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Original Article

Preparation of Hydrogel based on Starch, Carboxymethyl Cellulose, Methylene-bis-acralamide and its Application

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Abstract - *The article provides information about starch, carboxyl methyl cellulose, methylene bis-acralamide, and the technology of making highly elastic hydrogel based on them and their application. Brief information on the level of research on hydrogels is given using the literature. The synthesis process of the hydrogel, the amount of starch, and carboxyl methylcellulose were studied on the basis of percentages, and the swelling of the hydrogel was expressed in mg, and the water absorption was expressed in ml. IR spectrum, thermal analysis, thermogravimetry, Roman spectrum, and SEM analysis of hydrogel were obtained and analyzed. Simply put, 10 grams of the hydrogel can hold up to 2.5-4 liters of water. When used correctly, the hydrogel can save 20-40 percent of irrigation water for most agricultural crops. Finally, the importance of hydrogel in agricultural plants is summarized.*

Keywords - *Acrylamide, Acrylic acid, KMS, Montmorillonite, Superabsorbent hydrogel.*

1. Introduction

In our republic, a number of works are being carried out on the development of the chemical industry and the replacement of existing technologies in its branches with new ones and the management of production on the basis of new technologies, in particular, the growth of plants when used in agriculture on the basis of local raw materials. Much attention is being paid to the production of highly flexible polymer hydrogels, which serve to increase their efficiency to a certain extent, especially in the synthesis and use of polymer hydrogels based on new approaches; great results are being achieved. Biological polymer hydrogels are widely used in agriculture and other areas of society to increase the efficiency of water use. Provides the ability to retain plant nutrients and deliver them to plants. Studying the effect of hydrogels on productivity, saving water and mineral fertilizers, and soil fertility is to study the effect of hydrogels in preventing desertification and salinization of soil and groundwater [1].

2. Level of Study of the Problem

Scientific research on the production of new high-grade swelling polymer hydrogels based on polysaccharides and their practical application is carried out by the world's leading scientific centers and higher education institutions, including: Institute of Technology (USA), Sumitomo Chemical (Japan), State University of New Jersey American Chemical Society (USA), Beijing Forestry University (China), Russian University of Chemistry and Technology, Center of Creative Engineering Plastics (South Korea), School of Soil and Water Conservation, Tashkent Research Institute of Chemical Technology (Uzbekistan) is going In the world, a number of researches are being conducted on the development of technologies for obtaining high-swelling hydrogels based on polysaccharides and their derivatives, including in the following priority directions: obtaining environmentally friendly and inexpensive high-swelling hydrogels based on polysaccharides; to determine the effect of polysaccharides on the properties of highly elastic hydrogels. S. D. Zhang, Salih Muharam, S. K. Gulrez, Alessandro F. Martins, N. Dairi, R. Nuisin, S. Al-Assaf, A. Abidin, M. R. Lutfor, M. Z. Rahman on the development of the synthesis and production technology of hydrogels with high viscosity in water., by M. J. Zouhuriaan - Mehr, K. Kabiri, A. I. Jushman, D. E. Harley, C. Ivanov, V. V. Vasilevsky, and also in the Republic of Uzbekistan: A. T. Djalilov, Kh. Kh. Turaev, Sh. D. Shirinov, BA. Kholnazarovlar and others conducted scientific research [2]. Water absorption, swelling (or shrinking) of hydrogels and environmental factors affecting their use. Radiation treatment of hydrogels: Radiation treatment is a very convenient method for imparting desired effects to polymer materials and is one of the latest aroused great interest in several decades [3]. Polymerization of monomers is carried out by at least three separate stages, namely, monomer chain initiation, monomer chain growth, and monomer chain termination, and there is a possibility that they can be modified by additional reactions such as chain transfer [4]. Hydrogels are polymeric materials with a three-dimensional structure consisting of physically and chemically interconnected hydrophilic polymer chains. Hydrogels can hold a large amount of water due to surface tension and capillary forces [5].

The water absorption of hydrogels depends on the functional groups in the chain, the state of water and the molecular masses of polymer hydrogels [6]. A binding reagent is used during the reaction to form a three-dimensional structure, which increases the mass, mechanical properties, and thermal stability of polymer hydrogels. Binders improve the properties of elasticity, swelling and resistance to polymers' aggressive environments [7]. In 1976, the United States Department of Agriculture designated water-absorbing polymers as superabsorbent polymers (SAPs) and found them to be widely used in many fields, including horticulture, agriculture, cosmetics, pharmaceuticals, construction, and wastewater treatment.

These unusual polymeric materials consist of chains with hydrophilic groups and strongly interconnected chains, which allow them to absorb water up to a thousand times more than their own mass [8]. Starch-based hydrogels are rich in hydrophilic groups, biodegradable, inexpensive and non-toxic in nature. Therefore, starch-based hydrogels are used in tissue engineering, drug delivery systems, and blood purification [9]. Many scientific research works have been carried out on crosslinking and chemical modification of polysaccharides.

Modification of polysaccharides was carried out with various synthetic monomers, such as acrylic acid, acrylamide, methacrylamide, vinyl alcohol, 2-acrylamido-2-methylpropanesulfonic acid, styrene, vinylimidazole [10]. Hydrogels based on polysaccharides are environmentally friendly and cheap, and hydrogels based on petroleum products will replace hydrogels in the near future [11]. The above-mentioned scientists synthesized hydrogels with high swelling in water based on synthetic materials, natural polysaccharides and various gelatins used in agriculture, construction, packaging, medicine, the food industry and a number of other industries.

Carboxymethylation of polysaccharides is a widely studied transformation that leads to products with simple and diverse promising properties. As a rule, the polysaccharide is activated with various solutions of aqueous metal hydroxide, mainly sodium hydroxide. It is converted into monochloroacetic acid or its sodium salt according to the Williamson Yester synthesis, and the polysaccharide derivative gives carboxymethylcellulose (KMS) [12].

KMS is also the simplest cellulose ether that behaves like a typical polyelectrolyte. Although the commonly used form of KMS sodium salt is soluble in water, the conversion by treating the polymer with mineral acid leads to the free carboxylic acid of the polymer that is insoluble in water [13]. Based on the above, obtaining a new type of water-saving and environmentally friendly hydrogels based on local raw materials includes the following issues: studying the mechanism and optimal conditions of the interaction between polysaccharides, vinyl monomers and minerals; synthesizing hydrogels based on local raw materials, determining the dependence of physical-mechanical, technological and swelling properties on the nature and mass of reagents; to create a technology for obtaining inexpensive and effective environmentally friendly polymer materials based on copolymers of natural polysaccharides, to carry out research on their practical application on a large scale.

Six types of hydrogels were synthesized as a result of the copolymerization of resin with polyvinyl alcohol, salts, and vinyl monomers - acrylic acid, acrylamide, acrylonitrile, N, N-methylene bis-acrylamide, bentonite, kaolin and three montmorillonites and minerals [14]. Hydrogels based on cellulose and starches have many favorable properties such as hydrophilicity, biodegradability, biocompatibility, transparency, low cost and non-toxicity.

Thus, hydrogels based on cellulose and starch are widely used in tissue engineering [15], controlled delivery systems [16], through blood purification [17], sensors [18], agricultural plants [19], as well as in water treatment projects and chromatographic supports [20] various application prospects of cellulose- and starch-based hydrogels are shown [3]. It was found that the production of hydrogel contains several chemicals such as starch, polyacryl, dichlorohydrin, hippan, acrylinite, urea, and acrylic acid available in our country [21]. Hydrogels from copolymerization of starch with glutaraldehyde, citric acid and sodium carboxymethylcellulose.

During alkaline hydrolysis of hydrogels synthesized on the basis of starch copolymers with vinyl monomers, it was found that new hydrophilic groups are formed in the polymer chain: -ON, -SOONa, $-CONH_2$, -COOH, -CONH, -COO and because of these groups, the water absorption properties of hydrogels are improved. It was found that if bentonite, kaolin and montmorillonite minerals are added to the production of hydrogel, the water absorption properties of hydrogels increase by up to 30%, and the water retention time increases by up to 26%, and the water resistance of hydrogels increases up to 22% the effect of hydrogels on heat and mechanical effects increases, and the cost of the product decreases; it was found that with the increase in the concentration of NaCl, $CaCl_2$, and Na_2SO_4 salts in the solution, the swelling of hydrogels formed due to the high density of ions decreases. The technology of obtaining hydrogels based on starch, acrylic monomers, minerals and binding reagents has been developed. The degree of swelling depends on the local climate and soil conditions [22].

3. Experimental Part

3.1. Material and Methods

The study of natural gas-drying sorbent hydrogel shows that hydrogels based on carboxymethyl cellulose and starch have been confirmed to have natural gas-drying properties. Composition and physical properties of the hydrogel. Hydrogel does not lose its properties for up to five years; 40-50 kg of hydrogel was used on 1 hectare; as a result, the water deficit of plants was greatly reduced, and the productivity and yield of plants were greatly improved, and mineral fertilizers were used. The need is much reduced. For example, 10g of polyacrylic has been found to retain up to 2.5-3.0 liters of water through experiments. One ton of it today is 50-60 thousand soums.

So, it was found that when the hydrogel obtained on the basis of rice starch and KMS is 50/50, its swelling properties are much higher. Also, the hydrogel obtained on the basis of potato and corn starch and KMS in a ratio of 50/50 has been found to have high swelling properties.

Table 1. Physical properties of the hydrogel

Content	Swelling(mm)	Density
Composition of corn-based hydrogel		
Starch\KMS (50\50)	0.0460	0.012
Starch\KMS (25\75)	0.0425	0.016
Starch\KMS (75\25)	0.0315	0.033
The composition of hydrogel obtained on the basis of potatoes		
Starch\KMS (50\50)	0.0450	0.011
Starch\KMS (25\75)	0.0400	0.015
Starch\KMS (75\25)	0.0310	0.030
The composition of the hydrogel obtained on the basis of rice starch		
Starch\KMS (50\50)	0.0440	0.012
Starch\KMS (25\75)	0.0410	0.014
Starch\KMS (75\25)	0.0317	0.031

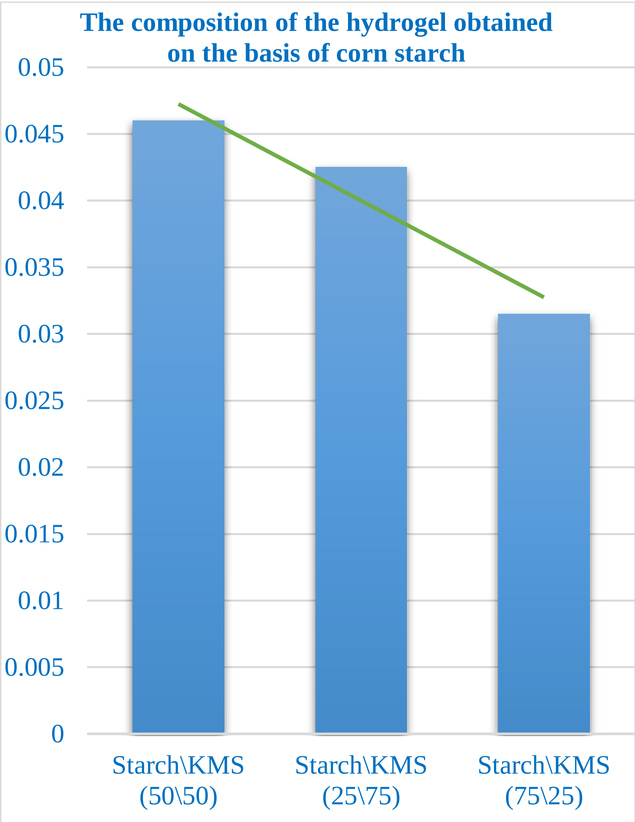

Fig. 1 Physical properties of corn starch-based hydrogel

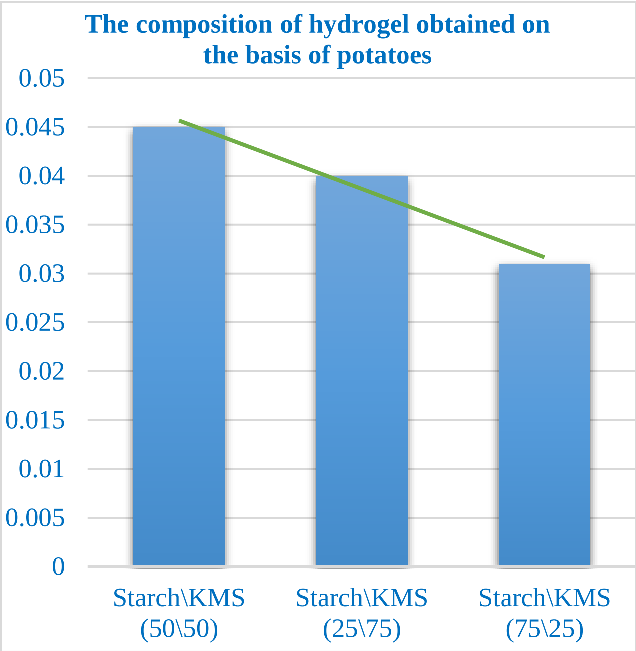

Fig. 2 Physical properties of hydrogel based on potato starch

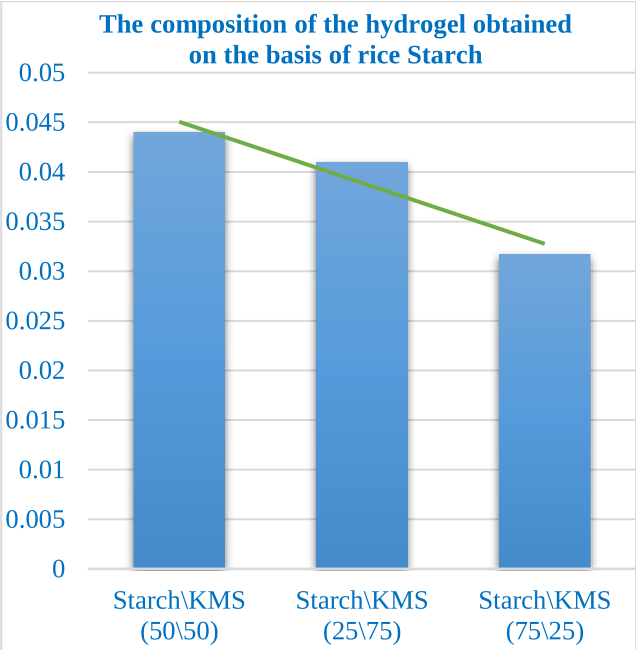

Fig. 3 Physical properties of hydrogel based on rice starch

3.2. Hydrogels Derived from Polysaccharides Starch/NaKMS/Bent/MBA/KPS

To obtain a hydrogel based on rice starch, 15 g of starch and 15 g of carboxyl methyl cellulose are mixed in 100 ml of water at a temperature of 70^0 C for 45 minutes, then bentonin, polyvinyl chloride, polyvinyl acetate and potassium salts of polyphosphoric acid, N-N-methylenebisacrylamide is added to the mixture in turn. The semi-finished product is mixed vigorously. Slowly raise the temperature to 70^0 C. It is worth noting that the change of potassium salt of polyphosphate acid and starch gels of polyvinyl acetate increases significantly.

For example, 6% starch - potassium salt of polyphosphoric acid - increases the viscosity of polyvinyl acetate by at least one factor. This process takes 150 minutes. Then, it is hydrolyzed for 90 minutes at a temperature of 95^0 C and placed in an oven to dry. The water absorption of the prepared hydrogel has been studied, and it has been found that it can absorb several times its own weight.

3.3. Starch/NaKMS/Bent/MBA/KPS Hydrogel

According to the IR spectra, the hydrogel obtained from Starch/NaKMS/Bent/MBA/KPS asymmetric valence v (OH) 3412 cm^{-1} belongs to the hydrogen bond to the valence vibration of the group. 2960.73 -2872.10 cm^{-1} symmetric valence (CH_3) bands belonging to the valence vibration of the methyl bond are located. The 2450 cm^{-1} valence vibration belongs to the V1716.65 cm^{-1} valence vibration of the potassium salt of polyphosphate acid and polyvinyl acetate unit in the superabsorbent.V.(C = O) group. V(CO)-ketone belongs to the valence vibration of the 1672.21 cm^{-1} group.

The plane strain vibrations of V(1276.88cm^{-1} (C-H) symmetric valence associated with starch changed absorption peak at 1496 cm^{-1} and 1456 cm^{-1}; during this reaction, OH in starch is OH and – The characteristic absorption peak of $CONH_2$ shows a shift due to the stretching of the C = O groups. Changed from $^{-1}$ to 1044 cm^{-1}.The absorption peaks at 1496 cm^{-1} and 1456 cm^{-1} are associated with starch change, which is characteristic of the OH and –$CONH_2$ groups in starch and shows a shift in the absorption reaction.

3.4. Analysis of Hydrogel Thermal Analysis

The differential thermal analysis of the rapid weight loss of the starch-based hydrogel due to the evaporation of water was proved by the analysis. The first weight loss stage starts at $27{,}86^0$ C at a rate of 0.24%/min and ends at $127{,}25^0$ C at a rate of 11.11 min. The weight loss in the first stage is -0.293mg or -3.793%, which is due to the low moisture content of the hydrogel.

The second stage of weight loss starts at a temperature of 126.820 C at a speed of 11.07 min and ends at a temperature of 306.62^0 C at a speed of 29.18 min. In the second step, the weight loss decomposition shows that the superabsorbent retains water even at high temperatures, with a mass loss of -3.306 mg or -42.857%.

The third stage of weight loss starts at a temperature of 306.62^0 C at a speed of 29.18 min and ends at a temperature of 306.62^0 C at a speed of 48.86 min. In the third stage, the mass loss is -3.306 mg or -42.857%. In this case, the complete decomposition of amine, carboxyl and carbonyl groups is observed due to the breakdown of polymer chains. The thermal analysis shows that the natural mineral bentonite is of great importance in the synthesis of highly flexible hydrogels, and the temperature resistance increased when we added bentonite to the hydrogel. As a result of thermogravimetric analysis, it was found that the thermal stability of hydrogel is very high.

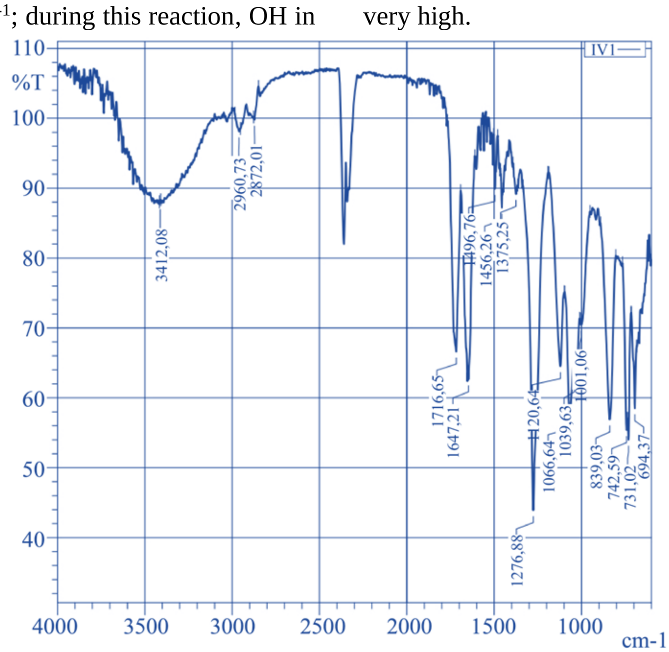

Fig. 4 Infrared spectrum of starch/NaKMS/Bent/MBA/KPS hydrogel

Table 2. Starch/NaKMS/Bent/MBA/KPS

Fluctuation in IR spectra frequencies, cm-$^{-1}$	Analysis
3412 sm^{-1}	*V(OH)* belongs to the hydrogen bond
2960,73 -2872,10 sm^{-1}	V(CH3) belongs to the methyl bond
2450 sm^{-1}	V(C = O) potassium salt of polyphosphoric acid and polyvinyl acetate
1672,21 sm^{-1}	V(CO)-ketone belongs to the valence vibration of the group
1496 sm^{-1}va 1456 sm^{-1}	V (C-H) deformation vibrations are related to starch
1120,64 sm^{-1}	C=C=O refers to the valence vibration of the ketone group

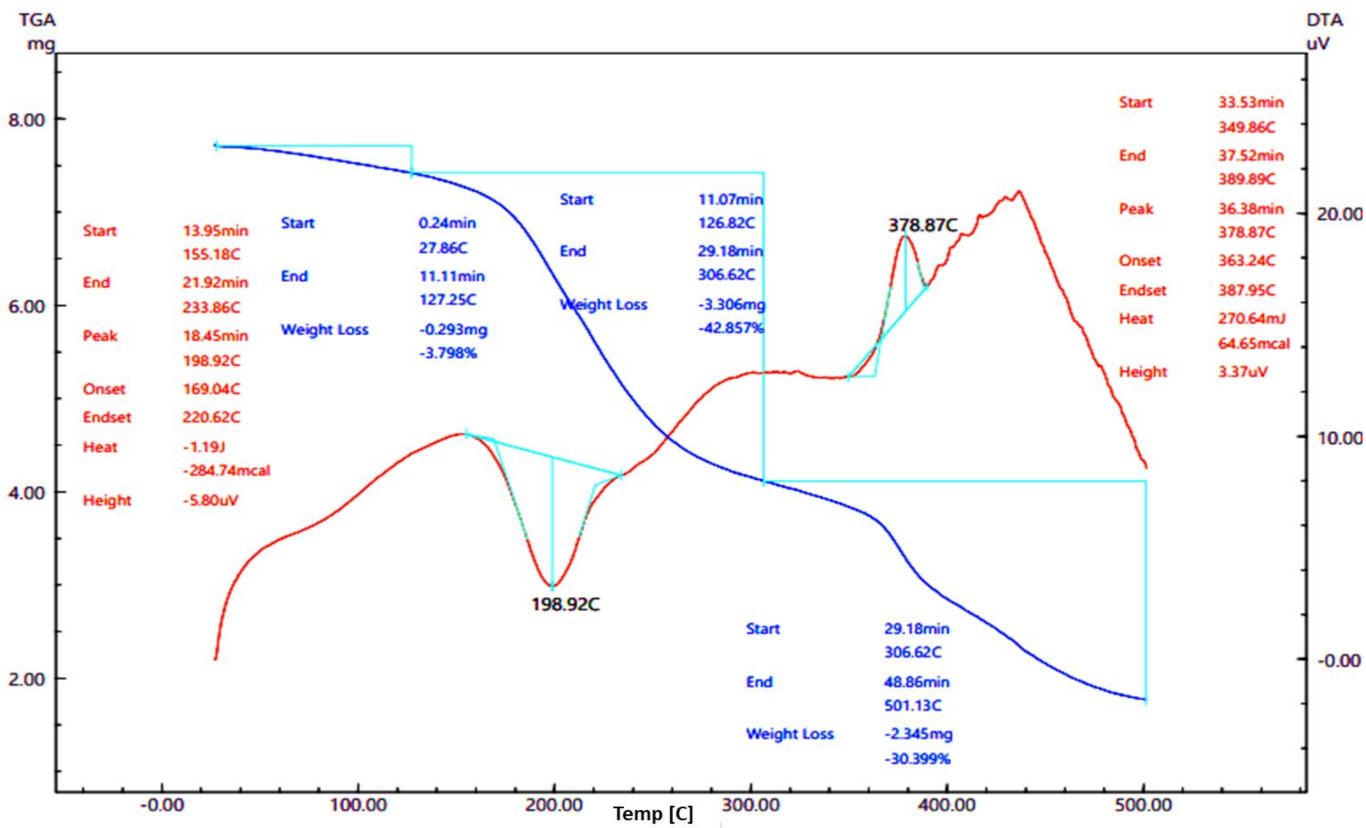

Fig. 5 Analysis of differential scanning calorimetric hydrogel thermal analysis

The exothermic process of the starch-based hydrogel is a chemical reaction with the release of heat proved by analysis of differential thermal analysis. Energy release takes place in two stages.

During the first differential thermal analysis, the energy absorption phase is completed at 13.95 min at 155^0 C and 21.92 min at 233.86^0 C. The highest completion is at 198.92 C, where a sharp weight loss is observed due to the low moisture content of the hydrogel, depending on what happens. In the second stage, the energy output starts at 349.86s at a speed of 33.53 min, ends at 389.89C at a speed of 37.52 min, and the peak is 36.38 min at 378.87C. As a result of the thermal analysis, it means that the natural mineral bentonite is of great importance in the synthesis of high-performance hydrogels, when we add bentonite to the hydrogel.

The 2626.65 and 2526.13 cm^{-1} bands in the spectrum belong to the asymmetric and symmetric vibrations of the methylene group, respectively. 2421 cm^{-1} valence vibration of potassium salt of polyphosphate acid and unit of polyvinyl acetate in superabsorbent. N=C=N belongs to 2134.10 cm^{-1} valence symmetric vibrations of carbamide. S=S Valence symmetric vibrations of vinyl ether are 1920 cm^{-1} and 1806 cm$^{-1,}$ respectively. Vibration involving the H-O bond. The deformation vibration O=N-O 1625.59 cm^{-1} and 1497.37 cm-^{1} is due to nitrites. The O-N strain vibration was associated with 491.39 cm^{-1} and 324.56 cm^{-1} of plain starch.

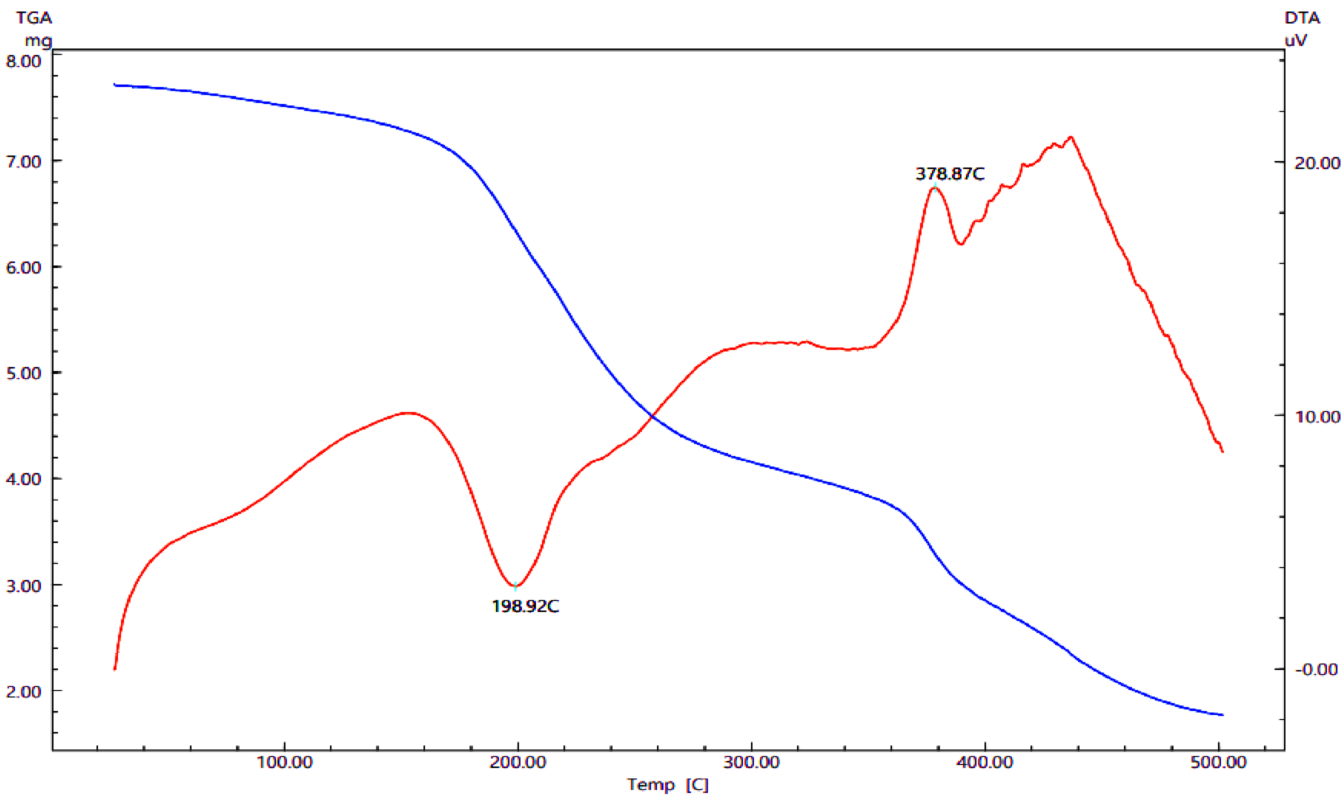

Fig. 6 Hydrogel thermogravimetry analysis

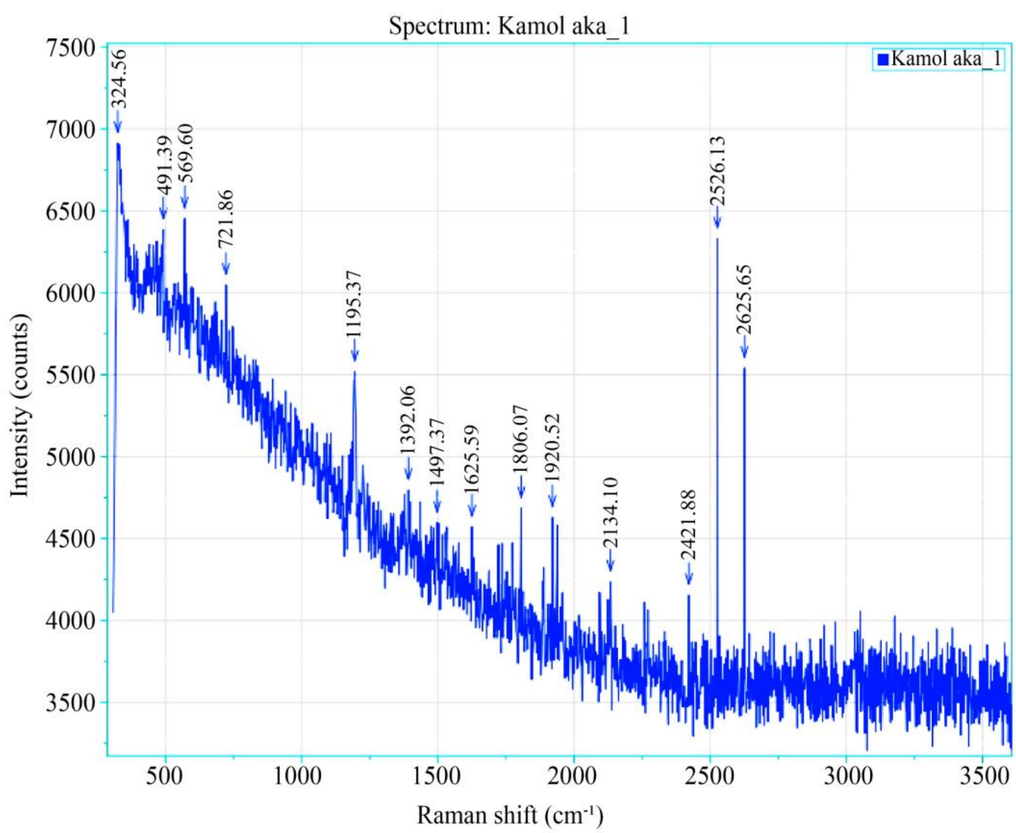

Fig. 7 Roman spectrum of a hydrogel based on Starch-/NaKSM-Bent/MBA-KPS

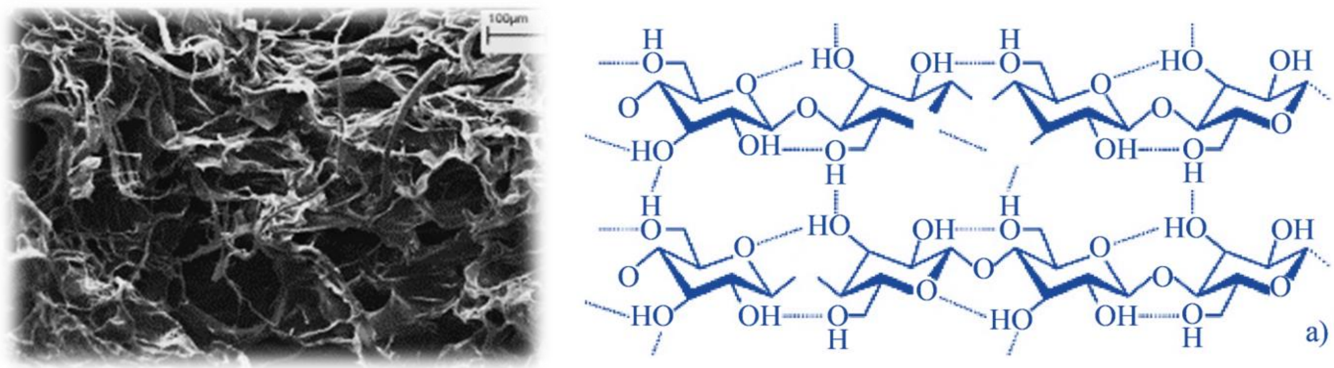

Fig. 8 Sem analysis Starch-/ NaKSM -Bent/MBA-KPS

Table 3. Hydrogel Synthesis based on Starch, Polyacryl, Acrylamide, Potassium Persulfate and Bentanite. Water absorption properties of hydrogel

No	Hydrogel quantity	% of water amount	Hydrogel sorption in water sources overall speed		
			Distilled water pH 7	Rainwater pH 7	River water pH 8.5
1	0.5	100	70%	60%	30%
2	1	100	100%	80%	40%
3	1.5	100	120%	100%	70%
4	2	100	130%	120%	90%
5	2.5	100	150%	140%	100%

SEM observations showed a change in the surface of the macromolecule formed after the crosslinking reaction. When studying the SEM images, we found a change in the pore size of the examined hydrogels, which indicates an increase in branching and crosslinking density. The denser network structures can be explained on the basis of the formation of intermolecular hydrogen bonds as a result of the increase in MBA content, as well as the formation of hydrogen bonds. That can clearly be seen in the analysis.

The polar oxygen-containing parts on the surface of the minerals can participate in the swelling by adsorption with the polymer, and the rest of the minerals are evenly distributed throughout the polymer to form small pores, as a result of which the water permeability, water return, and mechanical properties of hydrogels are improved. Epichlorohydrin and methylenebisacrylamide added during the polymerization of freeze-dried hydrogels have been proven to increase the molecular mass of the polymer product and prevent it from dissolving in water. Both samples are characterized by a sponge-like surface morphology, the presence of open channels interspersed with smoother interconnected domains, probably due to the growth of relatively large ice crystals during freeze-drying from water.

4. Results

In the experiment, the amount spent for 100% sorption of the hydrogel in distilled, rain and sewage water was determined. Accordingly, in the first variant, complete sorption was observed when 1 g of hydrogel was added to 200 ml of distilled water with a neutral pH of 7. 1.5 g of hydrogel was used in water with a pH of 8.5 and in rainwater with a pH of 7. The optimal hydrogel concentration in relation to the amount of water was determined. When 0.5 g, 1 g, 1.5 g, and 2.5 g of hydrogel were added to 100 ml of water, thick gels were formed from 50% sorption to 150%.

Due to the use of a small amount of hydrogel, apple, cotton, wheat, and barley fields gave very effective results and successfully passed the tests. As a result, it was observed that the experimental batches for apples, wheat, barley and cotton significantly reduced soil moisture retention and consumption of water and mineral fertilizers. During the experiment, we studied the availability of water, mineral fertilizers, agricultural resources, and plant growth; changes in the productivity reduction of soil hardening due to the effect, depending on the hydrogel, were studied.

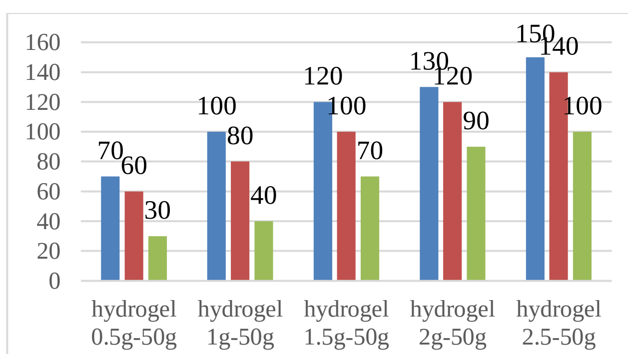

Fig. 9 Water absorption properties of hydrogel

Fig. 10 Hydrogel's water absorption processes and applications

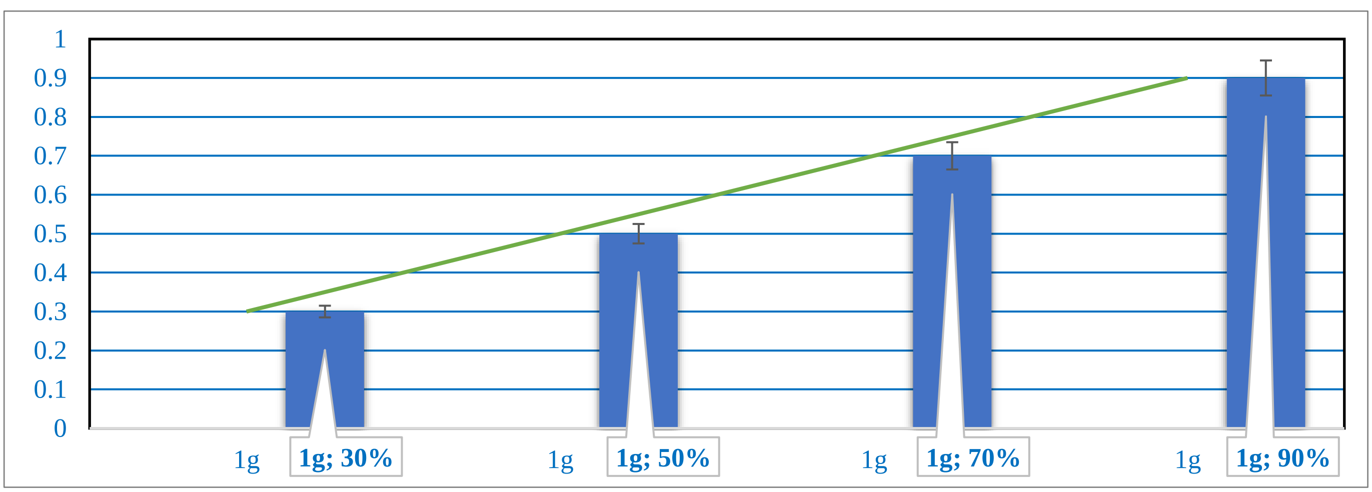

Fig. 11 Water absorption of hydrogel

Table 4. Water absorption of hydrogel

Hydrogel (g)	Dis.water (ml)	Time (min)	Water absorption (%)
1g	100 ml	30min	30%
1g	100 ml	90min	50%
1g	100 ml	180min	70%
1g	100 ml	240min	90%

Fig. 12 Difference between hydrogel-added and non-hydrogel-added fields

Rice starch, acrylamide and bentonite are placed in the three vessels obtained, and everything is mixed in a reactor to obtain a hydrogel based on these**.** Then, they are supplied with water from the bunker, and the heater is started. 12% rice starch solution is heated to 100°C for half an hour. Then, the starch solution is cooled to 20-24°C. First, acrylamide monomer is added, then bentonite is added, mixed thoroughly, heated to 50-60°C and mixed. Potassium persulfate (KPS) was prepared in an aqueous solution as an initiator, and methylenebisacrylamide (MBA) in water was added to the mixture. The mixture is kept under regular stirring at room temperature. The reaction temperature was gradually increased to 70°C.

The reaction process was carried out at 70°C for 3 hours. After the copolymerization process, the product is hydrolyzed in 1 M sodium hydroxide solution at a temperature of 100°C for 120 minutes. Then, the product is washed several times in water, neutralized in a constant weight column and dried in a vacuum drying oven. The product is crushed using a grinder and placed in special plastic bags to prevent moisture and dust. Technical and economic efficiency of the development As shown, according to the research results, it is proposed to obtain hydrogels based on starch, acrylamide and kaolin, as well as based on starch, acrylic acid and bentonite.

Hydrogels based on starch, acrylic and minerals are widely used in agriculture, industry, construction and other fields. The specific and general economic efficiency of the introduction of the obtained hydrogels was calculated.

5. Discussion

Hydrogels make it possible to obtain crops from lands that are suitable for irrigation, but it is impossible to use water and increase the yield of plants. It reduces the consumption of mineral fertilizers due to keeping them in the fertile layer of the soil, water due to the fact that the atmospheric precipitation on the soil through rain, snow or irrigation keeps the water in a state where it can easily pass to the roots of plants saves a lot of money. If hydrogel is used, it will not completely lose its properties for 2 years. The products formed from the destruction of expired hydrogels do not damage the earth's infrastructure but are easily absorbed by plants as a nitrogen fertilizer.

Farmers in some regions of China get 65-67 s of cotton per hectare. This is a much higher indicator compared to other countries. For example, in Uzbekistan in 2016, the average is 26.2 s per hectare, and in Kazakhstan, this indicator is 17 -18 s. For this reason, water-saving technologies, namely hydrogels, are used in China. 1. Saves up to 50% of water needed for crops. 2. Plants meet their need for water in drought conditions, are resistant to stress, and, as a result, increase productivity by 25-32%. 3. Ensures bigger crops and improves taste. It preserves nutrients in the soil, and as a result, it reduces the need for fertilizers by 22-28%. It keeps the soil from becoming lumpy and prevents it from being washed away.

6. Conclusion

The demand for these hydrogels is more than 12,000 tons in the Surkhandarya and Kashkadarya regions alone. Due to the fact that the average annual rainfall in the Surkhandarya, Bukhara, Kashkadarya, Khorezm regions and the Republic of Karakalpakstan is much less than the norm, there is no possibility of planting plants on dry lands on more than 6.6 million hectares of land.

Another 12 million hectares are suitable for irrigation but are not used due to water scarcity. Therefore, in the Republic of Uzbekistan, hydrogels that hold a lot of water and store mineral resources are very necessary. As the technical and economic efficiency of the development is shown, according to the research results, it is proposed to obtain hydrogels based on starch, acrylamide and kaolin, as well as based on starch, acrylic acid and bentonite. Starch, acryliccalculated. Simply put, 10 grams of polymer can hold up to 2.5-4 liters of water. If we talk about its effectiveness, if it is used correctly, it can save 20-40 percent of the water used for irrigation of most agricultural crops. As a result of the obtained experiments, 2450 m^3 of water was saved from 1 ha of cotton planted, and 4900 m^3 of water was saved from 1 ha of winter wheat.

In addition, 200 kg of mineral fertilizers were saved, and plant productivity increased by 10-14%. It also saves fuel and time. 10 kg of hydrogels were saved for the next season. Saturated from rain or irrigation water, the hydrogel gradually transfers moisture to the roots of plants, absorbing up to 250-

400 times its own weight in water. The study of natural gas drying sorbent hydrogel shows that hydrogels based on carboxymethyl cellulose and starch have been confirmed to have the properties of drying natural gas.

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