OPTIMIZATION OF LIQUID SOAP FORMULATION

ORIGINAL SCIENTIFIC PAPER

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ABSTRACT:
The quality of liquid soap for washing hands depends on its chemical composition, and it is evaluated by the physicochemical and functional characteristics of that product. The aim of the work is to prepare different series of liquid soap formulations by varying the concentrations of anionic and amphoteric surfactants and sodium chloride, and to optimize the formulation. The effects of the optimization were evaluated based on the results of the analysis of the physico-chemical parameters of the prepared formulations, such as density, viscosity, surface tension and critical concentration of micelles. As amphoteric and anionic surfactant concentrations in liquid soap increase, density and viscosity values increase. The value of the surface tension decreases with the increase in the concentration of the surfactants present, with the amphoteric surfactant making a greater contribution to the decrease. Regardless of certain advantages that the anionic surfactant shows in relation to the amphoteric one, the best characteristics of the liquid soap are shown by the formulation that contains a combination of both used surfactants. The addition of NaCl to the liquid soap formulation has multiple significance, but also a different effect on the physical and chemical characteristics at lower and higher concentrations. Functional and some physico-chemical characteristics of liquid hand washing soap depend on the pH value of the formulation. As the pH value increases, the surface tension and CMC increase to certain values, so pH=5.5 is taken as the optimal pH value of liquid soap, as a compromise between the values of the measured characteristics and the pH value of human skin. This research showed that the formulation containing 5.4 w/w% anionic surfactant, 1 w/w% amphoteric surfactant and 4 w/w% NaCl represents the optimal liquid soap formulation.

KEYWORDS: liquid soap; content of liquid soap; surfactants; electrolyte in liquid soap; characteristics of liquid soap; optimization of formulation

INTRODUCTION

To meet consumer expectations when buying liquid soap, it is necessary to take care of various characteristics such as washing power, pH value, density, visual appearance, and especially the viscosity of this product. Liquid soap should have an appropriate viscosity, enabling a smooth application process and ensuring even distribution and easier cleaning. Too low liquid soap viscosity values could lead to product leakage and give consumers the impression of poor cleaning. Excessively high viscosity of liquid soap affects difficult application and dispersion between hands during washing.

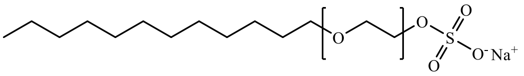
Liquid soaps are cleaning agents made of synthetic surfactants and auxiliary components, typically including anionic surfactants combined with amphoteric and nonionic surfactants. The type and relative concentration of the used surfactant determines the viscosity and other properties of the soap. In most cases, using a mix surfactants system leads to better interfacial properties, such as lower critical micelle concentration (CMC) and higher surface activity compared to single surfactants. This phenomenon is known as a synergistic effect [1], manifested by a significant increase in the viscosity of the solution [2]. Various additives, such as salts, can be added to liquid soap formulations to improve the properties of the final product. Some additives can enhance certain properties, while they can also have an adverse effect on other properties [3]. The addition of salt to an aqueous solution of surfactants changes the properties of the system, such as the critical micelle concentration, as well as the phase behavior of the surfactant solution [4].

MATERIALS AND METHODS

The experimental part of the work refers to the preparation of 5 series (A, B, C, D and E series) of liquid soap formulations and the determination of the optimal composition of the prepared formulations. The following components were used to prepare liquid soap formulations:

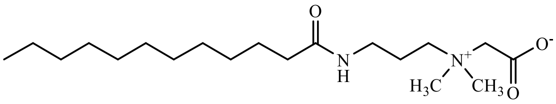
- sodium lauryl ether sulfate (Texapon N 70) – anionic surfactant (AS),
- cocamidopropyl betaine (Betadet HR) – amphoteric surfactant (AMS),
- sodium chloride (p.a.) and
- formaldehyde (36-38%, p.a.).

Sodium lauryl ether sulfate (Texapon N 70) is a highly concentrated anionic surfactant, a fatty acids alcohol derivative with 12-14 carbon atoms. It is a commercial product intended for the preparation of facial care and cleansing formulations, liquid soaps, shower gels, etc. Betadet HR is an amphoteric surfactant containing anionic (electronegative) and cationic (electropositive) polar groups. It is characterized by low toxicity, insensitivity to water hardness and compatibility with the skin. The basic roles of liquid detergents are thickening, foam reinforcement, emulsification, antiseptic action, and others.



(1)

Sodium lauryl ether sulfate



(2)

Cocamidopropyl betaine

Series A of liquid soaps was prepared as follows. A certain mass of anionic surfactant is weighed and dissolved in a glass beaker using a magnetic stirrer. A certain mass of amphoteric surfactant is measured in another glass beaker and mixed until homogenized. The prepared solutions are then mixed, and the pH value of the resulting solution is adjusted (with aqueous solution of NaOH or aqueous solution of HCl). The instrument used to measure the pH value of liquid soap formulations is a pH meter with a glass electrode (*Universal Meter, Multiline P4, WTW*). At the end, 2 drops of formaldehyde are added to the formulation to preserve it. The main goal of preparing this series of samples is to examine the influence of the pH value of liquid soap on the physico-chemical parameters and to determine the optimal pH value. Formulations of liquid soap series A are shown in Table 1.

Table 1. Formulations of liquid soap serie A

Sample	Texapon N 70 (w/w%)	Betadet HR (w/w%)	Formaldehyde	pH
A_1	5.4	1	2 drops	3
A_2	5.4	1	2 drops	5
A_3	5.4	1	2 drops	7
A_4	5.4	1	2 drops	9
A_5	5.4	1	2 drops	11

Series B of liquid soap formulations (Table 2) was prepared in a similar way as series A, with the fact that in these recipes, sodium chloride was present in different concentrations, which was added to the system after mixing the surfactant solution. All samples of serie B were adjusted to pH=5.5.

Table 2. Formulations of liquid soap serie B

Sample	Texapon N 70 (w/w%)	Betadet HR (w/w%)	NaCl (w/w%)	Formaldehyde	pH
B_1	5.4	1	1	2 drops	5.5
B_2	5.4	1	2	2 drops	5.5
B_3	5.4	1	3	2 drops	5.5
B_4	5.4	1	4	2 drops	5.5
B_5	5.4	1	5	2 drops	5.5
B_6	5.4	1	6	2 drops	5.5
B_7	5.4	1	7	2 drops	5.5
B_8	5.4	1	8	2 drops	5.5
B_9	5.4	1	9	2 drops	5.5
B_{10}	5.4	1	10	2 drops	5.5

Series of liquid soap formulations named C, D and E were prepared in the same way as series A and B. The pH value of these formulations was adjusted to pH=5.5 with the fact that these three formulations contain sodium chloride at a concentration of 4 w/w%. The main goal of preparing these groups of samples is to examine the effect of surfactant concentration on the characteristics of liquid soap formulations. Recipes of sample series from groups C, D and E are shown in Tables 3, 4 and 5.

Table 3. Formulations of liquid soap serie C

Sample	Texapon N 70 (w/w%)	Betadet HR (w/w%)	NaCl (w/w%)	Formaldehyde	pH
C_1	4.4	1	4	2 drops	5.5
C_2	6.4	1	4	2 drops	5.5

Table 4. Formulations of liquid soap serie D

Sample	Texapon N 70 (w/w%)	Betadet HR (w/w%)	NaCl (w/w%)	Formaldehyde	pH
D_1	5.4	0	4	2 drops	5.5
D_2	5.4	2	4	2 drops	5.5

Table 5. Formulations of liquid soap serie E

Sample	Texapon N 70 (w/w%)	Betadet HR (w/w%)	NaCl (w/w%)	Formaldehyde	pH
E_1	5.4	0	4	2 drops	5.5
E_2	0	5.4	4	2 drops	5.5

All samples of prepared liquid soap formulations were determined for physicochemical parameters, including density, viscosity, surface tension, and critical micelle concentration. Determining the density of liquid soap samples is based on measuring the mass *m* of a previously thermostated sample at 20 °C in a metal pycnometer with volume *V*. The density is calculated using the formula:

$$\rho = \frac{m}{V}\left[\frac{g}{cm^3}\right] \tag{3}$$

Cannon-Fensky viscometer was used to determine the viscosity of liquid soap samples. The determination of viscosity is based on measuring the time it takes for the thermostated liquid to efflux from the first to the second mark on the viscometer. The measurements were repeated 5 times, and the mean value of the liquid efflux time (t_{av}) was multiplied by the viscometer constant *C* to obtain the value for the kinematic viscosity of the tested sample, according to the formula:

$$\nu = C \times t_{av}\left[\frac{mm^2}{s}\right] \tag{4}$$

In this work, the stalagmometric method of determining the surface tension was applied. Surface tension was measured using a stalagmometer on samples prepared as solutions of liquid soaps with a concentration of 2 w/w%. After preparation, the samples were thermostated at a temperature of 20 °C. The lower part of the stalagmometer is immersed in the sample. The liquid is withdrawn above the upper mark on the stalagmometer. Counting drops starts when the liquid level coincides with the upper mark and ends when the liquid level coincides with the lower mark on the stalagmometer. The procedure is repeated several times, and the mean value is calculated.

The formula used to calculate the surface tension is:

$$\sigma_x = \sigma_{H_2O}\frac{\rho_x n_{H_2O}}{\rho_{H_2O} n_x}\left[\frac{mN}{m}\right] \tag{5}$$

where:

ρ_x - density of test liquid at the set temperature (kg/m^3),

ρ_{H_2O} - density of distilled water at the set temperature (kg/m^3),

n_{H_2O} - mean number of drops of distilled water and

n_x- mean number of drops of test liquid.

The critical micelle concentration (CMC) was determined from the graphical presentation of the measured values of electrical conductivity of liquid soap solutions of different concentrations, using a conductometer. For testing, 100 mL of liquid soap sample solution with a concentration of 1 w/w% was prepared, from which a series of dilutions of different concentrations was made. Electrical conductivity was measured from lower to higher sample concentrations at a temperature of 20 °C.

RESULTS AND DISCUSSION

The results of research are presented graphically using Microsoft Excel-prepared diagrams.

Samples of liquid soaps of series A show slight differences in the value of density and dynamic viscosity determined at a temperature of 20 ºC. The value of CMC is determined from the diagram of the dependence of electrical conductivity on the logarithm of the liquid soap concentration as a coordinate where two lines of different directions intersect. For both lines, the linear equations and the correlation factors are determined, which indicate their linearity. For sample A_1, the critical micelle concentration value is 0.203 w/w%. Sample A_1 was separated from the others as an example in order to explain the method of CMC determination (Figure 1). The following diagram (Figure 2) shows CMC values for the entire series of liquid soap samples of different pH values. This diagram shows that as the pH value increases, the CMC value increases until pH=5 (sample A_2). After pH=5, the CMC value starts to decrease again. The explanation for this can be found within the structure of the surfactants that make up the liquid soap formulation. Betadet is an amphoteric surfactant widely used in cosmetics and personal hygiene products, acting as a thickener, foam enhancer, and mildness enhancer [5]. This surfactant contains two polar groups, carboxyl (anionic) and quaternary ammonium group (cationic) [6], while anionic surfactant has one sulfated, negatively charged group. At higher pH values of liquid soap, the amphoteric surfactant behaves as an anionic compound because surfactants are deprotonated, and a negatively charged diffuse layer appears on their surface, which leads to the rejection of these substances. At lower pH values (acidic medium), the carboxyl group of the amphoteric surfactant is not ionized, but protonation of the tertiary amine of the amphoteric surfactant occurs, whereby a positively charged diffuse layer appears on the surface of this surfactant, so the surfactant behaves as a cationic surfactant in these conditions [2]. In the case of anionic surfactant, there is no change in the surface charge. The change in the surface charge of the surfactant at lower pH values causes their attraction, which results in a shift in the concentration of CMC formation to lower values [7].

The head charge of an amphoteric surfactant, combined with anionic surfactant, plays an important role in forming mixed micelles [2]. It is stated in the literature that the pH value of Betadet at which the strength of positive and negative charges are equal (isoelectric point) is 6.25, so at pH=5, it behaves slightly cationic. Therefore, the slightly positively charged molecules of the amphoteric surfactant are placed between the negatively charged head groups of the anionic surfactant, which promotes a tighter packing of the monomers [8].

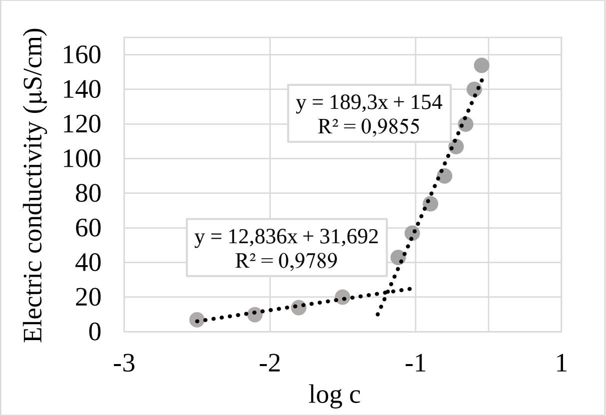


Figure 1. CMC of sample A_1

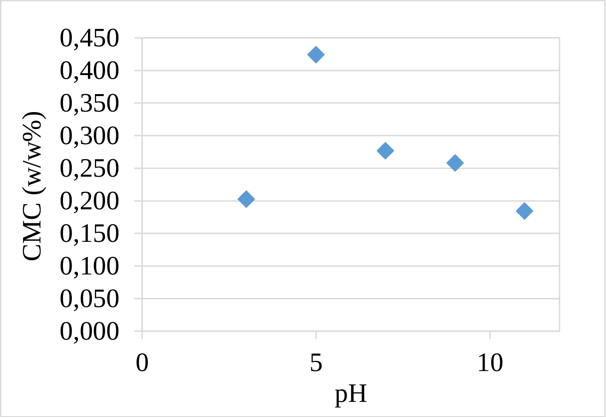


Figure 2. CMC of formulations serie A

Figure 3 shows that sample A_1 has the lowest value of surface tension, i.e., the sample whose pH value is set to pH=3. Also, it can be noticed that with an increase in the pH value, the surface tension increases to pH=7, after which it decreases slightly. In one study, it is stated that at low pH values, the CMC of sodium lauryl sulfate (anionic surfactant) decreases, and this decrease can occur either by replacing heavier ions with lighter ions or by reducing the charge on the surface of the micelle, thus changing the stability of the micelle [9]. A similar conclusion could be made for the SLES present in the samples of serie A.

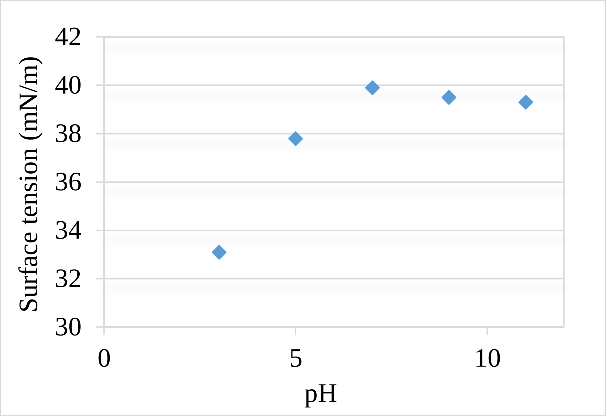


Figure 3. Dependence of surface tension on the pH value of the formulations of serie A

For the tested range of pH values, at pH=3, the liquid soap formulation has the lowest surface tension value. On the other hand, the pH value of the skin's balance is 5.5, so a soap with higher acidity would cause burning and irritation of sensitive skin. For this reason, pH=5.5 is taken as the optimal pH value of the liquid soap formulation. Therefore, all other samples were adjusted to that pH value.

Figures 4 and 5 show the dependence of density and viscosity on the concentration of sodium chloride present in the liquid soap formulation. It can be seen here that the density of liquid soap increases with the increase in sodium chloride concentration, and this growth is described by a linear function, which is also confirmed by the high value of the correlation coefficient R^2=0.9961. The change in viscosity with increasing NaCl concentration in the prepared formulations shows a characteristic shape similar to the curve of a Gaussian normal distribution. As stated in the literature, the change in viscosity is mainly a consequence of the transformation of their micellar morphology and structure [8]. The micellar structure changes from spherical to cylindrical, worm-shaped, and finally to branched micelles, which results in a non-monotonic viscosity trend in the form of the so-called "salt curve" [10]. Our research shows that the peak on the salt curve corresponds to the formulation containing 7 w/w% NaCl. Another study reported that formulations containing 7.5 w/w% sodium lauryl ether sulfate and 2.5 w/w% cocamidopropyl betaine showed a viscosity peak at 2 w/w% NaCl, where measurements were made at 22 °C [8]. Comparing them, it can be concluded that the viscosity of these systems is affected by the concentration of surfactants, temperature, the presence of co-surfactant, but also other factors such as the molecular structure of the surfactant, polarity, presence of other additives [10]. Such solutions of surfactants in which worm-like micelles are formed are used as viscosity modifiers and regulators [2].

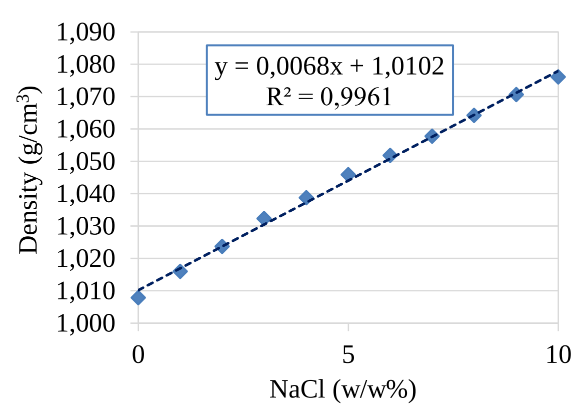


Figure 4. Density of formulations B serie

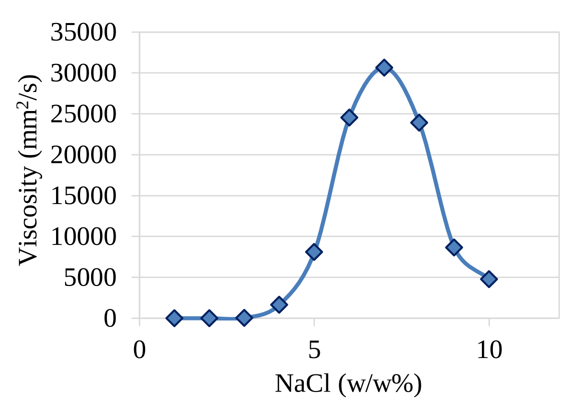


Figure 5. Viscosity of formulations B serie

After reaching the maximum on the salt curve, a further increase of NaCl concentration in the formulations leads to a decrease in viscosity due to the overlapping of worm-like micelles and the formation of a tangled network [11].

Figure 6 shows the dependence of the surface tension on the concentration of the liquid soap formulation of series B. The surface tension of solutions of liquid soaps decreases with increasing concentration of NaCl, so that at the highest concentration of NaCl it shows the lowest value of surface tension. The positive sodium cation and negative chloride anion of the electrolyte interact with the surfactant, reducing the electrostatic repulsion between the surfactant monomers in the solution. At the interface, there is more space for monomers that combine more compactly and further lower the surface tension [12]. On the other hand, when tested using a pressure piston pump, liquid soap formulations containing lower concentrations of sodium chloride showed a more uniform soap release, while the presence of higher concentrations of NaCl (5 w/w% and above) caused certain dosing disadvantages such as sliminess, stickiness, increased effort during extrusion, lumps, and the appearance of bubbles. For further tests, the formulation with 4 w/w% NaCl was selected as the formulation containing the optimum sodium chloride concentration because it performs better for user needs.

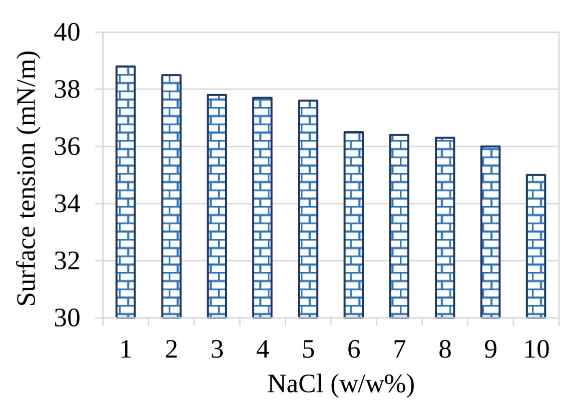


Figure 6. Surface tension of serie B

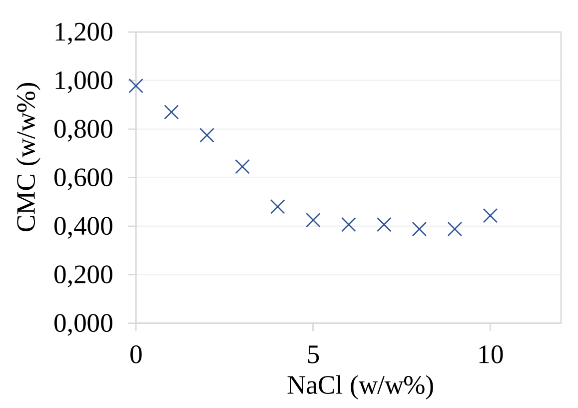


Figure 7. CMC of samples serie B

It is stated in the literature that the presence of electrolytes mainly causes a decrease in the critical micelle concentration, with this effect being most prominent with ionic surfactants. In contrast, with nonionic and amphoteric surfactants, this effect is smaller and often reversed [4]. This research showed an evident effect of the addition of NaCl on the reduction of the CMC of liquid soap made of a mixture of anionic and amphoteric surfactants (Figure 7). The addition of sodium chloride leads to a decrease in the electrostatic repulsion between the charged surfactant groups inside the micelle, which allows a denser packing of the surfactant molecules and, thus, a decrease in the CMC [12]. The change in CMC is mainly attributed to the "salting in" or "salting out" of hydrophobic surfactant groups by the electrolyte present in the aqueous system [4], whose effect depends on the charge and ion radius of the electrolyte itself [12].

The dependencies of the density and viscosity of the liquid soap on the concentration of the anionic surfactant SLES are shown using diagrams in Figures 8 and 9. With the increase in the concentration of sodium lauryl ether sulfate (SLES), the density and viscosity of the liquid soap increases. Diagram 9 shows a greater change in viscosity when looking at samples C_1 (4.4 w/w% SLES) and C_2 (5.4 w/w% SLES), where a greater interaction of the anionic surfactant with the electrolyte is assumed, in contrast to the higher concentrations, if we look at the samples C_2 (5.4 w/w% SLES) and C_3 (6.4 w/w% SLES).

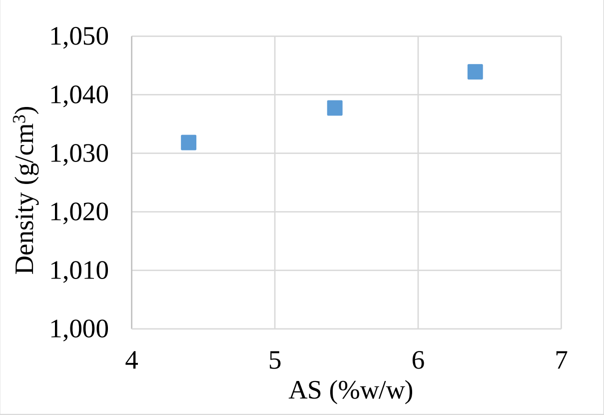


Figure 8. Density of samples serie C

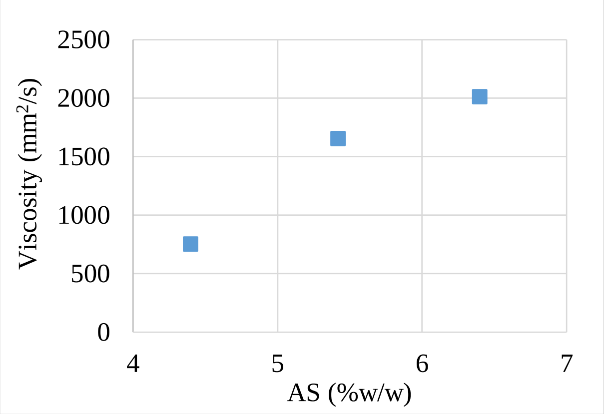


Figure 9. Viscosity of samples serie C

The surface tension measured for liquid soap formulations from series C shows very little change (Figure 10). The critical micelle concentration increases slightly with increasing SLES concentration (Figure 11). Of all tested samples of series C, sample C_3 shows the highest CMC value, which indicates that the present concentration of NaCl is not sufficient for maximum interaction with the increased concentration of anionic surfactant. In the work of Danov et al., 2004, formulations containing sodium dodecyl sulfate, alkylamidopropyl betaine and 10 mM NaCl (pH=5.5) were tested. The results of that research showed that with an increase in the concentration of anionic surfactant, the value of CMC increases, but the value of the surface tension is practically constant [13], as in this research.

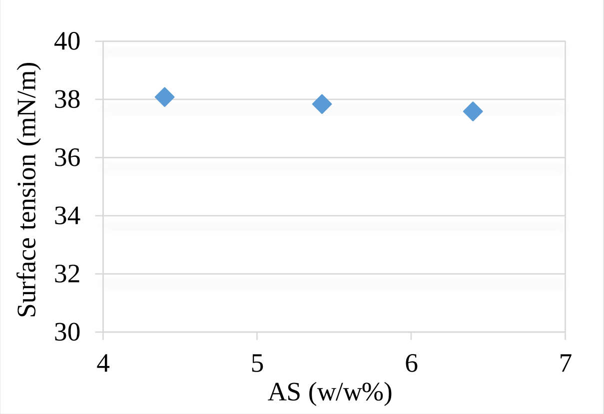


Figure 10. Surface tension of C serie

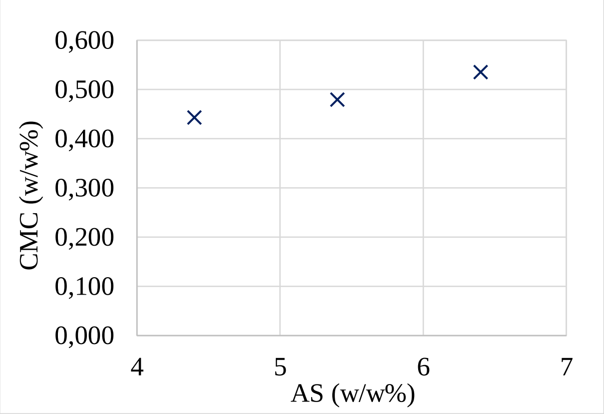


Figure 11. CMC of liquid soap C serie

Figures 12 and 13 shows that increasing the concentration of the amphoteric surfactant in the liquid soap formulation, density and viscosity increases. The synergistic effect of the mixture of anionic and amphoteric surfactants is particularly prominent in the case of an increase in the amphoteric surfactant concentration from 1 w/w% (1654 mm^2/s) to 2 w/w% (79 558 mm^2/s), which is a much higher value than the maximum viscosity on the salt curve (Figure 5). In the research of the group of authors, it was stated that the sudden increase in viscosity of mixed solutions of surfactants is attributed to the transition of spherical micelles into rod-shaped ones, as well as an increase in the size of the micelles during the transition [14]. Literature data show that the concentration of NaCl required to create a viscosity peak decreases with an increase in the concentration of the amphoteric surfactant alkylamidopropyl betaine [8],[15], which could be investigated in the continuation of the current research.

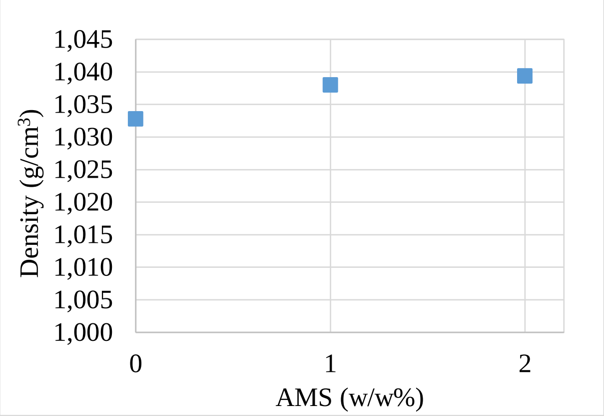


Figure 12. Density of samples serie D

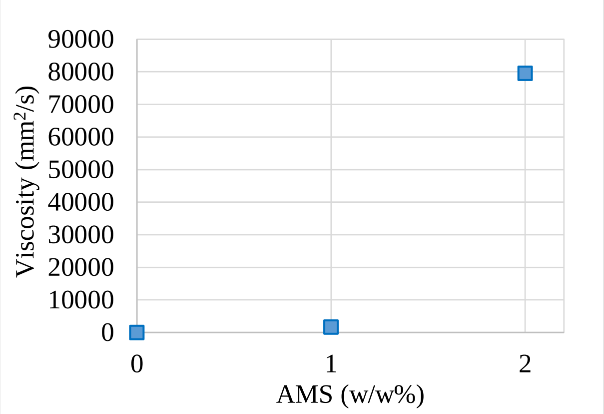


Figure 13. Viscosity of samples serie D

As Figure 14 shows, the surface tension of the solution decreases as the concentration of amphoteric surfactant in the formulation increases, while increasing the concentration of this amphoteric surfactant does not have a large effect on the reduction of CMC [12], as shown in Figure 15.

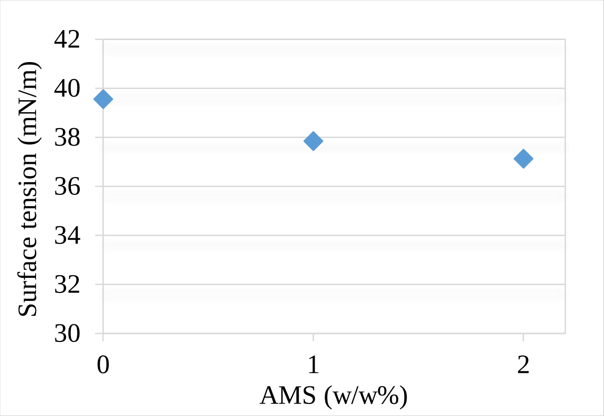


Figure 14. Surface tension of D serie

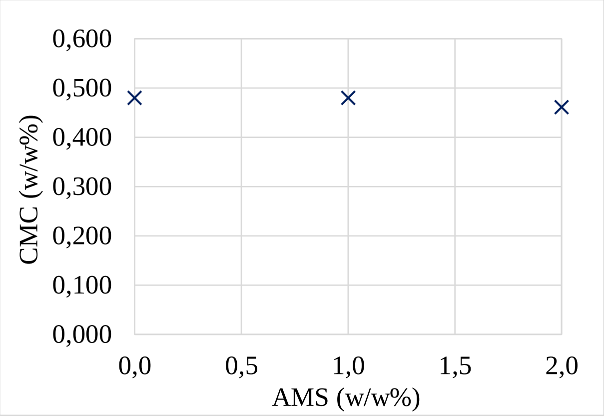


Figure 15. CMC of serie D

For the same concentration of one type of surfactant in the absence of another type of surfactant, it can be noted that the anionic surfactant contributes to a higher viscosity and density of liquid soap (Figure 16 and Figure 17) but also a higher surface tension and CMC than the used amphoteric surfactant (Figure 18 and Figure 19). Based on all the results of this test, it can be noted that mixed solutions of anionic and amphoteric surfactants in certain combinations show superior characteristics compared to solutions of individual surfactants, which coincides with the literature report [14].

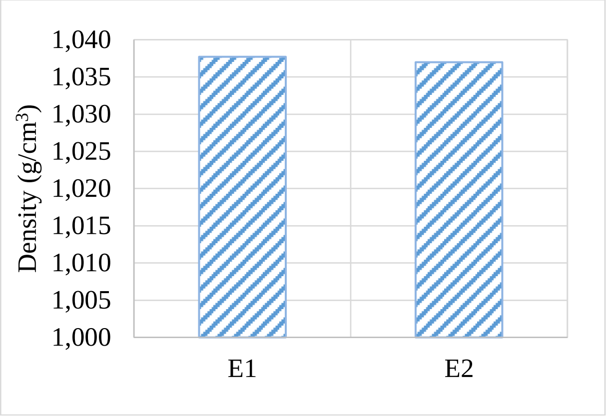


Figure 16. Density of samples serie E

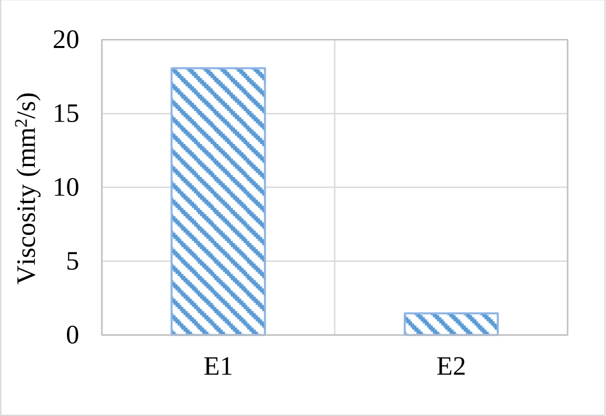


Figure 17. Viscosity of samples serie E

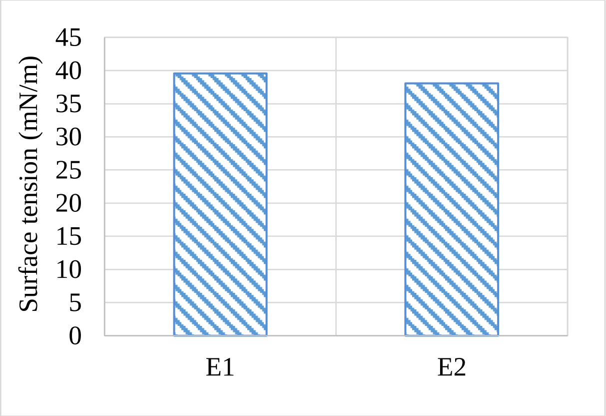


Figure 18. Surface tension of E serie

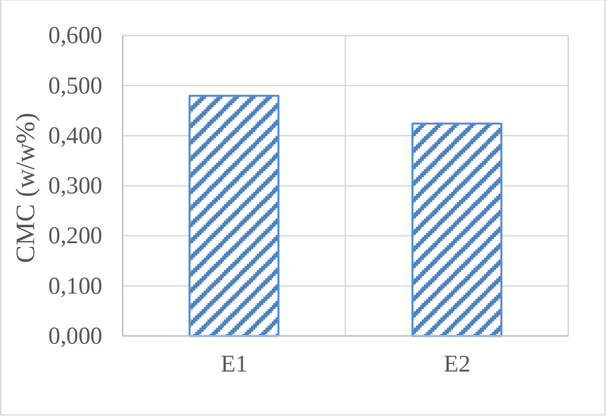


Figure 19. CMC of E serie

CONCLUSION

The functional and some physicochemical characteristics of liquid hand washing soap depend on several factors, including the pH value of the formulation. pH=5.5 is presented as the optimal pH value of liquid soap, as a compromise between the values of the measured characteristics and the pH value of human skin. As the concentrations of amphoteric and anionic surfactants in liquid soap increase, so do the density and viscosity values. The value of the surface tension decreases with the increase in the concentration of the present surfactants, with amphoteric surfactant making a greater contribution to the reduction. Regardless of certain advantages that the anionic surfactant shows in relation to the amphoteric one, the best characteristics of the liquid soap are shown by the formulation that contains a combination of both used surfactants. Adding NaCl to a liquid soap formulation has a different effect on the physico-chemical characteristics at lower and higher concentrations.

An increase in NaCl concentration leads to a change in the structure of the surfactant aggregates, which also changes the way the surfactant is packed in the micelles, which contributes to the transition of spherical micelles into rod-shaped micelles and then into wormlike micelles, which grow and overlap and create a transient interlaced network. These structural changes of the micelles are reflected in different effects on the physico-chemical characteristics, resulting in a liquid soap formulation containing 4 w/w% NaCl as the optimal concentration for an acceptable value of viscosity and stability of the formulation.

The desired properties of liquid soap require careful selection of the types and concentrations of appropriate surfactants and additives, where the formulation process still requires much time and research. In this research, the optimal formulation was presented as containing 5.4 w/w% of anionic surfactant Texapon N 70 and 1 w/w% of amphoteric surfactant Betadet HR, in the presence of NaCl (4 w/w%), at pH=5.5. For the optimal liquid soap formulation, the values of physico-chemical parameters are: density 1.0387 g/cm^3, viscosity 1654 mm^2/s, surface tension 37.7 mN/m and CMC 0.48 %w/w measured at a temperature of 20 ºC.

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