Chemical and Process Engineering: New Frontiers, 2023, 44(4), e42
DOI: 10.24425/cpe.2023.147401

Production of cosmetic emulsions based on plant biocomponents

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Presented at 24th Polish Conference of Chemical and Process Engineering, 13–16 June 2023, Szczecin, Poland.

Article info:
Received: 28 April 2023
Revised: 03 August 2023
Accepted: 01 September 2023

Abstract
Cosmetic emulsion bases containing extracts from natural plants were produced. The emulsifier was an aqueous solution of self-emulsifying base made from apricot kernel oil and soy lecithin, while the oil phase was based on coconut, almond or grape seed oils. In addition, mixtures enriched with vegetable glycerine were produced. It was found that for the emulsions with almond oil as the concentration of the oil phase increased, the value of the average Sauter diameter increased. In comparison, results for emulsions with coconut oil and emulsions with grapeseed oil did not give such a clear relationship. It was also shown that for stable emulsions, the self-emulsifying base of apricot kernel oil performed much better than soy lecithin. The addition of vegetable glycerine to the mixture resulted in a reduction of the average droplet diameter. Produced emulsions were also visually observed for 60 days to assess their stability and possible aging processes. In order to exclude the formation of microorganisms, periodic density control and microscopic examinations were carried out. The presence of microorganisms in the analysed emulsion was evaluated using microscopic and culture techniques. No tarnish was observed on the surface of the samples, indicating the formation of mould, which can lead to poisoning and the development of allergies, respiratory diseases, liver diseases, ulcers, or bleeding in the intestines.

Keywords
cosmetic emulsions, natural emulsifiers, vegetable oils, emulsifying and stabilising potential, eco-design

1. INTRODUCTION

Modern cosmetics are made in the form of single emulsions (oil-in-water, water-in-oil, water-in-silicone types) and multiple emulsions (water/oil/water, oil/water/oil) (Baki and Kenneth, 2015; Gilewicz, 1957; Mollet et al., 2000; Venkataramani et al., 2020). Depending on the application, cosmetic emulsions are prepared in liquid and semi-solid forms. Liquid cosmetics are called lotions, moisturisers or balsams, and are characterised by lower viscosity and higher water content than semi-solid forms. They are less greasy, easier to spread and wash off the skin. They are used as cleansing lotions, liquid face primers, aftershave lotions or sunscreens. Due to the possibility of combining hydrophilic and lipophilic cosmetic raw materials into a coherent system with a variety of effects, smooth consistency and application convenience, emulsions have found wide application in the cosmetics industry. Products which are available in the form of emulsions are products for makeup removal, washing of face, body and hands, skin moisturisers, cosmetics with targeted action (such as anti-wrinkle), sunscreen, antiperspirants and deodorants (Kichou et al., 2023; Marto et al., 2020).

Based on the analysis of compositions of individual cosmetics, regardless of the application or type of system, a cosmetic emulsion is formed by the same groups of raw materials. These are solvents that form the base of the emulsion, emulsifiers, moisturisers and thickening agents, active ingredients responsible for the functions of the cosmetic, and additives in the form of life-prolonging substances (preservatives) and modifiers of sensory properties (e.g. fragrances, dyes and pigments) (Arancibia et. al., 2016; Bhargava, 1987; Dammak et al., 2020; McClements et al., 2017). Note that many compounds have several functions in an emulsion system. For example, water is a solvent but also a consistency agent, while hyaluronic acid is a thickening and moisturising substance. Glycerine is a humectant, a viscosity-reducing agent, a fragrance-fixing agent for perfumed cosmetics and a preservative-enhancing agent.

Emulsifiers in emulsions are important components and their selection depends on the desired performance of the final products, by defining the HLB of the oil phase (the balance between the aqueous and lipid parts of the emulsifier), pH, electrolyte content, the presence of coemulsifiers and other factors. The numerical value of the HLB (*Hydrophilic Lipophilic Balance*) factor indicates the percentage of hydrophilic part in each emulsifier.

Vegetable raw materials have been recently used for numerous applications, including cosmetic emulsions (Dini and Laneri, 2021; Kunik et al., 2022; Tiwari et al., 2022). Natural emulsifiers are called surfactants isolated from renewable raw materials of animal, vegetable and microbial origins. These include phospholipids, biosurfactants, proteins, polysaccharides and molecular colloids, among others. Based on raw materials of natural origin, a number of commonly

used emulsifiers are obtained either in hydrogenation, catalysis or biocatalysis from vegetable raw materials such as jojoba oil, palm oil, coconut oil, soybean oil, cottonseed oil or other vegetable oils (Dănilă et al., 2019; Daugherty et al., 1958; Kavadia et al., 2017; Norhafipah et al., 2008). Examples of such emulsifiers include fatty alcohols (cetyl, lauryl, oleic) (Pratap, 2009), fatty acids (stearic), fatty acid esters (glyceryl stearate, isopropyl myristate, sorbitan laurate, sorbitan stearate) (Arniza et al., 2020; Barel et al., 2014; Vadgama, 2015).

Protective colloids are used to improve the system's stability, which contains hydrophilic molecules capable of adsorbing on the surface of hydrophobic fat phase molecules. The protective colloid layer further protects the droplets from coalescence due to collisions. The functions of protective colloids are often performed by thickening agents such as cellulose ethers (methylcellulose, carboxymethylcellulose, hydroxypropylcellulose), lecithins, cholesterol, gelatine, proteins, casein, gums, saponins, lignosulfonates (McClements et al., 2016; Mollet et al., 2000; Sinko et al., 2011).

The purpose of this study was to verify the possibility of preparing a cosmetic emulsion base on selected emulsifiers and oil phases of natural origin with the addition of vegetable glycerine. The work aimed to increase our knowledge of the possibility of replacing synthetic emulsifiers with natural substances. An allergy to many substances is a problem that has increased in scope in recent years. Therefore, the base of the proposed emulsion is on coconut oil, almond oil, grape seed oil and emulsifiers from the apricot kernel or soy lecithin. They can contribute to the development of more eco-friendly and skin-friendly cosmetic products. An evaluation of the effectiveness of these emulsifiers in stabilising emulsions and assessment of their effect on the shelf life of cosmetics was carried out, with application to scrubs, a popular cosmetic procedure for exfoliating dead skin and rejuvenating the skin. The work provides evidence that the use of natural emulsifiers in the production of cosmetic bases has the potential to create more eco-friendly and skin-friendly products. Further research in this area could contribute to the development of new methods of cosmetic production that are both effective and safe for users and the environment.

2. MATERIALS AND METHODS

2.1. Bio-components used

Apricot kernel oil is mainly abundant in unsaturated fatty acids, especially oleic and linoleic acid, and contains a small amount of palmitic acid, which is a saturated fatty acid. It is a source of bioactive compounds like tocopherols (vitamin E) belonging to powerful antioxidants and phytosterols (Bhanger et al., 2020; Durmaz et al., 2010; Saadi et al., 2022). It improves skin hydration and prevents water loss from the epidermis. It can be used as an emollient in dermocosmetics. It has an anti-ageing effect and can relieve skin inflammation in cases of atopic dermatitis and psoriasis.

Lecithin is a well-known emulsifier in the industry from the group of a low-molecular-weight phospholipids (Dickinson, 1993; McClements et al., 2017). Studies have shown that its stabilising properties depend on the concentration of individual phospholipids and the presence of emulsifiers from other groups like polysaccharides or proteins in the mixture (Dammak et al., 2020; McClements et al., 2016). The value of HBL in extreme cases can range from 2 to 8, but most often, it is 7. Therefore, lecithin can be used for the formation of both O/W and W/O dispersions. Emulsions produced on the basis of lecithin retain their properties with increasing temperature but show instability at pH below 3 and are very sensitive to the addition of electrolytes.

The experimental studies consisted of the production of emulsion bases using 10% solutions of two emulsifiers: apricot kernel and soy lecithin. On the other hand, the oil phase was coconut, almond and grape seed oils. In addition, mixtures were enriched with vegetable glycerine. All substrates for making the mixtures during the tests were sourced from the distributor company *Zrób Sobie Krem Natural Cosmetics*. However, 99.5% of vegetable glycerine came from the *BIOMUS* company. The emulsifier solution accounted for 0.4 (vol./vol.) of the total sample, while the remainder was oil and glycerine (Table 1). The densities of all components were obtained with the use of the volume-mass method and are reported in Table 2.

Table 1. Shares of individual components in the emulsion.

	The volumetric proportion of the various components								
oil	1	2	3	4	5	6	7	8	9
glycerine	9	8	7	6	5	4	3	2	1
ratio of oil in the mixture	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9

Table 2. The density of tested emulsion substrates.

Components	Density [g/cm^3]
Apricot kernel emulsifier (10 vol.% solution)	0.998
Emulsifier soy lecithin (10 vol.% solution)	1.039
Vegetable glycerine	1.240
Almond oil	0.912
Grapeseed oil	0.920
Coconut oil	0.946

2.2. Emulsification setup and process

A laboratory homogenizer ULTRA-TURRAX T 50 basic IKA-WERKE with a tip of 20 mm in diameter (Figure 1) was used to produce the tested emulsions. After preparing an emulsifier solution in distilled water in advance, an appropriate amount of each product was introduced into a container and placed in a homogenizer to produce an emulsion base. The process was carried out at a constant rotational frequency of 10000 rpm for 20 minutes. Previously, a series of tests was carried out in a homogenizer for different oils and emulsifiers, rotational speeds, and mixing times. It was evaluated whether the emulsion did not delaminate under the given operating conditions of the apparatus, as well as changes in droplet size with mixing time. Based on this, emulsification time and homogenizer speed were optimised to those used during the presented experiments.

Figure 1. Homogenizer tip used to produce test emulsions.

Stability analysis consisted of visual observation of the mixtures every 15 minutes during the first 5 hours, and then every hour for 3 days. After that period, samples were inspected every 24 hours for 60 days. Stability tests were conducted at 22 °C in a controlled room. The produced emulsions were stored at 22 °C in a room. In addition to visual observation, microscopic examinations were carried out after the first hour of receiving the emulsions. Then control examinations were carried out every 12 hours to evaluate any changes in droplet size.

All samples were also evaluated for changes in colour, odour and consistency. In order to exclude the formation of microorganisms, periodic density checks were conducted, and microscopic examinations were carried out. The presence of microorganisms in the analysed emulsion was evaluated using microscopic and culture techniques. Each time a sample was taken, every 7 days, it was checked under a microscope for detection of microorganisms. In addition, at the time of sampling, microbiological cultures were performed on solid media dedicated to the culture of bacteria, fungi, moulds and yeast. Isolation cultures were performed on Tryptic Soy Agar (Sigma Aldrich, Poland), Sabouraud Dextrose Agar (BLT, Poland), Malt Extract Agar (Sigma Aldrich, Poland) and Yeast Extract Peptone Dextrose Agar (Sigma Aldrich, Poland). Solid media were prepared with and without the addition of an antibiotic to inhibit bacterial growth. The culture was performed under sterile conditions using a laminar flow cabinet.

Bacteria were cultured at 30 °C for 24–72 h, while fungi, yeast and moulds were cultured for 96 h. After this time, the sterility of the agar media was checked. All solid media were sterile for up to 60 days.

2.3. Microscopic analysis

The cosmetic emulsion bases produced were subjected to microscopic analysis based on visual evaluation and taking a series of pictures. For this purpose, a microscope Nikon Eclipse 50i equipped with a camera OptaTech and a computer with software MultiScanBase and Image-Pro Plus companies Media Cybernetics were used.

Images taken with the microscope were processed in the Matlab software. The radii of individual droplets were determined using a C++ script applying an algorithm based on the Hough transform, which is used to detect circles in machine vision. The *imfindcircles* function in Matlab was used for analysis of each image to obtain the radius of the circle expressed as a range of values. As a result of the operation, the approximate size of the radius measured in pixels was obtained for each detected circle. The resulting data were exported to an Excel file in order to assess the quality of obtained droplets by calculating the volume-surface average droplet diameter (Sauter diameter, d_{32}, Eq. (1)), coefficient of variation (CV, Eq. (2)) and span (Sp, Eq. (3)).

$$d_{32} = \frac{\sum_{i=1}^{i=n} n_i d_i^3}{\sum_{i=1}^{i=n} n_i d_i^2} \tag{1}$$

$$CV = \frac{\sigma}{\overline{d}} \tag{2}$$

$$\mathrm{Sp} = \frac{d_{0.9} - d_{0.1}}{d_{0.5}} \tag{3}$$

3. RESULTS AND DISCUSSION

3.1. Analysis of produced emulsions with apricot kernel emulsifier

Based on the analysis of microscopic images taken during the tests, the quality of obtained droplets in terms of Sauter diameter, standard deviation (σ), median value, coefficient of variation and span were performed and are summarised

in Table 3. Note that in emulsion notations "C" stands for coconut oil, "G" for grapeseed oil, "A" for almond oil and the number represents the ratio of specific oil to glycerine. For example, the notation "C 0.1" means that the emulsion contained 0.1 units of volume of coconut oil with respect to glycerine. Data presented in Table 3 were obtained for the number of droplets ranging from 2975 up to 5710. As presented in Figure 2. the mean Sauter diameters achieved the smallest values for ratios of oils (i.e. 0.1 and 0.2), then increased, reaching their maximum for ratios 0.3, 0.4, 0.5, 0.6 and 0.7, and finally slightly decreased for ratios of 0.8 and 0.9. It is evident that despite the oil ratio, the d_{32} for apricot kernel emulsifier and all three oils dissolved in glycerine oscillate around 20 μm. The smallest value of mean Sauter diameter was obtained for the emulsion containing coconut oil (d_{32} = 7.2 μm). Regardless of which oil and in which ratio was added, the droplet sizes in terms of d_{32} varied between 7.2 and 34.4 μm with standard deviation in a range of 3.3 and 12 μm. In contrast, the coefficient of variation varied between 54% and 100%. Span values were between 0.8 and 7.0, which clearly shows that produced emulsions are not monodispersed, although their ranges of diameters are rather narrow. It is important to notice that at least half of the droplets in all emulsions containing apricot kernel emulsifier had a diameter of less than 13.2 μm.

The selected histograms were plotted for each sample and showed the distribution of droplets in given size ranges. Figure 3 shows the histogram for emulsions containing almond oil (Figures 3a and 4ab), grape seed oil (Figures 3b and 5ab) and coconut oil (Figures 3c and 6ab) for all studied oil-to-glycerine ratios. The largest number of small droplets was obtained in mixtures with the smallest ratio of coconut oil, less with grapeseed oil, while for almond oil it was down to circa 80% in comparison to circa 95% for coconut oil. Droplets with the smallest diameter range (1 ÷ 2 μm) were not found in all mixtures. It was also observed that increasing the oil content in each emulsion resulted in a small stability decrease after about 30 days, but only for the higher and medium oil content in the emulsion. This is related to the increase of droplets, as presented in histograms, to higher diameters and a substantial increase in the presence of droplets with diameters higher than 32 μm and the appearance of droplets bigger than 56 μm.

3.2. Analysis of soy lecithin emulsions produced

In the case of the cosmetic emulsion base productions, where the emulsifier was soy lecithin, no effect on the Sauter diameter was observed when changing the glycerine content in the mixture (Figure 7). There was a slight effect of the type of oil used, which was comparable to that observed for the emulsions produced with the emulsifier from apricot kernel. Noticeably, the smallest average droplet diameters were obtained for coconut oil. It is also important to notice that d_{32} values for lower content of oils were around two times higher than those obtained with apricot kernel. For higher content of oils these differences are smaller but with time they are increasing up to two times bigger than for apricot kernel.

It was also observed that the stability of emulsions with soy lecithin decreased after just 60 minutes. Repeated microscopic examinations were performed (Figure 8b), and the droplet sizes obtained after this time confirmed the impaired

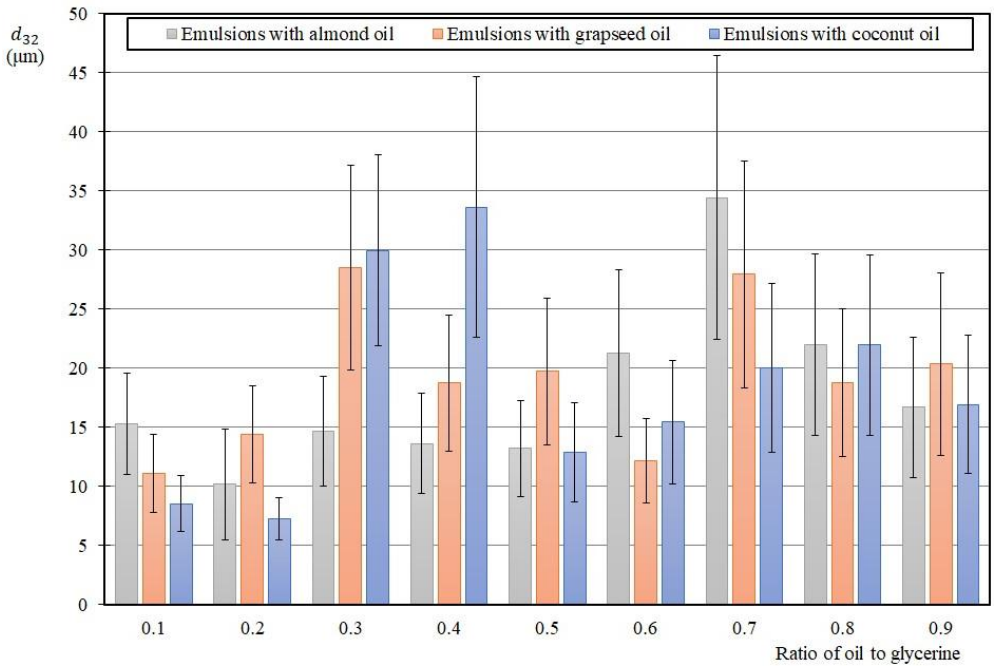

Figure 2. Dependence of average droplet diameter on the proportion of glycerine in the mixture.

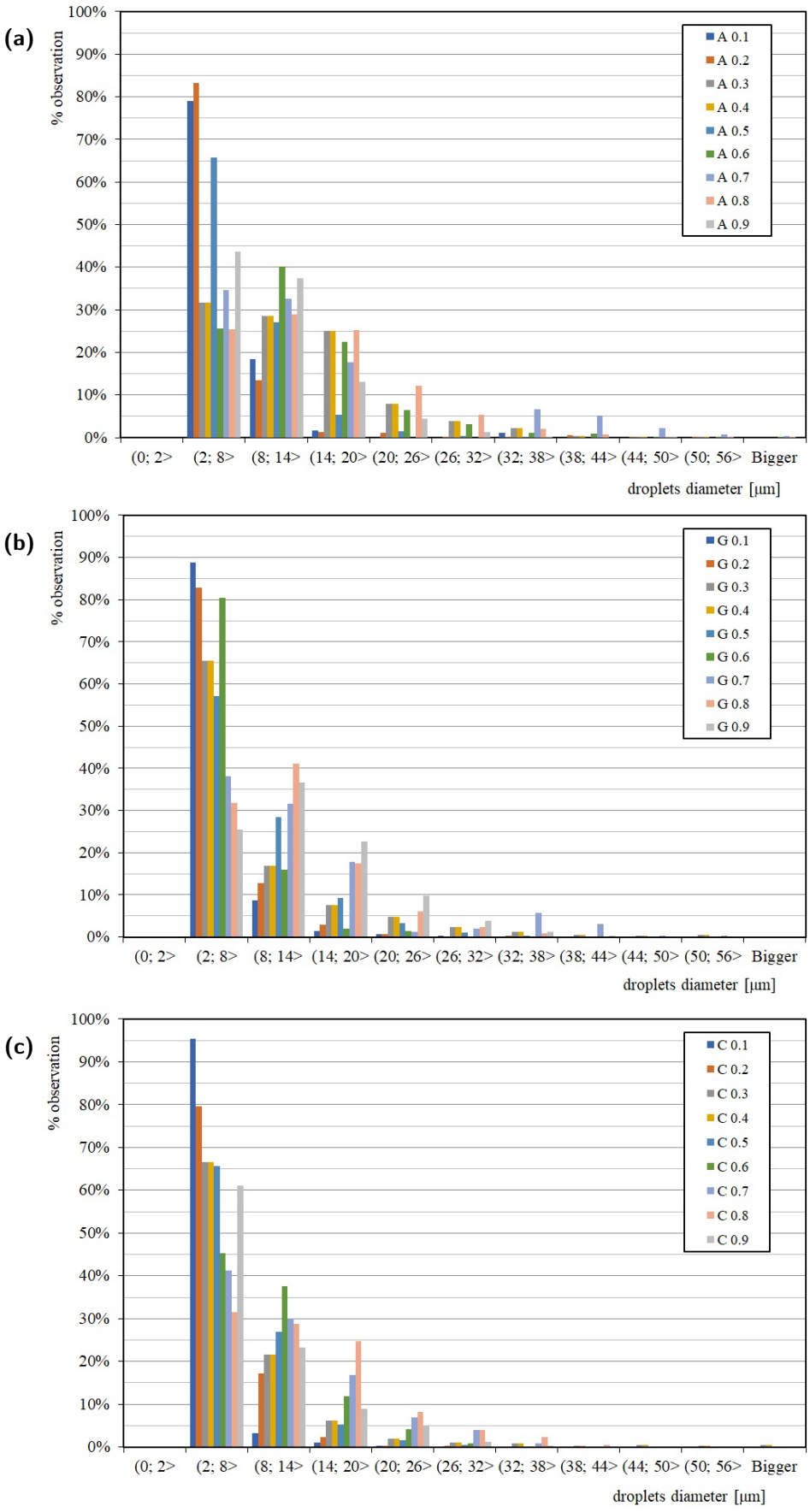

Figure 3. Droplet distributions for the tested mixtures for three glycerine to-oil ratios: a) almond, b) grape seed, c) coconut oil.

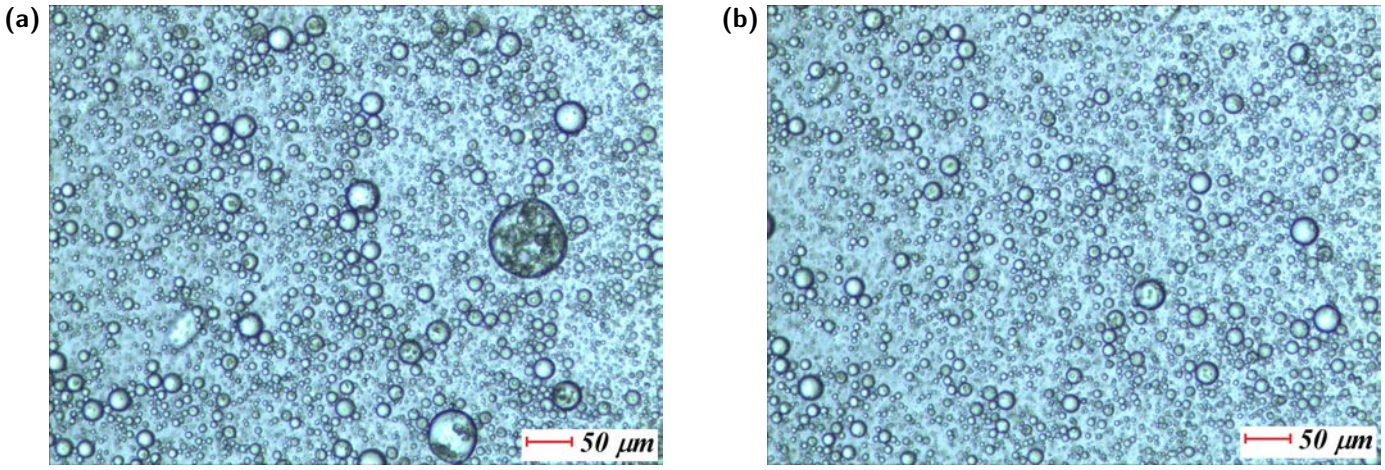

Figure 4. Exemplary microscopic images of almond for the proportion of glycerine in the emulsion: a) 0.9, b) 0.1.

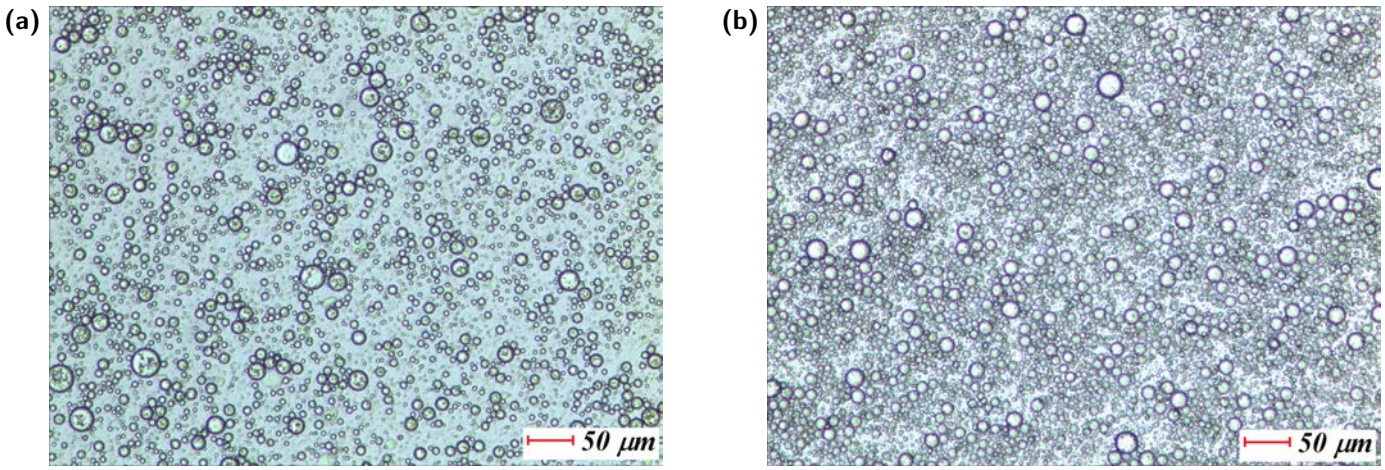

Figure 5. Exemplary microscopic images of grape oil for the proportion of glycerine in the emulsion: a) 0.9, b) 0.1.

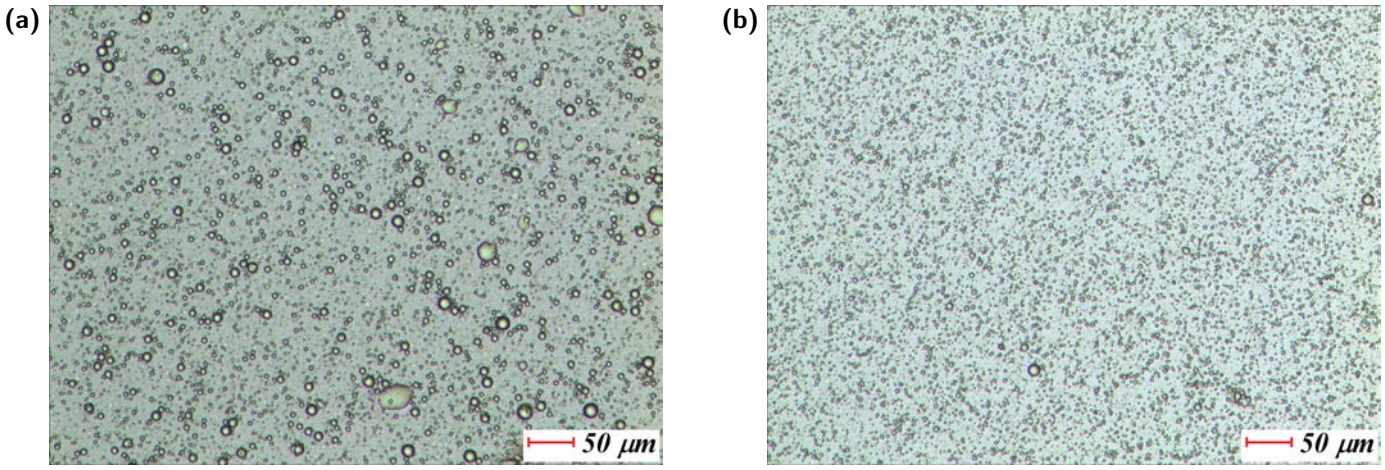

Figure 6. Exemplary microscopic images of coconut oil for the proportion of glycerine in the emulsion: a) 0.9, b) 0.1.

stability of these emulsions (Figure 7). As the oil in the mixture increased, the emulsions increased their tendency to delaminate. These results drive the hypothesis that there is a big difference in density between soy lecithin and oils to create stable emulsions compared to apricot kernel emulsifiers. That is manifested by ratios between specific oil to soy lecithin emulsifier varying between 87.78% (almond oil) to 88.55% (grapeseed oil) up to 91.05% (coconut oil) while for apricot kernel emulsifier respective values are higher and varied between 91.38%, 92.18% and 94.79%.

Table 3. Results of performed experiments with the production of emulsion containing water, apricot kernel, glycerine and coconut, grapeseed and almond oils.

Emulsion notation	d_{32} [μm]	Number of droplets	σ [μm]	Median [μm]	*CV* [%]	*Span* [–]
C 0.1	8.5	5710	2.4	3.3	59%	1.3
C 0.2	7.2	3849	1.8	2.9	55%	0.9
C 0.3	30.0	3106	8.1	5.8	100%	4.1
C 0.4	33.6	3795	11.0	7.6	95%	7.0
C 0.5	12.8	2975	4.2	6.5	56%	1.8
C 0.6	15.4	3407	5.2	8.5	55%	2.1
C 0.7	20.0	3792	7.1	9.2	64%	3.2
C 0.8	22.0	3118	7.6	10.4	59%	2.6
C 0.9	16.9	3335	5.9	6.8	68%	3.4
G 0.1	11.1	3155	3.3	3.7	69%	2.0
G 0.2	14.4	3816	4.1	4.4	74%	2.3
G 0.3	28.5	3274	8.6	5.7	98%	5.8
G 0.4	18.7	3284	5.8	7.1	69%	2.6
G 0.5	19.7	3346	6.2	7.2	71%	2.9
G 0.6	12.2	2985	3.6	5.5	56%	1.2
G 0.7	27.9	3602	9.6	9.6	76%	5.1
G 0.8	18.8	3210	6.3	10.4	54%	1.9
G 0.9	20.4	3244	7.7	12.1	58%	2.2
A 0.1	15.3	3694	4.3	5.2	71%	2.1
A 0.2	10.2	3858	4.7	5.2	78%	1.5
A 0.3	14.6	3212	4.7	5.9	65%	2.1
A 0.4	13.6	4236	4.3	6.5	58%	1.8
A 0.5	13.2	2975	4.0	5.4	66%	0.8
A 0.6	21.3	3973	7.0	12.3	55%	1.9
A 0.7	34.4	5664	12.0	10.8	83%	5.8
A 0.8	22.0	3182	7.7	13.2	55%	2.2
A 0.9	16.7	3120	5.9	8.9	61%	3.5

4. CONCLUSIONS

In the presented work, it was proven that it is possible to produce stable cosmetic emulsion bases from components of natural origin. The emulsions prepared with an emulsifier from the apricot kernel and a mixture of individual oils and glycerine were stable. In contrast, emulsions prepared with soy lecithin did not meet our expectations. They may prove to be more stable in a configuration with other ingredients.

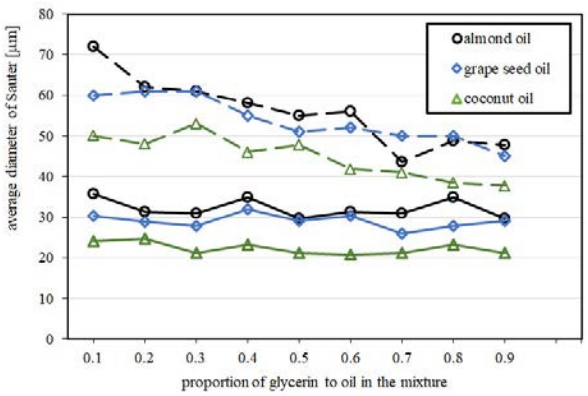

Figure 7. Effect of glycerine content in soy lecithin emulsion immediately after preparation and after 60 minutes.

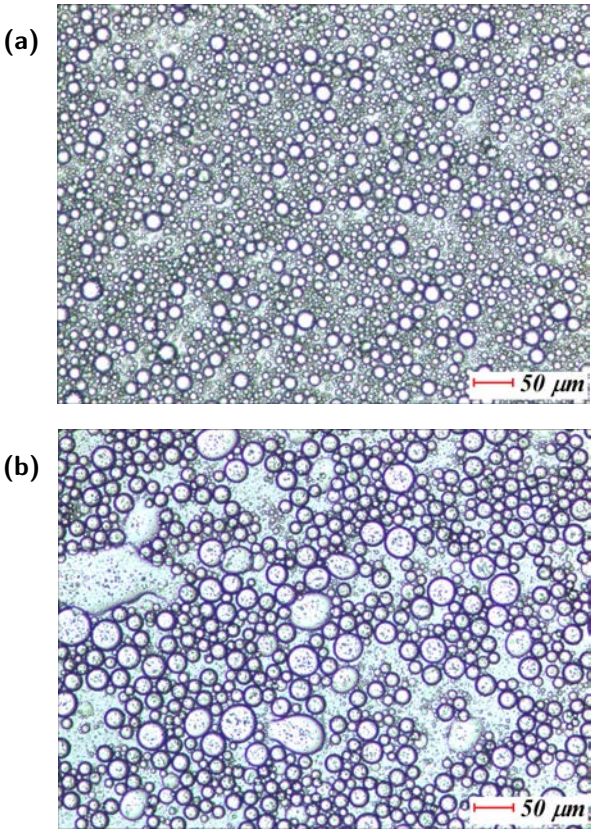

Figure 8. Exemplary microscopic images for 0.5 proportion of glycerol with coconut oil: a) immediately after receiving the emulsion, b) 60 minutes after receiving the emulsion.

As a bonus, after 60 days of testing with the apricot kernel emulsifier, the samples showed no microbial destruction characteristics that could affect bacterial or viral growth. The presence of microorganisms in the analysed emulsion was evaluated using microscopic and culture techniques to detect microorganisms. In addition, at the time of sampling, microbiological cultures were performed on solid media dedicated to the culture of bacteria, fungi, moulds and yeast. All solid media were sterile for up to 60 days. Unfortunately, this could not be stated for the emulsifier soy lecithin. Regard-

less of the glycerine content in the prepared emulsions, they proved to be unstable after only about 60 minutes. Their stability was slightly higher with higher glycerine content in the emulsion. Microscopic images confirmed this when both emulsifiers were used.

ACKNOWLEDGEMENTS

This research was funded by the Ministry of Education and Science (0912/SBAD/2302).

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