in microns

Dispersity index

Dispersity index (K) was calculated on the results obtained from laser diffraction measurements, according to the formula:

$$K = \frac{K90\% - K10\%}{K50\%}$$

Where:
K90%, K10%, K50% - percentile points (microns) show the given percent of the volume that is smaller than the indicated size.

The 50% is also known as the median diameter that is one of three measurements of average particle size.

Determination of skin capacitance

Skin capacitance was measured by means of a CM825 Corneometer (Courage+Khazaka Electronic). The obtained results indicate the degree of skin hydration. The values vary in the range of 0-130 arbitrary units (AU) [4].

To eliminate the impact of external conditions on the results, the measurements were performed under standard conditions of temperature and humidity (T° = 20-22°C, humidity 40-60%), away from direct sunlight. The testing was carried out on 15 respondents – women students of cosmetology from the University of Technology and Humanities in Radom, Poland. The respondents gave their written consent to having measurements of functional parameters of their skins taken before starting the study. The measurements were performed on designated forearm skin fragments before application, immediately after application and then every 15 minutes for 4.5 h. To obtain valid results, each measurement was taken three times.

Sensory analysis

Evaluation of the sensory properties is based on consumer feelings perceived by the senses (touch, taste, smell, sight). The sensory analysis allows to compare various formulations and determine levels of satisfaction after application of e.g. cosmetics.

Sensory analysis was performed by a group of respondents (15 females) who were trained and instructed on the methodology. A 8-point scoring scale was introduced, with 8 the maximum and 1 the minimum score. Six emulsions were subjected to the sensory analysis. The emulsions were assessed for the following characteristics: consistency (density and cohesion of the tested cosmetic), homogeneity (behaviour of the preparation when applied to the skin – absence of clots or air bubbles), cushion effect (palpability of the substance when rubbed between fingers), distribution (facility of spreading on the skin surface), smoothing (smoothing effect when applied to the skin), viscosity (degree of palpable viscosity left on the skin), greasiness (a fat film remaining on the skin) and absorption (rate of absorption by the skin). This analysis was carried out according to the procedure given in [15] and [19].

3. RESULTS AND DISCUSSION

Generally, emulsion destabilisation is a complex process. Properties of individual phases dictate the movements of dispersed phase particles, e.g. density or viscosity, droplet sizes and phenomena that occurs in the system as Van der Waals forces or Brownian movements [16]. To reduce interfacial surface tension occurring between aqueous and oil phase emulsifier is added [6].

Due to emulsions are thermodynamically unstable by nature, emulsion stability is one of the most important characteristics for the development and quality con-

trol of cosmetic emulsions. One of the main measurements in assessing emulsion stability is the monitoring of thermodynamic changes occurring in emulsion by determining their droplet size over time [14].
Analysing the results of laser diffraction determination, the smallest changes after 3 months of storage were noted for emulsion VI. The increase of average particle size after 3 months was only 0.2 μm (average particle size after 24h was 1.9 μm, after 3 months 2.1 μm). Moreover, it was the only emulsion for which dispersity index was unchanged after time (Figure 1). Additionally, this emulsion was characterised by a practically unchanged range of particle sizes after storage period, which suggests stable distribution of particle sizes (Table II, Figure 2).
Slight changes in the average particle size were observed in emulsion I during the whole storage period (Figure 1). Lower amounts of lecithin and carboxymethylcellulose resulted in obtaining the highest average particle size. These results agree with Authors [7] who concluded that not enough amount of emulsifier leads to rapid changes in particles distribution and instability of dispersion systems. The widest range of particle sizes was also noted for emulsion I (Table II).
In case of emulsions III and IV, similar increase of average particle size was observed after whole storage period. Average particle sizes after 3 months of storage were subsequently for emulsion III – 2.7 μm and for emulsion IV – 2.6 μm. It can be concluded, that additional amount of emulsifier had no impact on this parameter. The results agree with Author [20], who stated that an optimum amount of emulsifier stabilised the dispersion system and an additional amount of emulsifier did not improve stability. Thus, it can be concluded that a higher average particle size for emulsion V after 3 months of storage and also greater increase of particle size were dictated by a higher amount of carboxymethylcellulose. Average particle size initial value was the same as emulsion IV (1.7 μm) and close to emulsion III (1.8 μm) (Figure I). In consequence, additional amounts of carboxymethylcellulose and lecithin in this system resulted in greater changes of particle sizes than for the other two systems. Analysing the distribution of particle sizes of emulsions III, IV and V, similar displacements of the curves after storage period were observed (Figure 2). The ranges of particle sizes for those emulsions were congruous after the initial measurement as well as after 3 months of storage (Table II).
Emulsion II was definitely the most unstable system

Table II - Range of particles after 24h and 3 months from manufacturing

		Emulsion					
Particle size [μm] after:		**I**	**II**	**III**	**IV**	**V**	**VI**
24h	min*	0.69	0.69	0.58	0.69	0.69	0.58
	max	11.00	4.62	3.89	3.89	3.89	5.50
3 months	min	0.69	0.82	0.69	0.69	0.82	0.58
	max	15.56	22.00	13.08	11.00	11.00	6.54

* Minimum (min) is the smallest particles diameter determined in a sample.
Maximum (max) is the greatest particles diameter determined in a sample.

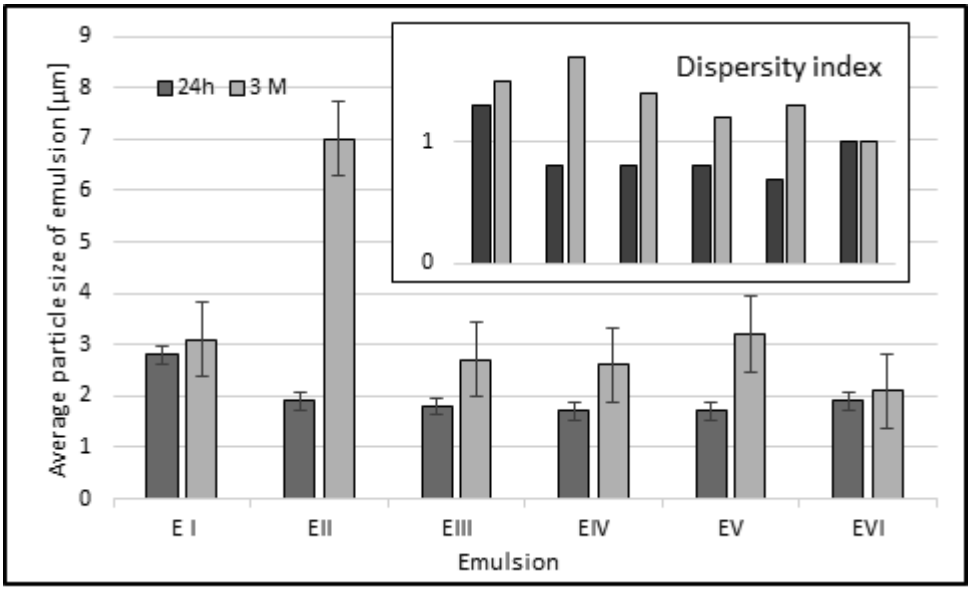

Figure 1 - Volume drop size distribution and value of dispersity index in emulsions I - VI

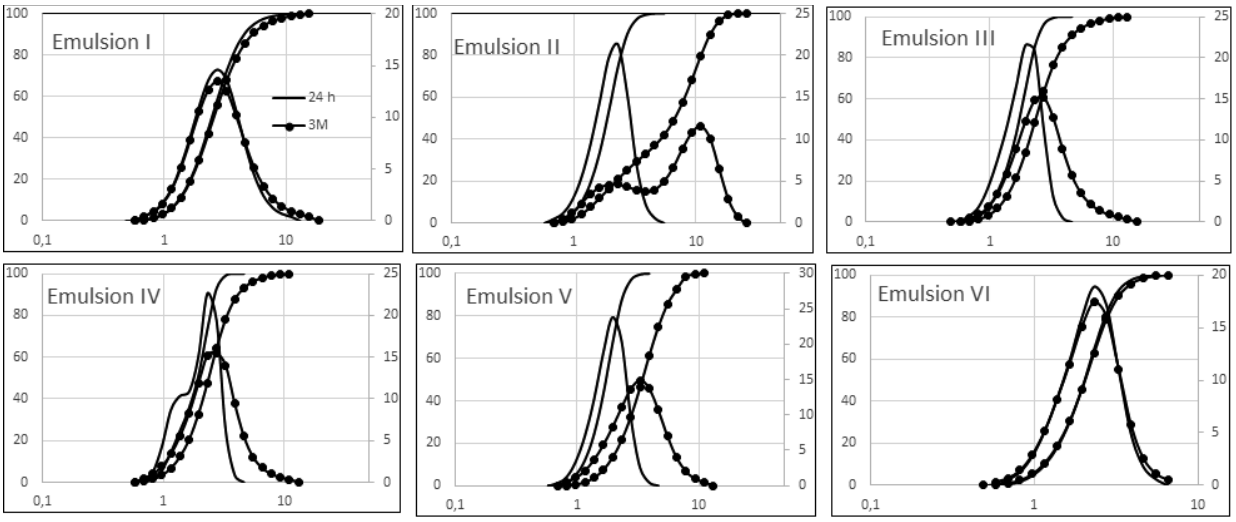

Figure 2 - Distribution of droplets in emulsion I-VI during 3 months period.
Legendary
24h: 24 hours; 3M: 3 months; X axis: diameters of particles [μm]; Y axis (left): cumulated distribution of particles [%]; Y axis (right): the percentage of a given particles diameter [%]

and had the most unfavourable composition. Variable value of average particle size in time was dictated by a too high amount of the thickener in relation to not enough amount of the emulsifier. According to Authors [5], a properly adjusted-ratio of emulsifier - thickener can improve stability; however, it is well known that a greater amount of thickener results in greater particle sizes for the dispersed phase [16].

Final value of the average particle size was 7 μm for emulsion II and was the highest of presented emulsions. This emulsion was characterised by the highest increase of average particle size (5.1 μm), the widest distribution of particle sizes, the highest change in dispersity index (0.9) and the greatest changes in distribution curves (after 3 months of storage the third fraction appeared) (Table II, Figure 1, Figure 2).

The certain range of the ratio emulsifier – thickener (E/T) was observed, in which similar changes of particle sizes were noted. The ratio for emulsions III, IV and V had values: 8.3:1; 12.5:1; 10.0:1 respectively; and, precisely, the increase of particle sizes for these emulsions was similar. In case of emulsion with the lowest E/T ratio 2.5:1 (emulsion II), the greatest difference of average particle sizes after 3 months of storage was observed. For emulsion with high E/T ratio 50.0:1 the differences were insignificant.

Figure 3 presents the skin capacitance before (control point) and after the application relative to time since application of the emulsions (as a mean value of 3 measurements for 15 respondents). The results show a significant increase of skin capacitance after the application of all emulsions, which was maintained for about 120 minutes. The highest level was reached for the skin areas after the application of emulsions II and III. The skin capacitance measurement was observed for the skin and after the application of emulsions I, II, III and IV they were higher than the control point for the whole time of the experiment. The lowest values were noted for emulsions V and VI, which contained the highest amounts of emulsifier.

The analysis of sensory properties shows that all the emulsions were homogenous and penetrated the skin well. Figure 4 shows that emulsions III and V showed the best distribution and skin smoothing effect. However, all the examined emulsions had a low score of greasiness, probably due to the type of emulsions (oil-in-water). The main role of this type of emulsions is the effective absorption, not leaving an occlusive layer on the skin. Emulsion VI, was rated as the lowest by the consumers. Regarding rheological properties of this emulsion, it was too runny to comply with the requirements of an appropriate cosmetic form.

4. CONCLUSIONS

Considering the presented results, it can be concluded that destabilisation changes occurred in emulsion VI. High content of emulsifier led to maintaining a constant average particle size, particle size distribution and dispersion index during the storage period. However, the respondents did not fully accept this emulsion. Emulsions III and IV were evaluated as higher. These systems showed the best distribution and skin smoothing effect. Skin capacitance measurements resulted in the conclusion that greater amount of lecithin had an unfavourable effect on this parameter. This clearly indicates that

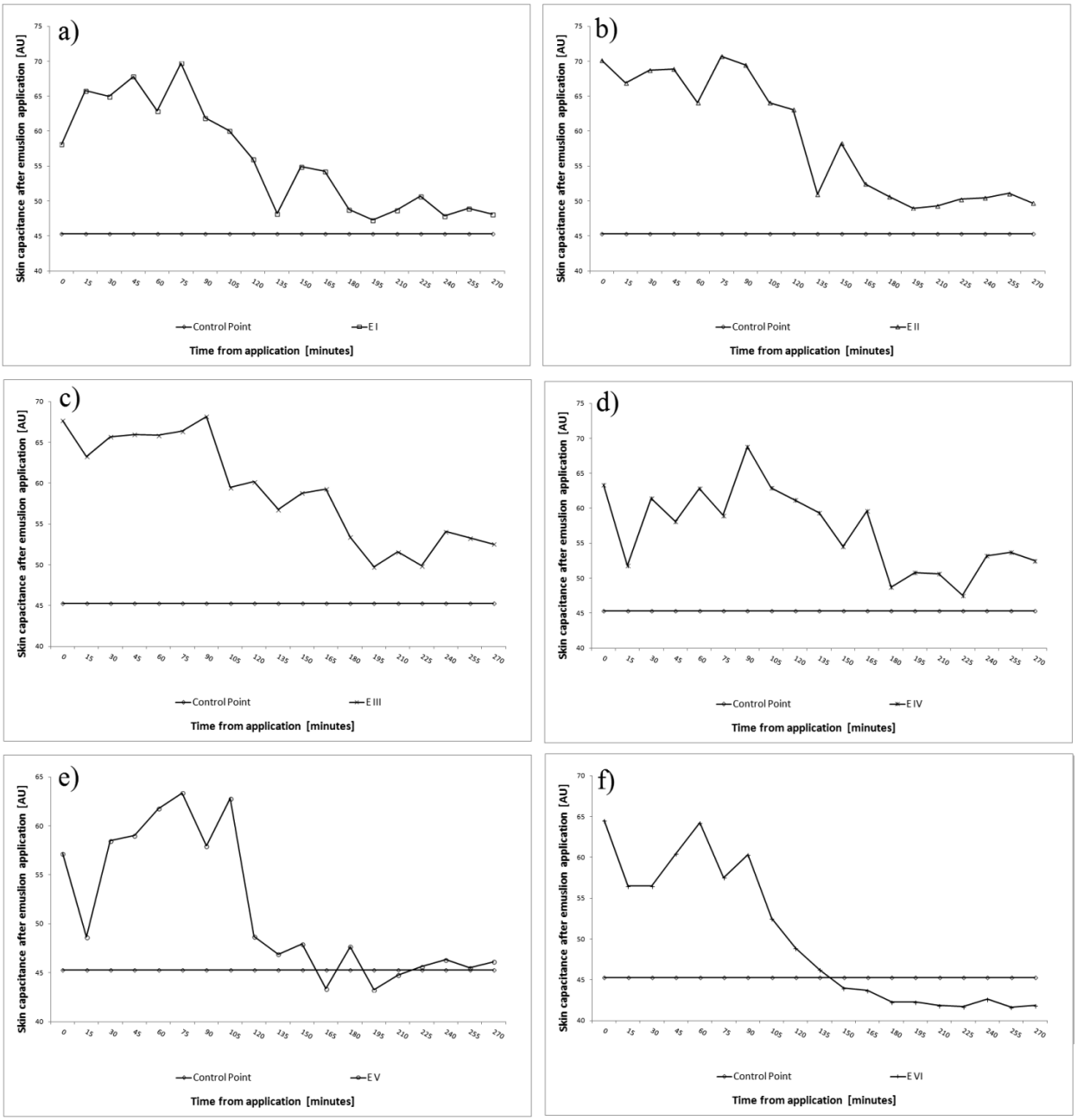

Figure 3 - Skin capacitance before (control point) and after emulsions application a) emulsion I, b) emulsion II, c) emulsion III, d) emulsion IV, e) emulsion V and f) emulsion VI

the addition of the thickener in the range 0.5-0.6 g and reducing the amount of the emulsifier at the same time, can change the emulsion properties and improve the results of the sensory evaluation performed by respondents. The emulsifier – thickener ratio indicated in the study (10:1) appears to be adequate for a proper stability of the emulsion in time and was also high rated by the respondents.

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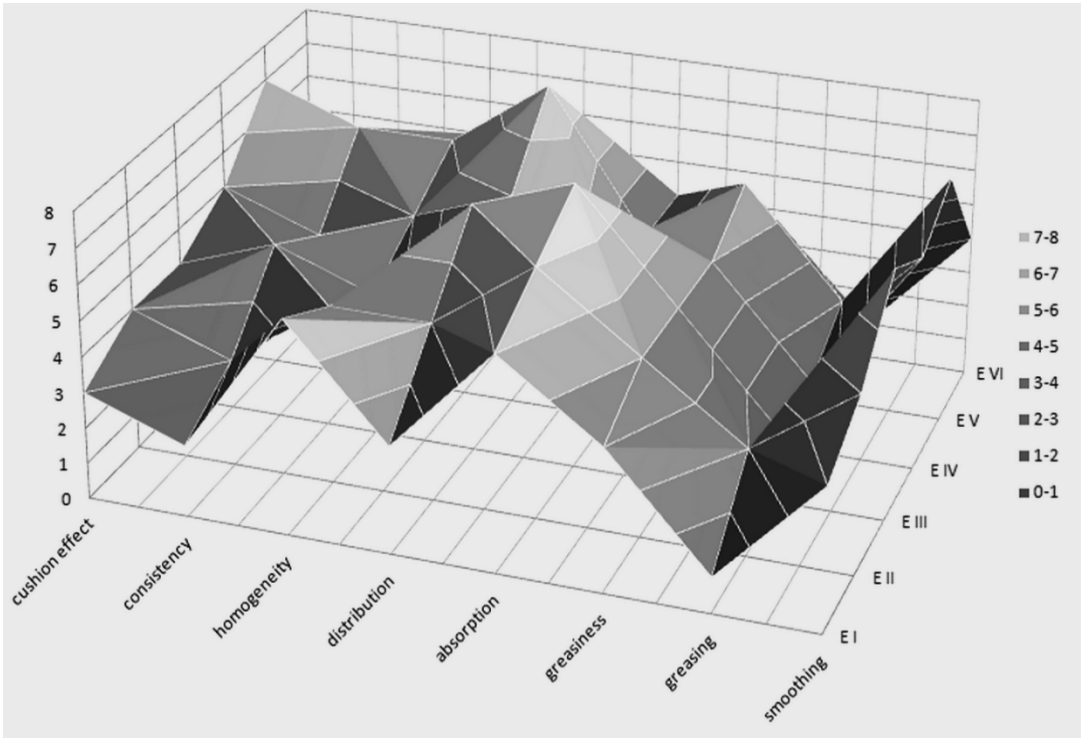

Figure 4 - Mean values of sensory assessment obtained as a result of the survey

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