

Some peculiarities of the kinetics of interaction of cationic hydrogels based on copolymers of vinyl esters of monoethanolamine and ethylene glycol with copper ions

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ABSTRACT

Over the past few decades, formation of polymer hydrogels complexes with metal ions, including transition ones, attracts the steady attention of researchers. The relevance of such study is due to the fact that the kinetics of swelling and contraction of hydrogels in various media has been studied in many works, however, this question remains insufficiently studied. Cationic hydrogels based on copolymer of vinyl ethers of monoethanolamine (VEMEA) and ethylene glycol (VEEG) as the study subject in the present work have been used. The kinetics of interaction cationic hydrogels based on copolymer of vinyl ethers of monoethanolamine and ethylene glycol with copper ions in aqueous solutions has been studied. It was shown that swelling behavior such hydrogel in this process is characterized two stages. At the initial stages of the interaction of the gel with the copper salt solution, the interaction in question is determined mainly by the displacement of water from the swollen mesh, due to the difference in osmotic pressures inside and outside the sample. At this stage, the kinetics of the gel contraction is described by the same laws as for polyelectrolyte hydrogel in the alkali metal solution. At the second stage, the network charge density increases due to the formation of a complex, and the degree of the gel swelling begins to increase with time. On the basis of the results obtained, it can be assumed that in systems of this kind, metastable states with differences in character from truly equilibrium states, persisting for a long time, can be formed.

Keywords: Coordination bonds, Cationic polymer hydrogels, Complex formation, Swelling behavior, Copolymer.

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1. Introduction

Over the past few decades, formation of polymer hydrogels complexes with metal ions, including transition ones, attracts the steady attention of researchers. The purpose of the article is to study the interaction of cationic hydrogels based on copolymer of vinyl esters of monoethanolamine (VEMEA) and ethylene glycol (VEEG) with copper ions in aqueous solutions. The relevance of such study is due to the fact that the kinetics of swelling and contraction of hydrogels in various media has been studied in many works, however, this question remains insufficiently studied.

An interest to this kind of complexes is traditionally associated with water purification technologies and enrichment technologies [1-8]. This is determined by the fact that the complexes between certain varieties of



hydrogels and ions of such metals as copper, nickel or cobalt are stabilized by coordination bonds, so that the sorption of these metals from the solution becomes a reversible process, which makes it possible to regenerate the adsorbent [9-16]. An important field of this application system type is catalysis by polymer-metal complexes [17-22], that can be considered as analogues of natural metal-containing enzymes. There are other promising areas of hydrogels application with grid density, artificially varied by complex formation; for example, to create new types of energy converters [23-27]. It is appropriate to emphasize that the manipulation of the hydrogel properties by means of complexes with ions such as copper ions can be realized not only through a change in the density of the net charge (for example, when ions are bound by non-ionic functional groups of the network), but also by varying the cross-link density [28-33]. It is important for the realization of a number of multifunctional systems based on polymer hydrogels [34; 35].

The kinetics of swelling and contraction of hydrogels in various media has been studied in many works [36-40]. Traditionally, it is believed that studying the kinetics of the swelling behaviour of hydrogels in solutions of low molecular substances is a more complex task than the study of the equilibrium state, which can be theoretically described in details by methods of classical thermodynamics. However, as it is shown in this paper, the study of swelling behaviour of hydrogels in solutions of low molecular substances provides additional possibilities, including studying the factors determining the degree of swelling / contraction of hydrogels in the equilibrium state. Namely, as it is shown in this paper, there are definite conditions when different factors (from the set that determines the equilibrium degree of swelling) dominate at different stages of interaction of the cationic hydrogel with low molecular weight salt of transition metals solutions. Cationic hydrogels based on copolymer of vinyl ethers of monoethanolamine (VEMEA) and ethylene glycol (VEEG) as the study subject in the present work have been used. These hydrogels have been obtained and their interaction with polyacrylic acid has been studied by G.A. Mun, Z.S. Nurkeeva, V.V. Khutoryanskiy, B.B. Yermukhambetova, S.M. Koblanov, and I.A. Arkhipova [18].

2. Materials and methods

VEMEA, VEEG and divinyl ether of diethylene glycol (DVEDEG) (Figure 1) are commercial products produced by Alash Ltd. (Temirtau, Kazakhstan). They were purified as described by Z.S. Nurkeeva, E.M. Shaikhutdinov, A.Z. Seitov, and C.Kh. Saikieva [41-47]. Hydrogels based on copolymer of VEMEA-co-VEEG were synthesized by g-irradiation polymerization VEMEA and VEEG in the presence of DVEDEG as a crosslinking agent [48-51]. The composition of the copolymer was determined by elemental analysis (with determination of nitrogen content) and potentiometric titration methods. Inorganic salts of the grade "ch.d." were used without further purification, distilled water was used to prepare aqueous solutions.

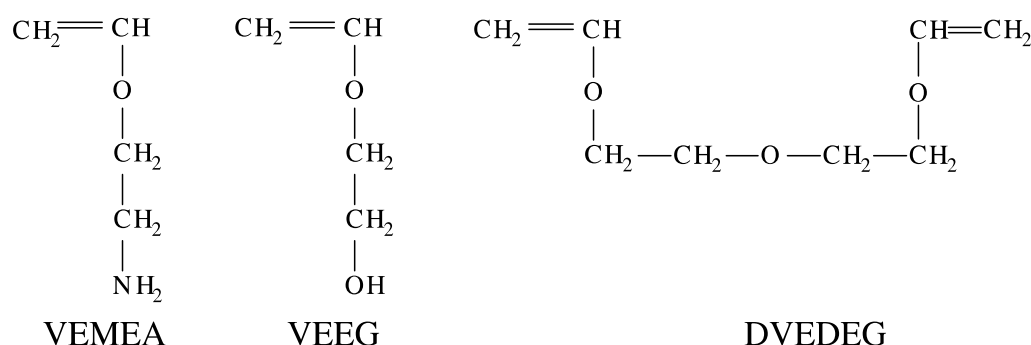


Figure 1. VEMEA, VEEG and divinyl ether of diethylene glycol structure

When studying the interaction of the resulting network polyelectrolytes with solutions of low molecular weight substances, samples of hydrogels in the form of tablets (2 mm in height, 3 mm in diameter) were used, which were previously kept in aqueous solutions for 7-10 days until the equilibrium state was established. The sample was then placed in the test solution. The swelling ratio V/V_0 ; where V_0 and V are the volumes of the polymer network immediately after synthesis and in the moment of measurements, respectively, was used for characterization of the hydrogels swelling in dynamics [52-58]. The diameter of samples was measured using V-630 cathetometer (Russian Federation) with the accuracy of measurements 0.01mm.

When a sample of the VEMEA-co-VEEG hydrogel that in equilibrium is swollen in water, and is placed in an aqueous solution of CuCl_2 , it acquires an intense dark blue colour, and the surrounding salt solution becomes

discolored, which unambiguously indicates the formation of a complex stabilized by coordination bonds. The conclusion about the formation of such complex was also made earlier [59-65]; as it is known, the interaction of transition metal ions with poly-ligands is accompanied by the formation of chelate structures.

3. Results and discussion

The kinetics of the interaction of the VEMEA-co-VEEG hydrogel that is in equilibrium swollen in water, with solutions of copper chloride and potassium chloride is reflected by the curves shown in Figure 2. On the basis of the theory developed by S.A. Vesnebolotskaya, N.G. Bel'Nikevich, and T.V. Budtova [16], the abscissa axis in Figure 2 is not a linear time, but a square root of the variable. It can be seen that swelling behavior of the VEMEA-co-VEEG hydrogel in the process its interaction with the copper chloride solution is characterized two stages nature [66-69]. At the initial stage there is a contraction of the gel, followed up by noticeable swelling (Figure 2, curve 1). At the same time, in the solution of the low-molecular-weight KCl, only the monotonous contraction of the sample of the VEMEA-VEEG hydrogels is observed (Figure 2, curve 2). It can be seen that the initial sections of curves 1 and 2 in the presented figure coincide with high accuracy [70-76].

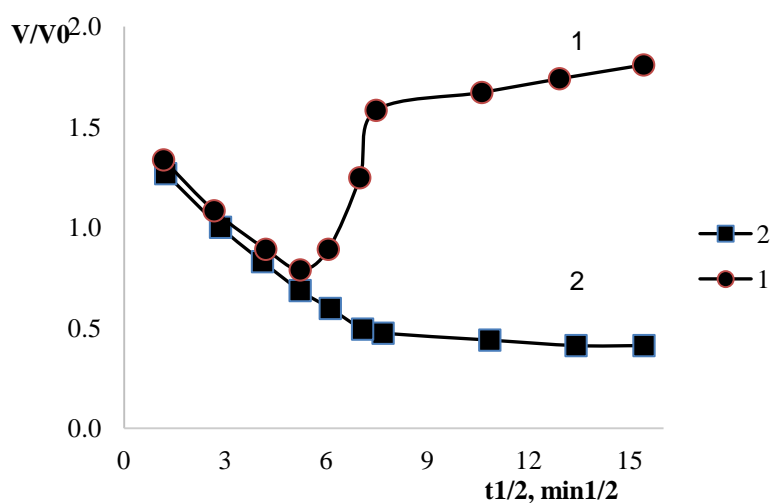


Figure 2. Swelling kinetics of VEMEA-co-VEEG hydrogel in solutions of low-molecular salts (dotted line is a linear approximation of the initial section)

Note: 1-CuCl₂, c = 0.05 mol/L; 2 – KCl, c = 0.05 mol/L; [VEMEA]: [VEEG], Mol. % = 13:87, pH = 5.5.

The medium acidity effect on the swelling behavior of VEMEA-co-VEEG hydrogels in the process its interaction with copper chloride solution is presented in Figure 3. It seen that in medium with low pH=2.4 value (curve 1) the monotonous contraction hydrogel is observed in the copper chloride solution; however, no appreciable staining of the sample occurs [77-83]. When the pH of the medium is increased to pH 6 (at higher pH, the copper hydroxide precipitates), the sorption proceeds more intensively, and the swelling of the hydrogel is observed already at the initial stages of the process (curve 2), i.e., the stage of contraction of the gel at such pH values does not take place [84; 85].

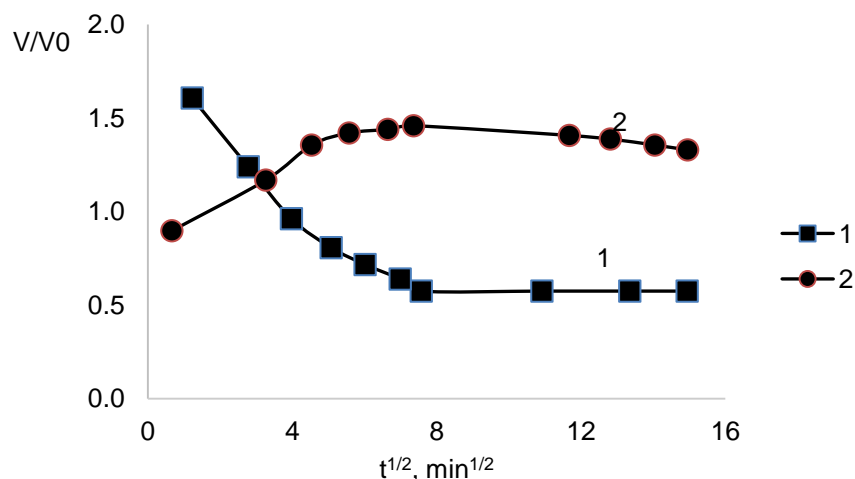


Figure 3. Swelling kinetics of VEMEA-co-VEEG hydrogel in CuCl_2 solution, at various pH values of 2.4 (1), 6.0 (2); (dotted line is a linear approximation of the initial section).

Note: $[\text{CuCl}_2] = 0.05 \text{ mol/L}$; $[\text{VEMEA}]: [\text{VEEG}] = 13:87 \text{ Mol. \%}$.

The swelling behaviour of the VEMEA-co-VEEG hydrogels studied in solutions of copper chloride of various concentrations is shown in Figure 4. It can be seen that there is a noticeable increase in the equilibrium degree of the network swelling as the concentration of copper ions increases. It can also be seen that as the concentration increases, the existence of two different gel swelling stages (the stage of contraction and re-swelling) becomes more pronounced.

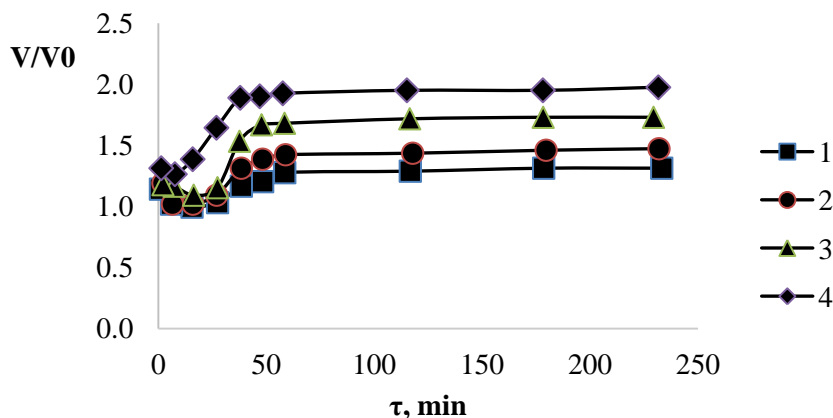


Figure 4. Swelling kinetics of VEMEA-co-VEEG hydrogel in CuCl_2 solutions of various concentrations

Note: $[\text{VEMEA}]: [\text{VEEG}] = 13: 87, \text{ mol. \%}$, $\text{pH} = 5.5$; $[\text{CuCl}_2], \text{ mol / L} = 0.001$ (1), 0.01 (2), 0.05 (3), 0.1 (4).

Significant swelling of VEMEA-co-VEEG hydrogels in water and aqueous solutions is certainly determined by its polyelectrolyte character [86-92]. The degree of polyelectrolyte hydrogel swelling of any type, placed in a solution of a low molecular weight salt, is influenced by factors of two types. Some of them are related to the peculiarities of the medium formation in the volume of the gel (in particular, if we talk about ions that do not enter into these or those reactions with functional networks, partial dissociation of functional groups and / or shielding of network charges by low-molecular ions can occur). Factors of the second type are associated with the features of the solution formation over the gel, more precisely, with its osmotic pressure. Thus, when a polyelectrolyte gel is placed in a solution of a low molecular weight salt, the effect of redistribution of concentrations takes place [93-100], which results in the concentration of the low molecular weight electrolyte in the solution above the gel substantially exceeding the concentration of this component in the bulk of the network. Simplifying, the interaction of low molecular weight electrolyte ions with water leads to the fact that the solvent is separated from the swollen network, and the gel itself decreases in volume.

Figure 2 unambiguously shows that the effects associated with osmotic pressure dominate in the first stage of the gel interaction with the solution of any low molecular weight salt: the initial sections of curves 1 and 2 coincide, which indicates an identical mechanism that causes the contraction. It is appropriate to emphasize that the interaction of copper and potassium ions with the network of the type under investigation proceeds according to completely different mechanisms and, consequently, in the first stage the factors associated with the peculiarities of the medium formation inside the gel do not appear [101-112]. This fact also indicates that the characteristic rates of processes associated with the influence of osmotic pressure on the hydrogel of the type being investigated significantly exceed the rate of formation of the polymer-metal complex. It should be emphasized that the actual rate of formation of such a complex is reduced because two counterflows develop during the contraction of the hydrogel: the flow of water from the volume of the hydrogel and the oppositely directed diffusion flux of low-molecular ions (Figure 5).

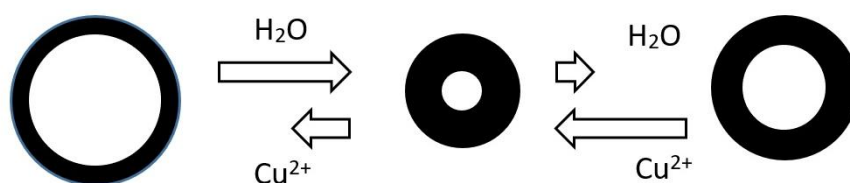


Figure 5. A diagram illustrating the existence of two different stages in the interaction of VEMEA-co-VEEG hydrogel with copper ions

The observed swelling of the gel in the second stage of its interaction with the copper chloride solution is obviously due to the formation of a complex between the metal ion and the -NH_2 groups stabilized by coordination bonds [113-119]. (The network acquires an additional charge due to copper ions.) However, the complex formed with the participation of the polyligand creates additional cross-links, as a result of which the equilibrium degree of swelling remains relatively low, Figure 6.

Figure 2 also shows that the contraction of the gel proceeds according to the mechanism of the frontal reaction; as this figure shows, at the initial stage of the interaction of the gel with solutions of both salts, its degree of swelling linearly depends on the square root of time, which corresponds to the regularity analysed by T. Budtova, I. Suleimenov, and S. Frenkel [13] Equation (1):

$$l \sqrt{tD}, \quad (1)$$

where l is the linear displacement of the hydrogel boundary under contraction, D is the diffusion coefficient of the solvent in the cross-linked network.

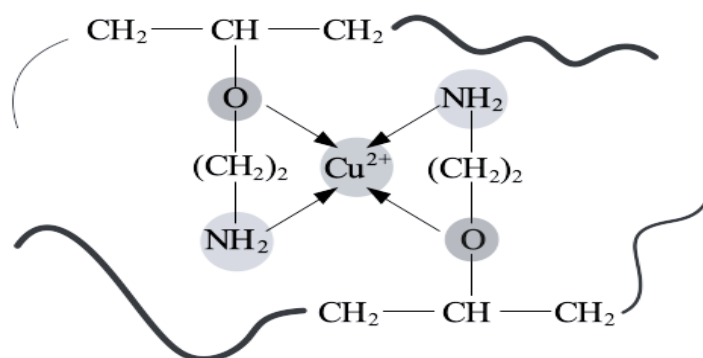


Figure 6. Formation scheme of complex in process of the interaction of VEMEA-co-VEEG hydrogel with copper ions

The contraction of the gel in a solution of copper chloride with low pH value (Figure 3, curve 1) also proceeds according to the mechanism of the frontal reaction, which follows from the linear dependence of the degree of gel swelling on the root of the square of the time in the initial section of this curve. The nature of the acidity

effect of the medium on the formation of the complex is reflected in Figure 6. The figure shows that in a strongly acidic medium, the monotonous contraction of the hydrogel in the copper chloride solution is observed, but there is no noticeable staining of the sample. This is due to the fact that in solutions with a low pH, the amino groups of the copolymer are predominantly in the protonated state, and accordingly, the number of copper ions forming the complex with the copolymer is small. The gel compression in this case proceeds by the same mechanism as in the solution of any other salt, the ions of which do not react with the functional groups of the network [120].

On the contrary, at pH values corresponding to a weakly acidic medium, when the number of protonated amino groups is relatively small, the process of interaction with the copper chloride solution proceeds in one stage: the complex is formed immediately. Figure 4 shows that the competition between the factors connected with the effect of the osmotic pressure of the solution on the hydrogel and the factors connected with the formation of the polymer-metal complex is determined primarily by the pH of the medium and not by the concentration of metal ions. The minimum of all curves in this figure is approximately the same time, whereas the equilibrium degree of the hydrogel swelling increases markedly as the concentration of copper ions increases.

4. Conclusions

Thus, the nature of the interaction of the hydrogel which is capable of forming complexes with copper ions stabilized by coordination bonds, is determined by two competing factors:

- the change in the difference in osmotic pressures inside and outside the sample, which leads to the displacement of water from the swollen mesh when placed in a solution of copper salt;
- the formation of a complex, which leads to the fact that the density of the network charge increases.

The first of these factors dominates the initial stages of the interaction of the gel substance with the copper salt solution, because the water separating from the network creates a flow that prevents the penetration of copper ions into the gel. The second factor dominates at the last stages of the process, since the contraction rate drops to low values and the formation of a complex stabilized by coordination bonds begins to proceed effectively. By this reason at the second stage, the network charge density increases due to the formation of a complex, and the degree of the gel swelling begins to increase with time. On the basis of the results obtained, it can be assumed that in systems of this kind, metastable states with differences in character from truly equilibrium states, persisting for a long time, can be formed.

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