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## FEATURES OF ELECTROCHEMICAL AND CONFORMATIONAL BEHAVIOR OF HYDROGELS POLYACRYLIC AND POLYMETHACRYLIC ACIDS, POLY-4-VINYLPYRIDINE AND POLY-2-METHYL-5-VINYLPYRIDINE IN AQUEOUS MEDIUM AND LANTHANUM NITRATE SOLUTION

**Abstract.** Electrochemical and volume-gravimetric behavior of rare-crosslinked polymer hydrogels of polyacrylic acid (hPAA), polymethacrylic acid (hPMAA), poly-4-vinylpyridine (hP4VP) and poly-2-methyl-5-vinylpyridine (hP2M5VP) were studied by methods of conductometry and pH-metry. It is found that in aqueous medium specific electric conductivity increases, pH decreases for polyacids and for polybases, swelling degree increases. The most significant changes occur during 6 hours, further increase is not so intense, what indicates that system reaches equilibrium state. In lanthanum nitrate solution electric conductivity, pH also decreases and swelling degree of hydrogels decreases a result of sorption lanthanum ions by polyacids and polybases.

**Keywords:** hydrogels, polyacrylic acid, polymethacrylic acid, poly-4-vinylpyridine, poly-2-methyl-5-vinylpyridine.

**Introduction.** The appearance of selectivity in functional polymers is due to the affinity of heteroatoms to metal ions and flexibility of polymer chain, allowing several ligands simultaneous interaction with complexing agent [1-2]. Heteroatoms in flexible chains can form spirals or spiral-like structures. Chains with volumetric side substituents are particularly prone to such conformational transformations. If the dimensions of pores of spirals or similar structures match the size of ion, maximum binding of the metal ion by the polymer is observed. The hydrated shell of metal ions is completely or partially replaced by the heteroatoms of the hydrogel units [3-9].

As known, in aqueous solutions and salt solutions there is an electrochemical equilibrium. Any intervention in the solution leads to a change in this balance.

Most hydrogels are polyelectrolytes [10]. The conformational behavior of polyelectrolytes is greatly influenced by degree of ionization of macromolecular globes [11-13]. Macromolecules interact with the ions present in the solutions.

Polyacids are prone to dissociation of carboxyl groups, sorption of lanthanum ions by its addition to the oppositely charged carboxylate anion. The polybases are subjected to protonization of nitrogen heteroatoms, leading to a change in their conformation.

## EXPERIMENTAL PART

*Equipment.* For measurement of electric conductivity a conductometer MARK 603 (Russia) was used, solutions pH was measured on pH-meter Metrohm 827 pH-Lab (Switzerland). Mass of swelled samples for further calculation of swelling degree ( $\alpha$ ) was determined by weighing on analytic scales SHIMADZU AY220 (Japan).

*Materials.* Studies were carried out in distillated water and in 0.005M solution of lanthanum nitrate. Hydrogels of polyacrylic (PAA) and polymethacrylic (PMAA) acids were synthetized in presence of crosslinking agent N,N-methylene-bis-acrylamide and redox system K<sub>2</sub>S<sub>2</sub>O<sub>8</sub>–Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. Hydrogels of poly-4-vinylpyridine (P4VP) was synthetized by «Sigma Aldrich» company (2% of crosslinking agent). Hydrogel of poly-2-methyl-5-vinylpyridine (P2M5VP) was synthetized in medium of dimethylformamide in presence of crosslinking agent epichlorohydrin. Swelling degrees of the hydrogels are:  $\alpha_{(hPAA)}$ =27.93 g/g,  $\alpha_{(hP4VP)}$ =3.27 g/g,  $\alpha_{(hP2M5VP)}$ =3.20 g/g.

*Experiment.* Experiments were carried out at a room temperature. Calculated amount of each hydrogel was put into distillated water and 0.005M solution of lanthanum nitrate. After that measurement of parameters (specific electric conductivity, pH and mass of the samples) was made during 48 hours. Measurements of conductivity and pH were made in absence of the hydrogels in solutions. Swelling degree was calculated in accordance with equation:

$$\alpha = \frac{\mathbf{m}_2 - \mathbf{m}_1}{\mathbf{m}_1}$$

where  $m_1$  – mass of dry hydrogel, g;  $m_2$  – mass of swelled hydrogel, g.

### **RESULTS AND DISCUSSION**

Study of electrochemical and volume-gravimetric properties of PAA hydrogel. Figure 1 shows dependence of change of specific electric conductivity of water solution and lanthanum nitrate solution in presence of PAA hydrogel from time. Electric conductivity increases with time, strong increase occurs during 6 hours after beginning of the contact of hPAA with water.

Sharp increase of electric conductivity points to the fact of absence of carboxylate anions in solution in initial moment, however, amount of them increases with time and carboxyl groups dissociation degree reaches threshold of dissociation. Taking in account slight increase of conductivity in time interval 24-48 h it is possible to conclude that at 48 h equilibrium is reached in an aqueous medium.

As seen from figure 1, electric conductivity of lanthanum nitrate decreases with time in presence of hPAA. Along with dissociation of carboxyl groups hPAA sorbes ions of lanthanum from the solution. During first 30 min there is a sharp decrease of conductivity. Minumum values of the parameter are observed at 48 h.



Figure 1 – Dependence on specific electric conductivity of PAA hydrogel in aqueous medium and lanthanum nitrate solution versus of duration

Figure 2 shows dependence of hydrogen ions concentration in water solution from time in presence of hPAA. Strong release of protons occurs during first 2 h. Decrease of pH after 24 hoccurs very slightly, minimum values are reached at 48 h. In turn it indicates to equilibrium in the solution. Such change of pH may evidence about occurrence of dissociation of functional carboxyl groups providing increase of protons concentration in an aqueous medium.



Figure 2 – Dependence on pH of PAA hydrogel in aqueous medium and lanthanum nitrate solution versus of duration

In lanthanum nitrate solution decrease of pH in presence of hPAA is observed. Overwhelming majority of protons is released during 6 hours. Increase of hydrogen ions concentration is due to sorption of lanthanum ions by polymer hydrogel of PAA, due to what there is an imbalance between carboxylate anions and protons and the equilibrium shifts to the right (proton formation).

Figure 3 shows change of swelling degree of hydrogel of PAA in time. During 6 hours in water there is a significant increase of swelling degree of the hydrogel. It is due to unfolding of the macromolecular globe. After that there is a slight increase of swelling degree in time interval 24-48 h. Maximum values of the parameter are observed at 48 h of contact of the polymer hydrogel of PAA with water medium.



Figure 3 – Dependence on swelling degree of PAA hydrogel in aqueous medium and lanthanum nitrate solution versus of duration

Swelling behavior of the hydrogel of PAA in salt solution is radically different comparatively to swelling in water medium. Main reason of this is sorption of  $La^{3+}$  ions. During first 30 min of interaction there is a strong increase of hPAA swelling degree, what is due to it's ionization during interaction with the salt solution. After that there is decrease of swelling due to folding of macro-molecular globe.

Study of electrochemical and volume-gravimetric properties of PMAA hydrogel. Figure 4 represents dependence of specific electric conductivity of water medium and lanthanum nitrate from time in presence of PMAA hydrogel. Strong increase of the parameter occurs during first 6 h of interaction of hPMAA with water. This is due to dissociation of functional groups on inter-node links of the macromolecule. The result of this phenomenon is release of hydrogen ions into the solution along with carboxylate anions formation. Further increase up to 24 h occurs not so intense. After that up to 48 hours electric conductivity increases very slightly, what points to electrochemical equilibrium.



Figure 4 – Dependence on specific electric conductivity of PMAA hydrogel in aqueous medium and lanthanum nitrate solution versus of duration

In lanthanum nitrate solution there is occurrence of hPMAA along with lanthanum ions sorption. Result of this is decrease of electric conductivity. Exact decrease is observed during 2 h. This provides conclusion that sorption mechanism is predominantly coordinating. In case of ionic exchange there should be increase of conductivity due to the fact that ionic mobility of hydrogen ions is much higher comparatively with ions, which formed during lanthanum nitrate dissociation.

Figure 5 shows dependence on pH of hydrogel PMAA in aqueous medium and lanthanum nitrate solution versus of duration. It initial moment of time there is occurrence of significant decrease of pH, what evidences about release of protons during electrolytic dissociation of carboxyl groups. Further increase of protons concentration is observed up to 24 h. Reason of it is dissociation of -COOH groups on H<sup>+</sup> and –COO<sup>-</sup>. After 24 hours dissociation occurs slowly, what indicates reaching of electrochemical equilibrium. Contact of hPMAA with the salt solution provides release of protons, main part of which is released during 6 h. Additional release of hydrogen ions occurs due to shift of electrochemical equilibrium to the right due to lanthanum ions sorption.

Dependence of hPMAA swelling degree from time is shown on figure 6. Sharp increase of swelling is observed during 2 h. This is due to dissociation of functional groups and unfolding of polymer globe. Further increase of swelling degree occurs slowly, what points to equilibrium in the system.

Behavior of hPMAA in the salt solution is radically different from interaction of hPMAA with water. During the 1 h there is an increase of swelling degree, which is accompanied by further decrease. Minimum values of the parameter are reached at 48 h of interaction of hydrogel of polymethacrylic acid



Figure 5 – Dependence on pH of PMAA hydrogel in aqueous medium and lanthanum nitrate solution versus of duration



Figure 6 – Dependence on swelling degree of PMAA hydrogel in aqueous medium and lanthanum nitrate solution versus of duration

with lanthanum nitrate. Decrease of swelling degree with time indicates the sorption of lanthanum by the polymer hydrogel.

Study of electrochemical and volume-gravimetric properties of P4VP hydrogel. Dependence of specific electric conductivity of water and lanthanum nitrate from time in presence of hP4VP is shown on figure 7. Obtained results show that electric conductivity of water increases with time. One of reasons of it is binding of protons, formed in result of water dissocitation, by nitrogen atoms



Figure 7 – Dependence on specific electric conductivity of P4VP hydrogel in aqueous medium and lanthanum nitrate solution versus of duration

and further shift of equilibrium to the right (to proton formation). Significant increase occurs during 6 hours after start of contact of hP4VP with aqueous medium. After that increase occurs not so intense up to 48 hours.

In lanthanum nitrate solution the dissocitated lanthanum ions binding with polybasis occurs as result decreased the electric conductivity. Significant decrease of it is observed during 2 h and further change was in significantly.

Figure 8 shows dependence of hydrogen ions concentration in water medium and lanthanum nitrate solution from time in presence of hydrogel of P4VP. As



Figure 8 – Dependence on pH of P4VP hydrogel in aqueous medium and lanthanum nitrate versus of duration

seen from figure, slight increase of pH values occurs due to association of protons by heteroatoms of polybasis. It should be noted that during all time of contact of the polymer with water medium decrease of hydrogen ions concentration is observed. In initial moment of time protons are intensively bind by nitrogen atoms in process of polybasis ionization, what is evidenced by significant decrease of their concentration. After 6 h degree of their association decreases due to what increase of pH becomes more slightly. Maximum values of pH are reached at 48 h.

As seen from figure 8, proton formation in the lanthanum nitrate solution occurs during all the time as result of interaction with polymer hydrogel P4VP. Sorption of lanthanum ions by the polybasis lead to formation coordination bonds of heteroatoms with the metal ions. Protonization of the basic hydrogel occurs due to binding of forming ions of hydrogen by heteroatoms of P4VP, due to this phenomenon the equilibrium shifts to the right, and, as a result, additional release of protons in solution occurs. Further interaction provides sorption of lanthanum in accordance with coordination mechanism due to what protons are released into the solution.

Swelling degree of hP4VP in water and in lanthanum nitrate has radically differrent character (figure 9). Swelling of the polybasis in water occurs very intensively during first 2 h. Further increase of swelling occurs more slightly. After 24 h swelling degree of P4VP almost does not change.



Figure 9 – Dependence on swelling degree of P4VP hydrogel in aqueous medium and lanthanum nitrate solution versus of duration

Behavior of the polybasis in lanthanum nitrate solution shows that significant increase of swelling degree of hP4VP occurs during 30 minutes after beginning of the contact. Further interaction is accompained by swelling decrease. Minimum values of swelling degree are observed at 48 hours.

Study of electrochemical and volume-gravimetric properties of P2M5VP hydrogel. In water solution significant changes of specific electric conductivity in presence of hydrogel of P2M5VP occur (figure 10). Strong increase is observed during first 6 h. Reason of such significant increase of conductivity is process of hydrogen ions binding, which were formed during water molecules dissociation, by nitrogen atoms of hydrogel of P2M5VP. Further increase of values of electric conductivity is not very intensive especially after 24 h, what may point to the fact that in system hydrogel of P2M5VP – water electrochemical equilibrium is set. Interaction of polymer hydrogel of P2M5VP with lanthanum nitrate provides binding of dissociated ions of the metal, what, in turn, provides decrease of values of specific electric conductivity of the solution. Strong decrease is observed at 30 min. Further decrease is slight up to 48 h of interaction of the polymer with the salt solution.



Figure 10 – Dependence on specific electric conductivity of P2M5VP hydrogel in aqueous medium and lanthanum nitrate solution versus of duration

Obtained results on electric conductivity provides conclusion that sorption of lanthanum ions by hP2M5VP occurs due to coordination mechanism.

Impact of presence of hydrogel of poly-2-methyl-5-vinylpyridine on pH of water medium and lanthanum nitrate solution in time is shown on figure 11. Change of pH of water occurs similarly to changes of hydrogel of P4VP. During first 6 h there is sharp decrease of protons concentration. It is due to predominance of binding process over protons release in the solutions. After 24 h pH increase occurs more slightly in comparison with initial time moment. Final values of pH of water solution are observed at 48 h of interaction of basic polymer hydrogel of P2M5VP with solution.

Proton release is also occurred during lanthanum sorption by hydrogel of P2M5VP. Ionization of this polybasis occurs due to formation of coordination



Figure 11 – Dependence on pH of P2M5VP hydrogel in aqueous medium and lanthanum nitrate solution versus of duration

bonds similarly to P4VP. Overwhelming majority of hydrogen ions is released during 6 h. After 24 h of interaction of polymer hydrogel of poly-2-methyl-5vinylpyridine with lanthanum nitrate solution proton release process is so insignificant that it is possible to conclude that electrochemical equilibrium is set.

Curves of swelling degree of hP2M5VP dependence from time in aqueous medium and lanthanum nitrate solution are shown on figure 12. As known, hydrogel of P2M5VP is weak polybasis and swells very slightly. Sharp increase



Figure 12 – Dependence on swelling degree of P2M5VP hydrogel in aqueous medium and lanthanum nitrate solution versus of duration

of the hydrogel's swelling in initial moment of time in water can be explained by the fact that there are conformational changes in structure of macromolecule in result of ionization of the hydrogel. It should be noted that significant increase of swelling degree of hydrogel is observed during first 6 hours of interaction. Maximum values of swelling degree are observed in water at 48 h.

Behavior of hydrogel of P2M5VP in lanthanum nitrate solution is rather different from it's behavior in water. There is an increase of swelling degree of the polybasis for the first 30 min of contact with further decrease of this parameter due to conformational changes in macromolecular structure in result of rare-earth metal sorption. Minimum values of swelling degree of basic hydrogel of P2M5VP are seen at 48 h of interaction of polymer hydrogel with the salt solution.

#### **Conclusion.**

1. Electrochemical behavior of hydrogels PAA, PMAA, P4VP and P2M5VP in aqueous medium shows that during the prosece there are occurrence of the following changes: specific electric conductivity increases due to dissociation of water molecules and carboxyl groups; pH decreases at presence of polyacids what points to carboxyl groups dissociation and pH increases at presence of polybases what points to partial binding of protons formed as result dissociation of water molecules.

2. Conformational behavior of hPAA, hPMAA, hP4VP, hP2M5VP in water medium points to the fact that rare-crosslinked polymer hydrogels of acid and basic nature undergo swelling. The most intensive swelling occurs during 6 h after beginning the contact of the macromolecules with water.

3. Electrochemical properties of lanthanum nitrate solution point to sorption of rare-earth metal by hPAA, hPMAA, hP4VP, hP2M5VP. This is evidenced by decrease the specific electric conductivity and pH. Swelling degree of hPAA, hPMAA, hP4VP, hP2M5VP in lanthanum nitrate solution also decreases due to sorption of lanthanum ion.

4. Study of behavior of individual rare-crosslinked hydrogels of PAA, PMAA, P4VP and P2M5VP provides conclusion that nature of medium (water or lanthanum nitrate) impacts on change of electrochemical and conformational properties of the initial macromolecules.

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#### Резюме

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### ПОЛИАКРИЛ ЖӘНЕ ПОЛИМЕТАКРИЛ ҚЫШКЫЛДАРЫ МЕН ПОЛИ-4-ВИНИЛПИРИДИН ЖӘНЕ ПОЛИ-2-МЕТИЛ-5-ВИНИЛПИРИДИН ГИДРОГЕЛЬДЕР ЕРІТІНДІСІНДЕГІ ЭЛЕКТРОХИМИЯЛЫҚ ЖӘНЕ КОНФОРМАЦИЯЛЫҚ ЕРЕКШЕЛІКТЕРІ СУДАҒЫ ЖӘНЕ ЛАНТАН НИТРАТЫ

Аннотация. Су ортасында және лантан нитратының ерітіндісінде полиакрил қышқылының (ПАҚг), полиметакрил қышқылының (ПМАҚг), поли-4-винилпиридиннің (П4ВПг) және поли-2-метил-5-винилпиридиннің (П2М5ВПг) сирек торланған гидрогельдердің электрохимиялық және көлемдік-гравиметрлік касиеттері кондуктомтерия және pH-метрия әдістерімен зерттелген. Су ортасында нақты электрөткізгіштігінің жоғарылауы, қышқыл гидрогельдер үшін pH төмендеуі, негізгі гидрогелдер үшін pH-нің ұлғаюы және ісік дәрежесінің жоғарылауы анықталды. Ең күшті өзгерістер 6 сағаттан кейін байқалады, содан кейін өсім қарқынды емес, бұл тепе-теңдікке көзқарасты көрсетеді. Лантаның нитратының ерітіндісінде электр өткізгіштігі азаяды, pH төмендейді, ал ісіну деңгейі төмендейді, бұл лантаның иондарының полиакидтер мен поли-негіздермен сорбциясын көрсетеді.

**Түйін сөздер:** гидрогельдер, полиакрил қышқылы, полиметакрил қышқылы, поли-4-винилпиридин, поли-2-метил-5-винилпиридин.

#### Резюме

#### Т. К. Джумадилов, Р. Г. Кондауров, А. М. Имангазы

## ОСОБЕННОСТИ ЭЛЕКТРОХИМИЧЕСКОГО И КОНФОРМАЦИОННОГО ПОВЕДЕНИЯ ГИДРОГЕЛЕЙ ПОЛИАКРИЛОВОЙ И ПОЛИМЕТАКРИЛОВОЙ КИСЛОТ, А ТАКЖЕ ПОЛИ-4-ВИНИЛПИРИДИНА И ПОЛИ-2-МЕТИЛ-5-ВИНИЛПИРИДИНА В ВОДНОЙ СРЕДЕ И В РАСТВОРЕ НИТРАТА ЛАНТАНА

Аннотация. Исследовано электрохимическое и объемно-гравиметрическое поведение редкосшитых полимерных гидрогелей полиакриловой кислоты (гПАК), полиметакриловой кислоты (гПАК), поли-4-винилпиридина (гП4ВП) и поли-2метил-5-винилпиридина (гП2М5ВП) в водной среде и растворе нитрата лантана методами кондуктометрии и рН-метрии. Установлено, что в водной среде происходит возрастание удельной электропроводности, снижение рН для кислотных гидрогелей, увеличение рН для основных гидрогелей и увеличение степени набухания. Причем наиболее сильные изменения наблюдаются на протяжении 6 ч, далее рост не столько интенсивный, что указывает на приближение к состоянию равновесия. В растворе нитрата лантана происходит снижение электропроводности, уменьшение рН и уменьшение значений степени набухания, что указывает на сорбцию ионов лантана поликислотами и полиоснованиями.

**Ключевые слова:** гидрогели, полиакриловая кислота, полиметакриловая кислота, поли-4-винилпиридин, поли-2-метил-5-винилпиридин.