Assessment of the Sensory and Moisturizing Properties of Emulsions with Hemp Oil

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Abstract: The aims of this work were a sensory analysis, an evaluation of moistening properties and establishment of emulsions stability. Emulsions were prepared with two variable parameters (hemp oil content and time of homogenization). 15 respondents assessed emulsions sensory and skin moisturizing properties. Kleeman's optimizing method was used to designate the most stable emulsion system with the variable parameters. Taking into consideration all properties of the presented emulsions, emulsion V (50g of hemp oil and homogenized for 6 minutes) was found to be the optimum composition with the best moistening and sensory properties. After the application of this emulsion, the skin was highly moistened. Consistency of the emulsions was homogeneous and free from clotting. According to Kleeman's method, emulsion V was also confirmed as the most optimum variant of the emulsion. The work confirmed that hemp oil can be successfully used as a component for cosmetic emulsions.

Keywords: hemp oil; emulsions; sensory evaluation; moisturizing parameters; emulsions stability

1 Introduction

Emulsions constitute an important part in many fields of different industries, including the pharmaceutics, cosmetics, food and petrochemicals [1, 3, 12, 13]. In food industry emulsions can be used in low-calorie products, as a taste masking ingredients or improved sensory characteristics. In pharmaceutical industry – as a drug delivery systems, while in cosmetics – as a creams with encapsulated ingredients. [11, 19].

The prosperity of a cosmetic emulsion formulation depends among others on the stability, an efficiency of the active ingredients but highly important is also the consumer assent, which is definitely caused by the sensory characteristics of the product. Actually, the aims of the studies in cosmetic field are to make products more innovative and successful, which will perfectly fulfill the sensory criteria of the customer. In general, customers choose a cosmetic emulsion for properties of active ingredients or effectiveness, but are mainly convinced by the positive feelings it brings to them, in particular in terms of texture. Therefore, sensory analysis dominates in the quality control of emulsions. This tool has rapidly been expanded and widely used to characterize and quantify texture properties of cosmetic dispersions [6, 14].

According to [15] sensory analysis is the evaluation of a product through the assessment of the properties perceptible by the following senses as taste, color, touch, texture, odor and noise, which results in receiving of the organoleptic profile of various products, for example cosmetics. Sensory evaluation interprets, measures and assess properties of a formulation, after stimulating users in connection to their main senses. So far, there is no instrumental methods, capable to estimate the human impressions, sensory analysis is a very effective method to receive data about a product in relation to consumer acceptance [4].

Vegetable oils, which contain unsaturated fatty acids, are frequently use in cosmetic dispersions to encourage customers and raise the sensory feelings. Hemp oil can be one of such oils and can also improve innovativeness of the cosmetic products. The attention directed to hemp for skin care is affected by great amount of oil in seeds of this plant (25-35%), but also a beneficial profile of unsaturated fatty acids consist of 50-60% linoleic acid (C18:2ω6), 20-25% α-linolenic acid (C18:3ω3) and a substantial portion of γ-linolenic acid (C18:3ω6). These three fatty acids constitute a structure of the cell membranes phospholipids. Moreover they have an effect on the immunology of the cells as well as on the several cell membrane functions such as the hormones activity, fluidity, or the transport of electrolytes [18, 20]. Linoleic and linolenic acids improve the skin structure and have a beneficial influence on dry and rough skin [20].

The aim of this work was to evaluate a sensory analysis and skin moisturizing properties of emulsions with hemp oil. To detect the most stable variant of emulsion, the optimization was performed using the software based on Kleeman's method [8]. The software indicated the variant with appropriate stability after only one measurement. It allows for reducing the time of the experiments and helps correctly design a stable dispersion.

2 Experimental

2.1 Materials

Emulsions were manufactured using the following components: distilled water, carboxymethylcellulose (Barentz Hoofddorp, Netherlands), cold pressed, unrefined (Gracefruit Ltd Stirlingshire, United Kingdom) (INCI: Cannabis Sativa Seed Oil) hemp oil containing the following fatty acids (FA): palmitic acid C16:0 – 5.7%, palmitoleic acid C16:1 – 0.15%, stearic acid C18:0 – 2.5%, oleic acid C18:1 9c – 11.4%, linoleic acid C18:2 9c12c – 55.4%, alpha linolenic acid C18:3 9c12c15c – 17.2%, gamma linolenic acid C18:3 6c9c12c – 3.6%, eicosenoic acid C20:0 – 0.6%, eicosenoic acid C20:1 – 0.4%, docosanoic acid C22:0 – 0.3%, tetracosanoic acid C24:0 – 0.2%, (Oleofarm company), sunflower lecithin (Lasenor, Emul, S.L. Barcelona, Spain) sodium benzoate (Orff Food Eastern Europe, Marki/Warsaw, Poland), aloe vera (FLP), Scottsdale, Arizona, United States, citric acid (Jungbunzlauer Basel, Switzerland).

2.2 Preparation of Emulsions

Lecithin (5.2%) was introduced to the oil phase (hemp oil). Carboxymethylcellulose (0.6%) was dispersed in distilled water, and then, aloe vera (0.2%) was added. Oil and aqueous phases were heated to 50–55°C in a water bath separately. Homogenization of the both phases was achieved by means of a high shear mixer at an equal speed of 36288 RCF for a suitable time (given in Table 1). Afterwards, the emulsions were cooled to room temperature, and sodium benzoate (0.25%) was added. Next pH was adjusted to 5.5 with citric acid using a pH-meter (Mettler Toledo) equipped with a calomel combined pH electrode. The content of changing components was presented in Table 1.

Table 1

Changing components/parameter of emulsions given according Kleeman's method

	Component [%w/w]/ Parameter	**EMULSION I**	**II**	**III**	**IV**	**V**	**VI**
Changing components /parameter	Water	83.75	43.75	63.75	63.75	43.75	83.75
	Hemp Oil	10	50	30	30	50	10
	Mixing time [min]	1.5	1.5	3.0	4.5	6.0	6.0

2.3 Methods

2.3.1 Determination of Skin Capacitance

The determinations of skin capacitance were performed using a CM825 Corneometer (Courage+Khazaka Electronic). The principle of this method concerns the difference between capacitance of the dielectric constant of water and other substances. Each change of the dielectric constant subsequent to the modification of skin hydration results in an impaired calculated capacitance of a capacitor. The skin hydration degree values are in the range 0-130 arbitrary units (AU) [5].

In order to eliminate the influence of external conditions on the results, the measurements were performed under standard conditions (T° = 20-22°C, humidity 40-60%), away from direct sunlight. Women students of cosmetology or chemical technology (specialty: biotechnology and technology of cosmetics and household products) from the University of Technology and Humanities in Radom, Poland participated in those measurements.

The test included measurements carried out immediately prior to the application and after 15 min, 30 min, 45 min, 60 min, 90 min and 120 min after application of emulsion (approximately 0.01 g) on the designated forearm skin fragments. To have valid results, each measurement was taken three times. The region of the skin was wiped with a clean cotton cosmetic swab before and after the measurement. Variation in the time of the skin capacitance, as a difference between the values before and after application (as a mean value of 3 measurements for 15 respondents), were calculated by the following formula:

$$\boldsymbol{C = C_t - C_0} \qquad (1)$$

where C_0 is a skin capacitance prior to the application and C_t is a skin capacitance over time t.

2.3.2 Sensory Determination

Sensory evaluation is based on the measurement and assessment of the product properties and consumer feelings by the senses (smell, taste, touch, sight).

Testing of the emulsion was performed by a group of respondents (15 females). The respondents were trained and specifically instructed on the methodology. A 5-point scoring scale was introduced, with 5 the maximum and 1 the minimum score. Details of the sensory assessments are presented in Table 2. The analysis was carried out at room temperature of 20°C (±2°C) and constant air humidity of 45% (±5%). Correctness of the testing was supervised by a researcher from the University of Technology and Humanities in Radom.

Table 2

Guidelines for sensory analysis of the cosmetic emulsions tested (study based on literature (Płocica et al. 2012; Płocica and Tal-Figiel 2009) and own experience (Kowalska et al. 2015)

Feature	Description of test procedure	Score (1–5)
C	Position hand at an angle of 60° and place 5 cm3 of the test substance there. Proceed to analyze its consistency by assessing the ability to keep the cosmetic adhering to the hand.	5. Cosmetic is easy to apply, not flowing 4. Easy to apply yet flowing can be observed 3. Cosmetic is hard to apply 2. Too thick to apply to the hand 1. Impossible to apply
H	Spread the substance on your hand and assess smoothness of its layer, presence of clots or air bubbles.	5. Completely homogeneous, no clots or air bubbles, forms a smooth layer on the skin 4. Homogeneous, no clots and few air bubbles, forms an uneven layer 3. Observable and palpable clots and air bubbles in the substance and on the skin when applied 2. Heterogeneous 1. Formulation components are not dissolved.
CE	Scoop 0.5 cm3 of the emulsion and rub between the thumb and index finger.	5. Imperceptible substance 4. Weakly perceptible substance 3. Somewhat perceptible substance 2. More perceptible substance 1. Highly perceptible substance
D	Spread 0.5 cm3 of the preparation on the forearm skin and observe its resistance to spreading.	5. No resistance to spreading 4. Little resistance to spreading 3. Incomplete cover, good spreading 2. Difficult to spread 1. Impossible to spread
SM	Apply 0.5 cm3 of the emulsion on the cleaned forearm skin and after an hour appraise the skin's smoothness in reference to a standard to which the substance has not been applied.	5. Very smooth, soft skin surface 4. Smoother and softer skin surface than of the reference standard 3. The skin surface is as smooth as that of the reference standard 2. Rough skin 1. Very rough skin
ST	Apply and spread the emulsion on the cleaned forearm skin, then press the other hand against this skin section and assess viscosity.	5. No palpable skin viscosity 4. Low skin viscosity 3. Palpable skin viscosity 2. Increased skin viscosity 1. High skin viscosity

G	Apply 0.5 cm3 of the substance on the cleaned forearm skin and assess formation of a greasy film	5. No sense of grease or film formation on the skin after application 4. Weak sense of greasiness, no film on the skin 3. Thin, greasy film on the skin after application 2. Greasy film on the skin directly on application 1. A compact, greasy film after application
A	Apply the substance on cleaned skin and assess the time of its absorption.	5. Very good absorption below 30 s 4. Good absorption from 30 s to 1 min 3. Average absorption from 1 to 3 min 2. Poor absorption from 3 to 5 min 1. Very poor absorption for more than 5 min

Legend:
- C - consistency (density and cohesion of the tested cosmetic),
- H - homogeneity (behavior of the preparation when applied to the skin – absence of clots or air bubbles)
- CE - cushion effect (palpability of the substance when rubbed between fingers),
- D - distribution (facility of spreading on the skin surface),
- SM - smoothing (smoothing effect when applied to the skin),
- ST - viscosity (degree of palpable viscosity left on the skin),
- G - greasiness (a fat film remaining on the skin),
- A - absorption (rate of absorption by the skin).

2.3.3 Determination of Viscosity

The viscosity of the emulsions were measured 24 h after manufacturing, at the speed of 10 rpm with spindle no. RV3, using a Brookfield Rheometer DV-I+. The measurements were performed at 25°C.

2.3.4 Determination of Mean Droplet Size and Droplet Size Distribution

The average droplet size and distribution were determined after 24 h using a Microtrac Particle Size Analyzer (Leeds & Northrup, Philadelphia, USA). Each measurement was repeated three times and given as the average value.

2.3.5 Dispersity Index

Dispersity index was calculated on the basis of laser diffraction droplet size measurements according to the formula:

$$k = (A - B)/C \qquad (2)$$

where A, B and C are the biggest sizes of oil droplets for 90%, 10% and 50% of all particles, respectively.

2.3.6 Optimization of Parameters of Emulsion Stability

To obtain a stable emulsion optimization of parameters was carried out, using the Kateskór software, which is based on the Kleeman's method. The software was created for the Department of Technology Footwear and Tanning at the Faculty of Materials Science, Technology and Design (University of Technology and Humanities in Radom). The analysis was based on the measurement of viscosity and mean droplet size (input parameters) and as well our previous studies [9, 10] (Table 3). According to above mentioned factors a emulsion model was adopted, described by the following parameters for optimization purposes: dispersity index – 1.2, viscosity – 2800 mPa s, number of fractions – 1, average particle size – 5 µm, maximum particle size – 13 µm, minimum particle size – 0.5 µm.

Table 3
Values of viscosity and average particle size of emulsions measured after 24h from manufacturing

Emulsion	I	II	III	IV	V	VI
Average particle size [µm]	3.5	10.8	3.8	3.6	5.8	3.7
Viscosity [mPa*s]	200.0	4100.0	900.0	980.0	4800.0	110.0

3 Results

The assessment of the skin hydration degree carried out using a corneometer showed that all respondents had very dry skin [7]. The measurement of skin capacitance before application of each emulsion was in the range 25.2 – 28.6 AU. Figure 1 presents the difference between the skin capacitance (before and after application) related to time from application of the emulsions (as a mean value of 3 measurements for 15 respondents).

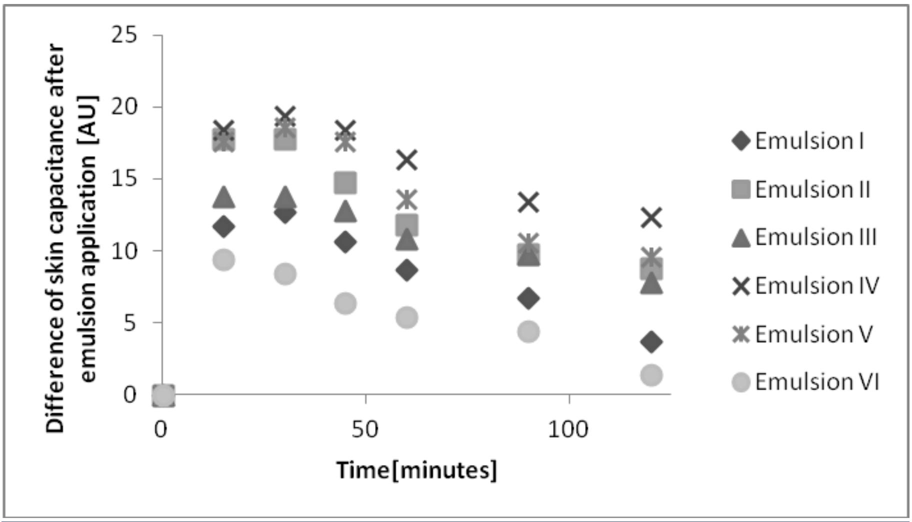

Figure 1
Variation in time of the skin capacitance after emulsions application as a difference between the values before and after application

After 15 minutes of application of each emulsion the corneometry values, compared to the previous measurements, significantly increased. The highest values were obtained after application of emulsions II, III, IV and V. While the lowest increase was recorded after application of emulsions I and VI. Thus, it can be assumed that the higher content of hemp oil in the emulsion caused higher degree of skin hydration. Obtained results clearly indicate a favorable effect of the high amount of hemp oil on the moisturizing properties of the examined emulsions. The corneometer showed a maximum increase of skin capacitance after 15 minutes to 50 minutes from the application of the emulsions. The highest increase of skin humidification was reported after application of emulsion V, which contained the highest amount of hemp oil and was homogenized the longest time. The proper homogenization (time and speed of homogenization) is the factor, which contributes to form an appropriate particle size of the emulsion and thus better penetration of the skin [2]. After 50 minutes from emulsions application the corneometry values decreased. In contrast, the Authors [5] obtained opposite results, because the maximum hydration was observed after one hour from application.

Sensory analysis provides the organoleptic evaluation of, among others, cosmetic products and is very helpful in providing the knowledge of how they are considered by the customers [15]. According to the results of the sensory analysis, emulsions II – V were evaluated the best, taking account the average value from all rated parameters. Generally, the respondents concluded, that emulsions had a regular, homogeneous structure, good rate of absorption, smoothing effect proper value of greasiness.

Emulsions IV and V obtained the highest average score of all assessed parameters. However, the respondents the least highly valued the following parameters: cushion effect, smoothing, smoothing, greasiness and absorption (Table 4). Remaining parameters received 5 points (Figure 2). According to respondents, emulsions I and VI were the least satisfying (average scores 3.8 and 3.6 respectively). Cushion effect, consistency and distribution were rated the lowest (Table 4, Figure 2).

Generally, for the presented emulsions, sensory analysis confirmed significance of applying larger amount of oil to the emulsion system, as well as the importance of homogenization time for this type of formulations. Emulsions V and II contained an equal amount of oil, although homogenization time of emulsion II was much shorter (1.5 min), which affected to the fact, that this emulsion (II) did not fully meet with the respondents' expectations. Emulsion (IV), which contained smaller amount of oil (30 g), but homogenized for a longer time (4.5 min) was assessed better. It was observed, that particle size depends on homogenization time and oil content, which affects the stability and quality of emulsion systems [9].

Table 4

Mean values (± sd) of sensory evaluation received as a result of the survey

Parameter	**Emulsion**					
	I	**II**	**III**	**IV**	**V**	**VI**
CE*	3 ± 0.6	4 ± 0.7	4 ± 0.8	4 ± 0.6	5 ± 0.5	3 ± 0.9
H	5 ± 0.3	5 ± 0.2	5 ± 0.3	5 ± 0.2	5 ± 0.4	5 ± 0.6
C	3 ± 1.0	5 ± 0.5	4 ± 0.4	5 ± 0.4	5 ± 0.4	3 ± 0.9
D	3 ± 0.7	4 ± 0.7	4 ± 0.8	5 ± 0.3	5 ± 0.3	3 ± 0.4
ST	4 ± 0.3	5 ± 0.6	4 ± 0.2	4 ± 0.4	5 ± 0.2	4 ± 0.3
G	4 ± 0.9	4 ± 0.8	4 ± 0.4	5 ± 0.2	4 ± 0.7	4 ± 0.8
A	4 ± 0.8	4 ± 0.5	4 ± 0.7	4 ± 0.7	4 ± 0.7	3 ± 0.5
SM	4 ± 1.0	4 ± 0.5	4 ± 0.9	4 ± 0.9	5 ± 0.3	4 ± 0.7
Average	**3.8** ± 0.7	**4.4** ± 0.5	**4.1** ± 0.4	**4.5** ± 0.5	**4.8** ± 0.5	**3.6** ± 0.7

*See legend in Table II

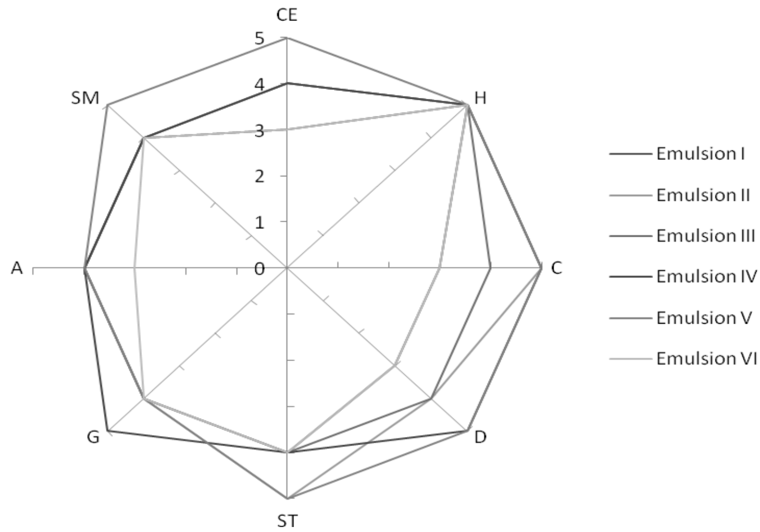

Figure 2
Sensory profile of emulsions (average values)

The optimization of the emulsions' stability demonstrated that the emulsions with two variables (the content of oil and time of homogenization) the optimum content of hemp oil should be in the range 30-50 g, while the optimum homogenization time should be in the range of 150 s to 360 s. Thus, emulsions indicated as optimal by the software were IV (contained 30 g of oil and homogenized for 4.5 minutes) and also V (contained 50 g of oil and homogenized for 6 minutes). Figure 3 presents a detailed study of the effect of the this two variables on the parameters of the emulsions.

Generally, it was observed, that increase of oil amount caused an increase of examined parameters' values. The exception was only dispersity index. In contrast, the increase of homogenization time leaded to increase of dispersity index and maximum particle size. However, the minimum and average particle size and viscosity remained practically unchanged (Fig. 3).

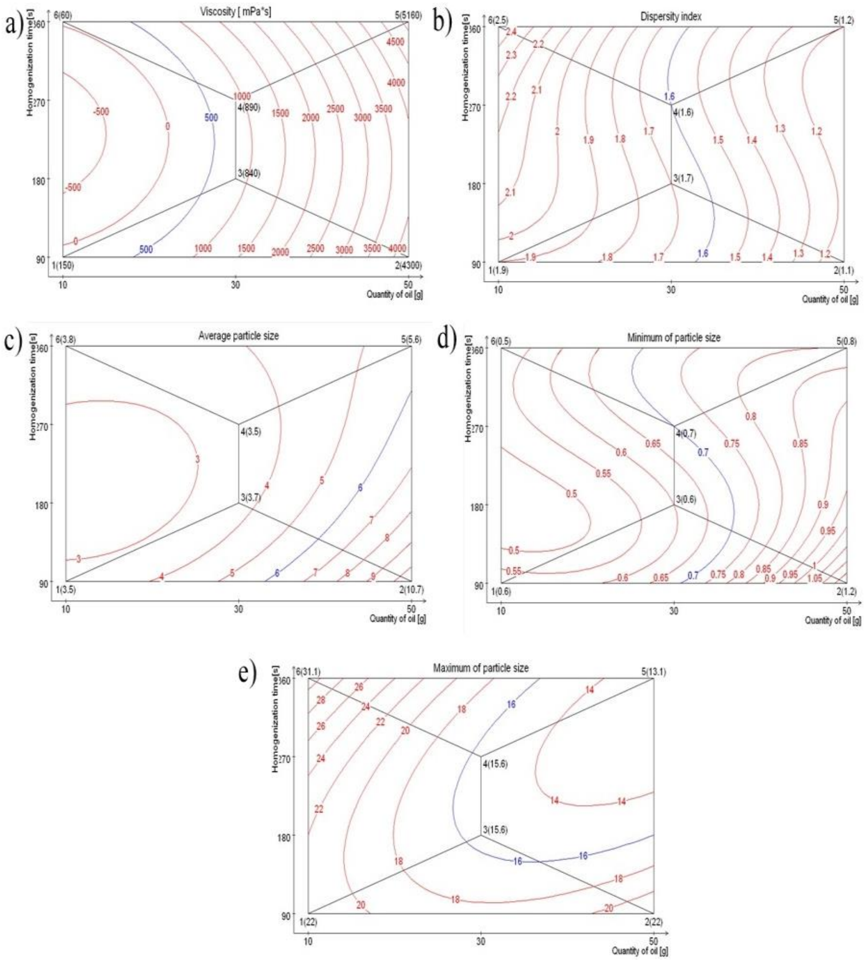

Figure 3
Effect of input parameters on: a) viscosity b) dispersity index c) average particle size d) minimum particle size e) maximum particle size

Conclusions

Hydration and improvements of the skin condition by the manufactured emulsions have been confirmed. Emulsions containing greater concentrations of hemp oil (50 g) and homogenized longer time (6.0 min) displayed maximum skin hydration. Consistency of the emulsion was homogeneous, smooth without clotting or air bubbles and was judged acceptable by the respondents. Additionally, all preparations retained a pleasant and had a delicate green color which makes such emulsions familiar for respondents.

The results corroborated the authors' hypothesis that hemp oil can be applied as an component of cosmetic emulsions with a low hydration.

The result obtained by computer analysis indicated that the most stable emulsion system is variant V (50 g of hemp oil, homogenized 6.0 min).

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