

Small-Angle X-ray Scattering in Sodium Dodecyl Sulfate Solutions and Micelle Clustering

O. G. Us'yarov

St. Petersburg State University, Universitetskaya nab. 7/9, St. Petersburg, 199034 Russia

e-mail: usyarov.oleg@mail.ru

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Abstract—The small-angle X-ray scattering (SAXS) in micellar sodium dodecyl sulfate solutions has been studied in the range of overall concentrations c from 8 mM (CMC_1) to 300 mM and the absolute values of scattering vector q from 0.07 to 3.0 nm^{-1} . The total intensity of isotropic scattering has been revealed to increase with solution concentration. At $c > 27$ mM, the SAXS spectra have been found to exhibit an interference peak, which testifies a correlation in the arrangement of micelles in the bulk solution. This peak corresponds to the magnitude of q close to 1.55 nm^{-1} . Using the position of this maximum, average distance r_0 between the centers of micelles has been determined, which is equal to 4.1 nm and remains almost unchanged upon an increase in the overall concentration of sodium dodecyl sulfate. The observed regularities have been explained in terms of the DLVO theory taking into account the electrostatic and molecular intermicellar interaction.

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INTRODUCTION

Electrical double layers (EDLs) of ionic surfactant micelles govern, to a large extent, the properties of micellar solutions, such as their electrical conductivity [1–4], average activity of ions [5–8], their self-diffusion coefficients [9, 10], osmotic pressure [11–13], etc. Usually an EDL and the dependence of its properties on the content of a background electrolyte and temperature are judged from the information obtained by the capillary and Doppler electrophoresis methods [14–18]. At the same time, there are reasonable assumptions confirmed by experimental data [19] that many properties of micellar solutions of ionic surfactants depend on the long-range interactions of micelles and the character of their spatial arrangement.

In the vicinity of the critical micelle concentration (CMC_1), when average distances r_0 between micelles are large and their EDLs do not overlap significantly, micelles represent individual kinetic units involved in Brownian motion. The situation changes, when distance r_0 becomes comparable with the EDL thickness. Two variants are possible in this case. When ion-electrostatic repulsion of micelles is essential, their arrangement in a solution exhibits ordering in some regions or in the entire system volume. According to the terminology proposed previously [20, 21], such “lattices” with fixed interparticle distances are referred to as second-type periodic colloidal structures (PCS_2). They exist only in a limited system volume

and are, as a rule, strongly defective and decomposed upon dilution. Of course, due to the weak ion-electrostatic interaction of micelles at long distances, one may speak only of the presence of liquidlike clusters in such systems.

Along with the formation of PCS_2 , micelles may, in principle, form compact ordered clusters. This may occur in the two following cases. First, the ordering in such clusters will take place, provided that micelles have similar shapes and a narrow size distribution. It may be believed that micellar solutions of ionic surfactants more or less obey this condition [22, 23]. Second, the clustering of micelles must not lead to their coalescence, i.e., they must retain their sizes and shapes. Such systems are referred to as the first-type periodic colloidal structures (PCS_1). Note that PCS_1 and PCS_2 are detected in different concentration ranges of a dispersed phase, although they may coexist with each other.

In the case of micellar solutions, the above considerations have a hypothetical character. To confirm (or rule out) them, it is necessary to use adequate methods for structural analysis. In our opinion, it is most reasonable to study small-angle X-ray scattering (SAXS) as a function of the surfactant concentration. X-rays are scattered due to spatial fluctuations of electronic density, which is proportional to the atomic number of a chemical element. Therefore, SAXS is especially informative for investigating counterions of heavy atoms contained in micelles. It provides information

about the sizes and shapes of micelles upon variations in surfactant and background electrolyte concentrations, as well as the nature of counterions [23–25]. However, it is of greater importance that the application of SAXS makes it possible to reveal an ordered arrangement of micelles in micellar solutions, structural transitions, and their concentration ranges, as well as to find the distances that characterize the spatial arrangement of micelles. It should be noted that SAXS seems to have been used for the first time relatively long ago by Norrish [26] while investigating the intracrystalline swelling and determining the interplanar distances in a layered clay mineral, montmorillonite.

The main goal of this work was to analyze the effect of long-range forces of molecular and ion-electrostatic interaction on structuring in micellar solutions of sodium dodecyl sulfate (SDS). Within the framework of its solution, we intended to study SAXS in micellar SDS solutions throughout a wide range of overall concentrations and compare the results with the DLVO theory.

EXPERIMENTAL

Micellar Solutions of SDS

SAXS was studied in micellar solutions of SDS (Sigma, L-4509, main component content $\approx 99\%$, used as received) with overall concentrations ranging from 8 mM (CMC_1) to 300 mM. The choice of SDS as an object of study was dictated by the large body of information available on the characteristics of micellization in its aqueous solutions (CMC_1 , degree of counterion binding, aggregation number of surfactant molecules in micelles, etc.).

Equipment for Measuring SAXS and Method for Data Processing

Experiments on SAXS in SDS solutions were carried out on an Anton Paar SAXSess mc diffractometer¹. A tube equipped with a copper anode was used as a radiation source. Wavelength λ of CuK_{α} -radiation was 1.5418 Å. The collimation system comprised two components that provided the instrument with high resolution, namely, a multilayer elliptical mirror, which focused the divergent beams of the tube, and a slit with a high-quality polished surface. A charge-coupled device (CCD) was used as a detector. The distance between a sample and the detector was constant and equal to 309 mm. Primary processing of experimental data was performed with the introduction of collimation correction for a height of the primary beam equal to 10 mm. The measuring time was 10 s, while the number of scans was 120. The experiments

were carried out at room temperature in the transmission manner. The examined solutions were placed into quartz capillaries 15 mm long. The measured scattering curves were preliminarily processed using the SAXSquant software supplied with the instrument. Therewith, the background was subtracted from the spectra of the surfactant solutions.

Micellar Solution Composition

To determine the intermicellar interactions as depending on the surfactant content, it is necessary to know the composition of the micellar solution, i.e., concentration c_{11} of free surface-active ions in the intermicellar solutions and their concentration c_m in the micellar form, as well as the average intermicellar distance. The composition was calculated based on quasi-chemical theory of micellization [27]. The calculation procedure has been described in detail elsewhere (see, e.g., [28]).

If the magnitudes of c_m and aggregation number n_1 of SDS molecules in micelles are known, the number of micelles in 1 cm³ of a micellar solution C_m may be calculated as follows:

$$C_m = \frac{c_m N_A}{1000 n_1} = \frac{(c - c_{11}) N_A}{1000 n_1}, \quad (1)$$

where N_A is the Avogadro number. Average distance r_0 between the centers of micelles upon their cubic arrangement in the space is

$$r_0 = \frac{1}{\sqrt[3]{C_m}}. \quad (2)$$

Calculation of Intermicellar Interaction Energy

Dimensionless intermicellar interaction energy \tilde{U} at distance r between micelle centers was found as an algebraic sum of molecular \tilde{U}_m and electrostatic \tilde{U}_i components. The value of \tilde{U}_m was calculated by the following equation [29]:

$$\tilde{U}_m = \frac{U_m}{\theta} = \frac{A}{6\theta} \left(\frac{2}{s^2 - 4} + \frac{2}{s^2} + \ln \frac{s^2 - 4}{s^2} \right), \quad (3)$$

where $s = r/a_e$ (a_e is the radius of a micelle that corresponds to the distance from the center of its hydrocarbon core to the Stern layer, θ is the product of the Boltzmann constant and absolute temperature, and A is the Hamaker constant. The precise value of the latter for the case of molecular interaction between micelles in an aqueous solution is, unfortunately, unknown. In our calculations, it was assumed that $A = 8 \times 10^{-20}$ J, which was close to the Hamaker constant calculated theoretically and found experimentally for a dodecane–water–dodecane system [30], and that radius a_e for SDS micelles was equal to 2.05 nm [7, 31].

¹ The measurements were carried out by A.A. Mistonov at the Methods for Analysis of Compound Composition resource center at St. Petersburg State University.

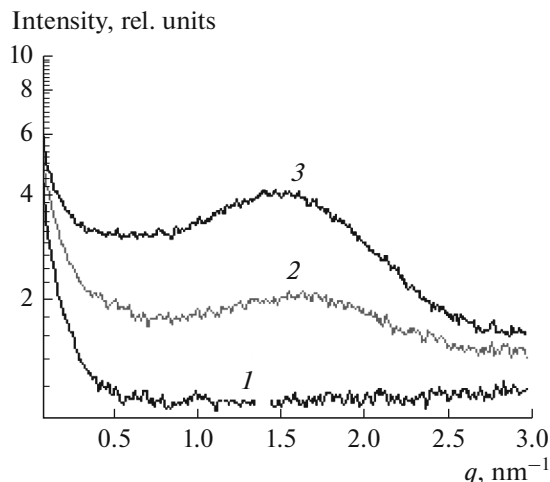


Fig. 1. SAXS spectra at different overall SDS concentrations: (1) 0.02, (2) 0.08, and (3) 0.3 M.

Dimensionless energy \tilde{U}_i of ion-electrostatic interaction was found under the condition of constancy for Stern layer potential ψ_d upon variations in the intermicellar distance. The corresponding equation for this case was derived by Deryaguin[32]. It was shown that, for identical spherical particles with potential $\psi_d \leq 50$ mV of the diffuse part under condition $\psi_d = \text{const}$, the following equation is valid for the energy of ion-electrostatic repulsion:

$$\tilde{U}_i = \frac{2\pi\epsilon_0\epsilon a_e\psi_d^2}{\theta} \ln\{1 + \exp[-\kappa(r - 2a_e)]\}, \quad (4)$$

where

$$\kappa = \sqrt{\frac{2e^2 c_{11} N_A}{10^3 \epsilon_0 \epsilon \theta}} \quad (5)$$

is the reciprocal Debye thickness of the EDL, ϵ is the dielectric permittivity of the medium, ϵ_0 is the dielectric constant, and e is the electron charge.

Knowing degree β of counterion binding by micelles, the ψ_d potential corresponding to the Stern layer may be calculated using the following relation [7]:

$$\beta = 1 + \frac{8\pi\epsilon_0\epsilon\kappa\theta a_e^2}{e^2 n_1} \left(\sinh \frac{\Phi_d}{2} + \frac{2}{\kappa a_e} \tanh \frac{\Phi_d}{4} \right). \quad (6)$$

Here, Φ_d is the dimensionless potential of the Stern layer, $\Phi_d = -e\psi_d/\theta$. When using formula (6), it was assumed that parameters n_1 , β , and a_e remain constant throughout the examined range of SDS overall concentrations. Therewith, for SDS micelles, it was accepted that $\beta = 0.81$ [7, 8, 33] and $n_1 = 60$ [31, 34, 35]. The calculation results showed that $\Phi_d = -2.6$ and $\psi_d = -65$ mV.

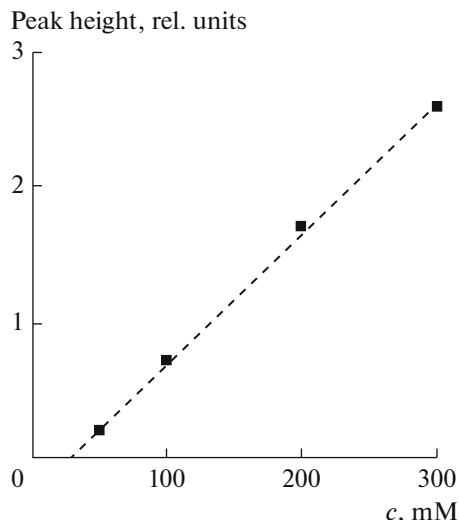


Fig. 2. Dependence of interference maximum height in the SAXS spectra on the overall SDS concentration.

Hence, the total dimensionless energy of intermicellar interaction may, as a first approximation, be determined from the following expression:

$$\tilde{U}_m = \frac{A}{6\theta} \left(\frac{2}{s^2 - 4} + \frac{2}{s^2} + \ln \frac{s^2 - 4}{s^2} \right) - \frac{2\pi\epsilon_0\epsilon a_e\psi_d^2}{\theta} \ln\{1 + \exp[-\kappa(r - 2a_e)]\}. \quad (7)$$

RESULTS AND DISCUSSION

Two-dimensional SAXS patterns for the SDS solutions are entirely isotropic. Although the intensity and angular range of scattering somewhat varied with an increase in the overall concentration of SDS from CMC₁ to 25 mM, the patterns of the SAXS spectra for this interval of c values did not differ significantly from each other (Fig. 1, curve 1). In the vicinity of 25 mM, a weak interference peak arose in the spectra, with its amplitude being close to the absolute value of scattering vector $q = 1.55 \text{ nm}^{-1}$. This maximum corresponded to the average distances almost equal to the micelle diameter [31]. The maximum amplitude monotonically increased as the SDS overall concentration grew, whereas its position in the abscissa axis remained almost unchanged in the whole range of the surfactant concentrations from 30 to 300 mM (Fig. 1, curves 2 and 3). The scattering spectra were approximated by the sum of the Lorentz functions with the zero position and the Gauss function at the point of peak appearance. The extrapolation of the linear dependence of the peak height on SDS concentration to its zero amplitude (Fig. 2) afforded the value of c equal to 27 mM.

There are two possible explanations for the observed spectrum. It is known [36, 37] that, in the

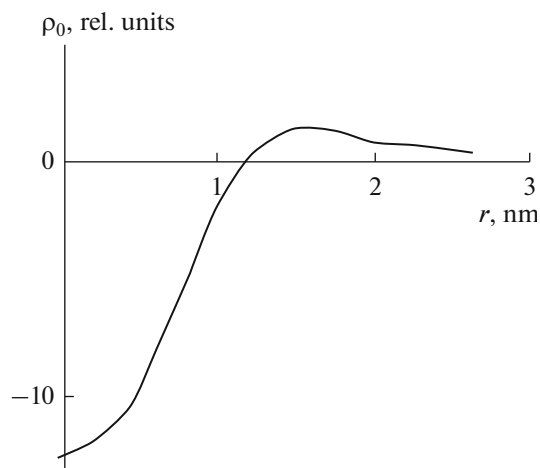


Fig. 3. Electron density profile for SDS molecules.

case of a narrow size distribution of spherical particles (variance of no more than 10–20%), the peak may arise due to the shape of the particles themselves. Some works [22, 23] have confirmed that the variance of micelle sizes is low indeed. When electron density ρ_0 of a micelle core (hydrocarbon radicals) is lower than the density of a medium (water) and the electron density of the shell (a layer of potential-determining ions and counterions) is higher than that of the medium (Fig. 3), the SAXS of micelles approaches the scattering observed in the experiments. Therefore, the appearance of such a peak in the micellar solutions can also be explained without involving the concepts of interparticle interference.

However, the simulation of scattering [37] by micelles with the profile of electron density ρ_0 presented in Fig. 3 leads us to conclude that the existence of this peak results primarily from the ordered arrangement of micelles in concentrated micellar solutions. This statement may be confirmed by four things. First, at CMC_1 , when the solution already contains a large amount of micelles, they are not reflected in the SAXS spectra. Second, at surfactant concentrations below 27 mM, the degree of micellization is high. For example, according to the quasi-chemical theory of micellization [27], at $c = 20$ mM, it is equal to 0.7. Therewith, micelle concentration C_m is equal to $1.4 \times 10^{17} \text{ cm}^{-3}$. In spite of such a high content of micelles in the system at $c = 20$ mM, scattering on them begins to be evident only at $C_m = 2 \times 10^{17} \text{ cm}^{-3}$. Third, the scattering peak (Fig. 4, curve 1) simulated even for a monodisperse model is essentially wider than the experimentally observed one (Fig. 4, curve 2). It is obvious that, at a high variance of micelle sizes, the difference between the experimental and simulated scattering will be even higher. Fourth, immediately on the left of the peak, the real spectra exhibit a wide range of a rather high scattered intensity, the presence of which

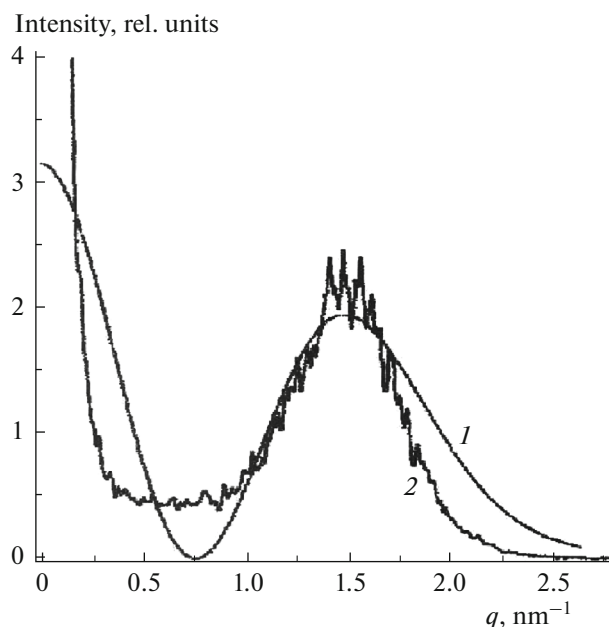


Fig. 4. SAXS spectra at an overall SDS concentration of 0.08 M: (1) simulated and (2) experimental data.

cannot be predicted using the model of scattering by individual spherical particles.

Hence, the performed simulation and the comparison of its results with the data of real experiments show that SAXS allows one to identify the formation of PCS_1 in concentrated micellar solutions of SDS.

To estimate the probability that ordered structures are formed in micellar solutions, let us consider the intermicellar interactions at different overall concentrations of SDS. Figure 5 presents the linear approximations for the dependences of the many-body interaction potential on intermicellar distance r at $c = 13.7$, 32.4, and 203 mM. Average distances r_0 between the centers of micelles for these concentrations are 260, 160 and 80 nm, respectively.

As follows from these results, at $c = 13.7$ mM (Fig. 5a), micelles must reveal a high relative mobility, which is characterized by the width of interval fg . In turn, the presence of a high ion-electrostatic repulsive barrier, which is five times higher than the energy of thermal motion, almost completely prevents the micelles from aggregation into large clusters. Hence, the appearance of both PCS_2 and PCS_1 seems to be impossible.

Upon an increase in the overall SDS concentration, the situation changes, primarily due to the possibility of mutual fixation of micelles at moderate distances from each other (Fig. 5b). According to the calculation results, the formation of PCS_1 , which is accompanied by a reduction in repulsive barrier ΔU , becomes already evident at $c = 30$ mM. Above this concentration, the probability that clusters are formed

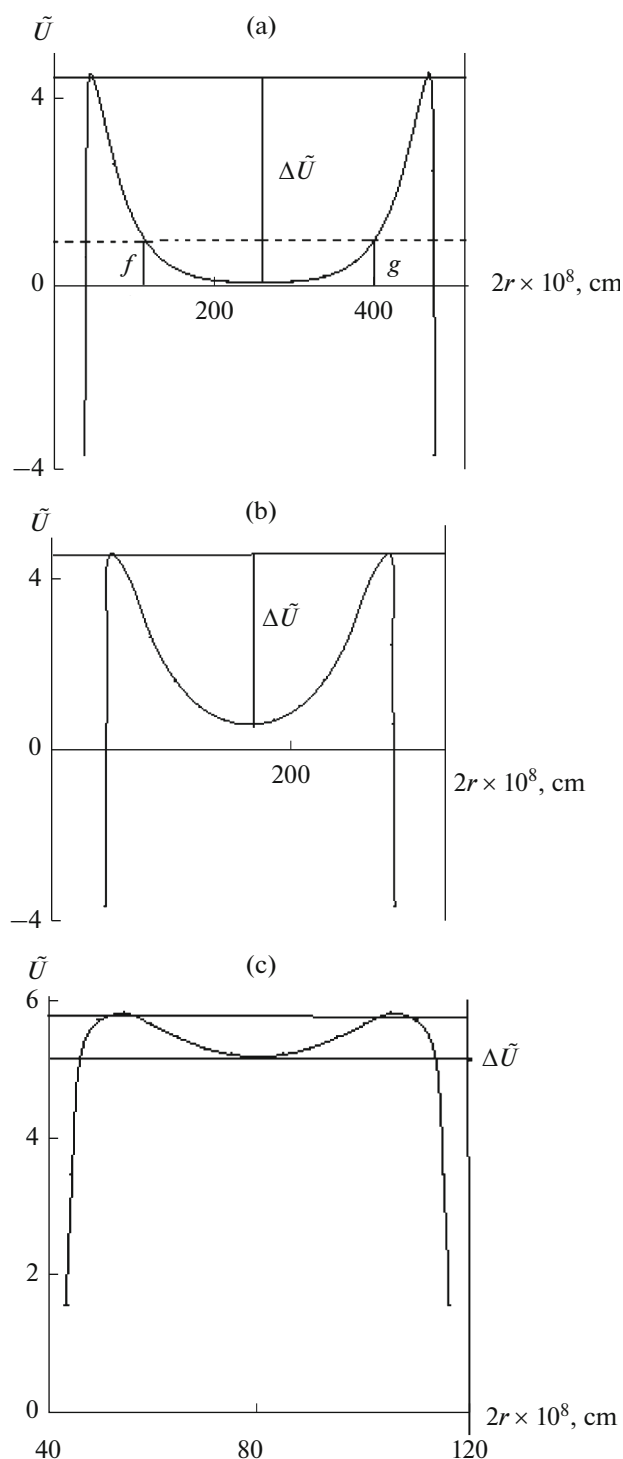


Fig. 5. Dependences of the potential of the interaction of two micelles with a third (probe) one (linear approximation) on the distance at overall SDS concentrations of (a) 13.7, (b) 32.4, and (c) 203 mM. The horizontal dashed line in Fig. 5a corresponds to the average energy of thermal motion of micelles, and the fg segment characterizes the interval of distances in which the motion of the probe micelle is not hindered.

in which micelles are located at a distance of 4.1 nm from each other essentially increases². A further growth in the SDS concentration causes the degeneration of repulsive barrier ΔU , thereby determining the high probability of micelle aggregation into large clusters of PCS₁ type (Fig. 4c).

The conclusion that micellar clusters may be formed owing to the molecular and ion-electrostatic intermicellar interactions agrees with the results of experimental investigating SDS micellar solutions by flow ultramicroscopy [39]. It has been found that, already in the vicinity of CMC₁, numerous microscopic particles with sizes of about 0.1 μm are formed in the system, with these particles being distinctly observed with an optical dark-field microscope. The clustering can also affect the conductivity of micellar SDS solutions [40]. It can probably also manifest itself as an increase in the slope of the dependence of the specific conductivity on the overall concentration of SDS. This bend is likely to be explained by the formation of percolation aggregates in the micellar solution.

The combined analysis of the SAXS spectra and intermicellar interactions allows one to outline the consecutive stages of structuring in the micellar solutions upon an increase in the overall SDS concentration. In the range of overall concentrations from CMC₁ to 27 mM, the ordered clustering accompanied by the appearance of PCS₂-type structures is almost invisible in the SAXS spectra. This seems to be due to the fact that the intermicellar distances in the clusters strongly vary, thereby leading to the appearance of a strong noise at small scattering angles³. The determination of average distances r_0 between the micelle centers has shown that they are large in the vicinity of CMC₁. Therefore, the Brownian motion causes rare collisions between the micelles. Hence, the formation of both PCS₁ and PCS₂ is unlikely in these systems. The state of such a system is illustrated schematically in Fig. 6a.

Further increase in the overall SDS concentration leads to a reduction in average intermicellar distances r_0 , which has great consequences. On one hand, at r_0 values comparable with the EDL thickness, overlapping of micelle EDLs takes place, i.e., owing to their ion-electrostatic repulsion, the ordered structures of the PCS₂-type can be formed (Fig. 6b). On the other hand, a reduction in the average intermicellar distances causes a reduction in repulsive barrier ΔU owing to the many-body intermicellar interaction. This may promote the passage from PCS₂ to PCS₁ and

²The probability of clustering is determined not only by the height of the repulsive barrier of micelles, but also by their bonding energy in the clusters. Nowadays, the theory of surface forces does not, unfortunately, allow one to estimate the depth of this energetic minimum for the formation of PCS₁. However, taking into account an insignificant time required to establish equilibrium in micellar solutions [38], it may be assumed that it is comparable with the energy of thermal motion.

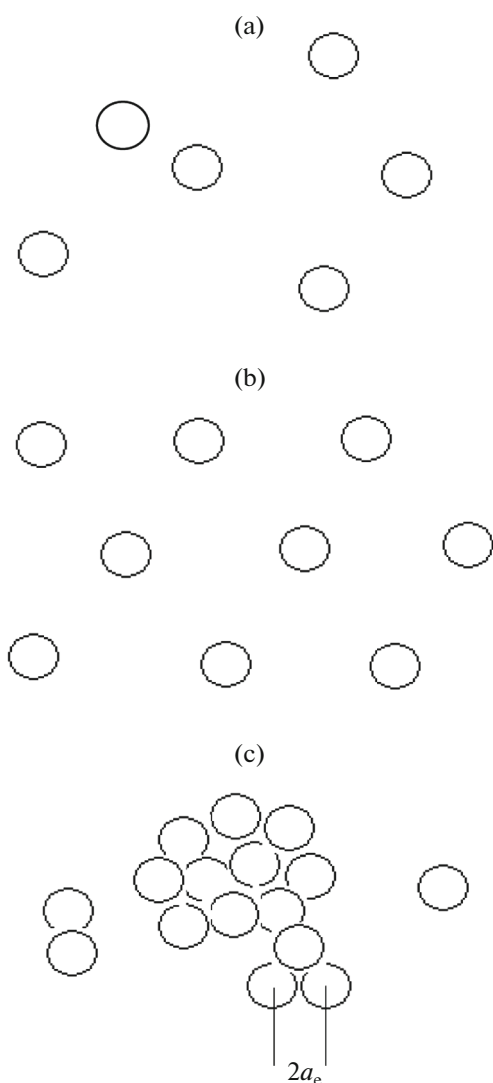


Fig. 6. Scheme characterizing the transformation of the structure of micellar clusters upon an increase in the overall concentration of ionic surfactants (see text for explanations).

the formation of clusters, in which micelles are in close contact with each other (Fig. 6c). Apparently, the passage from PCS_2 to PCS_1 may be evident only when the clusters, which make an essential contribution to the SAXS spectra, are rather large, with the shapes and sizes of micelles remaining, as a first approximation, unchanged. The existence of such a situation is evidenced by the presence of the maximum and its

³ The design features of the used instrument (wavelength, distance between sample and detector, etc.) may affect the fine structure of the SAXS spectra at small q values. As has been shown in [23], the SAXS spectra obtained using synchrotron radiation ($\lambda = 0.729 \text{ \AA}$ and a distance between the sample and detector of 397 cm) for solutions of cesium dodecyl sulfate at $c > 30 \text{ mM}$ distinctly show two peaks at $q = 0.5\text{--}07 \text{ nm}^{-1}$ (with location being dependent on the overall concentration of SDS) and $q = 1.8 \text{ nm}^{-1}$.

unchanged position. The complete passage of the micellar solution to PCS_1 is likely to correspond to CMC_2 , which simultaneously conforms to the onset of the appearance of supramicellar structures.

In conclusion, it should be noted that the agreement between the experimental data on the SAXS in SDS micellar solutions and the DLVO theory is satisfactory, at least at the qualitative level. Some quantitative deviations (in, e.g., the threshold concentration for the passage from PCS_2 to PCS_1) are likely due to the absence of reliable data on a number of parameters that determine the intermicellar interaction. Here, the uncertainty of the data on the Hamaker constant for molecular attraction of micelles and the degree of binding of counterions by micelles should primarily be emphasized. It is also necessary to take into account the absence of reliable information on the parameters of ion-electrostatic interaction (EDL thickness, Stern layer potential, etc.). The existing uncertainties cannot always be taken into account, thereby undoubtedly leading to an error in the corresponding estimates. At the same time, it is obvious that the experimental detection of clustering by the SAXS method is an important supplement to the results of investigating the rheological, electrophysical, diffusion, and other properties of micellar solutions of ionic surfactants.

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