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STUDY OF CHEMICAL TECHNOLOGY FOR THE PRODUCTION OF HIGH-SWELLING POLYMER HYDROGELS (HSPH)

Abstract. One of the ways of water management in agriculture is the use of highly swelling polymer hydrogels (HSPH). New hydrogels based on ech, ammonia and gipan were produced. An upward change in the amount of cross-linking agent in the gel, in our case from 0.1 to 9%, also affects the increase in cross-linking of the main chains of acrylic acid and, as a consequence, a decrease in sorption. Sorption is explained not only by gel-ion ion exchange, but also by complex formation between the polymer structure and the ions of these metals, or electrostatic interaction. During the study, it was found that the size of the polymer grain has a noticeable effect on the static exchange capacity. As the polymer grain decreases, the static capacity increases. The influence of factors (temperature, concentration, etc.) on the degree of swelling of hydrogel has been studied. As a result of systematic studies of the produced polymers, further directions for research of the developed complex structural polymers are shown. The proposed field of application: agriculture, metallurgical industry, medicine, cosmetology, household chemistry.

Keywords: hydrogel, epichlorohydrin, ammonia, hydrolyzed polyacrylic nitrile, swelling, polymers.

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Introduction. Hydrogels, as a three-dimensional cross-linked polymer, have a unique structure and are particularly responsive in response to internal and external stimuli. As a result, they have high hopes for drug discovery, cell therapy, and human tissue engineering. The properties of hydrogels are used for long-term treatment of the intraocular surface as medicines [1].

The studies describe the preparation of gel-like products from a solution of chitosan with silver nitrate. At high chitosan concentrations, continuous chitosan-silver hydrogels can be formed. At lower concentrations, nano- and micro-hydrogels are obtained. Sol-gel assays were performed to characterize the swelling properties of the hydrogels. In addition, mechanical testing of the hydrogels was carried out, with inductively coupled plasma optical emission spectroscopy to measure the silver 40 concentration, scanning electron microscopy to study the morphology of the products, dynamic light scattering and UV spectrophotometry to study the products formed at low concentrations of chitosan. Some hydrogels have been used to modify cotton fabric to impart antimicrobial properties. The resulting products acted against *E. coli subtilis* [2-4].

An experiment was conducted to evaluate the effect of hydrogel polymer levels (0, 10, 20, 30 g/m^2) and NO_3: NH_4 ratios (0:100, 25:75, 50:50, 75:25 and 100:0) on the yield of dill seeds and its main components. The results showed that when using 30 g/m^2 hydrogel polymer with O_3^-: 25 NH_4^+ ratio, maximum seed yield was obtained and the same plus - 100NO_3: 0NH_4^+ led to significant maximum oil productivity. GC-MS analysis showed that the main components of dill seed oil are limonene and α-phellandrene. The interactive effects of the simultaneous use of hydrogel polymer and NO^{3-}:NH_4^+ showed the interaction of the superabsorbent polymer with reduced components such as alpha-terpinene, limonene, dillapiol and trans-carvone, while its addition to compounds such as alpha-phellandrene, pcumene , carvone and transdihydrocarvone received a negative effect. In most cases,applying nitrate above 50% and adding ammonium had a positive effect on the compounds [5,6].

Various polymers with ultra-high absorption capacity were synthesized by radical copolymerization at 70°C using acrylic acid, potassium acrylate, N isopropyl acrylamide and sulfopropyl potassium methacrylate as monomers, bis methacryloyloxy ethyl phosphate as a crosslinking agent and potassium persulfate as an initiator. Optimization of the synthesis led to the preparation of a superabsorbent with a very high water absorption capacity, with a maximum swelling of 2618 g water/g dry hydrogel. The most promising superabsorbent was fully investigated and the absorption capabilities at different pH and ionic strength were studied. When superabsorbent was mixed with soil, the mixture was able to lose water more slowly. In addition, this material showed high good ability using urea as a fertilizer model. Due to these advantageous properties, the synthesized SAP can be used in agriculture [7].

The purpose of the study conducted controlled experiments is to introduce insulin into the production of smart hydrogels for the use of insulin for the human body. Insulin entrapped (HEMA-co-Eudragit L-100) hydrogels containing different ratios of 2-hydroxyethyl methacrylate and Eudragit L-100 were synthesized using ammonium persulfate as an initiator and ethylene glycol dimethacrylate as a cross-linker. New alternative methods for preparing such isulin hydrogels have been developed. The Taguchi method was used and the optimal synthesis conditions were determined. The resulting hydrogels were studied by Fourier transform infrared spectroscopy and scanning electron microscope analysis [8].

A new cross-linked hydrogel was prepared from sodium carboxymethyl cellulose and hydroxyethyl cellulose using ammonium persulfate as an initiator and methylene bis as a cross-linking agent to obtain drugs. The release of bovine serum albumin from drug hydrogels loaded under different pH conditions was studied to simulate gastrointestinal diseases. The amount of bovine serum albumin from the hydrogels at pH 1.2 was relatively low (17.8%), while 85.2% was obtained at pH 7.4. According to the results, the CMC – carboxymethylcellulose hydrogel has good potential for use in controlled release oral drug administration [9].

The aim of the study is:

– study and establishment of the structure of the compounds produced, determination of physico-chemical properties of polymers (swelling);

– creation and application of new highly swelling hydrogels for water conservation.

Materials and methods. *Materials. ECH -Epichlorohydrin* (3-chloro-1,2-epoxypropane) – before use, the fraction with a boiling point of 389.1 K and a melting point of 330 K was distilled and selected; molecular weight 92.53,d480 = 1, 181; n1320 = 1, 4381; solubility in water 6.55%, soluble in organic solvents; CPV 2.3-49.0% tvsp= 299 K, auto-ignition temperature 683 K. Application: in the production of epoxy resins, glycerin; vulcanizing agent ethylene propylene, rubber; solvent for cellulose ethers. MPC-1 mg/m^3. World production is 400 thousand tons/year.

GIPAN-Hydrolyzed polyacrylic nitrile – is a viscous dark yellowish liquid of 8-10% concentration with a density of 1.05-1.07 g/cm', pH = 12 or more, or a yellowish, cream or pink powder with a moisture content of 10%, which can be used in commercial form or in the form of a solution of 10% concentration with p = 1.05 g/cm'. GIPAN, like other acrylic polymers, forms strong polymer clay structures with clay particles due to chelate bonds and polymer membranes that partially prevent hydration of rocks

Method for studying the degree of water absorption. The water absorption capacity of hydrogels was studied in distilled water at room temperature. The hydrogel was taken from the water after it had swelled. The water absorption capacity of the hydrogel (Q_{H2O}) was determined using the equation [10,11]:

$$Q_{H2O} = (m_2-m_1)/m_1 \qquad (1)$$

Here m_1 and m_2 are the weight of the hydrogel in the dry and swollen state [12].

Starch $(C_6H_{10}O_5)n$ – used corn starch of «Golden Corn» Tashkent starch plant, solid, white powder, molar mass: 162.141* n g/mol, dense: 1.5g/cm^3.

The degree of swelling is determined by the mass of liquid absorbed per unit mass of a substance at a given stage of swelling at a given temperature:

$$a=(m_2-m_1)/m_1 \qquad (2)$$

where m_2, m_1 is the mass of the substance before and after swelling, respectively.

Swelling characteristics are similar to sorption characteristics. The main one is the swelling isotherm, which shows the dependence of the degree of swelling on the thermodynamic activity of the solvent (on its vapor pressure) at a given temperature.

Method IR spectroscopy. IR spectroscopy is a branch of spectroscopy, including the acquisition, study and application of emission, absorption and reflection spectra in the infrared region of the spectrum. IR spectra (SHIMADZU IRAFFINITI device, Japan) were taken from films of composite samples with a thickness of about 12 μm. The IR spectrum of the highly swelling hydrogel shows absorption bands corresponding to functional groups attached to the monomer units [13-15].

Research results and discussions. An important direction in the creation of hydrogels is the stitching of the hydrolyzed fibre product «nitron» in the presence of suturing agents. Up to 95-99% of the polymer can be sutured and the sutured polymer can swell, absorbing water 20-100 g/ml (Fig. 1). After drying the cross-linked polymer, the resulting glass-like mass is crushed to the powder state with the required particle size. GYPAN-based Hydrogel Swelling Kinetics with Ethylene

Glycol is showed in Figure 2. PAN macromolecules always contain ionized links of acrylic acid, and therefore the swelling of hydrogels based on it also depends on the ion force of the surrounding solution. The most important properties of hydrogels that determine their suitability as soil moisture absorbers are swelling, depending on the hydrogel structure and external conditions. Given the polyelectrolytic nature of most hydrogels, their structure should be characterized by the thermodynamic parameter of the polymer-water interaction, the density of mesh units, the proportion of ionogenic groups and the degree of their dissociation. The swelling process was experimentally investigated using methods, the optical measurement of the geometric dimensions of the visualized specimens of regular shape (cylinders, spheres), and the automatic recording of the volume of liquid remaining after absorption by the sample. Physicochemical tests of the synthesized polymers were carried out at various stages of polymerization.

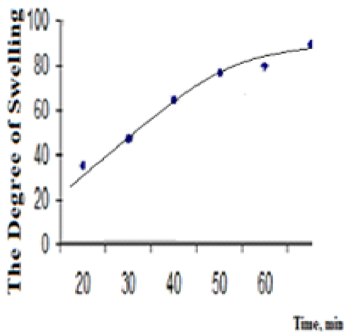


Fig. 1. Hydrogel swelling kinetics based on GYPAN with urea

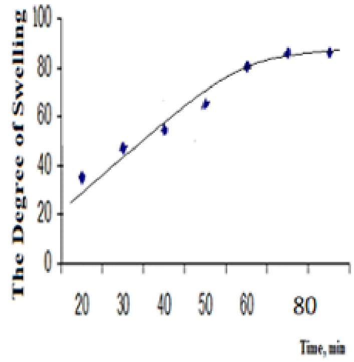


Fig. 2. GYPAN based Hydrogel swelling kinetics with Ethylene Glycol

The composition and structure of the synthesized products were studied using IR spectra using a spectrophotometer. In this way, the reaction products were studied at the initial stage of polymerization, then, at certain time intervals, the synthesis product was studied at the final stage upon completion of the process. Samples were examined according to standard method mentioned above. As a result of studying samples by IR spectroscopy, the following characteristics were obtained (Fig. 3). In the spectra taken immediately after mixing the components, it is observed the following picture: the region of absorption bands 2980-3000 and 1270 cm'1 includes $CH-CH_2$ groups of the epoxy ring and stretching vibrations of the ECH group. In the spectra of oligomeric reaction intermediates, a change in the shape of the peaks in the region of 1400-1490 and 2700-2800 cm^{-1} is detected, as shown, this relates to the vibrations of the CH groups of epichlohydrin and the groups of HIPAN. It follows that as a result of the polymerization reaction, first a dimer is formed, then an oligomer and then a polymer.

As can be seen from the spectra, the change in the intensity of absorption bands in theseareasindicates that there is a gradual reaction and opening of the ECH ring with HIPAN and the formation of quaternary ammonium salts due to the ionization of the amino groups of the nitrogen atom and interaction with the halogen. However, it should be noted that the nature of the changes in the X-ray and PMR spectra at the initial stages of polymerization indicates the presence in the system linear products and elementary links of the original components, since there is identity of the element-forming links. They can be distinguished only by their elemental composition and the number of active functional groups in structures of varying complexity (linear, branched, dimeric) can be determined, i.e. the content of chlorine ions will always be much lower, due to the presence of end groups that have not reacted with ECH. In the case of the formation of cross-linked structures, the calculations coincide with those practically found. Based on this, it was established that the epoxy cycle opens with the formation of a hydrophilic group, which simultaneously contains highly active amino groups and a mobile halogen (chlorine) atom, which subsequently leads to the formation of cationic polycomplexes of a linear structure [10-12].

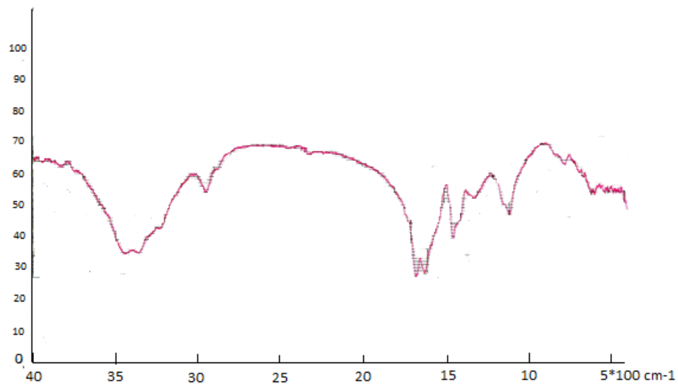


Fig. 3. IR spectra of the reaction products of HIPAN with ECH

Absorption bands in the region of 950, 866, 740 cm^{-1} refer to symmetric and asymmetric bending vibrations characterizing the epoxy cycle, and stretching vibrations of amino groups NH, NH2- in the region of 2820-2920 cm^{-1} are absent in the spectra of the resulting polymer. Thus, an intense absorption band in the region of 1270 cm^{-1} characterizing the CH_2-CI bond indicates that the synthesis reaction is accompanied by ring opening and interaction of the halogen with the amino group, and this absorption band is visible in the region of 1580 cm^{-1}. In addition, an absorption band appears in the spectrum in the region of 1580 cm^{-1} and then a band appears in the spectrum related to the associated OH group and the quaternary group 3090-3400 cm^{-1}. The obtained spectral analysis data provide ground sassume that the polymer has the following structure as shown above.

Considering the IR spectra of the studied diamine compounds with different structure, it can be noted that skeletal vibrations of the -CN bond give absorption maxima in the same regions of 1150-1030 cm^{-1}, and they are preserved in the spectra of the resulting polymers. Absorption bands in the region of 2950-2800 cm^{-1} belong to stretching vibrations characterizing the NH2-CH2- group. It should be noted that in the spectra of the starting amine an intense absorption band appears in the region of 3100-3500 cm^{-1}, and in the spectra the broad bands of amine and hydroxyl groups coincide. IR spectroscopy makes it possible to explain the process of the appearance of new absorption bands depending on the duration of polymer synthesis [13,14].

The composition of the polymer product during the interaction of ECH with HIPAN was also studied using IR spectroscopy. The rate of chemical activation is:

$$Wp = Kr[A]\ [ECH] = -d[n]/dt \qquad (3)$$

Rate of dimer...oligomer formation:

$$Wn = Kn\{d[A]/dt\}=Kn[M] \quad (4)$$

where, [A] and [M] are the molar concentration of active centers and monomer salt. Chain growth occurs as a result of the addition of a dimer, oligomer, etc.

The cessation of chain growth occurs, in all likelihood, due to a violation of the stoichiometry of the reacting active centers, and, asconsequence, the formation of non-functional centers at the ends of the growing chain.

A study of the influence of the nature of amino compounds on spontaneous polymerization showed that the highest rate of polymerization is achieved when using primary amines. It is assumed that here, apparently, there is a high rate of proton transfer from the ECH nitrogen atom with the formation of a complex containing activated groups, which subsequently participate in chain growth and, accordingly, polymerization. As established, with increasing basicity of amino compounds, the initial period of activation of monomersincreases from Ammonia to HIPAN i.e. from 9.21 to 9.5.

Studies have been carried out on the formation reactions of new water-soluble and water-insoluble polymer products when epichlorohydrin reacts with various amine compounds, in particular with ammonia and hexamethylenediamine. Since these monomeric compounds contain several epoxy- and chlorine-functional groups, their interaction with amines results in polymers with predictable properties, such as (cationic activity, anionic activity, amphotericity). For this reason, the chemical behavior of these compounds is determined by the reaction activities of the molecules, as well as directly by the conditions of the polymerization reaction. During the polymerization reaction, compounds of various structures are formed, both water-soluble and non-water-soluble and with a three-dimensional structure. As noted in the literature review, there is ungeneralized data on these compounds and there remain parameters for their further study, namely their solubility, swelling, and their performance as ion exchange resins in a dissolved state. Therefore, studying the process of obtaining these polymers, their nature, structure and mechanism is of particular scientific interest.

As is known, the presence of epoxy groups, halogens and amine compounds in the starting products will allow interaction with each other with the formation of viscous products, highly viscous substances and resinous high-molecular compounds with different solubilities.

When epichlorohydrin interacts with HIPAN, as well as ammonia, water-soluble oligomers, polymers, and non-water-soluble polymers of a network structure with valuable physical properties are obtained.chemical, physical and mechanical properties, which are widely used in various industries (chemical, textile, metallurgical, pharmaceutical), as well as in agriculture.

In this regard, the study of these products is of certain scientific and practical interest. To develop optimal conditions for the technology for producing polymers based on epichlorohydrin with HIPAN, comprehensive studies were carried out depending on the polymerization conditions. It is known that the reactivity of amine compounds depends on the nature of the activity, and the strongest bases are aliphatic amines in comparison with ammonia, in addition, in fact, the chemical activity of amine compounds depends on their donor-acceptor properties, where their activity as bases is manifested [15-17]:

$$N: +H^+ \qquad >N^+ -H \quad (5)$$

When ECH interacts with HIPAN, this ammonium base leads to the formation of an ammonium salt. As a result of the reaction, a quaternary salt is formed, which occurs through the donor-acceptor interaction of a free electron pair of a nitrogen atom with an electrophilic agent. Consequently, the introduction of functionally active groups (halogen compounds) as a substituent leads to a decrease in the activebasicity of the amino compound.

*Efficiency and ways of using hydrogels.*The data presented, as well as the extensive literature available, indicate the possibility of increasing the moisture capacity of light soils and sands by adding hydrogel additives.

The importance of these additives in the water balance of soils apparently lies in partial transfer of moisture from the gravitational drain to the state accumulated by the gel, since in thermodynamic terms the moisture in the hydrogel is closer to capillary. Otherwise, we can say that hydrogels transform large, easily drained pores into moisture-retaining ones. This an apparent redistribution was indeed detected. Thus, adding 0.1-0.5% hydrogel to sand reduces the proportion of drainable pores from 89.4 up to 49.6%, increasing the moisture-retaining content from 3.5 to 23.9%. In accordance with this, moisture utilization coefficients increase from 0.021 to 0.085 and transpiration ratios decrease from 4.9 104 to 1.2 104 [17].

The biological effect of adding hydrogels in a number of cases was found when such small doses (up to 0.001%) when the effect of the drug on physical the characteristics of the substrate are not yet apparent. A natural question arises, to which there is no answer yet: how effective is it to use hydrogels as moisture-retaining agents? Let us outline ways to evaluate the effectiveness of hydrogels. The differential (instantaneous) increase in useful moisture dw when adding hydrogel at a dose of D (both values in %) will be:

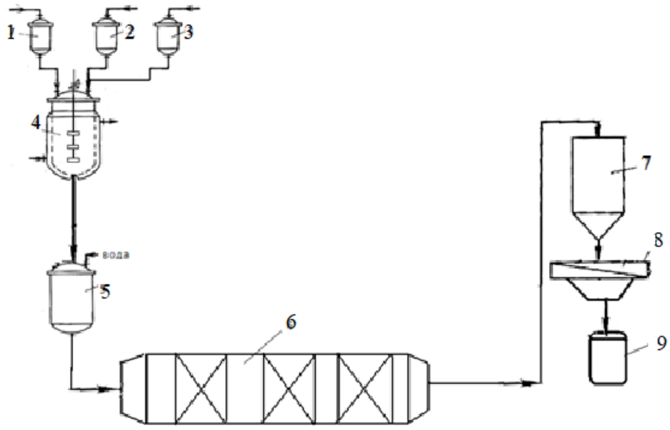
$$Dw = w \cdot Q \cdot D \qquad (6)$$

where w is the swelling of the hydrogel in the soil, and Q is the coefficient of moisture availability, close to unity (w and Q are dimensionless).

Obviously, the most important task is to achieve sufficiently high swelling of hydrogels directly in the soil. In addition to physico-chemical reasons, for example, destruction of the gel under the influence of swelling pressure at the beginning of the cycle, this factor will depend on the climatic and microbiological stability of the hydrogel, as well as its migration from the layer with streams of moisture. All these problems are currently unclear and await the attention of specialists.

Thus, the use of hydrogels is promising in the agrotechnical complex and land reclamation, although additional, serious research and development. Several social and environmental aspects of the use of hydrogels need to be taken into account. The possibilities of providing crop production to cities and towns in the arid zone and expressly solving the most pressing problems of this type must be deeply analyzed.

The technological process for producing hydrogel is carried out in a batchwise manner (Fig. 6). In accordance with the above diagram, the required amount of HIPAN, a cross-linking agent and water enters the reactor with a frame mixing device 4 (by gravity) from collectors 1-3 equipped with a dosing device. Then the recipe amount of initiator weighed on the scales is added. Mixing in the reactor is carried out at 70 0 C for 3 hours. Then the reaction mass enters a Werner-Pfleifer type mixer. The solution loading volume is 1/2 of the reactor volume. The mixer is heated by a tubular electric heater.



1,2,3 – collections of source materials; 4 – reactor; 5 – container for washing (or Nutsch filter); 6 – heater; 7 – crusher; 8 - vibrating sieve; 9 – packaging.

Fig. 6. Scheme of the technology for producing a hydrogel based on HIPAN

The temperature in the reactor, during the gelation process, is maintained at 70°C. The gelation process continues for 3 hours, after which the resulting hydrogel is unloaded into the tray by tilting the mixer using a special mechanism equipped with an individual drive. The washed hydrogel is unloaded into a tray and transferred for drying to a heating circulation dryer 6 type SP 32. At a temperature of 50°C, the hydrogel is dried for 24 hours to a residual moisture content of 0.2%. The dried hydrogel is fed into crusher 7 for grinding, and then to vibrating sieve 8 for separation into fractions. The resulting finished hydrogel is weighed on scales 9 and supplied for packaging in plastic containers. The technological process for producing hydrogel is s show great promise for applications in agriculture and land reclamation, though they still require extensive further research and development. Their use must also consider various social and environmental implications. A thorough analysis is needed to explore their potential in supplying crops to urban and rural areas in arid zones, as well as addressing the most critical challenges in this field.

Conclusion. An upward change in the amount of cross-linking agent in the gel, in our case from 0.1 to 9%, also affects the increase in cross-linking of the main chains of acrylic acid and, as a consequence, a decrease in sorption. Sorption is explained not only by gel-ion ion exchange, but also by complex formation between the polymer structure and the ions of these metals, or electrostatic interaction. During the study, it was found that the size of the polymer grain has a noticeable effect on the static exchange capacity. As the polymer grain decreases, the static capacity increases. In synthesis, as a result of the formation of the structure of mesh polymers, the main method is polymerization, since it determines the size of the net of the resulting hydrogel, and regulates the length of the chain, having a decisive influence on the properties of the polymer. The resulting hydrogel has bactericidal properties. The resulting materials are environmentally friendly, can be used in agriculture. The influence of factors (temperature, concentration, etc.) on the degree of swelling of hydrogel has been studied. As a result of systematic studies of the produced polymers, further directions for research of the developed complex structural polymers are shown. The proposed field of application: agriculture, metallurgical industry, medicine, cosmetology, household chemistry.

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ТЕЗ ІСІНЕТІН ПОЛИМЕР-ГИДРОГЕЛЬДЕРДІҢ ҚҰРЫЛЫМЫН ЗЕРТТЕУ

Аңдатпа. Ауыл шаруашылығында суды басқарудың бір жолы – жоғары ісінетін полимерлі гидрогельдерді (HSPH) пайдалану. ech, аммиак және гипан негізіндегі жаңа гидрогельдер алынды. Гельдегі айқаспалы байланыс агентінің көбеюі, біздің жағдайда 0,1-ден 9%-ға дейін, сонымен қатар акрил қышқылының негізгі тізбектерінің айқаспалы байланысының жоғарылауына және нәтижесінде сорбцияның төмендеуіне әсер етеді. Сорбция тек гель-ион ион алмасуымен ғана емес, сонымен қатар полимер құрылымы мен осы металдардың иондары арасындағы кешен түзілуімен немесе электростатикалық өзара әрекеттесумен түсіндіріледі. Зерттеу барысында полимерлі дәннің мөлшері статикалық метаболизм қабілетіне айтарлықтай әсер ететіндігі анықталды. Полимер дәнінің мөлшері азайған сайын статикалық сыйымдылық артады. Гидрогельдің ісіну дәрежесіне факторлардың (температура, концентрация және т.б.) әсері зерттелді. Алынған полимерлерді жүйелі зерттеу нәтижесінде дамыған күрделі құрылымдық полимерлерді зерттеудің келесі бағыттары көрсетілген. Болжалды қолдану саласы: ауыл шаруашылығы, металлургия өнеркәсібі, медицина, косметология, тұрмыстық химия.

Тірек сөздер: гидрогель, эпихлоргидрин, аммиак, гидролизденген полиакрилнитрил, ісіну, полимерлер.

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ИССЛЕДОВАНИЕ СТРУКТУРЫ БЫСТРО НАБУХАЮЩЕГО ПОЛИМЕРА ГИДРОГЕЛИ

Аннотация. Одним из способов управления водными ресурсами в сельском хозяйстве является использование высоконабухающих полимерных гидрогелей (HSPH). Были получены новые гидрогели на основе ech, аммиака и гипана. Увеличение количества сшивающего агента в геле, в нашем случае с 0,1 до 9%, также влияет на увеличение сшивки основных цепей акриловой кислоты и, как следствие, на снижение сорбции. Сорбция объясняется не только гель-ионным ионообменом, но и комплексообразованием между полимерной структурой и ионами этих металлов, или электростатическим взаимодействием. В ходе исследования было установлено, что размер полимерного зерна оказывает заметное влияние на статическую обменную способность. По мере уменьшения размера полимерного зерна статическая емкость увеличивается. Было изучено влияние факторов (температуры, концентрации и т.д.) на степень набухания гидрогеля. В результате систематических исследований полученных полимеров показаны дальнейшие направления исследований разработанных сложных структурных полимеров. Предполагаемая область применения: сельское хозяйство, металлургическая промышленность, медицина, косметология, бытовая химия.

Ключевые слова: гидрогель, эпихлоргидрин, аммиак, гидролизованный полиакрилнитрил, набухание, полимеры.