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Comparison of sensory and rheological properties of green cosmetic creams prepared on different natural, ECOCERT and BDIH certificated self-emulsifying bases

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ARTICLE INFO	ABSTRACT
Received 21 January 2021 Accepted: 07 December 2021	The objective of this paper was to evaluate and compare the rheological and sensory properties of six natural cosmetic creams that use ECOCERT certificated emulsifying
<i>Keywords:</i> cream, emulsifier, COSMOS, rheology, sensory analysis.	bases: Beautyderm, Dub Base Expert, Simulgreen [™] 18-2, Olivem 1000, Montanov L and Emulgin Succro against two synthetics: Granthix APP and Lanette W. The use of emulsifying bases in formulas helps to stabilize the product and improve its rheological and sensory attributes. In this study, the physical and chemical properties of several prepared cosmetic creams were determined. Apparent viscosity and thixotropy were evaluated by viscosimeter, while spreadability was ascertained by extensometer. Moreover, a group of 10 trained members evaluated the sensory properties of the formulas. The obtained formulas based upon natural emulsifiers are characterized by very good physical and chemical properties, high stabilization and good usage quality. The results were confirmed by sensory analysis. The cosmetic creams made with synthetic emulsifying bases were rated the worst by the study participants, which correlated with the physical tests.

INTRODUCTION

Production of formulates for body care are one of the fastest developments in the cosmeceutical industry, and it has made great progress in the development and incorporation of new and effective actives in their products. The growing public awareness of what is friendly and beneficial for their skin ensures the popularity of natural and ecological cosmetics.

In order for a product to be considered natural or ecological, it must satisfy certain stringent requirements. Today's cosmetic industry employs many advanced plant components: stable vitamins such as hydrogenated retinol and tetrahexyldecylascorbate [1], together with sophisticated technological developments such as transferosomes, liquid crystals, ethosomes and niosomy [2]. Cosmetics manufacturers try to entice potential customers not only with modern production technology, but also with the composition of cosmetics, which according to customers' expectations, should be based on natural, plant ingredients. However, currently

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Natural cosmetics are associated by the client with something better and healthier for the skin. However, there are no legal requirements that regulate the definition of natural, ecological and organic cosmetics. In the current Regulation of the European Parliament and the Council of Europe 1223/2009, there is only a definition of a cosmetic product, but there is no definition of what a natural cosmetic is and of what requirements it must meet (3). In order to reduce the false naming of cosmetics as eco-cosmetics, representatives of five World and European certification founders; EcoCert Greenlife SAS (France), BDIH (Germany), Soil Association (the UK), Cosmebio (France) and ICEA (Italy) have unified the requirements for labeling products as natural cosmetics. These requirements have been compiled into a standard, which is called the COSMetic Organic Standard - COSMOS. In natural cosmetics, in accordance with the requirements of COSMOS standards, the use of synthetic ingredients may not exceed 5% (weight/weight). Furthermore, the use of GMOs (genetically modified organisms),

© 2021 Author(s). This is an open access article distributed under the Creative Commons Attribution-NonComercial-No Derivs licence (http://creativecommons.org/licenses/by-nc-nd/3.0/) nanoparticles, plant ingredients extracted with petrochemical solvents and the use of substances such as EDTA (ethylenediaminetetraacetic acid), chlorinated based products, products based on ammoniac and phosphates is prohibited.

The Cosmetic Organic Standard defines precisely when a cosmetic can be considered natural and when it can be considered organic. According to the COSMOS standard, in 'organic cosmetics', the content of certified raw materials may not be less than 10%, in the case of cosmetics labeled only as 'natural content of certified ingredients', this may not be less than 5% [4,5]. In addition, COSMOS strictly defines the requirements to be met by the water used in the preparation of cosmetics, as well as the processes that are allowed in the preparation of plant raw material, and also determines the type of final packaging of cosmetics [5].

Cosmetic creams are one of the most frequently produced cosmetic formulations. Creams are semi-solid forms with multiphase surface dedicated for use on the outer skin. Physically, they are formed in the processing of thermodynamic, unstable dispersions of immiscible liquids that are stabilized in time owing to a variety of emulsifiers, thickeners and texture components [6]. The use of these substances not only enhances the stability of the product in time, but also allows modifying its sensory and rheological properties.

Cosmetic base cream (blank cream) has a significant influence on the bioavailability of cosmetic agents such as vitamins and plants extract. The degree of hydration of the stratum corneum, as well as its proper lubrication, affect the penetration of active substance into the skin [7].

Emulsifiers play the main role in obtaining stability within cream formulations. Their role is to reduce interphase tension and create uniform compositions [8]. Until recent times, cosmetics producers mainly used single synthetic emulsifiers (ionic or non-ionic) [7] such as Polisorbate 80 [9,10], Span 83 [10], Methyl glucose sesquistearate [11] and Poliacrylate natrium [12]. Applying single emulsifiers to stabilize emulsions usually entails the use of additive emulsifiers, i.e. co-emulsifiers – among others, fatty acids and monoglicerides [7]. In this way, it is possible to adjust appropriate levels of HLB (hydrophilic-lipofilic balance), which will help to obtain a desired type of emulsion [13].

The process of cosmetics production nowadays is largely enhanced with self-emulsifying bases (SEBs). These are ready compositions made of well-matched proportions of emulsifiers, co-emulsifiers and thickeners. Cosmetics prepared with SEBs are characterized by better stability, more beneficial rheological properties and better sensation. Ready-made SEBs have clearly defined HLBs, which definitely facilitates the procedure of creating desired cosmetic formulations. Standardized SEBs also allow for shorter time of component weighing and thus lowers production and storage costs [14].

Based on the overview of available references, one can conclude that there is very little research into the sensory and physical and chemical properties of creams made with natural and synthetic SEBs [15-20]. Krynicka *et al.* [17] researched the influence of moisturizing creams made with the use of two SEBs: TegoCare 150 (glyceryl stearate, steareth 25, ceteth 20, stearyl alcohol) and Wax Lanette W (sodium lauryl sulphate, cetostearyl alcohol) on the level of skin moisture. In comparative research, TegoCare 150 demonstrated higher value as a substrate in the production of moisturizing creams. Manson D. *et al.* [21] used SEBs with formulations of cetostearyl alcohol and sorbitan monostearate to create creams of the o/w emulsion type. The final formulation had pseudoplatic behavior with thixotropy properties. The influence of SEB on the stability of creams with sun protection filters was also evaluated in the paper by Gaspar L. and Campas M. [22]. The ionic phosphate base with the cetostearyl alcohol, diacyl phosphate, ceteth 10 – phosphate composition showed better physical stability and higher sun protection factor than did non-ionic base cetearyl alcohol and ceteareth 20.

In recent times, there has been a lot of interest in natural emulsifiers obtained from olive oil (OLIVEM) [23-28]. Olivem 1000 is a SEB with the formulation: cetearyl olivate and sorbinian olivate. Perfectly matched emulsifiers with the total of HLB 12 in base Olivem 1000 allow the stabilization of o/w emulsions without any other additives. Together with emulsifying properties, Olivem has many other qualities that are crucial in skin care products: it protects the skin from excessive loss of moisture, helps soften the skin and gives perfect spreadability [24,25].

Simulgreen[™] 18-2 (INCI: Hydroxystearyl alcohol and hydroxystearyl glucoside) is a new oil in water emulsifying base. It is made using ingredients of plant origin and in accordance with a process meeting green chemical principles. According to Roso A. *et al.* [26], creams which contain Simulgreen[™] 18-2 as an emulsifier have good sensory analysis without blanching of the skin. The soaping effect is quite often found in cosmetics based on natural emulsifiers. A growing interest in natural cosmetics and the increasingly popular use of self-emulsifying bases in the cosmetic industry have forced the need to analyze and compare the physicochemical and sensory properties of cosmetic formulations.

The objective of this paper was to evaluate and compare rheological and sensory properties of natural, green cosmetic creams using six different natural and ECOCERT certificated self-emulsifying bases against that of two synthetic bases.

MATERIAL AND METHODS

Cosmetic cream composition and sample preparation

Eight different samples of cream type oil in water (o/w) were created. Their compositions are presented in Table 1 and differ in the type of self-emulsifying base used (Table 2). The samples were prepared by warming to 80°C both phases and then adding the oil phase to the water phase. When the temperatures of the creams fell below 40°C, heat-sensitive components: squalene, sweet almond oil and vitamin E, as well as the preservative: GFecosafe (dehydroacetic acid and benzyl alcohol) were added and blended together by the use of stirrer IKA Microstar 7.5, at 700 rpm.

For the sake of better uniformity, the creams were subsequently homogenised for 2 minutes with the use of a Unquator e/s Gako homogenizer. The creams were then stored in glass containers at 4°C. The products obtained were given sensory analysis and physicochemical tests complying with current researches [6,29-31].

Table 1. Quantitative composition of the creams

INCI	Origin Concern (Supplie		Content %				
Oil phase							
Self emulsifying base*	Self emulsifying base*						
Cetearyl alcohol	derived from sustainable palm and coconut oil	LotionCrafter	3.0				
Shea butter crudum	Shea butter	Local supplier	1.5				
Squalene	vegetable origin from Olive oil (EcoCert)	LotionCrafter	1.0				
Caprylic/Capric Triglyceride	vegetable origin from coconut oil (EcoCert)	e-naturalne	1.5				
Sweet Almond Oil	Prunus Amygdalarus dulcis oil Cortex (gift)		8.0				
	Water phas	e					
Sodium pyroglutamate		Cornelius	1.0				
Glycerin	vegetable origin	Local supplier	4.0				
Sodium poliacrylate		Basf, (gift)	0.8				
Sodium Phytate	vegetable origin derived from rice bran	Lotion Crafter	0.1				
Aqua	Aqua destylata		72.1				
Tocopherol	vegetable orgin	e-naturalne	1.0				
Dehydroacetic acid, benzyl alcohol		Acros	1.0				

*The type and composition of used self-emulsifying base is given in Table 2

Table 2.	The type and	composition	of used self -	- emulsifying base
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Trade name and certifying organization		INCI	Concern
1	Beautyderm EcoCert	glyceryl stearate, cetearyl alcohol, stearic acid, sodium lauroyl glutamate	Enzym
2	Dub Base Expert (DBE) EcoCert	glyceryl stearate citrate, sucrose stearate, polyglyceryl-4 cocoate, cetyl alcohol, sodium ricinoleate	Azelis
3	Montanov L EcoCert and BDIH	C 14-22, C 12-20 alkyl glucoside	Seppic (gift)
4	Emulgin Succro EcoCert	Sucrose Polystearate, Cetyl Palmitate	BASF (gift)
5	Simulgreen™ 18-2 EcoCert	Hydroxystearyl alcohol and hydroxystearyl glucoside	Seppic (gift)
6	Olivem 1000 EcoCert	cetearyl olivate, sorbitan olivate	Local supplier
7	Lanette W Synthetic	Cetearyl alcohol; Sodium lauryl sulfate	BASF
8	Granthix APP (Granthix) Synthetic	isohexadecane, polyacyloyldimethyl taurate, polysorbate-80	IMCD (gift)

Sensory analysis of the formulations

The formulations obtained in the tests were analysed by sensory perception: smell, sight, and touch according to Płocica *et al.* [32]. Sensory analysis was carried out in the Department of Applied Pharmacy, Medical University of Lublin. The research was approved by the Bioethics Committee nr KE-0254/140/2017. The panel was formed by 10 experienced members (age range between 23-68). Eight sensory



Figure 1. Results sensory analysis of Beautyderm, DBE, Granthix, Lanette W

attributes were chosen: appearance, fragrance, cushions effect, adhesion to the finger, spreadability on the back of hand, stickiness of the cream, residual feeling on the skin after absorption, and absorption to the skin. The panellists evaluated the creams on 5-point scale. The experimental methods of the chosen attributes and the obtained effects of the creams are shown in Table 3. The average results of the analysis are given in Figure 1 and Figure 2.

Table 3. Experimental methods of the chosen attributes and effects of the prepared creams with interpretation by points-based assessment

Attribute and definition	Method	Interpretation
Cushion effect Cream is felt between a thumb and a forefinger while rubbing against each other	Apply the product with the forefinger then rub it against the thumb. Determine the perceptible amount of cream	5 p - not felt at all 4 p - hardly felt 3 p - not felt very well 2 p - felt 1 p - felt strongly
Adhesion	Apply a small amount of	5 p – easy to apply and keeps its size well
The cream can be applied on a finger	cream on a finger and find its consistency and how it keeps its shape	4 p - fairly easy to apply and keeps its shape quite well 3 p - difficult to apply and does not keep its shape 2 p - difficult to apply, it is too thin or too thick 1 p - product cannot be applied
Spreadability The cream is easily spread	Apply the cream on an inner side of arm and check the resistance during application	p – easy to spread, skin does not react with resistance 4 p – easy to spread, skin reacts with minimum resistance 3 p – fairly difficult to spread, skin reacts with resistance and bleaching effect 2 p – difficult to spread, it is too thin or too thick 1 p – difficult to spread, skin reacts with resistance during application or the product drops off the skin
Stickiness after application, the cream leaves a sticky layer on the skin	Apply the cream on an inner side of arm and check its stickiness with a hand	5 p - does not leave sticky layer $4 \text{ p} - \text{leaves sticky layer, it vanishes very quickly 3 \text{ p} - \text{leaves sticky layer, it vanishes after}5 minutes2 p - leaves sticky layer, which isunpleasant1 p - leaves sticky layer, which isunacceptable$
Residual after application, cream can leave greasy layer	Determine if the cream leaves a greasy layer	5 p - leaves a greasy layer which keeps up to 30 min 4 p - leaves a greasy layer but it vanishes with time 3 p - leaves a greasy layer but it vanishes quickly 2 p - leaves no greasy layer 1 p - product does not leave any layer
Absorption Duration of cream absorption into the skin	Monitor the time necessary for the product to be absorbed into the skin	5 p - absorbs very well 4 p - absorbs well 3 p - absorbs fairly well, there is a little bleaching, but it vanishes quickly 2 p -absorbs badly, there is a bleaching effect on application, absorption comes after a few minutes 1 p - absorbs badly or does not absorb at all



Figure 2. Results sensory analysis of Montanov L, Simulgreen, Olivem 1000, Emulgin

Physical analysis

Microscopic analysis

The analysis was made with optical microscope Leica ICC 50HD, equipped with $40 \times$ optical zoom and the computer program LAS V45. In order to determine the proper type of emulsion, each sample of the cream was stained by methylene blue as indicator and then observed. The size of 300 randomly chosen drops of the dispersion phase was measured and the results with calculated standard deviation are shown in Table 4.

Determination of density

Density was marked with digital densitometer DMA 500 (Anton Paar, Austria). The n=3 results are shown in Table 4.

Determination of pH

This measurement was undertaken by employing pH meter model CP-411 (Elmetron, Poland), equipped with an indicator electrode, at a temperature of $20^{\circ}C\pm2^{\circ}C$. The sample was introduced into a 100 ml beaker while taking the necessary precautions to fill the container evenly and to eliminate all air bubbles. The measurement was repeated six times. The results of n=3 are shown in Table 4.

Table 4. Summary statement of results: the droplet size of the oil phase, pH, density and spreadability of the preparation calculated as an area under the curves of spreadability (AUC)

Base	Droplet size (µm)		рН		Density (g cm ⁻³)		Spreadability AUC (g*cm ²)103	
		±SD		±SD		±SD		±SD
Beautyderm	10.55	2.24	5.52	0.01	0.978	0.003	21.91	0.685
DBE	8.46	2.19	5.53	0.00	0.991	0.002	21.15	0.581
Montanov L	10.21	2.76	5.56	0.01	0.983	0.002	18.84	0.48
Emulgin	7.82	1.49	5.60	0.01	0.956	0.005	27.65	0.076
Simulgreen	9.78	2.05	5.55	0.01	0.986	0.003	46.18	0.643
Olivem 1000	9.83	2.13	5.81	0.03	0.969	0.002	32.22	0.744
Granthix	8.57	1.68	5.75	0.03	0.998	0.001	22.96	0.850
Lanette W	11.59	2.72	5.57	0.04	0.978	0.003	22.78	0.373

Determination of the spreadability

The examination of spreadability of the prepared creams was made with an extensometer, at room temperature according to current literature [29,30]. With a syringe, 1 ml of the preparation was placed on the marked circle of the bottom disk, a measuring disk with a scale was attached and then loaded with 200 g increase of weight every 1 min. Maximum load was 1400 g. In order to analyse spreadability of the preparation, the areas below the spreadability curves AUC [j.u.] were calculated and then compared. The results of these measurements are shown in Table 4.

Rheological study

Rheological study is an essential part of quality control. Rheological parameters such as flow behaviour, apparent viscosity, yield point and thixotropy can make it easier to predict the reaction of the product during preparation, packaging and finally application on the skin. Rheological measurements were carried out 24h after preparation. A Rheolab OC (Anton Paar, Austria) rotational rheometer with coaxial cylinders CC27 was used for the determination of the relation between shear stress and shear rate. Flow curves were ascertained at shear rate range from 0,1 s⁻¹-300 s⁻¹ at constant temperature 20°C by the use of Peltier (Anton Paar, Austria). Prior to measurements, each sample was tempered for 240 s, and the time span was set to 5 min, during which, shear rate increased from 0,1-300 s⁻¹ and reverse measurements down to 0,1 s⁻¹ took additional 5 mins. RheoCompass[™] software version 1.19 was used to obtain flow properties parameters and thixotropy. Using regression analysis, the parameters of the power law model, Herschel-Bulkley model and Casson model were calculated. For the analysis, we chose the model with the highest regression coefficient R². All results are summarized in Table 5. Examples of flow curve with power law model for the three self-emulsifying bases are presented in Figure 3 and Figure 4.

Table 5. Parameters of the Power Law model

Base	K [Pasn]	n [-]	R ²	η _a (10s ⁻¹) [mPas]	AUC [Pas ⁻¹]
Beautyderm	71.81	0.276	0.994	14439	3501
DBE	152.70	0.226	0.996	27034	8336
Montanov L	122.53	0.250	0.980	25375	6567
Emulgin	123.6	0.261	0.980	22660	5101
Simulgreen	32.14	0.368	0.998	8599	4396
Olivem 1000	25.01	0.170	0.984	3743	2116
Granthix	204.26	0.217	0.981	37976	11079
Lanette W	127.50	0.216	0.982	23463	9203

(consistency parameter – K, flow index – n) with regression coefficient – R^2 , apparent viscosity – η_a calculated at shear rate 10 s 1 and hysteresis loop area AUC



Figure 3. Flow curves of the samples with different self-emulsifying base according to Ostwald de Waele's-Power Law model



Figure 4. Example of ascending and descending flow curves with closed hysteresis loop area of cream – made with Simulgreen base

RESULTS

Sensory analysis

All except Granthix-based formulations were snow-white in colour and had pleasant scents typical of unrefined shea butter. The aforementioned had an unpleasant, chemical smell. All the preparations had a fine and homogenous textures, as well as smooth shiny surfaces without lumps and air bubbles. In sensory analysis, all creams had excellent performance. They applied easily and kept their shape. Creams based on Olivem 1000, however, failed to keep their shape on being applied on a finger. All the formulations were easily felt while rubbing against fingers. The preparations spread well on the forearm and absorbed quickly. The creams prepared on bases: Granthix and Lanette W showed slight bleaching effect while being rubbed into the skin, but they absorbed quickly. All the preparations left a characteristic light and greasy layer on the skin. The results of the sensory analysis of the preparations are typical of normal skin caring creams.

The study of physical characteristics of the formulations proved that the preparations were type w/o emulsions, the size of the balls in the oil phase ranged from approx. 7,82 μ m for the Emulgin base, to 11,59 μ m for the Lanette W base. The density of each preparation was very similar; practically the same. The determination of pH allowed for a comparison to physiological pH value of the skin. In the analysis, the pH values of the preparations were slightly acid.

Rheological study

Rheological study of the preparations involved the evaluation of viscosity, thixotropy and spreadability. Recognizing rheological parameters gives important information as it helps to predict the reaction of the formulation not only in processing, but also in later use [20]. The experiments were conducted at 20°C and allowed to the drawing of flow curve shear stress [Pa] versus shear rate [s⁻¹]. The analysis confirmed that samples of the creams are pseudoplastic fluids with shear thinning behaviour. Ostwald de Waele's model (Best Power Law model) (Tr=k Drⁿ) was used to determine consistency parameter, K – [Pasⁿ], flow index – n [-]. Apparent viscosity η_a was calculated at shear rate 10 s⁻¹. Note: Soriano Jimmenez M.M. (20), in his research on moisturizing properties of the cream with base NEO PLC, obtained a flow curve that he described with the power equation Tr=55.53 Dr^{0.524}. The flow type in accordance with Ostwald de Waele's model is typical of light creams [7,20,33]. Cosmetic creams described by Moravkova T. and Filip P [33] also followed Ostwald de Waele's model. Depending on the emulsifier used (Arlacel 989, Arlatone T), the values of viscosity coefficient and flow index were: (k=28,12, n=0,46), (k = 13.90, n= 0.52), respectively. Very low values of apparent viscosity at Dr – 10.0 s⁻¹ were obtained only for the cream with base Olivem 1000 (3743 mPas), which gives rise to the suspicion that with its prescribed composition, it is much better for body balms [8,33,34]. At shear rate 10 s⁻¹, the other preparations had viscosity comparable to the research data [20,35]. The analyses conducted at ascending and then descending shear rate helped to obtain at a temperature of 20°C, a closed histeresis loop area on all the preparation rheograms. Creams prepared with Granthix and Lanette N showed high thixotropy, which coincide with the results of sensory analysis. All probants was evaluated as difficult to spread and absorb into the skin. The value of thixotropy for the other creams is comparable to the research data [20], which confirms the researchers' opinion that the formulations will be easy to spread on and absorb into the skin.

A supplementary examination that completes the rheological study of the cosmetic preparations is an extensibility test under the action of applied load [20]. The analysis of areas under curves AUC [j.u.] enabled a comparison of the preparations that is presented in Table 4. The highest level of extensibility was noticed in the cream prepared with Simulgreen base and Olivem 1000, which is in connection with their low structural viscosity. Extensibility of the other preparations was similar, which might suggest that their spreadability on the skin will be high.

DISCUSSION

The growing interest in natural cosmetics has prompted manufacturers to look for organic ingredients that meet the requirements of COSMOS, such as eco-emulsifiers, ecooils, eco-butter. Cosmetics containing natural and certified vegetable ingredients or products obtained from animals, such as honey, are more friendly to our skin than cosmetics containing synthetic products or petroleum derivatives. However, it is not only the safety of cosmetics that is crucial for manufacturers. In order for the product to appeal to the consumer, it must also have a nice appearance, a pleasant smell and have very good skin spreadability. Cosmetic consistency, together with its scent and appearance, is important in consumer choice. Without emulsifiers and other stabilizing components, creams cannot have perfect quality and texture. Using natural or synthetic emulsifying bases that are prepared from especially composed mixture of emulsifiers, co-emulsifiers and stabilizing components is advantageous for manufacturers, as it enables a shorter time of weighing the components, minimizes storing costs and, at the same time, helps to prepare a product that is stable in time. The choice of emulsifying bases has a crucial influence on the production of stable cosmetic creams with favourable utility.

The cosmetic creams obtained in this study, for the preparation of which self-emulsifying bases of various natural origin were used, had similar sensory and physicochemical properties. None of the creams significantly differed in terms of their properties. Creams made with synthetic bases were, however, noted by the testers to be slightly worse, due to the chemical smell and weaker absorption into the skin.

Among the creams prepared with the use of natural, certified emulsifiers, all creams were rated very positively by the testers. However, the cream with Simulgreen was rated the best.

CONCLUSION

1. Creams made with the use of natural emulsifying bases had very good physicochemical properties; that is, they

spread very well over the skin, were quickly absorb into the skin, and did so without superficial whitening effect.

- 2. Creams made of natural emulsifying bases had lower thixotropy values, thanks to which they spread better on the skin and absorbed into it faster.
- 3. Creams made with the use of natural self-emulsifying bases were accepted by probants better than creams made of synthetic emulsifiers.
- 4. Sensory evaluation of cosmetic preparations allows completing the physicochemical tests.

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