

Review

Characterization of Liquid Adsorption Layers Formed from Aqueous Polymer–Surfactant Solutions—Significant Contributions by Boris A. Noskov

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Abstract: In many modern technologies, surface-active compounds, such as surfactants, polymers, proteins, particles and their mixtures, are essential components. They change the dynamic and equilibrium properties of the inherent interfaces, which is mostly visible in foams and emulsions. The interfacial dilational visco-elasticity is probably the most informative quantity due to its direct interrelation to the equation of state of the corresponding interfacial layers as well as the mechanisms governing the interfacial molecular dynamics. The scientific field of interfacial visco-elasticity, although quite young, has been inspired by the pioneering work of Marangoni, Levich, Lucassen, Lucassen-Reynders, Hansen, van den Tempel and Krotov, and during the last decades, also significantly by Boris Noskov. His contributions to the theoretical foundation and experimental analysis of polymer and mixed surfactant–polymer interfacial layers in particular are essential.

Keywords: adsorption layers; surfactant; polymer; protein; surface dilational visco-elasticity; diffusion-controlled exchange of matter; kinetic-controlled exchange of matter



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1. Introduction

Disperse systems consisting of different fluid phases are widespread in nature [1,2] and can be applied in various technological processes, like extraction, froth flotation, wastewater treatment, tertiary oil recovery, etc. [3–5]. Parts of living organisms and many consumer products in food, cosmetic and pharmaceutical industries exist in the form of foams and emulsions [6–8]. Their properties strongly depend on the properties of liquid/fluid interfaces [9]. Moreover, liquid surfaces can be a template for the formation of two-dimensional structures [10,11]. Therefore, the comprehension of processes in the surface layer and the capability to design properties of the liquid surface play an important role in multiple areas [12,13]. For example, the formation and destruction of foams or emulsions depend on the stability of thin films and, especially, the dynamic properties of the liquid/fluid interface play a key role [14–17]. In the twentieth century, the application of macromolecules instead of conventional low molecular weight (LMW) surfactants allowed the obtention of more stable dispersed systems. Nowadays, complexes of surfactants with macromolecules and different types of molecular aggregates or nanoparticles are attracting significant attention as stabilizers for foams and emulsions [18–23]. The possibility of achieving extremely stable dispersed systems allows the development of new materials [24,25].

Surfactant molecules decrease the surface tension and form a rigid surface layer that prevents the destruction of thin liquid films and coalescence at deformations [26,27]. The rigidity of surface layers depends on the nature and concentration of added surfactants [28,29]. Moreover, the surface layer's response to deformations depends on the magnitude and

way of deformation. In particular, the stability of foams and emulsions is governed by the dynamic dilational surface elasticity of the surface layers [30,31]. The dilational surface elasticity is a thermodynamic property of surface layers, which is connected with both the equilibrium characteristics and the kinetic coefficients of the relaxation processes in the surface layer [26]. Therefore, the application of methods of surface rheology for measurements of dynamic surface properties represents a powerful tool for the investigation of surface layers [32,33]. While modern experimental techniques, like x-ray reflectivity, neutron reflectivity and various kinds of spectroscopy, are usually applied to surface layers in the equilibrium state, some important questions regarding for example protein unfolding and aggregate formation in surface layers remain unanswered for many systems [34–37]. Investigations of dynamic surface properties in the course of adsorption give additional information about the surface layer's structure, conformation of macromolecules and mechanisms of complex formation. Figure 1 provides an overview of the various fields in which studies on the dilational visco-elasticity of complex liquid interfacial layers were performed by B.A. Noskov and his team.

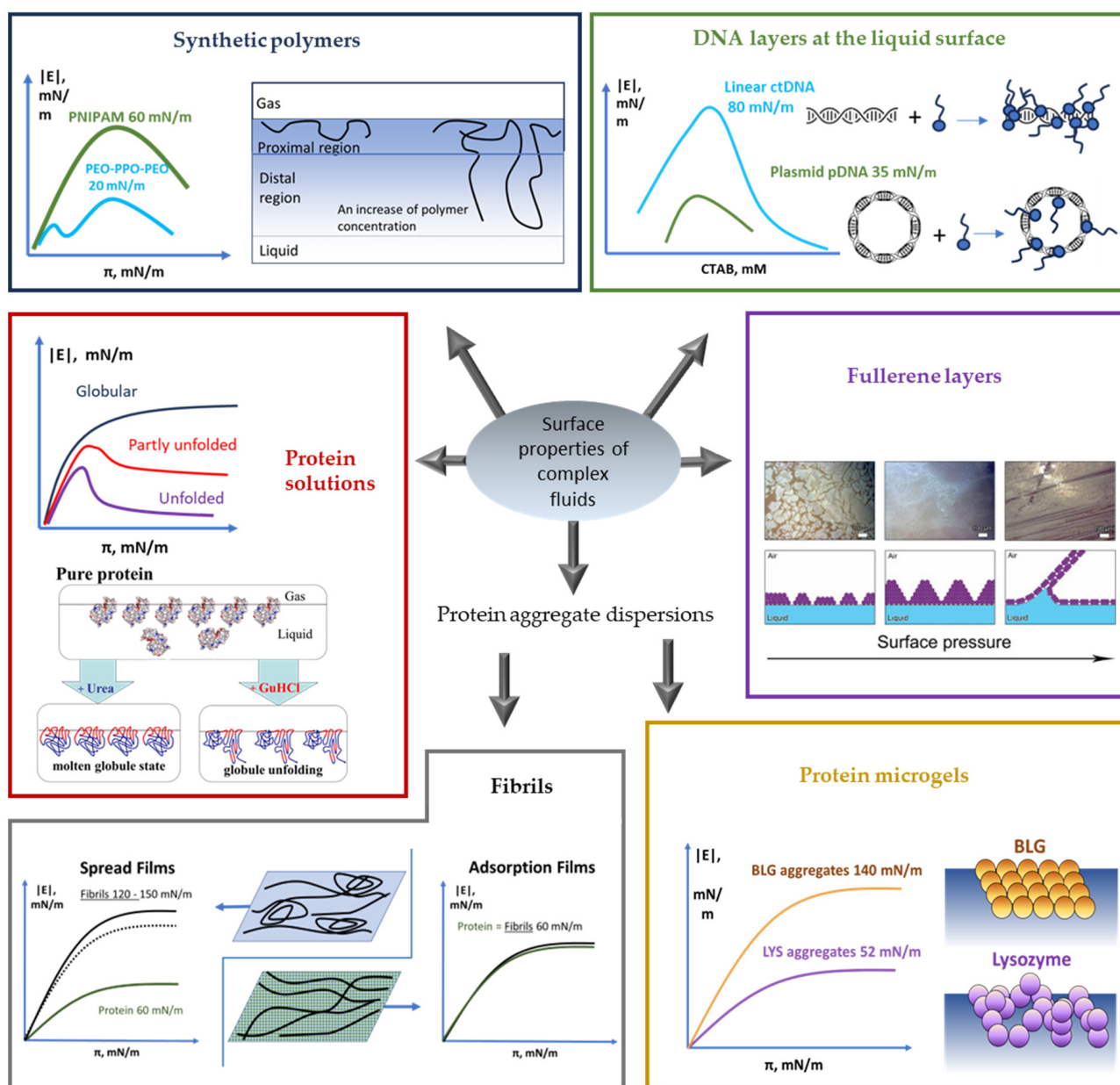


Figure 1. Schematic representation of the main research directions.

In this study, we provide a short historical review of the work of Boris Noskov on the thermodynamics and the kinetics of surface phenomena of complex liquids. He started his work under the supervision of Anatoly I. Rusanov at Leningrad State University (now Saint Petersburg State University) where a strong thermodynamic school existed for many decades. The first works of B. Noskov on fast surfactant adsorption at the surface of liquid jets and on capillary waves were marked by the first prize for young researchers of the university and by the first prize of the All-union Chemical Society named after Mendeleev. The results of subsequent theoretical investigations of the dilational surface visco-elasticity of surfactant and polymer layers at the liquid–gas interface proved to be useful in further studies of more complex systems—protein solutions, polyelectrolyte/surfactant solutions, spread and adsorbed layers of soft and rigid nanoparticles including protein aggregates and fullerenes. The main focus of this review will be the results of the last ten years.

2. Theories and Experimental Methods

The first theory of surface dilational elasticity goes back to the middle of the twentieth century when the influence of surface tension gradients on the damping of capillary waves was realized. All methods of surface rheology are based on the deformation of the surface and the registration of the system's response, which consists in surface tension changes. It should be noted that the system's response at the given surface age depends not only on the surface area at this moment but also on the prehistory of the system, i.e., the surface layer should be considered as a system with memory. Therefore, a strict relation between a surface tension change and a deformation can be written only for infinitesimal deformations in an integral form. In the case of small sinusoidal oscillations, the increments in surface tension $\Delta\gamma$ and the surface area ΔA can be presented as complex functions of time:

$$\Delta\gamma(t) = \Delta\gamma_0 \exp(i\omega t + i\varphi), \quad (1)$$

$$\Delta A(t) = \Delta A_0 \exp(i\omega t), \quad (2)$$

where t is the time, ω is the angular frequency of oscillations, φ is the phase difference between the oscillations of surface tension and surface area, ΔA_0 and $\Delta\gamma_0$ are the amplitudes of surface area and surface tension oscillations, respectively. Then, the dilational surface elasticity can be defined by the following equation:

$$\varepsilon(\omega) = A \frac{\Delta\gamma_0}{\Delta A_0} \exp(i\varphi) = \varepsilon_{re} + i\varepsilon_{im}, \quad (3)$$

where ε_{re} and ε_{im} are the real and imaginary parts of the dilational surface elasticity. Thus, the dynamic surface elasticity is a complex quantity and depends on the frequency of the surface area oscillations. The ratio of the real and imaginary parts of the dilational surface elasticity depends on the difference between the characteristic time of relaxation processes and the period of oscillations. However, unlike the case of bulk rheology, the expression for the dynamic surface elasticity usually includes kinetic parameters which characterize matter exchange between the surface layer and the bulk phase. The first relation for solutions of a single surfactant with diffusion-controlled adsorption kinetics was derived by Lucassen and Hansen [38]:

$$\varepsilon = \varepsilon_0 \frac{(1+i)/2\zeta}{1+(1+i)/2\zeta}, \quad (4)$$

where $\varepsilon_0 = -\frac{d\gamma}{d\ln\Gamma}$ is the surface elasticity within the limit of high frequency, Γ is the adsorbed amount, $\zeta = \left(\frac{\omega_D}{\omega}\right)^{1/2}$, $\omega_D = \frac{D}{2} \left(\frac{dc}{d\Gamma}\right)^2 = \frac{1}{\tau_D}$, D is the diffusion coefficient, c is the surfactant concentration in the bulk phase and $\tau_D = 1/\omega_D$ is the diffusion relaxation time.

Based on the approach of non-equilibrium thermodynamics, Noskov obtained more general relations when the number of soluble surfactants exceeds one or an adsorption

barrier has to be taken into account [39]. In the last case, the expression for the dilational surface elasticity takes the form:

$$\varepsilon = \varepsilon_0 \frac{i\omega\tau + (1+i)/2\zeta}{1 + i\omega\tau + (1+i)/2\zeta'} \quad (5)$$

where τ is the relaxation time of the barrier-controlled adsorption process.

The first measurements of the dynamic surface elasticity were performed for solutions of LMW surfactants [40]. To measure the dilational surface elasticity for solutions of these surfactants, methods working at high frequencies (>100 Hz) were usually applied, since the adsorption is rather fast and the relaxation time consequently short [41–43]. These methods are based on measurements of the wavelength and damping coefficient for capillary waves. Spontaneously generated due to thermal fluctuations, capillary waves on liquid surfaces allow measurements of the dynamic surface elasticity in the frequency range from 10 kHz to 2 MHz by the quasi-elastic light scattering method, which has been recently reviewed [44,45]. Capillary waves excited by a mechanical or electrical oscillator are used for measurements in the range from 50 to 1000 Hz. The theory of these methods was reviewed by Noskov in the monograph devoted to interfacial rheology [26]. The application of capillary wave methods to solutions of LMW surfactants gave the possibility of estimating the micellization kinetics and their influence on dynamic surface elasticity [46–48].

The application of the relation derived by Lucassen-Hansen to the adsorption of macromolecules can lead to incorrect conclusions, since amphiphilic polymers adsorb essentially irreversibly; also, the assumption of diffusion-controlled kinetics is invalid. For this case, Noskov proposed a relation for the dynamic surface elasticity based on a kinetic model for a polymer adsorption film consisting of a relatively dense region proximal to the gas phase and a diluted distal region where loops and tails protrude into the solvent [49,50]. Under dilation (compression) of the polymer film, the change in surface tension takes place due to corresponding changes in the concentration of chain segments in the proximal region. The exchange of polymer segments between the proximal and distal regions caused by surface deformations proceeds as a result of two processes: the relaxation of the inner stress of a polymer chain, which can be described by the Rose model; squeezing in/out a chain as a whole, which is in agreement with the reptation model of de Gennes [51]. Then, the relation for the dynamic surface elasticity of polymer adsorption layers can be presented in the following form:

$$\varepsilon = -\frac{\partial\gamma}{\partial\ln\Gamma} \left[\frac{2\Gamma_3}{\pi\Gamma} \sum' \frac{8i\tau_B\omega}{p^2\pi^2 \left(1 + \frac{i\omega\tau_B}{p^2}\right)} + \left(1 - \frac{2\Gamma_3}{\pi\Gamma}\right) \frac{i\omega\tau_2}{1 + i\omega\tau_2} \right], \quad (6)$$

where p changes from 1 to infinity and the sum includes only the odd terms, Γ is the number of moles of monomers per unit surface area in the proximal region, Γ_3 is the number of moles of those monomers per unit area in the proximal region that belong to trains with two transitional regions from proximal to distal, τ_B is the relaxation time of the inner stresses of a polymer chain and the relaxation time τ_2 corresponds to the motion of a train in the proximal region as a whole.

According to Noskov's model for the adsorption of flexible polymers, the initial accumulation of macromolecule chains in the proximal region leads to an increase in surface elasticity [49]. When the magnitude of adsorption exceeds some critical value, loops and tails are formed and additional mechanisms for surface stress relaxations appear. In this case, the surface stress relaxation occurs at the expense of the exchange of chain segments between the proximal and distal regions of the surface layer. It leads to a decrease in the surface elasticity and the appearance of a maximum in the concentration and kinetic dependencies of the dynamic surface elasticity while corresponding dependencies of surface tension remain monotonic [50].

For investigations of the dynamic surface elasticity of polymer solutions, low frequency methods are usually used due to the slower diffusion of polymer molecules as compared to conventional surfactants [52]. These methods are based on the excitation of longitudinal surface waves, which have a wavelength larger than the dimension of the liquid surface (Langmuir trough or drop) when the oscillation frequency is sufficiently low (<0.2 Hz) [53,54]. Thus, the surface strain proved to be homogeneous and can be easily measured using different techniques, such as the Wilhelmy plate in the oscillating ring or the barrier method [50], via the surface profile or the capillary pressure using oscillating bubble/drop methods [55–57]. At present, new experimental tools appear for generating purely dilational deformations with a minimum contribution by shear deformations [58,59] suitable for measurements of the surface dilational elasticity in the range of extremely low surface tensions [60–62]. The development of surface rheology methods allows their application as diagnostic tools even for the analysis of biological liquids in medicine [6,63].

All relationships for dynamic surface rheology presented above are valid only for low strains and not for large deviations from equilibrium, i.e., only when the system's response is linearly proportional to the deformation [64,65]. The conditions for their applicability in the surface layer were discussed in the study of [43]. When deformations become sufficiently large, the oscillations of surface tension can deviate from a purely sinusoidal form and include contributions of higher harmonics. The degree of non-linearity can be estimated based on the calculation of the total harmonic distortion parameter, which is defined as the ratio of the amplitudes of the main harmonic to the higher harmonics [66]. For the description of a non-linear response of a system, several methods can be applied [64,67,68]. It was shown that the measurement of surface tension changes at large deformations for spread monolayers of lipids or adsorbed layers of pulmonary surfactant makes it possible to determine the efficient surface elasticity and to estimate the relaxation time in regions of low surface tensions [69,70]. Also, for heterogeneous interfaces, the contribution from the surface layer structure to the system response on deformations has to be considered [43,71].

For the description of the adsorption kinetics of charged globular proteins, an additional model was proposed [72]. It considers the formation of an adsorption barrier due to electrostatic repulsive forces between globules approaching the surface from the bulk with a similar charge. Moreover, this model takes into account steric hindrances due to the previously adsorbed globules using a blocking function taken from random sequential adsorption theory [73]. Note that the relationship for calculating the kinetic dependence of the surface coverage has a single fitting parameter—the real charge of the protein globule.

$$t = \frac{1}{Dc_0} \sqrt{\frac{4RT}{\alpha}} \int_0^\theta \frac{e^{\frac{\alpha\theta}{\pi a^2 RT}}}{\sqrt{\theta} \left(1 - 4\theta + \frac{6\sqrt{3}}{\pi} \theta^2 + 1.4069\theta^3\right)} d\theta, \quad (7)$$

where θ is the surface coverage, c_0 is the concentration of globules and a is the radius of globules. α is defined as follows:

$$\alpha \equiv \sqrt{\frac{2\pi RT}{\epsilon c} \frac{z^2 F}{z_b}} e^{-2\kappa a}, \quad (8)$$

where c is the concentration of indifferent electrolytes, z_b is its electrochemical valency, κ is the Debye length and z is the charge of globules.

The application of this relationship for the description of adsorption of protein/surfactant complexes has shown good agreement with experimental data obtained from ellipsometry measurements [72].

3. Synthetic Polymers

The results of dynamic surface elasticity measurements for synthetic polymers confirm the surface layer structure model proposed in [50]. For spread layers of poly(ethylene oxide)

(PEO) or adsorbed layers of poly(ethylene glycol) (PEG) and poly(N-isopropylacrylamide) (PNIPAM), the dependence of dilational surface elasticity on surface concentration pass through a maximum [74–76]. According to the model, the maximum in dilational surface elasticity is connected with changes in the conformation state of the macromolecules in the surface layer and the formation of loops or tails protruding into the subphase. For PNIPAM solutions, a maximum was also observed in the kinetic dependences of surface elasticity [76]. The increase in the surface layer's thickness corresponds to the formation of a distal region for PNIPAM solutions, which was confirmed with ellipsometry measurements [77]. For block copolymers, such as PEO-poly(propylene oxide)-PEO (PEO-PPO-PEO), the dependence of surface elasticity have two maxima corresponding to the transition of each type of block to the subphase [78]. The position and magnitude of the maxima of surface elasticity depend on the block size and their degrees of hydrophobicity. The formation of the distal layer with loops and tails for solutions of PEO-PPO-PEO was confirmed by neutron reflectivity [79].

The behavior of microgel particles with a core-shell structure, when a relatively dense core is surrounded by a soft shell, has attracted significant attention due to its multifunctionality [80,81]. It was shown that for adsorbed layers of PNIPAM microgel particles with a cross-linked core, the dependence of the surface dilational elasticity on surface pressure is non-monotonic, and the maximum almost coincided with the corresponding dependences for solutions of PNIPAM homopolymers [82,83]. Therefore, it was assumed that at the initial step of adsorption, the chains of the shell would be approximately parallel to the surface; however, they are squeezed out into the subphase when the surface concentration increases, and the particles begin to interact with each other. Similar behavior was observed not only for adsorbed but also for spread monolayers of a PNIPAM microgel [76]. Results of cryo-TEM and AFM on surface layer morphology agree with the proposed model [82,83]. However, for spread monolayers after a significant compression, the surface elasticity starts to increase again and reaches higher values than those in the first maximum [84]. The second maximum corresponds to core-core interactions and the collapse of the monolayer structure. High surface elasticities in the range of core-core interactions agree with the behavior of solid particles at the liquid-fluid interface [85,86]. Solid nano- and microparticles with a contact angle around 90° can form very rigid monolayers with a surface elasticity larger than 500 mN/m. The collapse upon compression of the monolayer for nanoparticles can be explained by particle displacement from the surface; however, for microparticles, a compression can lead to the bending or wrinkling of the surface layer [87]. Since the energy required for displacement of particles from the surface strongly depends on the size and contact angle of the particles, the monolayer behavior of the nanoparticles can be intermediate between monolayers of microparticles with extremely high surface elasticity (up to 1000 mN/m) and monolayers of macromolecules with intermediate elasticity values around 50–100 mN/m [80].

Synthetic polyelectrolytes are usually characterized by a lower surface activity than non-ionic polymers, and they can hardly be used as surfactants [88]. Moreover, their adsorption from the solution's bulk to the surface is very slow due to the electrostatic adsorption barrier. Therefore, much more attention has been paid to the surface properties of complexes between polyelectrolytes and oppositely charged LMW surfactants [89]. It was discovered several decades ago that the formation of surface active complexes between surfactant ions and oppositely charged polyelectrolyte molecules leads to a surface tension decrease in a concentration range much lower than that for pure surfactant solutions [90]. At low surfactant concentrations, the complex formation occurs as a result of non-cooperative interactions. However, the increase in surfactant concentration in the solution up to the critical aggregation concentration at a constant concentration of polyelectrolytes leads to cooperative interactions between the macromolecules and the surfactants in the solution's bulk. For different polyelectrolyte/surfactant complexes, one can observe high dynamic surface elasticities in the range of low surfactant concentration [89]. It drops abruptly to low values with increasing surfactant concentration after passing a local maximum and

finally approaches zero. It was assumed that the decrease in the dynamic surface elasticity is connected with the formation of three-dimensional aggregates in the surface layer and the appearance of a new mechanism of surface stress relaxation due to the matter exchange between aggregates and the surrounding film [91,92]. The formation of three-dimensional aggregates (microgel) is confirmed by the strong increase in the ellipsometric signal for solutions of poly(styrenesulfonate) PSS complexes with alkyltrimethylammonium bromides (ATAB) in the mixed adsorption layers due to light scattering by the aggregates [93]. Moreover, the formation of microaggregates was discovered directly by atomic force microscopy for adsorption films [94]. Comprehensive investigations of the dynamic surface properties allow the observation of non-monotonic kinetic dependences in the dynamic surface elasticity of polyelectrolyte/surfactant solutions [95,96]. These dependences indicate that the aggregates are formed directly in the surface layer, and they do not adsorb from the bulk phase. In the range of relatively high surfactant concentrations, the kinetic dependences have two local maxima. The appearance of the second maximum of the surface elasticity's dependence can be explained by the formation of a multilayer structure in the adsorption films of polyelectrolyte/surfactant complexes, which was confirmed using neutron scattering [97]. The addition of ATAB to the dispersion of hydrophilic solid silica nanoparticles allows the enhancement of their surface activity [98]. Hydrophobized silica particles adsorb at the air–water surface and form rigid surface layers with surface elasticities of up to 800 mN/m [99].

4. Protein Solutions

The large number of hydrophobic groups on the surface of protein globules causes the adsorption of these macromolecules at the air–water interface and leads to the formation of a durable adsorption layer [100]. For globular proteins, such as bovine serum albumin (BSA), β -lactoglobulin (BLG), lysozyme or myoglobin, the dynamic surface elasticity monotonically increases up to relatively high values of 60–90 mN/m when approaching the equilibrium [101–105]. The absence of any maximum in the dependences of the dynamic surface elasticity points at the preservation of the tertiary globular structure of the protein molecules in the surface layer.

For non-globular or denatured proteins, the dependences of the dynamic surface elasticity are non-monotonic [102,103,106–108]. For the non-globular β -casein, two local maxima, associated with the displacement of relatively hydrophilic and relatively hydrophobic parts of the molecule into the distal region of the surface layer, appear in the dependences of the dynamic surface elasticity. The destruction of the protein's tertiary molecular structure and at least partial destruction of the secondary structure results in an increased flexibility of the macromolecules and thereby leads to the formation of loops and tails in the surface layer [100]. In some cases, proteins could demonstrate some resistance against the action of denaturants. Lysozyme disulphide bonds help the molecules to preserve a molten globular state in the presence of denaturants [103,104,109].

Not only typical denaturants like Guanidine hydrochloride (GuHCl) and urea cause the unfolding of protein molecules in the surface layer but also some polyelectrolytes and ionic surfactants [34,72,106,107,109,110]. Ionic surfactants cause the destruction of the protein's tertiary structure at concentrations a few orders of magnitude lower than the corresponding concentrations of GuHCl and urea [107,111]. According to the features of the kinetic dependences of the dynamic surface elasticity and dynamic surface tension, the entire surfactant concentration range could be divided into three parts: before critical aggregation concentration; after critical aggregation concentration and before critical micelle concentration; after critical micelle concentration. In the first concentration range, the addition of surfactant does not influence the dynamic surface properties of the protein solution significantly. In the second concentration range, the addition of surfactant leads to the formation of surfactant–protein complexes. A local maximum in the dependences of the dynamic surface elasticity appears if the surfactant concentration is higher than the critical aggregation concentration (about 0.01 mM) and lower than the corresponding CMC.

When the concentration is higher than the CMC, the surfactant monomers displace the complexes from the interface and the surface elasticity tends to zero. For the non-globular β -casein, the addition of surfactant changes the height of the local maxima and increases the corresponding surface pressures needed to displace the macromolecules into the distal part of the surface layer [106,112].

The rate of change in the dynamic surface properties of protein–surfactant complexes depends on the charge of the resulting complex. If it has a charge lower than the protein molecule, the electrostatic adsorption barrier decreases and the rate of change of the dynamic surface elasticity increases. If the components are similarly charged, the opposite situation is observed unless the ionic strength becomes high enough to shield any electrostatic interactions [105,107,111].

5. DNA Layers at the Liquid Surface

The surface activity of DNA is very low. Therefore, to study its surface properties, it is necessary to use complexes of DNA with amphiphilic substances. Such complexes are well known. Their formation in the bulk phase has been studied in some detail, primarily in connection with problems of transferring genes and drugs into the cells of living organisms. It has recently been shown that fibrillar aggregates form at the surface of aqueous solutions of a mixture of DNA and amphiphilic polyelectrolytes and surfactants [113–118]. The first step of adsorption layer formation corresponds to the adsorption of free surfactant molecules. At the second step at low surface coverage, some fibrillar aggregates appear in the surface layer. They do not form a continuous network and are present in the form of islands at the air–water interface, which can be observed by AFM microscopy. On a third step, a continuous network of aggregates is formed at the interface. At the final step, a polylayer is formed. The surface layers of the DNA/polyelectrolyte networks turn out to be fragile. Network formation is a relatively slow process compared to the rapid adsorption of weakly charged complexes. The driving force of this process is the increase in the entropy of the system due to the release of counterions. The study of DNA adsorption on the air–water interface covered with a previously deposited polyelectrolyte monolayer [117,118] shows that for the formation of DNA/PDAHMAC complexes in the surface layer, the system must overcome an energy barrier corresponding to the formation of a nucleus of a DNA aggregate and polyelectrolyte molecules. After the formation of a critical nucleus, the process continues like an avalanche. The formation of networks of mesoscopic aggregates results in an increasing surface elasticity.

6. Fullerene Layers

Fullerene C_{60} spread layers at the water–air interface demonstrate strong adhesion to the water [119]. The dynamic dilational elasticity reaches extremely high values at medium surface pressures, indicating strong interactions between the two-dimensional aggregates in the surface layer. Spread layers of fullerene C_{60} are actually bilayers or trilayers and can also contain large aggregates in the Z-direction with a size of up to approximately 100 nm. The increase in surface pressure upon compression leads to layer destruction and a decreased surface elasticity.

Fullerenes form soluble complexes with amphiphilic polymers and proteins in the bulk. The surface pressure isotherms for mixed films of C_{60} fullerene and polymers [119] or proteins [120] differ noticeably from the isotherms for films of the individual components. For C_{60} /BSA films, the surface properties indicate the formation of mixed aggregates and a strong interaction between fullerene and protein in the surface layer. In both cases, mixed deposited layers contain sections of a network formed by contiguous aggregates with an extension in the Z-direction of less than about 20 nm. This network apparently also contains an amphiphilic component especially in the case of proteins. Even in a highly compressed state, the films consist of curved regions and non-collapsed regions. The formation of such a complex structure can be explained by the strong inhomogeneity of the films and its strong adhesion to the water's surface.

Fullerenols demonstrate relatively weak surface activity [121,122] which is almost independent of the number of hydroxyl groups in the molecule. However, the dynamic surface elasticity increases significantly. For $C_{60}(OH)_{20}$ fullereneol solutions, the surface elasticity reaches very high values of about 300 mN/m [122]. Despite the relatively low molecular weight of fullereneols, the surface properties of their solutions are closer to those of solid nanoparticle dispersions than to those of surfactant solutions. The adsorption layer of fullereneols is microscopically inhomogeneous and contains microaggregates comprised of several layers of molecules. Surface properties of carboxyfullerenes differ significantly from those of fullereneol films [123]. Carboxyfullerenes act more like typical surfactants, in contrast to fullereneols [121,122].

7. Surface Properties of Protein Aggregate Dispersions

Proteins can form different types of aggregates when heated. Among them, fibrils and protein nanoparticles are most frequently observed. In spite of the high importance of dynamic surface properties for the stabilization of dispersed systems by protein aggregates and for their interactions with cell membranes, so far they have attracted less attention than the study of bulk properties [124–126]. It has been shown that polypeptides, which are formed along with aggregates due to protein hydrolysis at high temperature, influence the surface properties of the corresponding adsorption layers. The presence of polypeptides makes the dynamic surface properties of solutions containing aggregates indistinguishable from the properties of pure proteins [125]. However, the purification by dialysis or centrifugation can reduce the impact of admixtures. Spread layers of purified BLG, lysozyme fibrils and microgel layers are characterized by high surface elasticity values [124,125]. For microgels, such values are reached when it is possible to provide a uniform distribution of particles along the surface. It is harder for lysozyme microgels since they tend to form compact three-dimensional clusters at the interface [124].

Probably, the main difference between microgel and fibril layers consists in the collapse mechanism [124–126]. For spread and adsorbed layers of BLG microgel particles, fluctuations of the surface pressure at strong compression are observed. At medium compressions, BLG microgels form bunches which develop into macroscopic three-dimensional structures with increasing surface pressure. In contrast, a collapse caused by fluctuations in fibril layers is uncertain. Their behavior resembles that of amphiphilic polymers. However, the properties of BLG microgel layers resemble an intermediate between the properties of soft and rigid nanoparticles.

8. Summary

The interfacial dilational visco-elasticity is a quantity that includes the essential properties of liquid interfacial layers, formed by various types of surface-active compounds, such as surfactants, polymers, proteins, particles and their mixtures. It is directly interrelated with the equilibrium equation of state representing the dependence of the interfacial pressure as a function of the adsorbed amount. Depending on the rate of deformation, i.e., compression or expansion, it also provides insight into the relaxation mechanisms within the interfacial layer as well as between the interface and the adjacent bulk phase, the so-called sublayer. These mechanisms include diffusion to and from the interface, adsorption/desorption barriers and interfacial conformational changes.

The given overview includes, in particular, the damping of capillary and longitudinal interfacial waves as a very efficient methodology for the measurement of the dilational visco-elastic behavior of interfacial layers, including the early work of the pioneers in the field as well as the remarkable contributions by Boris A. Noskov. Some selected interfacial layers, built by surfactants, proteins and mixtures of surfactants with polyelectrolytes, are discussed in detail. Also, some particular systems, relevant in biophysics and biomedicine and dealing with DNA and fullerenes, are presented and their evidence for other scientific disciplines emphasized.

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