Physics of Complex Systems, 2024, vol. 5, no. 2
www.physcomsys.ru

Condensed Matter Physics.
Physics of thin films.
Surfaces and interfaces

UDC 54.057

EDN AEKJUI
https://www.doi.org/10.33910/2687-153X-2024-5-2-49-59

Molecular structure and dynamics of a water–ethanol solution of sodium dodecyl sulfate

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For citation: Lunev, I. V., Galiullin, A. A., Zuev, Yu. F. (2024) Molecular structure and dynamics of a water–ethanol solution of sodium dodecyl sulfate. *Physics of Complex Systems*, 5 (2), 49–59. https://www.doi.org/10.33910/2687-153X-2024-5-2-49-59 EDN AEKJUI

Received 12 April 2024; reviewed 26 April 2024; accepted 26 April 2024.

Funding: The work was partially carried out as part of the state assignment of the FRC Kazan Scientific Center of RAS (Yu. F. Zuev — general analysis of the results). Dielectric measurements were carried out with support from the Strategic Academic Leadership Program of the Kazan (Volga Region) Federal University (Priority 2030).

Abstract. The processes of dielectric relaxation of sodium dodecyl sulfate (SDS) solutions were studied in a range of concentrations in a binary water–ethanol solvent at various alcohol concentrations. It was shown that ethanol at concentrations below 40% does not interfere with the formation of SDS micelles, and at a higher ethanol content, surfactant micelles do not form. However, NMR data show the presence of small associates, most likely SDS dimers, the properties and mobility of which depend on the composition of the water–ethanol medium, in solutions with high alcohol concentrations. Transformations in the structure and size of the complexes observed upon changing the ethanol content in the solution are discussed.

Keywords: binary water–ethanol solutions, surfactant, SDS, structure formation, solvation, micelles

Introduction

Ethanol is present in biological systems as a product of various chemical and biochemical reactions in living systems. The same applies to a variety of amphiphilic compounds, for example, lipids and fatty acids, the spontaneous association of which is an integral step in various biochemical processes. From the point of view of fundamental and applied research, it is necessary to represent the dynamic behavior and self-association features of various chemical compounds not only in aqueous, but also in water–ethanol solutions, which can be considered as a local environment for the functioning of biomacromolecules with different polarities and microstructures. In addition, we should not forget about the use of ethanol in technological schemes, where it is used with various functional implications. Given the development of renewable energy and the increasingly widespread use of ethanol as a biofuel, interest in regulating the properties of ethanol mixtures through various structuring additives is growing. This, in particular, explains the continuous interest in water–ethanol compositions as a medium in which various organic compounds acquire new properties.

It is known that the molecular structure of binary water–ethanol mixtures does not correspond to ideal solutions. This is indicated by nonlinear variations in the physicochemical properties of water–alcohol mixtures depending on the ethanol ratio (Beddard et al. 1981; Brai, Kaatze 1992; Dutt, Doraiswamy 1992; Soper et al. 2006; Wakisaka et al. 2001). Based on data from various methods, it has been shown that microscopic phase layering of the solvent occurs in such systems (Asenbaum et al. 2012; Mashimo et al. 1991; Mijaković et al. 2011; Nishikawa, Iijima 1993; Sato, Buchner 2004). Binary water–ethanol mixtures are prone to microlayering, leading to different types of structural states of the solvent, depending on the water/ethanol ratio. For example, it was suggested (Halder, Jana 2018) that as the ethanol concentration increases, the structure of the binary solvent changes, which at low alcohol concentrations is cyclic and at high alcohol concentrations is a chain structure of hydrogen-bonded molecular associates. It was found (Hu et al. 2010a; 2010b) that the ethanol/water binary solvent consists of four components: water associates, ethanol associates, and two types of water–ethanol complexes of different composition and structure.

Due to a continuous thermodynamic process of formation and destruction of hydrogen bonds in the volume of such liquids, the properties of ethanol/water systems are determined by the relative average contribution of at least four components with different types of ordering, the predominant composition of which changes as the composition of the binary medium changes. Such microheterogeneity affects both the solvation (Faizullin et al. 2017; Konnova et al. 2013) of solute molecules and the association processes of dissolved molecules. Therefore, in the solutions of organic substances in binary media, competition between hydrophilic and hydrophobic groups of each of the three components (water, alcohol and solute) depends on the ratio between them and affects the formation of supramolecular structures in such a solution.

The study of the structural-dynamic behavior of relatively simple amphiphilic surfactant molecules in aqueous ethanol media allows us to deepen our understanding of the behavior of more complex systems, for example, macromolecules of biological nature in living systems (Gubaidullin et al. 2016). Using the example of one of the most common surfactants, sodium dodecyl sulfate (SDS), this work examines the features of structure formation in SDS/ethanol/water systems.

Sodium dodecyl sulfate is a convenient model sample that makes possible a thorough study of the surfactant micelle formation process (Zueva et al. 2022). It has been studied in detail by NMR and conductometry methods, with the CMC and Krafft point values known for it (Gnezdilov et al. 2011; Zuev et al. 2007; Zueva et al. 2020). Some works (Buchner et al. 2005; Zuev et al. 2024) have obtained the values of dielectric parameters for aqueous SDS solutions. The properties of SDS in binary water–ethanol solvents have also been studied by NMR (Zueva et al. 2021). An analysis of reference literature shows that SDS solutions in binary solvents have been mainly studied by NMR methods and much less frequently using dielectric spectroscopy.

Below the critical micelle concentration (CMC), SDS molecules are predominantly in the molecular form, which is characterized by the highest self-diffusion coefficient D. As the surfactant concentration increases to CMC, the solution begins to form SDS micelles with an aggregation number N and a shape close to spherical (Zuev et al. 2007; Zueva et al. 2021). Based on the CMC of SDS, we selected surfactant concentrations of 3, 8, 20, 50 and 100 mmol×L^{-1}, which enabled us to quickly assess structural and dynamic changes in the surfactant solution during transition from a molecular to a micellar solution.

It is known (Wakisaka, Ohki 2005; Wakisaka et al. 2001; Zueva et al. 2021) that water–ethanol mixtures have three characteristic concentration ranges of 0–10%, 25–40% and 60–92.3%, which share the existence of the binary water–ethanol solvent in different energetic and structural states. These states differ in the microheterogeneous structure of the solvent and in the geometry of the hydrogen bond network. This can affect the process of surfactant micelle formation and the structure of its associates. For this reason, corresponding ethanol concentrations in our samples were chosen as 0, 10, 40 and 80 vol.%.

The purpose of this work is to study the dynamic structure of water–ethanol SDS solutions using dielectric spectroscopy.

Materials and methods

Materials

We used sodium dodecyl sulfate (SDS, L-4509, Sigma-Aldrich) with the main component accounting for at least 98.5% without additional purification. To prepare water–ethanol solutions, we used distilled water, additionally purified using the Milli Q system, and ethyl alcohol (95%, Medkhimprom OJSC, Russian

Federation). Samples were prepared with an SDS content of 3, 8, 20, 50 and 100 mmol×L^{-1} at ethanol concentrations of 0, 10, 40 and 80 vol.%.

Dielectric spectroscopy

All measurements of the dielectric spectra of aqueous alcohol solutions containing surfactants were carried out using a PNA-X Network Analyzer N5247A (Agilent Technologies, USA) (Fig. 1). The experiment was carried out in the temperature range of +20 to +50 °C in increments of 3 °C. Temperature was regulated using a LOIP LT 900 thermostat, into which test tubes with the solution were immersed. The accuracy of maintained temperature was ±0.1 °C.

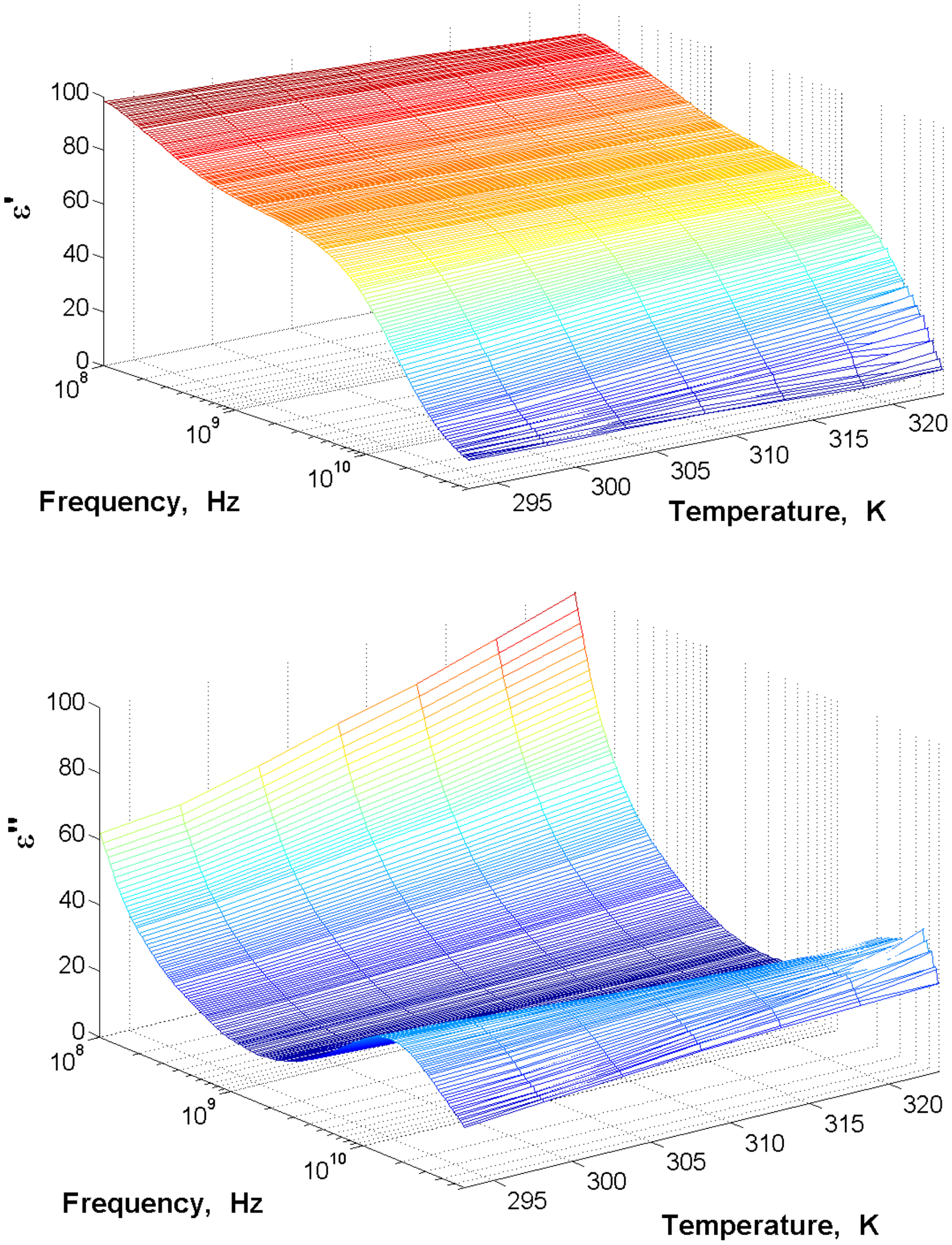

Fig. 1. Spectra of the real and imaginary part of the permittivity of an aqueous solution with an SDS concentration of 100 mmol×L^{-1} at different temperatures

Fig. 2 shows typical dielectric spectra of an aqueous solution with an SDS concentration of 100 mmol × L^{-1} at a temperature of 35 ˚C. Dielectric spectra were approximated by the superposition of two Cole–Cole functions and through conductivity in the Datama software package (Axelrod et al. 2004):

$$\varepsilon^*(\omega)=\varepsilon'(\omega)-i\varepsilon''(\omega)=\varepsilon_\infty+\frac{\Delta\varepsilon_{solvent}}{1+(i\omega\tau_{solvent})^{\alpha_{solvent}}}+\frac{\Delta\varepsilon_{SDS}}{1+(i\omega\tau_{SDS})^{\alpha_{SDS}}}+\frac{\sigma}{i\omega\varepsilon_0}, \quad (1)$$

where $\Delta\varepsilon$ is the increment of permittivity, τ is the relaxation time, α is the distribution coefficient of relaxation times, with $(0 < \alpha < 1)$, ε_∞ is the maximum high-frequency dielectric constant, σ is the conductivity of the sample, ε_0 is the dielectric constant of vacuum and ω is circular frequency.

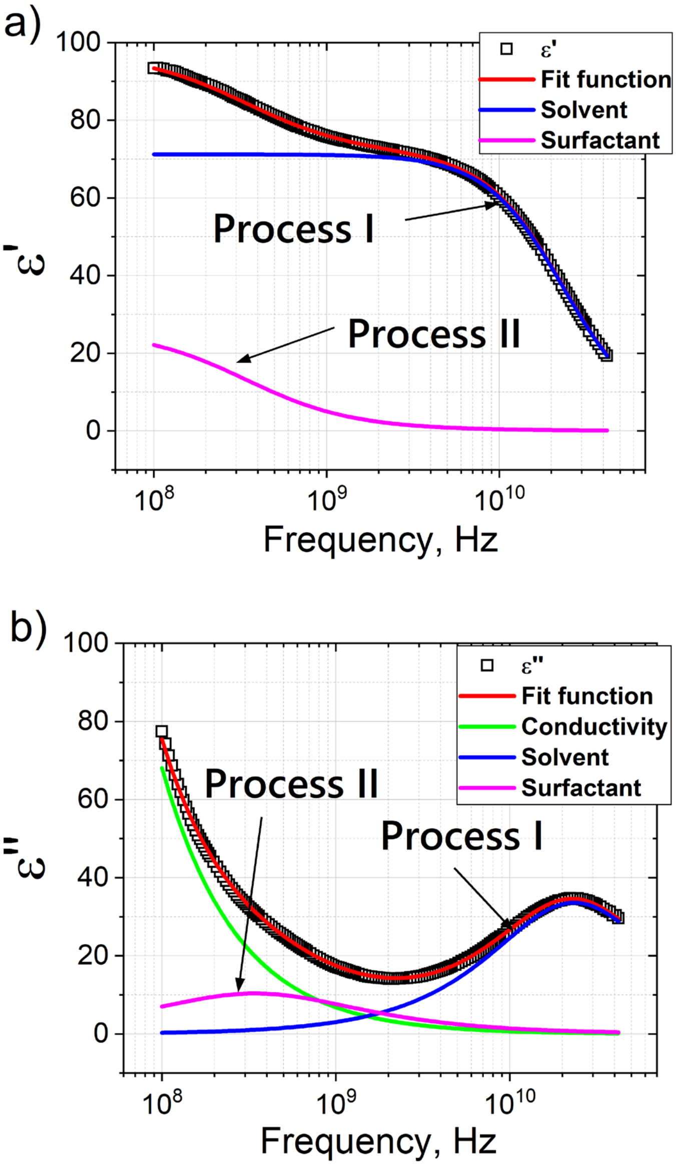

Fig. 2. Real (a) and imaginary (b) parts of the dielectric spectrum of an aqueous solution with an SDS concentration of 100 mmol × L-1 at a temperature of 35 ˚C. Hollow squares show experimental data; the red line denotes the fitting function (see equation (1)); the blue line — relaxation process 1, which corresponds to the solvent; the magenta line — relaxation process 2, which corresponds to SDS relaxation; and the green line — through conductivity

Discussion

Polarization

As a result of approximating the experimental data by equation (1), the dielectric increment $\Delta\varepsilon$ and relaxation time τ were determined. Fig. 3 shows the dependences of $\Delta\varepsilon(C_{SDS})$ at different ethanol proportions in the solvent. Let us analyze the obtained dependencies.

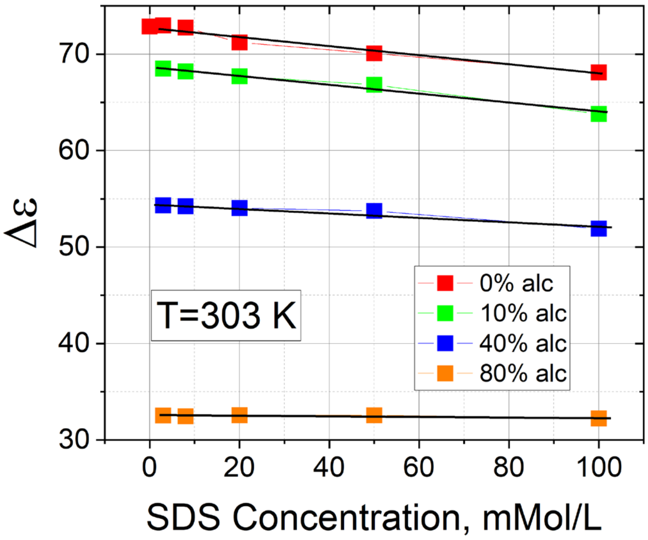

Fig. 3. Dependence of the parameter Δε (solvent) on SDS concentration in a solution with a different ethanol content; red squares — 0% ethanol; green squares — 10% ethanol; blue squares — 40% ethanol; orange squares — 80% ethanol. Data are given for a temperature of 303 K

Let us consider the dependence of Δε of the solvent on the SDS concentration in the absence of alcohol (Fig. 1a, red symbols). We can see that at a low SDS content (3 mmol × L^{-1}, 8 mmol × L^{-1}), the value of Δε practically does not change (*Δε* ≈ 73); with an increasing concentration, Δε decreases. Apparently, after passing the CMC point (~8.3 mmol × L^{-1}), micelles that bind part of the water (hydrate water) are formed, excluding it from the polarization process, which leads to a decrease in the Δε value.

Let us analyze the dependence of Δε of the solvent on the concentration of SDS with an alcohol content of 10% (Fig. 3a, green symbols). The presence of alcohol reduces permittivity of the solvent (*Δε* ≈ 68). It is known that surfactant micelles are formed due to a hydrophobic effect, when non-polar hydrocarbon radicals of a surfactant 'hide' from a polar solvent, forming spheroidal particles isolated from the solvent by a monolayer of polar head groups of the surfactant and a layer of hydration water (Romsted 2014; Rosen, Kunjappu 2012; Zueva et al. 2022). The presence of 10% ethanol in the water does not prevent the formation of micelles. The resulting micelles hydrate the solvent molecules, which leads to a decrease in *Δε* of the solvent. We cannot say which molecules, water or alcohol, are bound by micelles; however, it is known from literature that alcohol molecules can also be incorporated into the structure of micelles (Arkhipov, Idiyatullin 2012).

Now let us consider the dependence of Δε of the solvent on the concentration of SDS with an alcohol content of 40% (Fig. 3a, blue symbols). Alcohol concentration of 40 vol.% leads to a decrease in Δε of the solvent (Δε ≈ 54). The value of Δε practically does not change at SDS concentrations of 3 mmol × L^{-1}, 8 mmol × L^{-1} and 20 mmol × L^{-1}. Apparently, at this alcohol content, the formation of micelles is difficult. Therefore, a decrease in Δε of the solvent occurs only at SDS concentrations of 50 mmol × L^{-1} and 100 mmol × L^{-1}, i. e. at such an SDS content a number of micelles are still formed, which hydrate some of the solvent molecules.

Finally, let us analyze the dependence of Δε of the solvent on the concentration of SDS with an alcohol content of 80% (Fig. 3a, orange symbols). The dependence $\Delta\varepsilon(C_{\text{SDS}})$ shows that at this alcohol content, Δε of the solvent has a fairly low value (Δε ≈ 32). The *Δε* value remains practically unchanged over the entire range of SDS concentrations. This may indicate that micelles are not formed in this case, so the solvent molecules remain in a free state and all participate in the polarization process.

The effect of the solvent on micelle formation can be assessed by the slope of the $\Delta\varepsilon(C_{\text{SDS}})$ dependence in Fig. 3. The efficiency of interaction between a pair of ions depends on the permittivity of the solvent (Holmberg et al. 2002):

$$u_{eff}(r) = q_i q_j \big/ (4\pi\varepsilon_0 \Delta\varepsilon_r r), \tag{2}$$

where $u_{eff}(r)$ is the interaction potential of two-point charges, q_i and q_j are the magnitudes of the charges, ε_0 is the dielectric constant of vacuum, $\Delta\varepsilon_r$ is the increment of permittivity, and r is the distance between the charges.

Therefore, a decrease in $\Delta\varepsilon$ leads to an increase in electrostatic repulsive forces and complicates the process of micelle formation. Accordingly, the greater the slope of the $\Delta\varepsilon(C_{\mathrm{SDS}})$ dependence, the more efficient the micellization process. Fig. 4 shows the dependence of the slope of the $\Delta\varepsilon(C_{\mathrm{SDS}})$ curves on the concentration of ethanol in the solvent.

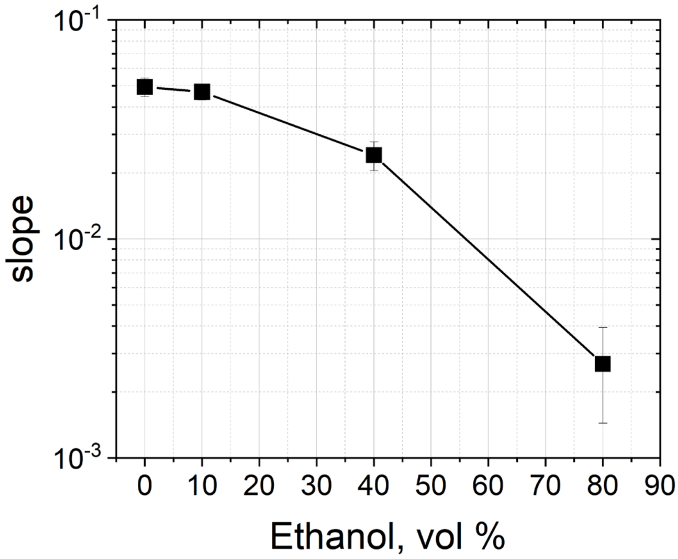

Fig. 4. Dependence of the slope of the $\Delta\varepsilon$ (C_{SDS}) curves on ethanol concentration in the solvent

Based on the dependence shown in Fig. 4, one can judge the efficiency of micellization.

Relaxation time

Let us consider the dependence of the relaxation time τ of the solvent on the SDS concentration in a solution with different ethanol content shown in Fig. 5. We can see that with increasing ethanol content the curves shift to the region of longer times. Depending on the SDS content, the τ values fluctuate around a certain average value. Apparently, the relaxation time of the solvent is practically independent of the SDS content, at least in the concentration range of 3–100 mmol × L^{-1}.

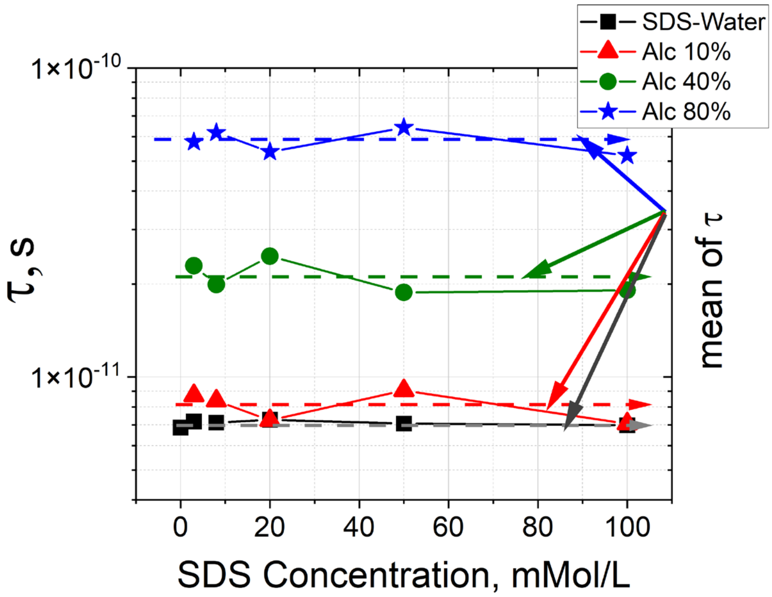

Fig. 5. Dependence of the relaxation time τ (solvent) on SDS concentration in a solution with different ethanol content; black squares — 0% ethanol; red triangles — 10% ethanol; green circles — 40% ethanol; blue stars — 80% ethanol. Data are given for a temperature of 303 K. The dotted line indicates the average values

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To assess the effect of SDS content on the relaxation time of the solvent, the averaged values of τ over the SDS concentration were calculated. Fig. 6 shows a comparison of the dependences of the average relaxation time $<\tau>$ of a solvent with different SDS content on the ethanol content in the solution with the concentration dependence of the relaxation time τ for a water–ethanol mixture (T = 303 K). We can see that with an alcohol content of 80%, the $<\tau>$ and τ values are practically the same, within the error of determining $<\tau>$. With a further decrease in the proportion of alcohol, the $<\tau>$ values are slightly shorter than τ values. This can be explained by the fact that when the alcohol content decreases, a larger number of micelles are formed, and the number of counterions correspondingly increases, which, in its turn, increases the through conductivity of the system. For the hydrogen bond network of the solvent, ions are a kind of defects that break the bonds, which leads to the so-called 'blue shift' (Feldman, Ben Ishai 2021; Levy et al. 2012), i. e. shortening of the relaxation time, as observed in Fig. 6.

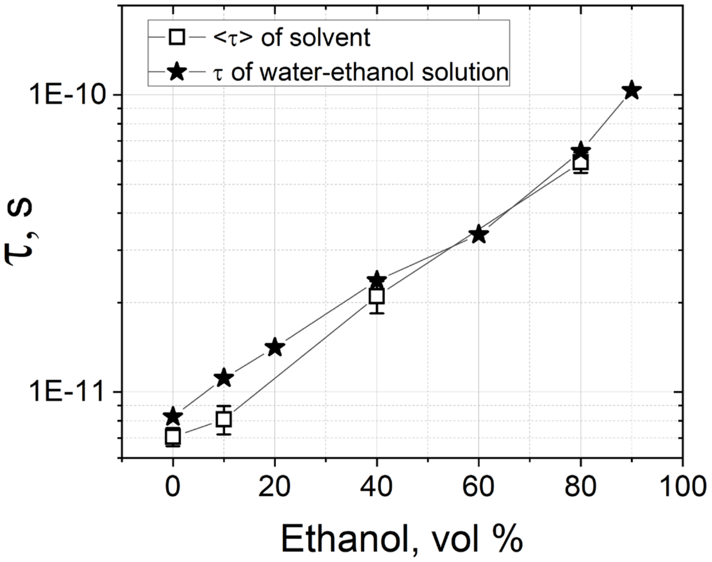

Fig. 6. Comparison of the dependences of the average relaxation time $<\tau>$ of a solvent with different SDS content on the ethanol content in the solution with the concentration dependence of the relaxation time τ for a water–ethanol mixture (Sato et al. 1999). Data are given for a temperature of 303 K

Let us consider the behavior of conductivity in aqueous solutions of SDS with different ethanol content, shown in Fig. 7. Indeed, with an increase in the proportion of ethanol in aqueous SDS solutions, the conductivity of the system decreases, which confirms the assumption that the proportion of ethanol influences the number of micelles in an aqueous SDS solution.

Some works (Wakisaka, Ohki 2005; Wakisaka et al. 2001; Zueva et al. 2021) show that water–ethanol mixtures have three characteristic concentration ranges of 0–10%, 25–40 and 60–92.3%, which share the existence of the binary water–ethanol solvent in different structural and energetic states. These states differ in the microheterogeneous structure of the solvent and in the geometry of the hydrogen bond network.

The values we obtained for the activation energy ΔE of the dielectric relaxation process of the solvent depending on the SDS concentration in a solution with different ethanol content are shown in Fig. 8. It is clearly seen that the ΔE values, depending on the ethanol proportion, fluctuate around a certain average value. There is no clear dependence on the content of SDS. Therefore, the average activation energies $<\Delta E>$ were calculated. Fig. 9 shows the dependence of the average value $<\Delta E>$ on the proportion of ethanol in the solvent.

With an increase in the ethanol proportion, the $<\Delta E>$ values increase, reaching a maximum at a volume fraction of ethanol of 40%, after which the $<\Delta E>$ value decreases. This behavior of the activation energy correlates well with the behavior of the dynamic viscosity of water–ethanol mixtures described in some works (Wakisaka, Ohki 2005; Zueva et al. 2021). Table 1 shows the averaged $<\Delta E>$ values we obtained and the already available dynamic viscosity values (Zueva et al. 2021).

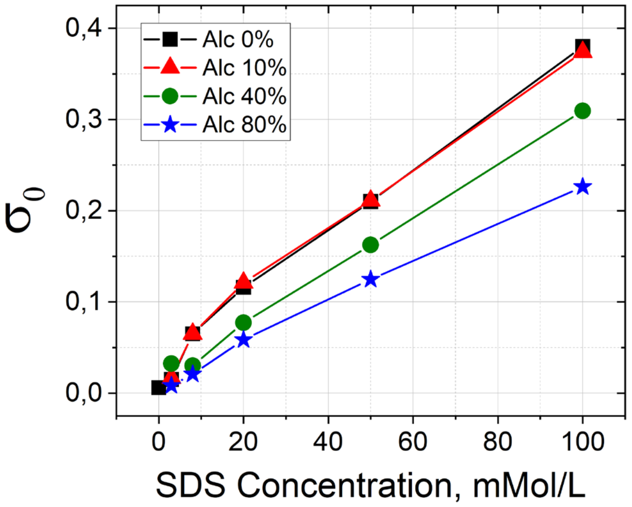

Fig. 7. Dependence of through conductivity on SDS concentration for different ethanol contents. Black squares — 0% ethanol; red triangles — 10% ethanol; green circles — 40% ethanol; blue stars — 80% ethanol. Data are given for a temperature of 303 K

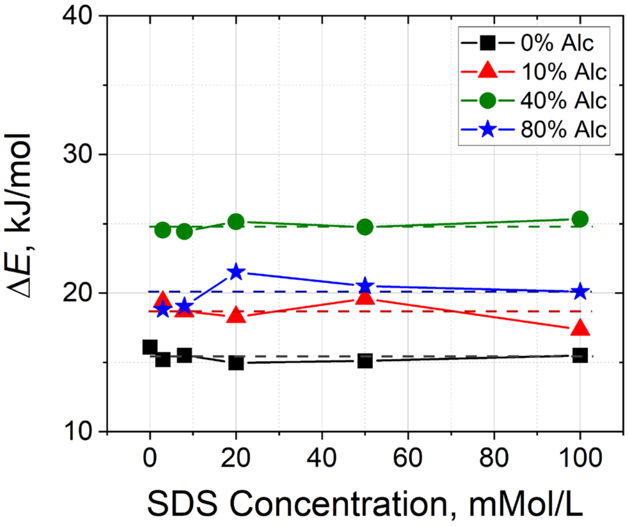

Fig. 8. Dependence of the values of activation energy Δ*E* of the dielectric relaxation process of the solvent on SDS concentration in a solution with different ethanol content; black squares — 0% ethanol; red triangles — 10% ethanol; green circles — 40% ethanol; blue stars — 80% ethanol. The dotted line indicates the average values

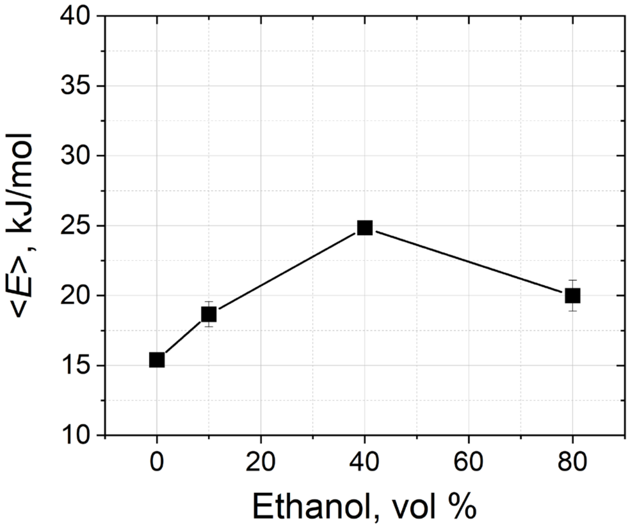

Fig. 9. Dependence of the average activation energy <Δ*E*> on ethanol proportion in the solvent

Table 1. Comparison of <Δ*E*> values obtained in this work and the values of dynamic viscosity of water–ethanol mixtures taken from the work (Zueva et al. 2021)

Volume fraction of ethanol in solvent	**<Δ*E*>, kJ/mol**	**Viscosity, mPa×s**
0	15.4 ± 0.4	0.8 ± 0.016
10	18.7 ± 0.9	1.1 ± 0.02
40	24.8 ± 0.4	1.8 ± 0.02
80	19.9 ± 1.1	1.7 ± 0.02

Fig. 10 shows a comparison of the dependences of average <Δ*E*> values and dynamic viscosity values on the ethanol fraction in the solvent; good agreement is observed between the presented data. Both dependences have maximum values in the alcohol concentration range of 40–50%. One work (Wakisaka, Ohki 2005) has obtained a similar dependence of viscosity on the ethanol content in a water–ethanol mixture. Its authors explain the increase in viscosity by the formation of ethanol self-association clusters; ethanol clusters grow when mixed with water, and their formation reaches a maximum of about 40–50 wt.% ethanol.

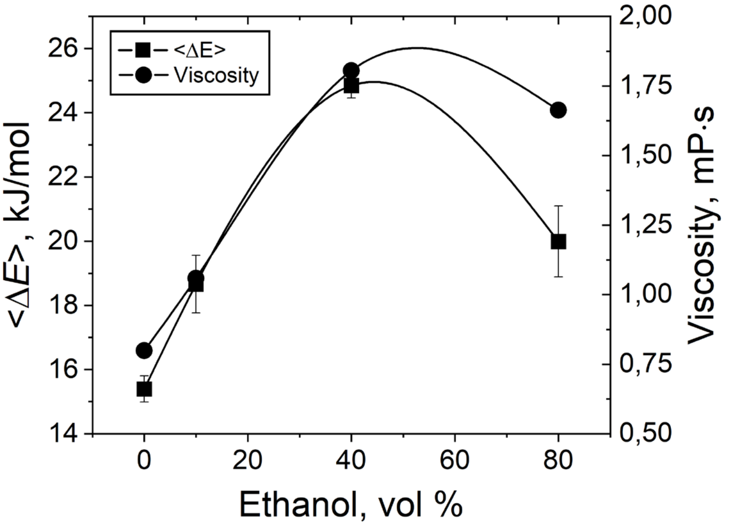

Fig. 10. Mutual correlation between averaged <Δ*E*> values and dynamic viscosity values

Viscosity is related to the molecular dielectric relaxation time by the Debye relation:

$$\tau_\mu = \frac{4\pi a^3 \eta}{kT}, \tag{3}$$

The relationship between molecular time τ_μ and macroscopic time is given by the Pauls expression (Powles 1953):

$$\tau = \frac{3\varepsilon_s}{2\varepsilon_s + \varepsilon_\infty}\tau_\mu, \tag{4}$$

The relaxation time τ is related to the change in the dipole relaxation activation energy ΔE by the following relationship (Greinacher 1948; Lewis, Smyth 1939):

$$\tau = \frac{hN}{RT}\exp\left[\frac{\Delta E}{RT}\right], \tag{5}$$

where h — Planck's constant; R — the gas constant; N — Avogadro's number.

Thus, the activation energy of the dielectric relaxation process is related to the structural features of the solution.

Conclusion

We have shown that in binary water–ethanol mixtures the process of SDS micelle formation changes significantly, which clearly follows from the characteristics of dielectric relaxation. In the region of high water concentrations, when the structure of water dominates, the process of SDS micellization in an aqueous ethanol medium does not differ significantly from an aqueous surfactant solution. In the region of average water concentrations, the micelle-forming properties of the surfactant are only partially preserved. In the region of high ethanol concentrations, the tendency of surfactant molecules to associate remains, but the sizes of associates are obviously small, which we have shown previously using other methods (Zueva et al. 2021). Based on the results obtained, the following conclusions can be drawn:

1. The formation of micelles leads to an increase in the through conductivity of the solution and a slight decrease in permittivity, proportional to the increase in the SDS concentration (the number of micelles);
2. An increase in the proportion of ethanol in the solvent complicates the process of SDS micellization since the presence of alcohol reduces the polarity of the solvent, which accordingly reduces the apparent value of the hydrophilic–lipophilic balance (HLB) of the surfactant. It has been established that with an increase in the ethanol fraction, the slope of the $\Delta\varepsilon(C_{SDS})$ dependence decreases; this parameter can be used to judge the efficiency of micellization;
3. Analysis of the dependences $<\Delta E>(C_{alc}, \%)$ and $\eta(C_{alc}, \%)$ presented in Figs. 8, 9 and 10 allows us to conclude that the structural state of the binary water–ethanol solvent is mainly determined by the composition of the solvent (ethanol content) and weakly depends on the SDS content.

Conflict of Interest

The authors declare no potential or apparent conflicts of interest.

Author Contributions

All the authors discussed the final work and took an equal part in writing the article.

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