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The Emulsifying Properties of Compositions Based on Stearic Acid and Sodium Carboxymethyl Cellulose Synthesized at the Interface of Vaseline Oil/Water Solution of KOH

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Abstract

Emulsifiers of natural origin play an important role in producing stable emulsions for applications in food, pharmaceuticals, cosmetics, and other industries. In this work, the emulsifying properties of a new composite based on stearic acid, its salt (potassium salt of stearic acid) forming at the water/vaseline oil and polymer sodium carboxymethyl cellulose (Na-CMC) were investigated. Potassium stearate was formed directly at the interface of water and vaseline oil: a solution of stearic acid in vaseline oil was used as the oil phase, and a solution of potassium hydroxide in water was used as the polar phase. It was shown that at a 1% concentration of stearic acid, high stability was achieved for a 50% emulsion. The stability of the emulsion increased with the concentration of both the surfactant and Na-CMC. The effect of the composite emulsifier on the stability of the emulsion is associated with the interaction of its components and the formation of a mixed interfacial adsorption layer. It was found that the lifetime of the vaseline oil-in-water emulsion at the optimal concentrations of 1% potassium stearate and 0.25% Na-CMC exceeded three days. The results of this study contribute to understanding the formation of emulsions where a surface-active emulsifier is formed directly at the oil/water interface and stabilization occurs through the participation of polymers.

1. Introduction

Obtaining stable emulsions using of emulsifiers of natural origin is important for the production of cosmetics, food products, pharmaceuticals, and other materials [1–4]. Emulsions, as colloidal and microheterogeneous systems, are aggregative unstable due to excess free energy at the interface. The instability of emulsions is caused by the spontaneous formation of droplet aggregates, followed by phase separation into two liquid layers. Emulsion stability is determined by rate of emulsion separation or time of contact of individual droplets with each other (droplet coalescence) or with the interfacial surface. Thermodynamically, an emulsifier adsorbs at the interface and reduces interfacial

tension. Alternatively, the stabilization of emulsions can be explained by the repulsive forces that arise between droplets [5]. An emulsifying agent, or emulsifier, is a compound that adsorbs at the interface between two immiscible phases. It reduces the interfacial tension at the interfaces, and forms an adsorption film or barrier around the droplets, preventing the coalescence of droplets. Various substances of different nature are used as emulsifiers: ionic surfactants with charged polar groups, non-ionic surfactants, high-molecular compounds of amphiphilic structure, and highly dispersed powders or Pickering emulsions [6]. The type and stability of emulsions are determined by the nature of the emulsifier. The efficiency of an emulsifier is characterized by a specific parameter – the hydrophilic–lipophilic balance (HLB) – which can be calculated using established formulas. Hydrophilic emulsifiers promote the formation of o/w emulsions, while

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hydrophobic (or oleophilic) emulsifiers stabilize w/o emulsions (Bancroft's rule) [7, 8]. Stability of an emulsion strongly depends on the properties of the emulsifier (nature, HLB, length of a hydrocarbon radical etc.) in a system.

Emulsifiers of natural origin like biopolymers, natural surfactants are of special interest, since they are characterized by low toxicity and can be used to obtain emulsifying compositions that stabilize cosmetic and food emulsions. Natural emulsifying agents are mostly hydrated lipophilic colloids that are obtained from plant and animal tissues. These emulsifiers affect the protective layer around the droplets, giving them a charge that aims to repel one another and cause them to swell, increasing the fluid's consistency. Natural emulsifiers are safe, non-toxic, and mild, and they can be less expensive than synthetic ones [5, 9]. Soaps can be considered natural occurring emulsifiers [10–12] because they are synthesized from plant and animal oils through the saponification reaction. They are derived from natural fats and oils such as stearic, palmitic, and oleic acids. When fatty acids react with alkalis, they form sodium or potassium salts, which are the actual molecules of soap. The development of natural emulsifiers has attracted increasing interest due to the growing emphasis on sustainability [13].

Currently, there is growing interest in composite materials capable of effectively modifying the key properties of various types of dispersed systems. This interest arises from their broad application in oil and petroleum refining, medicine, food production, the paint and coatings industry, light industry, and other sectors of the economy. In practice, emulsifiers are usually composed of mixtures of several components that together provide the required set of physicochemical properties [14–15].

Composite emulsifiers consisting of surfactants and polymers are of great interest. They are promising for preparation of new highly effective emulsions due to the possibility of formation of structures at the interface. Therefore, to regulate the properties of emulsions, studying the features of adsorption of polymers, surfactants and their complexes at the interface is relevant for modern colloid chemistry. The polyelectrolytes used are capable to form a strong protective layer on the droplet surface [12]. Polyelectrolyte complexes of surfactants at the oil/water interface exhibit high surface activity and they generate gel-like structured adsorption layer. The study of synthetic polyelectrolytes and surfactant associates is important for determination of their influence on the conformational state of

macromolecules [16–18]. Since common polymers cannot fully satisfy the needs of growing production and economy, there is a great demand in studies aimed at obtaining new types of emulsifying polymers and surfactants. One of the interesting novel approaches is the synthesis of an emulsifier directly at the interface like in heterophase polymerization. The key aspect of this process is the formation of surfactants at the interface between an organic solvent and water during polymerization [19, 20]. The solubility of surfactants in the different phases (water and organic phase) affects the stability and particle size distribution of the polymers suspensions [21, 22].

In this study, the synthesis of a vaseline emulsion, including emulsion polymerization of vaseline with potassium cations of stearic acid as an emulsifier was investigated. Stearic acid, a typical fatty acid, is an inexpensive, naturally occurring surfactant with a wide range of applications [23]. The potassium salt of stearic acid was obtained directly by adding potassium hydroxide to the aqueous phase and stearic acid to the oil phase, leading in their neutralization at the interface.

Sodium carboxymethyl cellulose (Na-CMC) was used as a water-soluble polymer to study the emulsifying properties of the surfactant/polymer composition. Na-CMC is a biopolymer of natural origin and is applicable in cosmetic and food emulsions. Soaps are more natural than synthetic surfactants (alkyl sulfates, alkyl sulfonates) and considered readily biodegradable [24, 25]. In this work, we examined the emulsifying properties of the polymer and surfactants with non-toxic properties suitable for use in the cosmetic industry and the production of detergents. Vaseline oil, used as the oil phase, can also serve as a component in cosmetic and pharmaceutical emulsions. The development of new approaches aiming to reduce toxicity and stabilize emulsions is a major area of interest within the field of cosmetic and pharmaceutical industries [25, 26].

The aim of this work is to study the emulsification process using natural emulsifiers – stearic acid and sodium carboxymethyl cellulose – at the interface between vaseline oil and an aqueous KOH solution. The distinctive feature of this study is the synthesis of the surfactant, potassium stearate, at the oil-water interface. The formation of the emulsifier at the interface makes it possible to control interfacial tension, dispersion, and the microemulsification of water-insoluble components, as well as to form a structured interfacial adsorption layer.

2. Materials and Methods

2.1. Materials

Sodium carboxymethyl cellulose Na-CMC- $[C_6H_7O_2(OH)_{3-x}(OCH_2COOH)_x]_n$ (JSC Karbokam, Russia) was used. Vaseline oil (JSC Pharmacy, Kazakhstan) was used without additional purification. Stearic acid ($C_{17}H_{35}COOH$) and potassium hydroxide (KOH), both of analytical grade, were also used without further purification.

2.2. Methods

To obtain composite emulsifiers, oil in water emulsions of vaseline were obtained at different ratios of oil and water phases (9%, 20%, 33%, 44%, 50%). The stability of emulsions is determined by the volume of phases separated from emulsion, which determines its "lifetime". Test tubes containing the emulsions were placed on a stand, and a stopwatch was started. The volumes of water and oil separated into distinct layers over a period of time (τ) were monitored for several hours.

The experimental data are presented a graph of H versus time (τ), where H represents the percentage of the dispersed phase separated relative to the total emulsion volume, and α denotes the absolute amount of the dispersed phase separated.

$$H = \frac{\alpha \cdot 100\%}{V},$$

where H is the percentage of the separated phase (water), %; α is the volume of the separated phase (water), ml; V is the total volume of the emulsion, ml.

By extrapolating the initial linear portion of the obtained curves to the ordinate at H = 100%, the point corresponding to the emulsion lifetime (A) is determined on the abscissa (Fig. 1) [27].

Interfacial and surface tensions were measured using the stalagmometric method. The viscosity of the aqueous solutions was determined with an Ubbelohde viscometer (suspended level type) at 298 K. The efflux time of the solvent (water) was 120 sec.

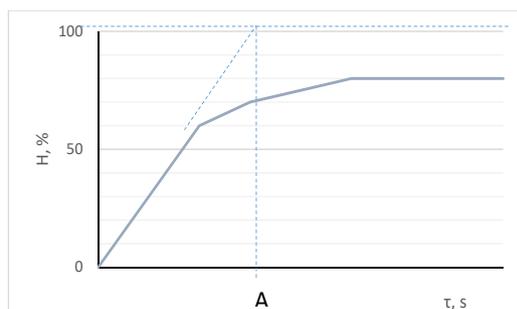


Fig. 1. The determination of A (the lifetime of emulsions).

3. Results and Discussions

The surface and interfacial tension values between the contacting phases (vaseline oil and water) are critical for emulsion preparation. Therefore, the surface and interfacial tensions of the surfactant and Na-CMC polymer solutions were measured at 60 °C (Figs. 2–4).

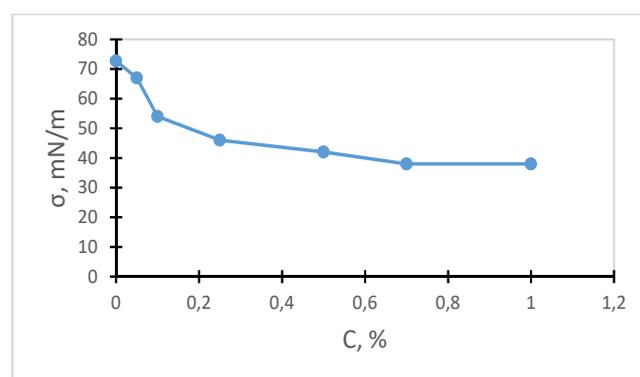


Fig. 2. Interfacial tension of stearic acid at the vaseline oil/water interface. T = 60 °C.

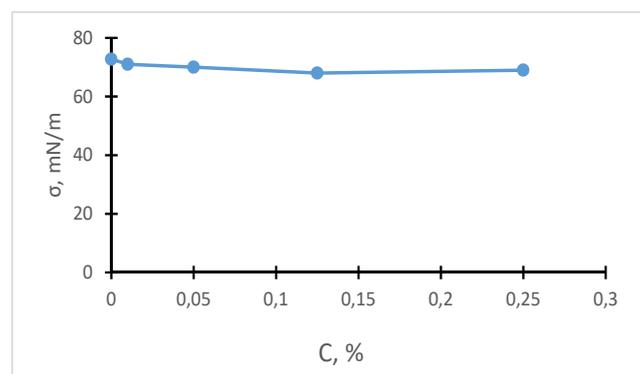


Fig. 3. Isotherm of surface tension of aqueous solutions of Na-CMC. T = 25 °C

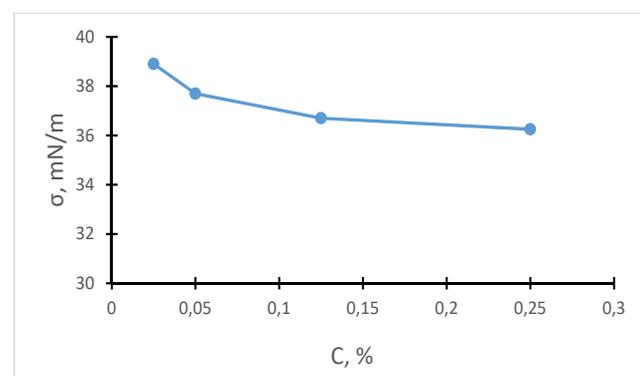


Fig. 4. Isotherm of interfacial tension of NaCMC at the interface of vaseline oil/water in the presence of 1% stearic acid. T = 60 °C.

As shown in Fig. 4, a 1% stearic acid solution mixed with different concentrations of Na-CMC reduces the interfacial tension of the system, like an individual surfactant (Fig. 2). The decrease of interfacial tension occurs due to the formation of an adsorption layer consisting of Na-CMC macromolecules and surfactant molecules adsorbed from the oil phase. When a polyelectrolyte is added, surfactant molecules interact with the polymer chain with their hydrophobic radicals, forming micellar aggregates, and as a result, a decrease in the critical micelization concentration of the surfactant is observed.

To obtain emulsions, a solution of stearic acid in vaseline oil was used as the oil phase, and a solution of potassium hydroxide in water was used as the polar phase. Vaseline oil was used as the oil phase because of its availability due to its low cost and toxicity. In this regard, water-in-vaseline oil emulsions stabilized with surfactants and polymer-surfactant compositions were obtained.

To study the stability, emulsions stearic acid dissolved in vaseline oil and KOH dissolved in water were prepared (Figs. 5–9).

As shown in Fig. 5, at a 1% concentration of stearic acid, high stability was achieved for the 50% emulsion. At this surfactant concentration, the emulsion stability can be attributed to the formation of an adsorption layer on the surface of the dispersed phase droplets, which prevents coalescence and enhances overall system stability.

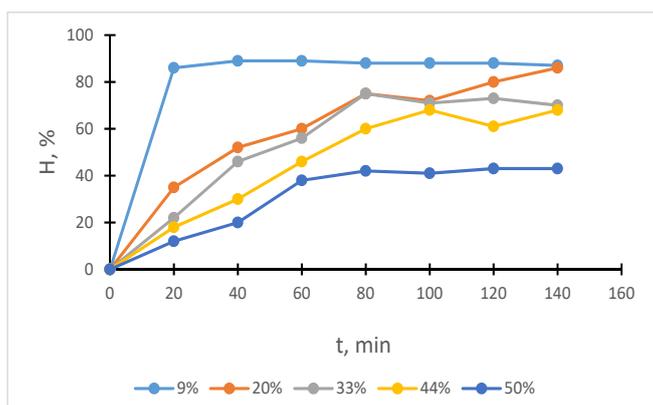


Fig. 5. Kinetics of breaking of emulsions of 1% stearic acid in vaseline oil with 0.5 N aqueous KOH solution.

The data presented in Figs. 5–8 show that the emulsion stability increases with the rising concentration of stearic acid in the emulsion system. The stability of emulsions is explained by the formation of a structural-mechanical barrier in the interphase layer, as well as the presence of an electrostatic factor affecting the stability of model emulsions.

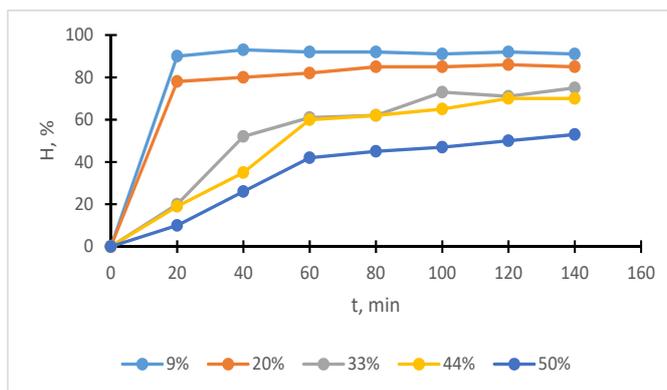


Fig. 6. Kinetics of breaking of emulsions of 0.5% stearic acid in vaseline oil with 0.5 N aqueous solution of KOH. Emulsion concentrations: 1 – 9%; 2 – 20%; 3 – 33%; 4 – 44%; 5 – 50%.

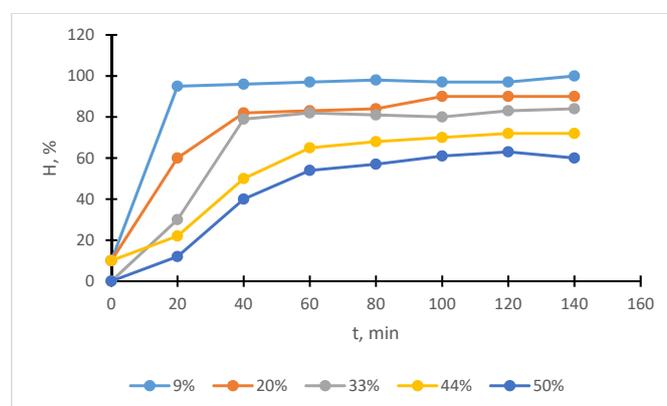


Fig. 7. Kinetics of breaking of emulsions of 0.25% stearic acid in vaseline oil with 0.5 N aqueous KOH solution.

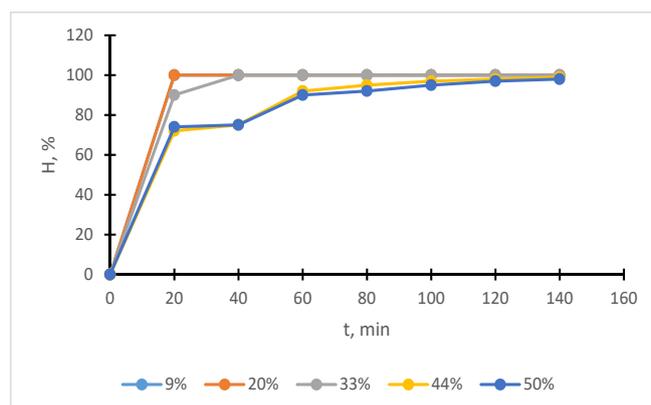


Fig. 8. Kinetics of breaking of emulsions of 0.1% stearic acid in vaseline oil with 0.5 N aqueous KOH solution.

Figure 9 illustrates that the stability of the 50% emulsion is influenced by the stearic acid concentration. It can be seen that increasing the concentration of stearic acid leads to a corresponding improvement in emulsion stability.

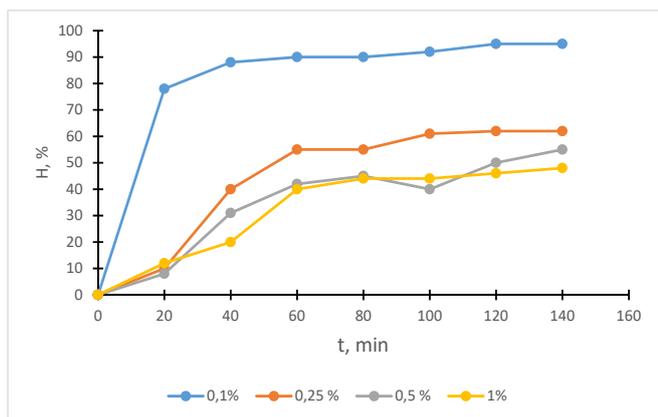


Fig. 9. Kinetics of breaking down of 50% emulsions of stearic acid of different concentrations in vaseline oil with an aqueous solution of KOH. Concentration of stearic acid in vaseline oil: 0.1% (1); 0.25% (2); 0.5% (3); 1% (4).

According to the initial kinetics of the destruction of emulsions (Fig. 10), it is shown that the emulsions initially begin to quickly separate into two phases, and then the value of K stabilizes, where K is the rate of emulsion destruction ($K = h/t$), K is calculated as the ratio of the emulsion separation height (h) to the time (t) during which this separation occurred.

In the polymerization of surfactants at the interface, alkalis are usually introduced by pre-dissolving the initial emulsion component – a long-chain carboxylic acid – in a monomer and then mixing it with the aqueous phase [20]. In this case, a highly dispersed monomer emulsion can be obtained due to a strong decrease in surface tension as a result of the neutralization reaction at the interface (Fig. 3) and the formation of salts. The emulsifying agent formed at the interface spreads depending on solubility between a monomer and aqueous phase, which leads to intensive microemulsification of a monomer.

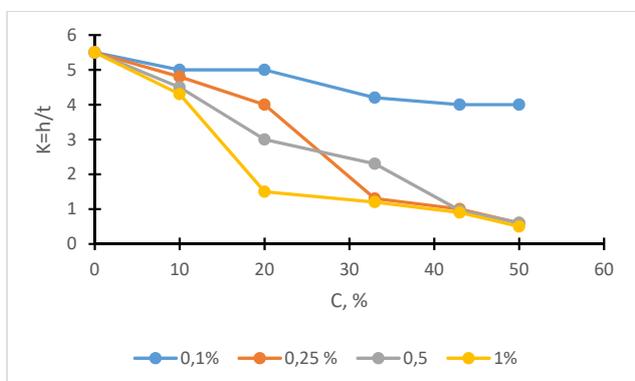


Fig. 10. Initial kinetics of destruction of emulsions of stearic acid of different concentrations in vaseline oil with 0.5 N aqueous solution of KOH. Concentration of stearic acid in vaseline oil: 0.1% (1); 0.25% (2); 0.5% (3); 1% (4).

Figure 11 shows the kinetics of emulsion breakdown for 1% stearic acid in vaseline oil at various Na-CMC concentrations in a 0.5 N aqueous KOH solution. The results indicate that increasing the Na-CMC concentration enhances the emulsion stability.

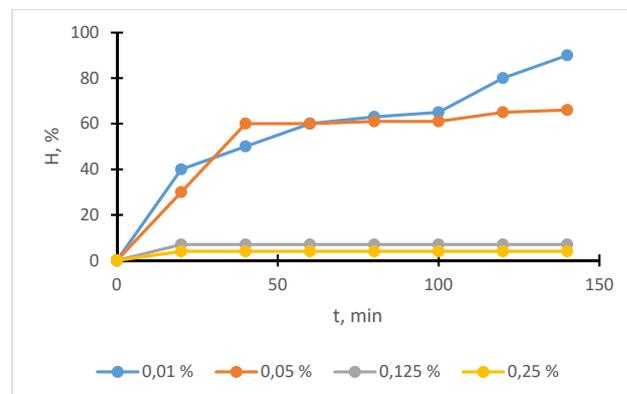


Fig. 11. Kinetics of breaking of emulsions of 1% stearic acid in vaseline oil with different concentrations of NaCMC in 0.5 N aqueous solution of KOH. Concentrations of Na-CMC: 0.01% (1); 0.05% (2); 0.125% (3); 0.25% (4).

The kinetic curves of emulsion breakdown were used to determine their lifetime (A , min). The complex formed through hydrophobic interactions between stearic acid and Na-CMC exhibits properties that differ from those of the individual components, enhancing the emulsion's stability through a synergistic effect. It was found that increasing the surfactant concentration leads to greater emulsion stability. The lifetimes of vaseline oil-in-water emulsions stabilized with individual surfactants and with the Na-CMC-surfactant composite were determined (Figs. 12–13). As can be seen, the stabilizing effect of the surfactant-polymer composite is significantly higher. Specifically, the lifetime (A) of emulsion stabilized by 1% stearic acid equals is 160 min, whereas the A for emulsions in the presence of 0.25 % NaCMC and 1 % stearic acid increases by 3.5 times ($A = 600$ min).

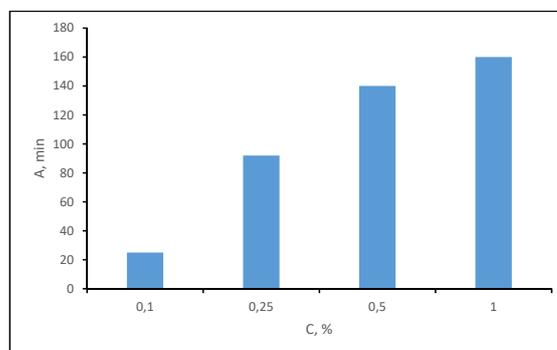


Fig. 12. The lifetime of the emulsion of a solution of stearic acid of different concentrations in vaseline oil in a 0.5 N aqueous solution of KOH.

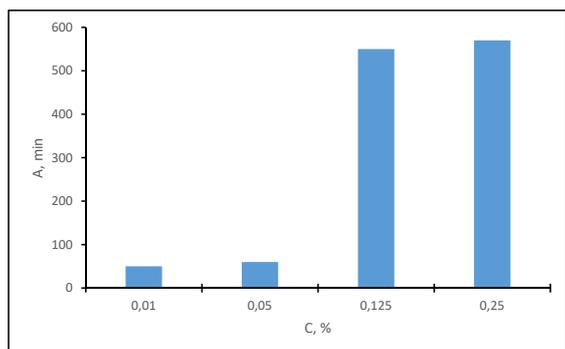


Fig. 13. The lifetime of 1% stearic acid emulsion in vaseline oil with different concentrations of NaCMC in 0.5 N aqueous KOH solution.

To characterize the obtained emulsions, the viscosities of emulsions were studied by the Ubellode method at different temperatures. Figure 14 shows the results of the dependence of emulsion viscosity stabilized by the polymer-surfactant composition on concentrations of the polyelectrolyte.

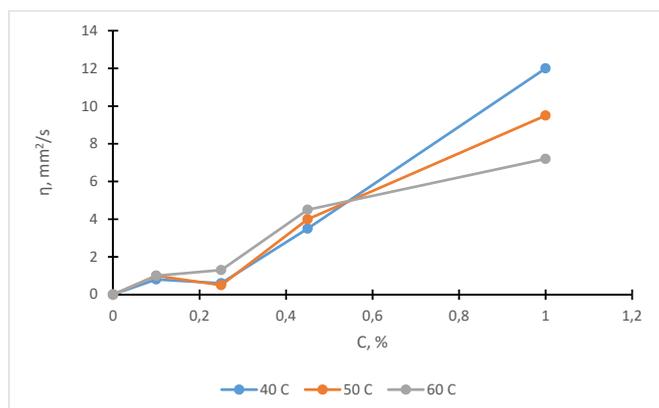


Fig. 14. Viscosity of the emulsion of stearic acid solution in vaseline oil with different concentrations of NaCMC in 0.5 N KOH solution in water: 40 °C (1); 50 °C (2); 60 °C (3).

The data clearly show that the dependence of viscosity on concentration reaches its maximum at the highest composition concentration. As the polymer concentration increases, the viscosity rises markedly, whereas an increase in temperature leads to a noticeable decrease in viscosity. With higher viscosity, the frequency of particle collisions is reduced. Thus, from the results of the study it is evident that the structural-mechanical factor plays the main stabilizing role in stabilizing the model emulsions. The surface charge of the oil droplets is determined by stabilizer adsorption. Both the structural-mechanical and electrostatic factors act in the same direction, contributing to an overall increase in emulsion viscosity.

4. Conclusions

Vaseline oil-in-water emulsions were obtained at different ratios of oil and water phases during the synthesis of emulsifiers at the vaseline oil/water interface. It was shown that with an increase in the concentration of the emulsion, its aggregation stability increases.

The effect of stearic acid and Na-CMC concentration on the stability of vaseline emulsion in water was studied. It was shown that the stability of the emulsion increases with an increase in the concentration of the surfactant and Na-CMC. It was shown that the stability of this system depends on the composition of the polymer and surfactant mixture. The effect of the composite emulsifier on the stability of the emulsion is associated with the interaction of its components and the formation of a mixed interfacial adsorption layer. The mechanism of stabilization by Na-CMC and potassium stearate at the interface is complex and can be explained by combined stabilizing effect. Potassium stearate forming *in situ* at the boundary reduces interfacial tension and allows fine droplet formation. Na-CMC being a high-molecular anionic polymer adsorbs at the interface provides the steric barrier and forms mixed adsorption layer with the surfactant molecules due to possibility of hydrophobic interaction between Na-CMC backbone and stearate alkyl chain. In addition, Na-CMC thickens the aqueous phase reducing droplet movement and sedimentation. Both Na-CMC and potassium stearate are anionic, their combined negative charge creates the repulsive forces between droplets, preventing coagulation [29]. It was found that the composition of Na-CMC and potassium stearate has a positive effect on the stability of the emulsion.

The lifetime of the vaseline oil-in-water emulsion at the optimal ratio of concentrations of 1% potassium stearate and 0.25% Na-CMC exceeded three days. The results of this work contribute to a better understanding of the emulsifying properties of the surfactant-polymer composite formed at the oil/water interface.

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