

INVESTIGATION OF THE POSSIBILITY OF THE SYNTHESIS OF SUCROSE ESTERS FROM BIODIESEL

ORIGINAL SCIENTIFIC ARTICLE

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ABSTRACT:

Growing awareness of the negative environmental impacts of synthetic surfactants and their limited biodegradability require the search for sustainable and environmentally acceptable alternatives. In this context, sucrose esters represent a promising class of green surfactants due to their renewable nature, biodegradability and low toxicity profile. In this work, the synthesis of sucrose derivatives was investigated using sucrose and a mixture of methyl esters of fatty acids (biodiesel), with the use of potassium carbonate as an effective catalyst. The reactions were carried out in the presence of methanol or *N,N*-dimethylformamide as a solvent, with the aim of examining the influence of reaction conditions on the formation of products. The FTIR technique was used in combination with the appropriate physico-chemical analysis to characterize the obtained synthesis products. The obtained results indicate a significant influence of the choice of solvent on the character of the product, while the use of biodiesel and sucrose as reactants and potassium carbonate as a catalyst provides a perspective for the development of new environmentally friendly surfactants as a promising alternative to synthetic surfactants.

KEYWORDS: biobased surfactants; synthesis of surfactants; renewable raw materials; sucrose; biodiesel

INTRODUCTION

Surface active agents are an amphiphilic group of molecules that can reduce the surface and interfacial tension between two immiscible phases. They are also known as surfactants, tensides, micellar colloids, associative colloids, and detergents. Their chemical structure consists of a hydrophilic (polar) head and a hydrophobic tail that is nonpolar (Figure 1).

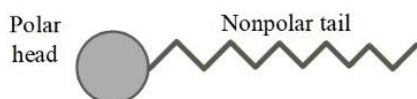


Figure 1. Molecule of surfactant

The hydrophobic tail is a linear or branched hydrocarbon, fluorocarbon or siloxane chain of 8-18 carbon atoms in length, while the polar head group can be nonionic or ionic [1]. Depending on the nature of the charge of the polar head, surfactants are divided into anionic, cationic, nonionic and amphoteric surfactants. The proportion and nature of the groups

present in the molecule can vary widely, which affects the properties of the surfactants.

The amphiphilic nature of surfactants makes them suitable for use in numerous products, and they are often found in various products where they perform the functions of detergents, emulsifiers, wetting agents, corrosion inhibitors for the protection of steel and various metals, pour point depressants, and others [2]. The total global production of surfactants is constantly increasing, of which approximately 54% is used in households in the form of laundry detergents, while 32% is used in industry [3].

Most classical surfactants are produced from raw materials based on petrochemical sources, whose biodegradability and toxicological properties can leave a negative mark on the environment [4]. In addition to environmental requirements, there are problems of stricter legal regulations in various industries, an evident trend of consumer preference for more natural products, instability and fluctuations in the prices of fossil sources, as well as scientific interest in new surfactant structures that can have improved

performance while reducing unwanted effects [5]-[7]. A possible alternative to classical synthetic surfactants are „green surfactants”, which can be classified into: bio-based surfactants obtained chemically and bio-based surfactants produced by living cells (biosurfactants) [8].

One of the most studied and promising classes of bio-based surfactants are sucrose esters, which are the product of transesterification of fatty acid esters and sucrose or direct esterification of sucrose with fatty acids [5]. Sucrose has 8 hydroxyl groups that can be esterified, of which three are primary and five are secondary, so depending on the degree of substitution and the length and saturation of the hydrophobic tail of the fatty acid, a wide range of surfactants with different properties can be obtained [9].

The most common fatty acids used for the synthesis of sucrose esters are lauric, myristic, palmitic, stearic, oleic, behenic and erucic acids [10]. In some studies, methyl esters of lauric acid [11], methyl esters of stearic acid [12], ethyl esters of octanoic acid [13], but also mixtures of fatty acid methyl esters previously produced by transesterification of coconut oil [14], used as a source of hydrophobic raw material for the synthesis of sucrose esters. A group of authors [15], showed that sucrose esters can be synthesized using lower-value raw materials, such as molasses and methyl esters of fatty acids obtained from waste cooking oil. Sucrose transesterification reactions are most often catalyzed by catalysts such as potassium carbonate [14], sodium carbonate [15], potassium methoxide [12], sodium hydrogen phosphate [16], Amberist resin [17]. The reactions are carried out at different temperatures in the presence of different solvents such as dimethyl sulfoxide [13], [16], N-methylpyrrolidone [18], methanol [11]. As stated in the research of the author's group [19], the synthesis of sucrose esters is possible even without the presence of solvents, with the use of emulsifiers (sucrose ester, potassium palmitate and glycerol monostearate). In order to increase sustainability, methods with prior treatment such as grinding sucrose have also been developed, for better phase interaction, which contributes to a significant increase in yield and reducing reaction time [20]. The

sucrose transesterification reaction can also be assisted by microwave irradiation, thus avoiding sugar pyrolysis, as evidenced by the retention of the white color of the surfactant [12]. In the study [11], sucrose esters were synthesized using ultrasound at temperatures of 40 °C and 65 °C, for 1 h.

The aim of this work is to examine the possibility of synthesizing sucrose esters under laboratory conditions from table sugar (sucrose) and biodiesel (mixture of fatty acid methyl esters) in the presence of methanol or *N,N*-dimethylformamide solvents.

EXPERIMENTAL

Materials

The following substances were used for the synthesis of the surfactant and the separation of the products: table sugar (*Aragold, technical purity*), biodiesel - methyl esters of fatty acids (FAME) whose basic characteristics and fatty acid composition are given in Tables I and II, methanol (*VWR International, p.a.*), *N,N*-dimethylformamide (*VWR International, p.a.*), potassium carbonate (*Lach-Ner, p.a.*), sodium chloride (*Lach-Ner, p.a.*), *n*-hexane (*VWR International, p.a.*) and *n*-butanol (*VWR International, p.a.*).

Equipment

For the synthesis, an apparatus consisting of a glass reaction vessel with a lid containing five openings was used, to which the stirrer propeller, water cooler and thermometer were connected, and the remaining two openings were used for adding reactants. The reaction mixture was stirred using a mechanical stirrer with direct drive (*Witeg Direct Driven Stirrer HS-120A*), and heated using a water bath (*Inko*). Evaporation of the solvent was performed using a rotary vacuum evaporator (*Heidolph LABOROTA 4000*). The dissolution of the samples was assisted by mixing on a magnetic stirrer. Thermostating of the samples for the purpose of some product analysis was performed in an incubator (*"VELP SCIENTIFICA FOC Cooled Incubator"*).

Table I Basic characteristics of biodiesel

Parameter	Unit	Method	Result
FAME	(% m/m)	BAS EN 14103	93.45
FAME	(% v/v)	BAS EN 14078	92.50
Flash point	(°C)	ASTM D 93	160.50
Pour point	(°C)	ASTM D 97	1.50
Sulfur content	(mg/kg)	BAS EN ISO 20846	8.14
Iodine number	(g I ₂ /100 g)	ISO 3961:2019	96.54
Kinematic viscosity (20 °C)	(mm ² /s)	ISO 3104	6.9869
Kinematic viscosity (40 °C)	(mm ² /s)	ISO 3104	4.3741
Density (15 °C)	(kg/m ³)	ASTM D 4052	881.0

Table II. Fatty acid composition in biodiesel determined by gas chromatography

Fatty acid	C12:0	C14:0	C16:0	C16:1	C17:0	C18:0	C18:1 (trans)	C18:1 (cis)	C18:2	C18:3	C20:0	C22:1
%	0.14	0.60	19.03	0.65	0.14	5.33	0.46	36.23	32.18	4.22	0.35	0.24

Synthesis procedure

A certain mass of table sugar ($c=40$ g/L) was transferred to a reaction vessel and then 100 mL of solvent (methanol or *N,N*-dimethylformamide (DMF)) was added. The rest of the apparatus was assembled and placed on a water bath where heating was performed, up to 65 °C. Mixing was performed for 30 minutes, in order to dissolve the sucrose. Then, a certain amount of K₂CO₃ catalyst (CAT:SU=0.25:1) was added through the opening and heating and mixing were continued for the next 30 minutes. After the time elapsed, a certain amount of biodiesel, equivalent to the amount of sucrose (SU:BD=1:1), was added and the entire content was mixed while heating at a temperature of 65 °C for 2 h. After the reaction was completed, the reaction mixture was subjected to product separation, which differed for the syntheses performed with different solvents (Supplementary Materials - Figure 1SM and Figure 2SM).

Product testing methods

In order to characterize the synthesized products, the following characteristics were examined: pH value, electrical conductivity, surface tension, critical micelle concentration and foaming power. Also, the values of saponification, acid and ester number were determined for both the reaction products and the initial reactant biodiesel. Structural analysis was carried out using the FTIR spectroscopy method, which recorded FTIR spectra for the reactants and the main products in the reaction in order to compare the composition and quality.

A Fourier transform infrared spectrophotometer (FTIR), model "Tensor 27" manufactured by "Bruker", with a scanning range of 400-4000 cm⁻¹, with a resolution of 4 cm⁻¹, was used to identify characteristic functional groups in the reactants (biodiesel and table sugar) and synthesized products. In order to determine the values of pH, electrical conductivity, surface tension and foam height, series of solutions with different concentrations were prepared ($c=0.01\%$ - 1%). Measurements were performed in solutions at temperature of 20 °C. A pH meter (WTW Multiline P4) was used to determine the pH of the synthesized products and a conductometer (Consort C861 Multi Parameter Analyzer) for measure electrical conductivity.

The stalagmometric method was used to determine H₂O the surface tension. Based on the number of liquid drops (n_x), the number of distilled water drops (n_w), the density of water ($\rho_w(20^\circ\text{C})=0,9982$ g/cm³), the density of the liquid ($\rho_x(20^\circ\text{C})$) the surface tension of water ($\sigma_w(20^\circ\text{C})=72,75$ mN/m), the surface tension of the specific liquid ($\sigma_x(20^\circ\text{C})$) was calculated according to the following formula:

$$\delta_x = \delta_{H_2O} \frac{\rho_x n_w}{\rho_w n_x} \left[\frac{mN}{m} \right] \quad (1)$$

The density of the liquid and distilled water was determined at 20 °C using a pycnometer, by dividing the mass of the tested liquid (m_l) by the volume of the pycnometer (V_p).

$$\rho_x = \frac{m_l}{V_p} \left[\frac{g}{cm^3} \right] \quad (2)$$

The critical micelle concentration (CMC) values for the synthesized products were determined graphically from the dependence of surface tension on the logarithm of concentration. The CMC value was determined as the point of intersection of two adjacent straight lines, for which the equations of the line and the correlation factors were determined. By solving the system of two equations with two unknowns, the CMC value is obtained for the x coordinate ($\log c$) of the intersection point.

To test the foaming ability, 5 mL of the solution was measured into a measuring cylinder and covered with a stopper, followed by intensive shaking for 20 seconds. The foam height (mL) was read immediately after the shaking stopped and then after 30 min, 60 min, 90 min and 120 min.

The weighed sample (m) was first dissolved in a previously neutralized solvent mixture of diethyl ether:ethanol (2:1), and then titrated with an alcoholic solution of KOH ($c=0.1$ mol/L) using phenolphthalein, and based on the titrant consumption, the acid number (AV) was calculated according to the formula:

$$AV = \frac{V \times c \times 56,1}{m} \left[\frac{mg \text{ KOH}}{g} \right] \quad (3)$$

The determination of the saponification number (SV) is based on the saponification of the weighed mass of the sample (m) with an alcoholic solution of KOH ($c=0.1$ mol/L), heating with a reflux condenser in a water bath for one hour, with frequent shaking. After saponification is complete, a few drops of phenolphthalein are added to the hot solution and the excess KOH is immediately retitrated with HCl solution ($c=0.1$ mol/L) until the pink color disappears. In parallel, the consumption of HCl for the blank sample (V_0) was determined and the value of the saponification number was calculated as follows:

$$SV = \frac{(V_0 - V_1) \times c \times 56,1}{m} \left[\frac{mg \text{ KOH}}{g} \right] \quad (4)$$

The ester number (EV) is calculated from the difference between the saponification and acid numbers. The ester number indicates the mg of KOH consumed to saponify the esters present in 1 g of sample.

$$EV = SV - AV \left[\frac{mg \text{ KOH}}{g} \right] \quad (5)$$

RESULTS AND DISCUSSION

To facilitate the characterization of the resulting product, Figure 2 provides a graphical representation of the FTIR spectra of the reactants used for the synthesis and the products of both experiments (S-MeOH and S-DMF).

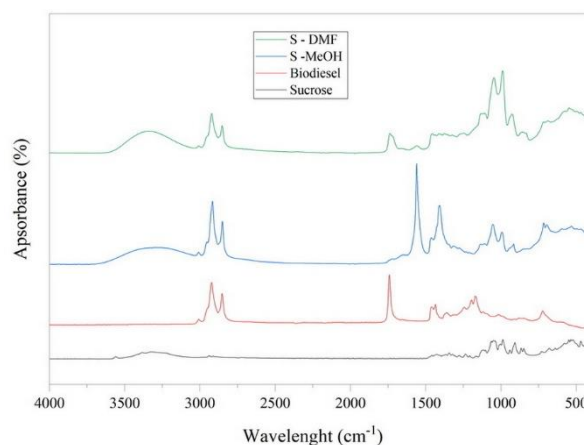


Figure 2. Comparison of FTIR spectra of reactants and synthesis products

In the FTIR spectrum of biodiesel (mixture of fatty acid methyl esters – FAME), absorption maxima were identified that confirm the presence of characteristic functional groups and are in accordance with other literature sources [21]. Absorption maxima in the lower wavenumber region of 987 cm^{-1} indicate vibrations of $=\text{C-H}$ functional groups, which originate from unsaturated bonds in the acid chains of biodiesel. A specific peak at 721 cm^{-1} indicates rocking vibrations of the methylene functional group in biodiesel ($-(\text{CH}_2)_n-$). Characteristic peaks found in the region of $1016\text{-}1245 \text{ cm}^{-1}$ indicate C-O and C-O-C stretching vibrations as well as O-CH₃ bending vibrations, while absorption peaks in the region of $1360\text{-}1460 \text{ cm}^{-1}$ are attributed to C-H bending vibrations of methyl groups. The most significant peak is located around 1741 cm^{-1} , which corresponds to the stretching of the carbonyl ester group, or methylated carboxylate groups, which is a clear indication of the presence of esters formed by transesterification. Additionally, the spectrum shows pronounced peaks at 2852 cm^{-1} and 2922 cm^{-1} , which are attributed to symmetric and asymmetric C-H stretching in the alkyl chains of biodiesel.

In spectrum of sucrose, absorption maxima can be observed at: 3561 cm^{-1} , 3319 cm^{-1} and 3383 cm^{-1} , which correspond to the stretching of the O-H group from water, as well as the hydroxyl groups in sucrose; 2939 cm^{-1} and 2914 cm^{-1} , which correspond to asymmetric and symmetric stretching of C-H, while 1475 cm^{-1} , 1459 cm^{-1} and peaks in the region $639\text{-}867$

cm^{-1} correspond to the change in the angle between the C and H atoms in the C-H bond; the region $1200\text{--}900\text{ cm}^{-1}$ corresponds to the "fingerprint" of sucrose, and the region $579\text{--}470\text{ cm}^{-1}$ corresponds to low-frequency skeletal modes.

The FTIR spectra of the synthesized products S-MeOH and S-DMF differ, which shows that the synthesized products in two different solvents are not the same. The aim of these experiments was for sucrose to react with biodiesel and form a nonionic surfactant (sucrose esters). The spectrum of the surfactant S-DMF shows the presence of characteristic absorption maxima for sucrose esters, i.e. the stretching of the ester carbonyl group at 1738 cm^{-1} , and the region $3200\text{--}3500\text{ cm}^{-1}$ corresponding to the stretching of free hydroxyl groups characteristic of the sucrose structure. A set of absorption maxima characteristic of the "fingerprint" sucrose is also noticeable, of which the peaks at 1002 cm^{-1} corresponding to the disaccharide bond of the $\alpha\text{-D-glucopyranosyl}$ and $\beta\text{-D-fructofuranosyl}$ groups are particularly important; 1050 cm^{-1} and 1235 cm^{-1} corresponding to C-O stretching in C-OH groups, as well as C-C stretching in sucrose; and 1114 cm^{-1} corresponding to C-O band stretching in C-O-C bond [22]. The peaks at 2922 cm^{-1} and 2852 cm^{-1} originate from C-H stretching of the hydrocarbon chain, characteristic of biodiesel. All this indicates that sucrose esters (nonionic surfactants) have been formed.

However, in the case of using methanol as a solvent, the transesterification reaction was not as successful, so the absorption maximum of the ester group of sucrose esters was much weaker in intensity. Due to its poor solubility in methanol, sucrose did not react efficiently. Instead, K_2CO_3 caused saponification of the biodiesel, which formed potassium carboxylates. Potassium carboxylates are soaps and are anionic surfactants, which is noticeable by the appearance of absorption maxima at 1560 cm^{-1} and 1410 cm^{-1} that originate from the asymmetric and symmetric stretching of the carboxylate groups (COO^-). These absorption maxima are also observed in the spectrum of the S-DMF product, which indicates that soaps are also present in addition to sucrose esters, but here to a much lesser extent.

With increasing concentration in the solution, the pH value increases for both synthesis products (Figures 3 and 4), with the product S-MeOH showing a much higher pH value. This is consistent with the finding that the product S-MeOH is mostly composed of soap (potassium carboxylate), the hydrolysis of which produces carboxylic acid and potassium hydroxide, which gives alkalinity to the solution. The

diagram (Figure 3) shows a clearly expressed trend of increasing electrical conductivity with the concentration of the product S-MeOH, because with the increase in the concentration of this product in water, the concentration of anionic surfactant (soap) ions also increases, and thus the value of electrical conductivity.

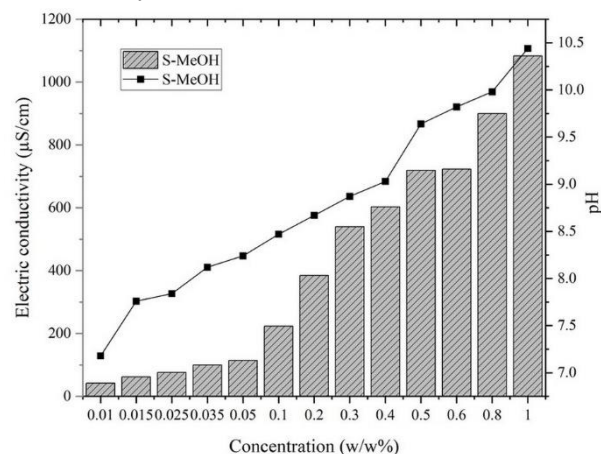


Figure 3. Dependence κ and pH on concentration of S-MeOH

Nonionic surfactants do not contain ionizing groups, so they should not show elevated values of electrical conductivity. However, the surfactant S-DMF shows low but positive values of electrical conductivity (Figure 4), which originate from smaller amounts of soap formed during the synthesis.

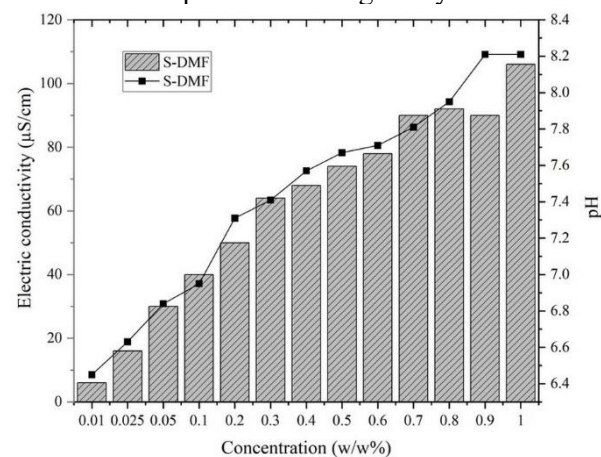


Figure 4. Dependence κ and pH on concentration of S-DMF

The surface tension of both synthesized products decreases with increasing concentration up to a certain value, after which they maintain an approximately constant value (Figure 5). This is a consequence of the adsorption of the synthesized products molecules on the phase boundary. The surface tension values of a 1% solution of the product S-MeOH (29.64 mN/m at $20\text{ }^\circ\text{C}$) and the product S-DMF (34.04 mN/m at $20\text{ }^\circ\text{C}$) indicate a significant decrease in surface tension

compared to the surface tension of pure water (72.75 mN/m at 20 °C), which confirms that both products exhibit the property of a surfactant capable of effectively reducing the surface energy at the water-air interface.

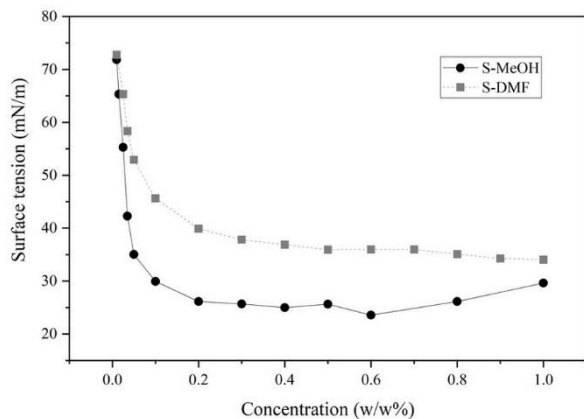


Figure 5. Dependence of σ on the concentration of S-MeOH and S-DMF

In order to determine the lowest concentration at which micelle formation occurs, surface tension values are presented as a function of the logarithm of concentration.

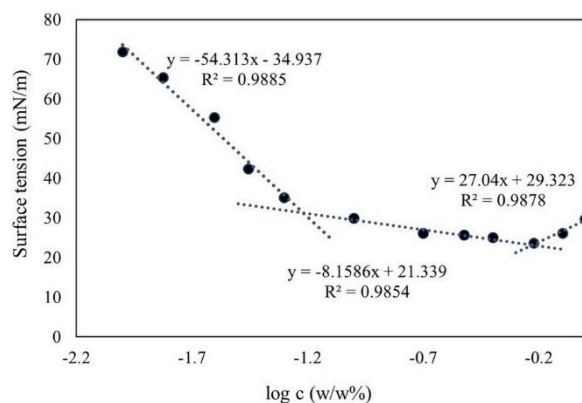


Figure 6. CMC of product S-MeOH

The mixed system of soap and sucrose esters in the solution of the synthesized product S-MeOH shows a complex behavior of surface tension depending on the concentration (Figure 6). In the region of low concentrations, a steep drop in surface tension is observed, which is attributed to the dominant adsorption of surfactant monomers at the air-water interface. With a further increase in concentration, the slope of the curve changes, indicating the beginning of the formation of mixed micelles and defining the critical micellar concentration at $c \approx 0.06$ w/w%. At concentrations higher than the CMC, the surface tension remains almost constant, but a pronounced minimum is observed ($c \approx 0.59$ w/w%), after which a

slight increase in surface tension occurs. The increase in surface tension is attributed to the post-micellar reorganization of mixed micelles, a change in their composition, as well as the possible formation of higher-order aggregates. The increase in surface tension may be a consequence of the partial withdrawal of surfactants from the interphase and a change in the efficiency of molecular packing on the surface. The dependences of pH and electrical conductivity on concentration further confirm the conclusions obtained from surface tension measurements. In the low concentration range, there is a sharp increase in pH value with concentration as a result of soap hydrolysis and the basic character of the system, but also an increase in electrical conductivity with increasing concentration due to the appearance of a large number of free ions. The change in the slope of the electrical conductivity curve with concentration in the range above 0.20 w/w% can be associated with the formation of mixed micelles, i.e. the binding of counter-ions to micelles. In this range, the increase in pH value is also slowed down, because part of the anionic groups are incorporated into the micelles and the availability of soap for hydrolysis is lower. In the higher concentration range, the increase in surface tension together with pH and conductivity indicate post-micellar reorganization of aggregates.

The diagram of the dependence of surface tension on $\log c$ of the S-DMF sample shows three linear segments, with different slopes that are a sign of a change in the dominant mechanism in the solution (Figure 7). In the low concentration range, the adsorption of surfactants at the air-water interface is dominant and the surfactant molecules are mainly present as monomers. After saturation of the interface, stable mixed micelles are formed, at a concentration $c \approx 0.18$ w/w%, which is denoted as CMC. A significantly smaller slope of the surface tension is observed at concentrations higher than the CMC, which indicates that the added surfactant participates in the formation of micelles, and no longer dominantly in the packing on the surface. The formation of micelles leads to a reorganization of the molecules, which changes the balance between free surfactant molecules and those in the micelles, and this leads to a change in the ion distribution and therefore to a gentler slope of the change in pH and electrical conductivity than the concentration. At higher concentrations, the system is in the post-micellar region, where the surface tension does not show an ideally constant value with increasing concentration, but a slight decrease, which can be attributed to the structural reorganization of the mixed micelles and a change in their composition.

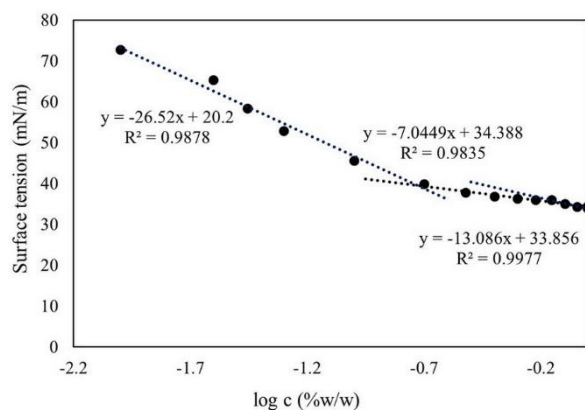


Figure 7. CMC of product S-DMF

Considering the literature data on CMC values for individual sucrose esters ($C_{12}SE$ 0.3 mM; $C_{14}SE$ and $C_{16}SE$ 0.02 mM; $C_{18}SE$ <0.01 mM [23]), it could be said that the CMC values for the products S-MeOH and S-DMF are in accordance with the literature data. However, it is difficult to find an adequate literature reference for comparison because these products were synthesized from a FAME mixture, whose CMC value depends on the ratio and type of acid residues present in the biodiesel, the degree of esterification, as well as the presence of other substances, such as soap, in these experiments.

Both synthesized products show foaming ability (Figures 8 and 9), but it is more pronounced in the S-MeOH product, in which the soap content is dominant compared to sucrose esters. In contrast, the S-DMF product is characterized by lower foaming ability, which is expected given that this product is dominated by sucrose esters, which as nonionic surfactants do not show significant foaming ability.

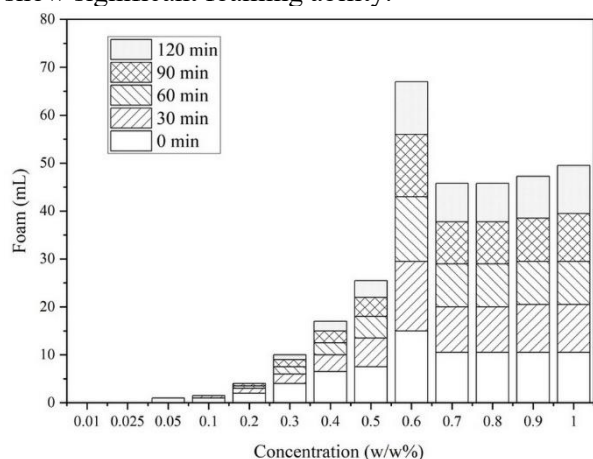


Figure 8. Dependence of foam height on time and S-MeOH concentration

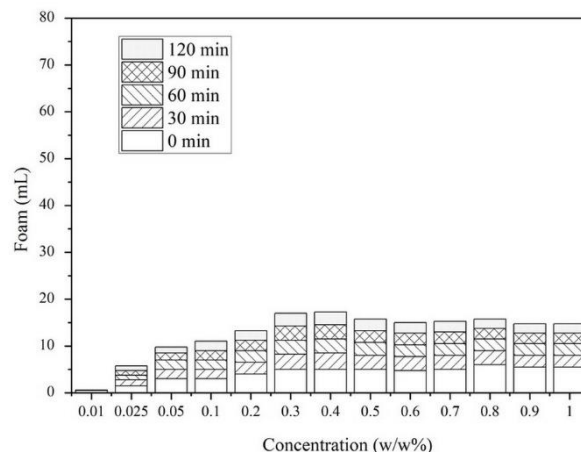


Figure 9. Dependence of foam height on time and S-DMF concentration

The height of the formed foam generally increases with increasing concentration of the product in the solution, although there are points on the diagram that deviate from this dependence. In the area close to the CMC, a pronounced increase in the height of the formed foam is observed, which can be attributed to the optimal availability of surfactant monomers in the surface layer and the beginning of micelle formation, which favors foam formation. At concentrations above the CMC, the growth of the formed foam is much weaker, which suggests that surface saturation and aggregation of surfactant molecules have occurred. The second, more pronounced foaming maximum corresponds to the post-micellar region, where the combination of minimal surface tension, stable pH and favorable ionic strength leads to maximum foam stability. Over time, the height of the formed foam decreases, but without sudden changes, which indicates its stability. Soaps mostly foam well, but their foams are often unstable, which would mean that here the small amount of nonionic surfactant present (sucrose ester) contributes a lot to the stabilization of the foam.

Figure 10 shows the values of AV, SV and EV of biodiesel and the products S-MeOH and S-DMF. The AV of biodiesel indicates the presence of free fatty acids, which may be a consequence of ester hydrolysis during the biodiesel production process or storage. A negligibly higher acid number value is observed for the product S-DMF, while the product S-MeOH has a significantly higher AV. The drastic decrease in the EV of the product S-MeOH compared to the initial reactant biodiesel indicates that most of the esters from the biodiesel reacted during the synthesis and produced already saponified products (soaps), which are not capable of further saponification reaction with KOH which was used for EV determination. The

remaining part of the product that reacted with KOH (EV=30,36 mg KOH/g) originates from the produced nonionic surfactants, specifically sucrose esters. The high SV, more importantly, high EV, along with the low AV, indicate that in the S-DMF product, most of the ester bonds of biodiesel have been replaced by newly formed ester bonds of sucrose esters.

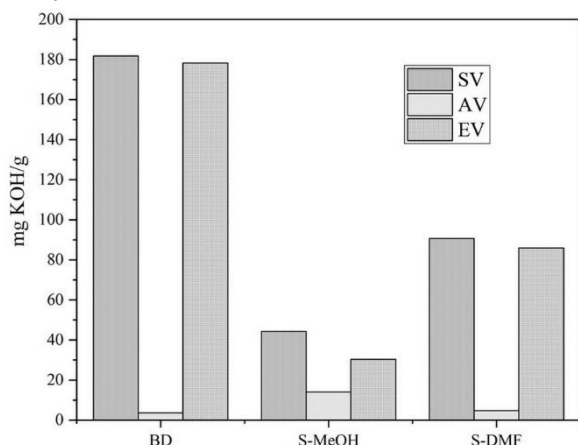


Figure 10. Results determination of SV, AV and EV

CONCLUSION

The synthesis of surfactants from table sugar and biodiesel is possible in both solvents used. The choice of solvent greatly influences the composition and behavior of the final product. During the synthesis in methanol as a solvent, the formation of soaps is dominant over sucrose esters, while in DMF as a solvent the situation is reversed, and more desirable, as sucrose esters are mostly formed. Both synthesized products showed pronounced surface activity and foaming ability, with a clearly defined critical micellar concentration, which confirms their surfactant properties and indicates their potential for application in certain formulations.

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