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STUDY OF THE CHEMICAL TECHNOLOGICAL PROCESS FOR OBTAINING HYDROGELS BASED ON ACRYLIC ACIDS

Abstract. It is known that one of the main physical and chemical properties of absorbents is their water-absorbing capacity, which depends on the degree of neutralization of the carboxyl groups of the main monomer and the concentration of the cross-linking agent. In this study, novel crosslinking agents based on divinyl quaternary ammonium salts of dialkylaminoalkyl methacrylates (DMAEMA and DEAEMA) with dihalogen compounds (such as 1,2-dibromoethane and 1,3-dichloropropanol) were successfully synthesized and characterized. These salts demonstrated spontaneous polymerization in aqueous and ethanol media at 20-50°C, forming water-soluble products at low conversion levels (8-10%). Further polymerization led to the formation of crosslinked structures. Kinetic studies revealed a first-order reaction with respect to monomer concentration, with the rate significantly increasing as concentration rose from 0.25 to 0.5 mol/L. Furthermore, the detailed technological processes for both crosslinker and hydrogel production were established, including temperature, timing, and purification steps.

Keywords: hydrogels, acrylic acids, process for obtaining, swelling dynamics, acrylate hydrogel, technological process.

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Introduction. The use of hydrosorbent polymers as moisture adsorbents has been widely developed in agriculture. However, polymer gels have a number of properties, the use of which would allow expanding the areas of their application. The most interesting, from the point of view of practical use, is the behavior of hydrogels in solutions of various polyvalent metals. Polymer hydrogels based on acrylic acid are capable of exchanging ions with soil structures, which was noted in the early stages of the work [1,2].

Cation exchange capacity is one of the functional properties of soil. Cation exchange is a special case of a more general ion exchange in soil, i.e. its ability to absorb and exchange ions at the boundary of two phases. K.K. Gedroits called the part of the soil capable of exchanging calcium and magnesium for ammonium the soil absorption complex [3,4]. The importance of the soil absorption complex is unusually great. It is "the most valuable part of the soil and as it is destroyed, the soil increasingly passes from a set of very complex and comparatively unstable compounds that determine its life into a mixture of simple and stable compounds, i.e. into a dead body". In this aspect, it is of interest to study the ion exchange properties of materials based on acrylic acid cross-linked with cellulose derivatives [5,6].

At present, ion exchange methods of extraction, purification and separation of complex mixtures have become widespread and have become an integral part of most technological processes and scientific research. With the expansion of the scope of application of ionites, continuously increasing requirements are imposed on them [7,8].

Usually the quality of ionites is characterized by their sorption and physicochemical properties, the study of which is the subject of numerous works by scientists, both domestic and foreign. The efficiency of ion exchange on ionites is determined by a number of factors: the degree of dissociation, ionogenic groups, chemical and thermal resistance, mechanical strength, cost price [9,10].

In this aspect, the synthesis of new water-swelling ionites based on acrylic acid and its derivatives is of interest. There are a large number of works devoted to the synthesis of ionites based on these materials, which differ from some known ion sorbents by greater exchange capacity, stability to high-temperature operating modes, and relative cheapness. In the Republic, acrylic acid derivatives are produced on an industrial scale.From the above it follows that the study of the sorption properties of gels, on the one hand, and the synthesis of new ionites based on acrylates, on the other, is of certain practical and scientific interest [11-14].

Materials and methods. In the work, polymers of partially neutralized acrylic acid cross-linked with carboxymethylcellulose were used. Water-absorbing hydrogels are spatially cross-linked natural or synthetic polymers, as well as their compositions, possessing high hydrophilicity capable of retaining a large amount of water in a swollen state 1000-10000 g/g. A number of hydrogels possessing valuable properties were synthesized, the most interesting are polymers of the type Hydrout-1, Hydrout-2 and Hydrout-3. Table 1 presents data on the n_{sw} properties of these hydrogels.

Table 1

Main characteristics of the obtained hydrogels

Polymer name	Degree of swelling in distilled water (g/g)	Degree of swelling in 1% aqueous NaCl solution (g/g)
Acrylic acid + starch (Hydroyut-1)	500-1000	100-150
Acrylic acid (50-60% saponification) + CMC (Hydroyut-2)	1000-1500	150-200
Acrylic acid (40% saponification) + CMC (Hydroyut-2)	1000-1500	150-200

It should be noted that cationites based on acrylic acid derivatives are particularly distinguished by high sorption activity for non-ferrous metals, and in the early stages of water purification development, ordinary cotton wool was used to sorb heavy metals (for example, lead). This is obviously explained by the structure of the framework of these substances, as well as the presence of characteristic groups included in their composition.

In the practice of ion exchange technology, there are frequent cases of contact of pontoons with hot solutions. An increase in temperature leads to acceleration of diffusion processes during sorption and desorption, to a decrease in the influence of non-exchange sorption forces and to acceleration of reaching equilibrium in the system. Therefore, the thermal stability of ionites is of great practical importance.

In the study of metal sorption by gels, polymer hydrogels based on acrylic acid with a degree of saponification of 10%, 20%, and 30% were used. Saponification was carried out with potassium hydroxide. The amount of crosslinking agent a for all samples was the same, with a ratio of 1:7 relative to the monomer. Some physicochemical characteristics of gels, studied as cation exchangers, are presented in Table 2. Based on literature data, the physicochemical properties of the polymer hydrogels are summarized in Table 3.

Table 2

Physicochemical characteristics of gels

Polymer name	Appearance	Degree of swelling in distilled water (g/g)	Degree of swelling in 1% aqueous NaCl solution (g/g)
Acrylic acid (10% saponification) + CMC	White or yellowish powder	400-450	50-65
Acrylic acid (20% saponification) + CMC	–	500-550	65-70
Acrylic acid (30% saponification) + CMC	–	600-650	70-75

Table 3

Physicochemical properties of polymer hydrogels

Polymer name	Mechanical strength	Bulk weight (g/cm^3)	Thermostos Bone (SO)	Chemical durability
Acrylic acid (10% saponification) +CMC	Satisfactory in swollen state	0.60	Higher 100	Does not decompose in solutions of alkalis (LO 211) and organic acids
Acrylic acid (20% saponification) + CMC	Satisfactory in swollen state	0.60	Higher 100	Does not decompose in solutions of alkalis (up to 2N) and organic acids
Acrylic k-that (30% saponification) + CMC	Satisfactory in swollen state	0.62	Higher 100	It does not collapse in solutions of alkalis (up to 211) and 1 organic acids

Research results and discussion. Monomeric divinyl quaternary salts based on dialkylaminoalkyl acrylates N,-N-dimethyl aminoethyl methacrylate (DMAEMA) and N,-N-diethylaminoethyl methacrylate (DEAEMA) and dihalogen-containing compounds: α, γ-dichloroglycerol and dibromoethane were proposed as crosslinking agents.

It was found that the synthesized salts spontaneously polymerize in aqueous and ethanol solutions at 20-50°C with the formation of water-soluble products only at low (8-10%) conversion degrees. With further polymerization, polymers of a cross-linked structure are formed. In this regard, all kinetic studies were carried out up to 8% conversion. It was established by elemental analysis and PMR spectroscopy that the isolated polymers correspond to monomeric quaternary salts (MQS) in composition and structure. A study of the effect of the initial concentration of monomeric quaternary salts on the polymerization rate showed that with a decrease in the monomer concentration, the rate drops, and below 0.1 mol/l it is completely absent, and with an increase in concentration from 0.25 to 0.5 mol/l it increases 11 times. The salt polymerization reaction is of the first order, which proves the straightening of the kinetic dependences in logarithmic coordinates. The effect of the temperature regime and the nature of the solvent was established. Kinetic curves measured in water and ethanol showed that the reaction rate in water is significantly higher than in ethanol, which is due to its high ionizing capacity. The characteristic viscosity values of the polymers formed change similarly.

At this ratio of crosslinking agent: acrylic acid is 20% saponified carboxyl groups. At this ratio of reagents the polymer is strong, with maximum exchange capacity to most of the metals studied.

During the study, it was found that the polymer grain size has a noticeable effect on the static exchange capacity. With a decrease in grain diameter, the exchange capacity for all the studied metal salt solutions increases accordingly. This effect of grain diameter on exchange properties is obviously associated with the structural features of acrylate hydrogels. Therefore, gels with such a degree of saponification are advantageously used as swelling ion-exchange materials with a small grain diameter.

It has been established, that hydroquinone 2,2,6,6-tetramethylpiperidine-1-oxyl does not suppress the reaction, and styrene and methyl methacrylate do not form homoo- and copolymers under the specified conditions with the monomers under study. Thus, based on a set of kinetic studies and the revealed practical absence of influence of inhibitors on polymerization, a zutter-ionic mechanism of polymerization of the salts under study was proposed.

Further studies were devoted to the synthesis of hydrogels based on AK and its sodium salts using the above-mentioned MChS as crosslinking agents. The synthesis was carried out in an aqueous medium in the presence of an initiator and polymerization of potassium persulfate at a temperature of 40-50°C. The amount of the crosslinking agent varied from 0.1 to 0.5wt% relative to the monomer mass. With an increase in the concentration of the crosslinking agent DMAEMA-DBE, the induction period decreased, and the polymerization rate increased; in all cases, the reaction proceeded to nearly 100% monomer conversion.

Based on the presented experimental and literature data, the formation of three-dimensional networks by the polymerization method can be divided into two stages. At the first stage, a linear copolymer of AA with DMAEMA-DBE or DEAEMA-DBE is formed. These copolymers contain a certain amount of unused double bonds C = C of the crosslinking agent, which is attached only by one double bond to the main chain. The probability of interaction of the active center with the double bonds of another molecule is not high, while the probability of intramolecular interaction under these conditions is significantly higher. Such interaction leads to the formation of microgels that contain unreacted double bonds and the system still retains solubility. At the second stage, under the action of radicals, the microgels react with each other, forming insoluble macrogels. The process of radical polymerization occurs according to the classical scheme, supplemented by initiation at the second functional group and chain growth. The proposed polymerization scheme fully corresponds to the experimentally observed dependence of the reaction rate on the concentration of AA and crosslinking agents.

It is known that one of the main physical and chemical properties of absorbents is their water-absorbing capacity, which depends on the degree of neutralization of the carboxyl groups of the main monomer (AM) and the concentration of the cross-linking agent. In this connection, gels based on partially or completely neutralized acid with different degrees of cross-linking were synthesized and their properties were determined. It can be assumed that the conclusions made about the structure of polyacrylic gels equally apply to the corresponding hydrogels based on sodium acrylate.

To identify the relationship between the physicochemical properties, the content of neutralized groups and the concentration of the cross-linking agent, the water absorption of hydrogels was determined at different ratios of the initial reagents. It was found that for the entire series of cross-linking agents obtained, the optimal degree of saponification is 60-90%.

A study of swelling dynamics in deionized water showed that the highest water absorption was observed in samples containing the smallest amount of crosslinking agent. However, it was found that, all other factors being equal, the use of crosslinkers based on o-phthalic acid dichloride and 1,1-dimethyl- or 1,1-diethylaminoethyl methacrylates allowed the preparation of hydrogels with higher water absorption in distilled water. For instance, a gel with 0.1 % DMAEMA-DBE absorbed up to 1000 g of water per gram of polymer, whereas a gel with DMAEMA-DCH at the same concentration swelled up to 1600 g/g. This difference can likely be explained by variations in the chain lengths of crosslinking agents between vinyl groups. Under laboratory conditions, a hydrogel based on sodium polyacrylate and DMAEMA-DCH demonstrated a water absorption capacity of 5000 g/g in deionized water. Further reduction in the concentration of the crosslinking agent may yield samples with an even higher equilibrium swelling degree; however, such polymers exhibit unsatisfactory mechanical strength in the swollen state.

Thus, the above-listed features of the synthesized hydrogels allowed us to recommend them for use in arid zone agriculture, as well as for solving water and economic problems. The physical effect of hydrogels on soil moisture capacity and plant development is that the gel particles located in the soil pores swell when moisture enters, prolonging its stay in the soil and then transferring it to plants, in addition, the use of gels contributes to greater plant resistance to water stress and drought. The use of hydrogels allows for significant water savings, which allows them to be recommended for implementation in industrial production (Table 4).

Characteristics of manufactured products. The hydrogel is obtained from the reaction of partially saponified acrylic acid with a divinyl monomer salt derived from dialkylaminoethyl methacrylate and either dibromoethane or dichloroethane. Primary application: the hydrogel is intended for use as a moisture absorbent and as a skin-filling composition. Product properties. Color: white to light yellow

Table 4

Proportions of starting materials for obtaining crosslinking agents

Components	Crosslinking agent			
	1	2	3	4
Dimethylaminoethyl methacrylate	160		312	-
Diethylaminoethyl methacrylate	-	175	-	375
Dibromoethane	52	52	-	-
Dichloroethane	-	-	101	101
Hydroquinone	12	12	12	12
Dimethylformamide	200	200	250	250

Description of the technological process. The process of obtaining the crosslinking agent is carried out periodically as follows: the required amount of DMF was charged from vertical collector 1 into the cooled reactor 5, followed by DMAEMA (DEAEMA) from the tank 2. Hydroquinone, weighed on the scales 4, was then added. Then, while stirring, the required amount of DBE or DCE was gradually added from the collector 3. The temperature is maintained at approximately –10°C. After the monomers are completely consumed, mixing was continued for another 3 hours. After this, the solution entered the crystallizer bath. Cooling brine was supplied to the jacket of the crystallizer bath and the reactor. A water-ammonium nitrate mixture was used as brine in a 5:3 ratio. The solution in the crystallizer bath was maintained for 4 days at a temperature of –5 to –10°C. The solution was then filtered under vacuum using filter 7, and the filtrate was sent for regeneration. The filtered crystals were transferred to a mixing device (washer) using a tray. During mixing, the SA crystals were washed with the required amount of acetone for 30 minutes, after which they were subjected to vacuum filtration using a Nutsche filter 10. The resulting crosslinking agent was moved to a TsVSh-24 vacuum drying cabinet (12) using tray 11. Drying was carried out for 24 hours at 15-20°C under a residual pressure of 20 mm Hg. Finally, the crosslinking agent was packed in polyethylene containers and stored at 0–+5°C (Table 5).

Table 5

Standards for the technological process of crosslinking agent synthesis

Operation name	Duration	Temperature, °C	Pressure, hPa	Quantity reagent
1	2	3	4	5
Loading into the reactor				
DMF	5 min	–5 +–10	1010.8	47.2
DEAEMA	5 min	–5 +–10	Same	39,00
hydroquinone	5 min	–5 +–10	Same	3.1
2 Stirring	15 min	–5 +–10	1010.8	-
3 Loading DBE	90 min	–5 +–10	1010.8	24.00
4 Mixing	180 min	–5 +–10	Same	20.5
Draining the solution into a vacuum crystallizer	10-15 min	–5 +–10	1010.8	-
6 Holding in a crystallizer bath	64 hours	Same	Same	-
7 Unloading from the crystallizer and loading into the Nutsche filter	30 min	0	1010.8	
8 Filtration under vacuum	60 min	15	824.6	
9 Unloading the product onto a Nutsche filter	30 min	15-20		

Table 5 (continued)

1	2	3	4	5
10 Loading the product into the washer	20 min	20		
11 Rinsing with acetone	60 min	20	1010.8	180
12 Unloading the product into the nutsch filter	30 min	20		
13 Filtration under vacuum	60 min		824.6	
14 Unloading from the Nutsche filter	20 min			
15 Loading in vacuum drying camera	20 min			
16 Vacuum drying	day	20	26.6	
17 Unloading from A	30 min	20	1010.8	-
18 Packing CA	40 min	20	1010.8	-

Technological process for producing acrylate hydropolymer (Table 6). Proportions of the initial monomers for obtaining acrylate hydropolymer: Acrylic acid – 45; 50% sodium hydroxide solution – 35; crosslinking agent – 0.02; initiator – 0.03; water – 65.

Table 6

Standards for the technological mode of obtaining hydrogel

Name operations	Time	Temperature, ℃	Quantity downloadable component, kg
1. Loading (into the reactor)			
Acrylic acids	5 min	20	70
Caustic soda	5 min	20	95
water	5 min	20	80
Persulfate potassium	5 min	20	0,1
Stitching agent	5 min	20	0.07
2. Stirring	30 min	20	
3. Draining the mixture into the mixer	5 min	20	1/3 volume
4.Stirring with heating	60 min	60	-
5. Unloading	30 min	30	-
6. Loading into a Nutsche filter and vacuuming	120 min	30	
7. Rinsing with water	120 min	30	
8. Unloading from the Nutsche filter and loading into the drying chamber	60 min	30	-
9.Drying	48 hours	50	-
10. Unloading from the drying chamber	30 min	25	-
11. Crushing	120 min	25	“
12. Sifting	120 min	25	
13. Packaging	60 min	25	

Description Of The Technological Process. The technological process of obtaining acrylate hydrogel is carried out periodically. In accordance with the given scheme, the required amount of caustic soda, acrylic acid and water are fed into the reactor with a frame mixing device 5 (gravity flow) from collectors 1-3, equipped with a dosing device. Then the prescribed amount of the initiator and crosslinking agent, weighed on scales, is added. Mixing in the reactor is carried out at 25℃ for 30 minutes. Then the reaction mass is fed to a Werner-Pfleifer mixer. The volume of the solution loading is 1/3 of the reactor volume due to strong foaming at the initial stage of polymer formation. Heating of the mixer is carried out by a tubular electric heater. The temperature during the gelation process is maintained at 60℃. The gelation process continues for 60 minutes, after which the obtained hydrogel is unloaded into the tray 7 by tilting the mixer using a special mechanism equipped with an individual drive. Then the hydrogel is loaded into the Nutsche filter 8. The hydrogel is washed with water until a neutral reaction is achieved for 1-2 hours and dried under vacuum. The resulting wash water enters the ion exchange column 9, after purification the water is again fed into the Nutsche filter 8 using a pump 10. The washed hydrogel is unloaded into a tray 11 and transferred for drying into a heating circulation dryer 12 of the SP 32 type. At a temperature of 50℃ the hydrogel is dried for 24-48 hours until the residual moisture content is 0.2%. The dried hydrogel is fed to a crusher 13 for grinding and then to a vibrating screen 14 for separation into fractions. The resulting finished hydrogel is weighed on scales 15 and fed for packaging into polyethylene containers.

Conclusion. In this study, novel crosslinking agents based on divinyl quaternary ammonium salts of dialkylaminoalkyl methacrylates (DMAEMA and DEAEMA) with dihalogen compounds (such as 1,2-dibromoethane and 1,3-dichloropropanol) were successfully synthesized and characterized. These salts demonstrated spontaneous polymerization in aqueous and ethanol media at 20-50°C, forming water-soluble products at low conversion levels (8-10%). Further polymerization led to the formation of crosslinked structures. Kinetic studies revealed a first-order reaction with respect to monomer concentration, with the rate significantly increasing as concentration rose from 0.25 to 0.5 mol/L. The synthesized crosslinkers were applied to produce hydrogels based on acrylic acid and its sodium salts. The polymerization followed a two-stage mechanism, forming microgels in the first stage and insoluble macrogels in the second, governed by radical polymerization principles. The swelling behavior of the hydrogels depended on both the degree of neutralization of carboxylic groups and the concentration of the crosslinking agent. Optimal swelling was observed at 60-90% neutralization, with lower crosslinker content yielding higher water absorption. Hydrogels crosslinked with DMAEMA-DXG showed superior performance, absorbing up to 5000 g of deionized water per gram of polymer. The resulting hydrogels demonstrate strong potential for agricultural applications, particularly in arid zones. Their ability to retain and gradually release water enhances soil moisture retention and plant resilience under water stress. Furthermore, the detailed technological processes for both crosslinker and hydrogel production were established, including temperature, timing, and purification steps. These hydrogels offer promising prospects for scaling up in industrial settings due to their favorable water-absorbing capacity and environmentally beneficial application.

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АКРИЛ ҚЫШҚЫЛДАРЫ НЕГІЗІНДЕ ГИДРОГЕЛЬДЕРДІ АЛУДЫҢ ХИМИЯЛЫҚ ТЕХНОЛОГИЯЛЫҚ ПРОЦЕСІН ЗЕРТТЕУ

Аңдатпа. Сіңіргіштердің негізгі физика-химиялық қасиеттерінің бірі – олардың су сіңіру қабілеті екені белгілі, ол негізгі мономердің карбоксил топтарының бейтараптану дәрежесіне және тігуші агенттің концентрациясына байланысты. Осы зерттеуде диалкил-аминоалкилметакрилаттардың (DMAEMA және DEAEMA) дивинилді төртіншілік аммоний тұздары негізінде, дигалогенді қосылыстармен (мысалы, 1,2-дибромэтан және 1,3-дихлорпропанол) жаңа тігуші агенттер сәтті синтезделіп, сипатталды. Бұл тұздар 20-50℃ температура аралығында су және этанол ортасында өздігінен полимерлену қабілетін көрсетті, төмен конверсия деңгейінде (8-10%) су ерітіндісінде еритін өнімдер түзді. Полимерленудің ары қарай жүруі тігілген құрылымдардың түзілуіне алып келді. Кинетикалық зерттеулер мономер концентрациясына қатысты бірінші ретті реакцияны көрсетті, әрі концентрация 0,25-тен 0,5 моль/л-ге артқан сайын реакция жылдамдығы айтарлықтай өсті. Сонымен қатар, тігуші агенттер мен гидрогельдер өндірудің толық технологиялық процестері – температуралық режим, уақыт және тазарту сатылары – анықталды.

Тірек сөздер: гидрогельдер, акрил қышқылдары, алу процесі, ісіну динамикасы, акрилат гидрогелі, технологиялық процесс.

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ИЗУЧЕНИЕ ХИМИКО-ТЕХНОЛОГИЧЕСКОГО ПРОЦЕССА ПОЛУЧЕНИЯ ГИДРОГЕЛЕЙ НА ОСНОВЕ АКРИЛОВЫХ КИСЛОТ

Аннотация. Известно, что одним из основных физико-химических свойств абсорбентов является их водопоглощающая способность, которая зависит от степени нейтрализации карбоксильных групп основного мономера и концентрации сшивающего агента. В данном исследовании были успешно синтезированы и охарактеризованы новые сшивающие агенты на основе дивинильных четвертичных аммониевых солей диалкил-аминоалкилметакрилатов (DMAEMA и DEAEMA) с дигалогенсодержащими соединениями (такими как 1,2-дибромэтан и 1,3-дихлорпропанол). Эти соли проявляли способность к самопроизвольной полимеризации в водной и этанольной средах при 20-50℃, образуя водорастворимые продукты на низких степенях конверсии (8-10%). Дальнейшая полимеризация приводила к образованию сшитых структур. Кинетические исследования показали реакцию первого порядка по концентрации мономера, причем скорость значительно возрастала при увеличении концентрации с 0,25 до 0,5 моль/л. Кроме того, были разработаны детальные технологические процессы как для получения сшивателей, так и гидрогелей, включая температурный режим, продолжительность и этапы очистки.

Ключевые слова: гидрогели, акриловые кислоты, способ получения, динамика набухания, акрилатный гидрогель, технологический процесс.