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STRUCTURAL FEATURES
OF LAMELLAR-CHAIN HYDROGELS

The possibility of creating hydrogels of a new lamellar-chain type in the framework of increased rigidity on the basis of ternary aqueous solutions of polymeric and oligomeric molecules has been considered. A hypothetical model of such a framework whose structural elements are oligomeric lamellas and polymer chains is proposed. These elements are connected, because the ends of the polymer chains are embedded into the pores of lamellas. A formula for the free energy of such a system is obtained. It is shown that an increase in the polymer concentration should lead to a mutual approach of the lamellas. This conclusion is consistent with the results of a small-angle X-ray scattering experiment in which ternary aqueous solutions with various concentrations of a polymer (polyethylene glycol) or an oligomer (sodium dodecyl sulfate) are studied.

K e y w o r d s: hydrogel, lamellar-chain network, small-angle X-ray scattering.

1. Introduction

According to modern ideas [1–3], gel is a dispersed system, where

1) particles of the dissolved substance form a porous framework;

2) elastic shear stresses may arise, but the elastic shear modulus of the gel is several orders of magnitude lower than that of solids.

A gel in which the dissolved substance is a polymer is called the polymer gel. Its generally accepted model [4] is shown in Fig. 1. As one can see, the framework of a polymer gel is a network formed by polymer chains connected with one another by long-term chemical or physical bonds. A space region, where such connections are located, is called a node. In Fig. 1, those regions are encircled.

In the case where the solvent is water, the terms "hydrogel" and/or "polymer hydrogel" are used.

The physics of polymer hydrogels has a long history [5–7]. Today, this scientific direction continues to be developed intensively. This is its second birth (see reviews and books [8–11] and the references therein). The main reason is that the structure of polymer networks in hydrogels is similar to that of the intercellular matrix in biological tissues and can im-

itate its physical, chemical, and mechanical properties [8]. As a result, hydrogels are widely applied in various areas of medicine, such as ophthalmology [12, 13], controlled drug delivery systems [14, 15], wound treatment [16, 17], tissue engineering [18, 19], and others.

In view of the aforesaid, we performed a cycle of works aimed at researching the hydrogels based on cellulose derivatives [20–28]. The presented work is aimed at studying the possibility of creating hydrogels based on a ternary aqueous solution of polymeric and oligomeric molecules of a new type, namely, hydrogels with a framework of increased rigidity. A necessity in such hydrogels is associated with a probable application of those polymers in tissue engineering. In this case, their main function will consist in the creation of a space, where all necessary conditions for the biological tissue development would be provided: the organization of the cell structure, its nutrition, delivery of bioactive substances, and so forth. An important role in the implementation of the relevant tasks is played by the hydrogel framework. In work [29], it was noted that the majority of polymer hydrogels used in tissue engineering cannot provide conditions for the development of biological tissues with enhanced mechanical properties, such as bones, muscles, and blood vessels; the obvious reason is the insufficient rigidity of the polymer framework.

To solve this problem, in this article, we consider a possible variant of creating a hydrogel based on polyethylene glycol (PEG) possessing a framework with increased rigidity. The properties of PEG-based hydrogels are widely discussed in the modern literature, in particular, concerning their possible application in industry (water vapor absorption [30], oil production [31], heating/cooling systems in buildings [32], and biomedical technologies (see works [33–35] and the references therein). PEG is an attractive material for tissue engineering, because, as a rule, it does not invoke an immune response [34], although this is not always the case [33]. An idea of creating such a hydrogel consists in the introduction, along with the polymer, of another dissolved substance with a lamellar structure into the solution: this substance, when becoming a part of the framework, could increase its rigidity. For example, a method was suggested in work [36] to create mesostructured hydrogels based on PEG-diacrylate with the addition of the block copolymer P123 [37], which forms lamel-
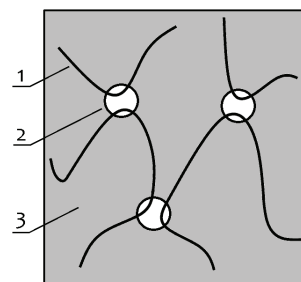
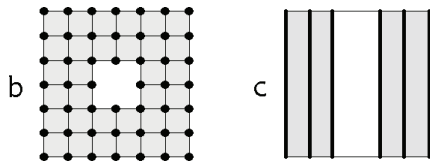**Fig. 1.** Polymer gel structure: polymer chain (*1*), node (*2*), solvent (*3*)
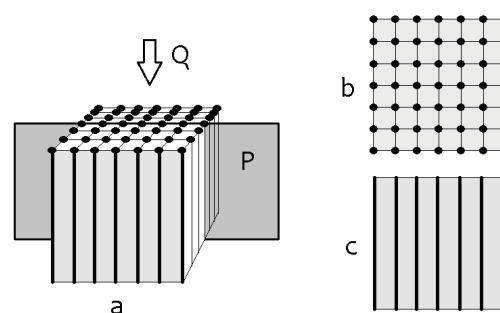
Fig. 2. Oligomeric lamella: general view (*a*), view along the arrow Q (*b*), cross-section of the lamella in the P-plane (*c*)

lar domains. In this work, a simpler model system (water-PEG-sodium dodecyl sulfate) was studied for the first time for the existence of lamellar structures.
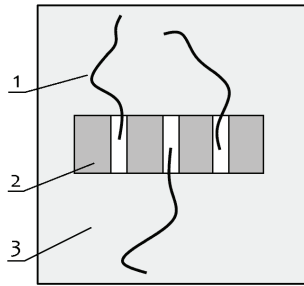
2. Mechanism of Lamellar-Chain Hydrogel Structure Formation

When studying the physical properties of an oligomer, its chains in the totally extended configuration are considered as rods [4]. Being arranged in parallel to one another, those rods form ordered layers, lamellas (Fig. 2). Figure 2, *a* illustrates the general view of a lamella, Fig. 2, *b* demonstrates its top view, and Fig. 2, *c* shows the lamella cross-section along the plane P.

In work [38], on the basis of the concepts of solid state physics about defects in crystals, the issue of the existence of a similar phenomenon for lamellas was considered. The vacancy is one of the defects discussed in solid-state physics. By definition [39], the vacancy is an empty site in the crystal lattice. As one can see from Fig. 2, *b*, the ends of the rods composing a lamella form a plane lattice, and Fig. 3 demonstrates a vacancy in such a lattice. It is clear that this vacancy arises as a result of the removal of the cor-

Fig. 3. Vacancy in an oligomeric lamella: view along the arrow Q (*b*), lamella cross-section in the P-plane (see Fig. 2) (*c*)
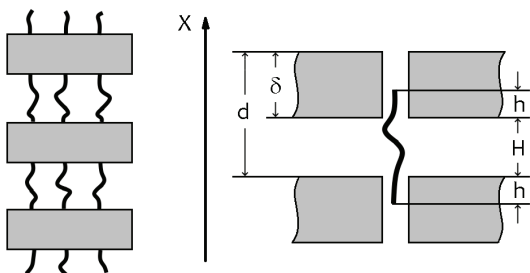

Fig. 4. Node of the lamellar-chain hydrogel framework: polymer chain (*1*), lamellas (*2*), water (*3*)
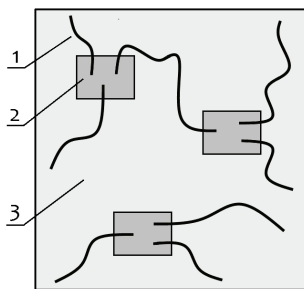
Fig. 5. Lamellar-chain hydrogel structure: polymer chain (*1*), lamellas (*2*), water (*3*)

Fig. 6. Approximate model of the lamellar-chain hydrogel structure. See explanations in the text

responding rod from the lamella. In this case, there appears a pore in the lamella the size of which is equal to the size of the removed rod (Fig. 3, *c*). That is why this defect was called the supervacancy [38].

From the viewpoint of thermodynamics, it is beneficial for the end sections of the polymer chains to enter the pore (the supervacancy) in a lamella, because the total energy of the system decreases at that. If several chains are penetrated into the pores of the same lamella (Fig. 4), a node of the framework of a polymer network is formed in effect. The structure of a hydrogel with such nodes is schematically depicted in Fig. 5. Since the hydrogel structural elements shown in this figure are lamellas and chains, it is logical to call it the lamellar-chain (LC) hydrogel. The framework of the LC hydrogel contains lamellas. Therefore, it is obvious that its rigidity should be higher in comparison with that of the hydrogel (the structure of the latter is shown in Fig. 1).

3. Lamellar-Chain Hydrogel Restructuring Induced by the Polymer Concentration Variation

It is clear that, like any hypothetical model, the model of the LC-hydrogel structure depicted in Fig. 5 has to be verified experimentally. The main structural element of our model is the lamella, which suggests that the application of the small-angle X-ray scattering method [40] to study, for example, the influence of various physical factors on the spatial period in the hydrogel lamellar structure could be an effective tool for such a verification.

Bearing in mind the application of this experimental method, let us determine the dependence of the spatial period in the LC-hydrogel on the polymer concentration in it, i.e., the dependence inherent to the model shown in Fig. 5. Let us consider an approximate model of the LC-hydrogel structure exhibited in Fig. 6. This model is a set of parallel lamellas connected by chains and arranged at the same distance from one another. Let us denote this distance as H.

Obviously, the approximate model corresponds to a periodic structure with the period d that satisfies the equality

$$H = d - \delta, \tag{1}$$

where δ is the lamella thickness. Let us draw the X-axis perpendicularly to the surfaces of the lamellas. The length of the polymer chain projection on the X-axis is equal to

$$\ell = H + 2h, \tag{2}$$

where h is the length of the chain segment located in the lamella's pore. It is known [4, 41] that if the ends

of a polymer chain are moved apart to a distance ℓ, there appears a force of an entropic origin, which is determined by the equality

$$f = 3k_{\mathrm{B}}T\frac{\ell}{Na^2}, \tag{3}$$

where k_{B} is the Boltzmann constant, T the temperature, N the number of chain links, and a the link length. Therefore, the work that must be done to move the chain ends apart to the distance ℓ is equal to

$$A = \frac{1}{2}f\ell = \frac{3}{2}k_{\mathrm{B}}T\frac{\ell^2}{Na^2}. \tag{4}$$

If the chain ends are drawn into the lamellar pore, the energy gain equals

$$U = -2hW, \tag{5}$$

where W is the energy of intermolecular interaction per unit molecular length. The change in the free energy density F_1 due to the drawing of the chain ends into the pore amounts to

$$F_1 = Z(A + U), \tag{6}$$

where Z is the number of chains per volume unit. This quantity is determined by the equality

$$Z = \frac{\phi}{Nv}, \tag{7}$$

where ϕ is the volume fraction of polymer and v is the volume of a polymer link.

The appearance of forces f that attract the lamellas to one another gives rise to the deformation of the LC-hydrogel structure. The corresponding deformation energy density F_2 is equal to

$$F_2 = \frac{E}{2}\left(\frac{d-D}{D}\right)^2, \tag{8}$$

where E is Young's modulus of the lamellar structure, and D is the spatial period in the absence of deformations (at $\phi = 0$). The total change in the free energy density F induced by the appearance of polymer chains in the solution is equal to

$$F = F_1 + F_2. \tag{9}$$

Substituting the quantities F_1 and F_2 into Eq. (9), we have

$$F = \frac{E}{2}\left(\frac{d-D}{D}\right)^2 +$$

$$+\frac{\phi}{Nv}\left\{\frac{3k_{\mathrm{B}}T}{2Na^2}(d-\delta+2h)^2 - 2hW\right\}. \tag{10}$$

At a fixed ϕ, the free energy (10) is a function of two variables, h and d. The equilibrium values of those variables can be determined from the solution equilibrium conditions

$$\frac{\partial F}{\partial h} = 0, \qquad \frac{\partial F}{\partial d} = 0. \tag{11}$$

Substituting Eq. (10) into Eqs. (11), we obtain the following formula for the equilibrium value of the period d:

$$d = D\left(1 - \frac{\phi WD}{ENv}\right). \tag{12}$$

From whence, it follows that the period d should decrease, if a polymer is introduced into the oligomeric solution, and, correspondingly, ϕ increases.

4. Experimental Part

Ternary aqueous solutions of the oligomer sodium dodecyl sulfate (SDS, $C_{12}H_{25}SO_4Na$, Sigma-Aldrich, a molar mass of 288.8 g/mol, a density 1.16 g/cm^3) and the linear water-soluble polymer polyethylene glycol (PEG, $HO(C_2H_4O)_nH$, Merck, a molar mass of $44.05n + 18.02$ g/mol, a density of 1.13 g/cm^3) were studied using the method of small-angle X-ray scattering. For the experiment, four specimens were prepared with the following SDS and PEG volume fractions: 1) $\phi_{\mathrm{SDS}} = 0.1$ and $\phi_{\mathrm{PEG}} = 0.02$, 2) $\phi_{\mathrm{SDS}} = 0.26$ and $\phi_{\mathrm{PEG}} = 0.04$, 3) $\phi_{\mathrm{SDS}} = 0.29$ and $\phi_{\mathrm{PEG}} = 0.2$, and 4) $\phi_{\mathrm{SDS}} = 0.31$ and $\phi_{\mathrm{PEG}} = 0.38$.

The experiment was performed at the Institute of Experimental Physics of the Slovak Academy of Sciences (Košice, Slovak Republic). The measurements were carried out in the air environment at room temperature and the fixed wavelength $\lambda = 1.54$ Å of monochromatic X-ray radiation corresponding to the K-alpha line of the copper anode of the X-ray tube. The distance between the specimen and the detector was 0.69 m, which made it possible to register the scattering vector magnitudes within an interval from 0.01 to 0.3 Å^{-1} with a resolution of 0.005 Å^{-1}. The research specimens were in borosilicate capillaries with a diameter of 1 mm and a wall thickness of 0.001 mm. The specimen exposure time was equal to 2 h.
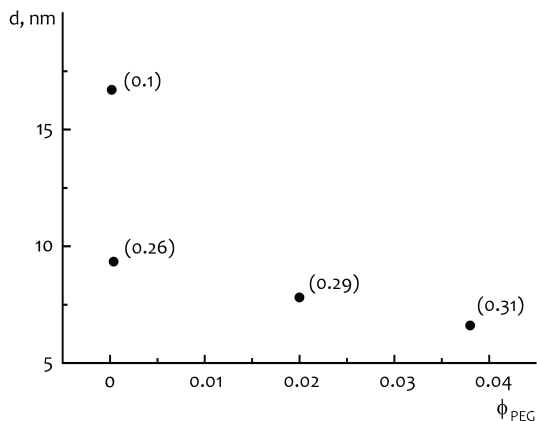
Fig. 7. Dependence of the period d in the structures of examined solutions on the polymer volume fraction ϕ_{PEG}. The corresponding values of the oligomer volume fraction ϕ_{SDS} are given in parentheses

The values of the period d determined from the experimental data for four specimens are shown in Fig. 7. As one can see, the period d decreases with the polymer introduction into the oligomer solution and the corresponding polymer concentration growth. This result agrees with formula (12), which can be considered as a confirmation of the existence of the structure depicted in Fig. 5, i.e., the reality of the LC-hydrogel existence.

As was already mentioned, the hydrogel framework is formed as a result of drawing the ends of polymer chains into the pores (supervacancies) of oligomeric lamellas. Which physical factors are responsible for such a behavior of the chains?

It is known [4] that the decisive role in the physics of polymers belongs to the model of freely-jointed chains. According to this model, a chain in the equilibrium state acquires, on average, the form of a sphere whose radius R undergoes substantial fluctuations. Such a sphere is called the statistical ball. The average value of its squared radius equals [42]

$$\langle R^2 \rangle = Na^2/6. \tag{13}$$

The entropy of a chain in the statistical ball state is maximum. When lamellas appear in the solution, the thermodynamic situation for the chain changes. Now, two factors act on the chain. On the one hand, the drawing of the chain ends into the lamellar pore decreases the system energy. On the other hand, such a drawing decreases the number of possible chain con-

formations, i.e., decreases the system entropy. The framework structure shown in Fig. 5 is a result of establishing a balance between the actions of those factors.

5. Conclusions

In this work, the simple liquid system water-PEG-SDS is considered theoretically and experimentally for the first time in order to verify the existence of lamellar structures. Hydrogels formed in ternary aqueous solutions of polymer and oligomeric molecules have a framework in the form of a network of polymer chains connected with one another by oligomeric lamellas. Such a connection is realized by drawing in the ends of polymer chains into the lamellar pores.

The presence of oligomeric lamellas in the framework of the mentioned hydrogels makes them principally different from hydrogels formed in aqueous polymer solutions, which allows the former to be considered as a new type of hydrogels. Taking the structure of their framework into account, it is reasonable to call such hydrogels lamellar-chain (LC-hydrogels). Note that, due to the presence of lamellas in the framework of LC-hydrogels, such hydrogels have a higher rigidity. This circumstance is promising for the application of LC-hydrogels in tissue engineering to restore or replace such biological tissues as bones, muscles, and blood vessels.

Surely, the researched system cannot be directly applied in tissue engineering. However, the obtained results may be useful in further studies with more complicated systems suitable for biomedical applications.

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ЛАМЕЛЯРНО-ЛАНЦЮГОВІ
ГІДРОГЕЛІ: ОСОБЛИВОСТІ СТРУКТУРИ

Розглядається можливість створення на основі бінарного водного розчину полімерних та олігомерних молекул гідрогелів нового типу – ламелярно-ланцюгових – із каркасом підвищеної жорсткості. Запропоновано гіпотетичну модель такого каркасу, структурними елементами якого є олігомерні ламелі та полімерні ланцюги. Ці елементи з'єднуються завдяки тому, що кінці полімерних ланцюгів втягуються в пори ламел. Отримано формулу для вільної енергії такої системи. Показано, що збільшення концентрації полімера має привести до взаємного зближення ламел. Цей висновок узгоджується з результатами проведеного методом малокутового розсіяння рентгенівських променів експерименту, в якому досліджувались бінарні водні розчини полімера (поліетиленгліколя) та олігомера (додецилсульфата натрію) з різними концентраціями.

К л ю ч о в і с л о в а: гідрогель, ламелярно-ланцюгова сітка, малокутове рентгенівське розсіяння.