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Loosely Crosslinked Hydrogel with Combined Water-Retaining and Anti-Erosion Effect

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Abstract—A new loosely crosslinked network copolymer consisting of acrylamide, potassium acrylate, starch, and bis(acrylamide) as a crosslinking agent is synthesized. The physicochemical properties of the synthesized copolymer are compared with the properties of a commercial network copolymer of sodium acrylate and acrylamide which is used in agricultural technologies as a water-retaining agent. Both copolymers are water swellable; when swelling in air, the first copolymer absorbs more water than the second one; when swelling in the pores of fine-grained quartz sand, the opposite picture is observed. An aqueous suspension of easily deformable hydrogel particles of the synthesized copolymer (with a particle concentration of 1 wt %) can be applied to the sand surface by spraying, which is impossible for a suspension of elastic weakly deformable particles of the commercial hydrogel. After drying the sand with the synthesized copolymer applied to its surface, a coating is formed that protects the sand from the action of wind and water. The synthesized copolymer at a concentration of up to 0.5 wt % does not have antimicrobial action against bacteria and yeast. On the basis of its physical, chemical, and biological properties, the copolymer can be recommended as a multifunctional soil conditioner.

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INTRODUCTION

Water-soluble polymers with groups dissociating into ions, polyelectrolytes, are widely used in agricultural technologies; they are used as soil conditioners and materials for reclamation of soils and increase in their resistance to erosion. The spectrum of application of polyelectrolytes includes preservation (improvement) of the physical properties of arable land, stabilization of the soil structure, reclamation and detoxification of disturbed and contaminated soils, combating desertification, retention of moisture in the soil, and more [1-13].

When treating the soil with solutions of linear polymers, a protective soil-polymer coating (crust) is formed on its surface which prevents mechanical destruction, erosion of agronomically valuable soil aggregates, and wind entrainment of fine dust [14, 15]. The effectiveness of protective coatings is determined by the properties of both components, soil substrates and polymer binders. Polymer stabilizers of an amphiphilic (dual) nature exhibiting an affinity for hydrophilic and hydrophobic areas on the surface of soil

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particles have a pronounced advantage over traditional polyelectrolytes [16-18].

Linear polymers poorly bind water; tillage with such polymers does not have a noticeable effect on its water-retaining properties [19, 20]. For improving the water regime of the soil, covalently crosslinked polyelectrolytes with a three-dimensional network structure and a high content of functional groups capable of ionization are used [11-13, 21]. Water-swollen network polymers (hydrogels) accumulate a large amount of water which can be used to eliminate the lack of fresh water in agriculture, improve soil fertility, and save natural resources [22-24]. The ability to absorb and retain a significant amount of water and aqueous solutions in the volume of the swollen network and to adsorb metal ions and biologically active compounds makes hydrogels unique materials for many agrotechnical applications [25, 26].

In recent years, there has been an increasing interest in the synthesis of new soil conditioners based on polyelectrolytes, with special attention paid to polymers of natural origin which are capable of degradation under the action of soil microorganisms and enzymes [27–29]. However, in most works, traditional air conditioners are discussed that perform any one main function: water retaining or structure forming; and soil conditioners that solve both of these problems are hardly considered. Meanwhile, the combination of both functions in one preparation is attractive from several points of view: reducing the "chemical" (often ambiguous) effect on the soil, simplifying the procedure for applying the conditioner, and the possibility of quantitative control of the physicochemical behavior of the polymer carrier and encapsulated biologically active substances and, as a result, a cheaper technology for the use of polymer conditioners.

In this work, we synthesized an anionic loosely crosslinked copolymer containing biodegradable starch fragments (copolymer I); obtained a hydrogel by swelling the copolymer in an aqueous salt solution; investigated the effect of the hydrogel on the waterretaining properties of quartz sand, the main component of most types of soils, and the anti-erosion properties of protective polymer sand crusts; and compared the physicochemical properties of the synthesized hydrogel with the properties of the commercial hydrogel based on anionic three-dimensionally crosslinked copolymer (copolymer II). In addition, we estimated toxicity of the synthesized copolymer with respect to soil bacterial and yeast cultures.

EXPERIMENTAL

We used acrylic acid, potassium hydroxide, and potassium persulfate from Vekton (Russia); acrylamide (**AM**) and crosslinking agent N,N-methylenebis(acrylamide) from Acros Organics (United States); starch (Vekton); and sodium hydrogen phosphate and sodium dihydrogen phosphate (both from Khimmed (Russia). Fine-grained quartz sand with a particle size of 0.1–0.2 mm (ORT-6, Russia) was repeatedly washed with bidistilled water before use.

Anionic loosely crosslinked copolymer I was synthesized according to the following procedure. Starch (5.5 g) was dissolved in 30 mL of distilled water at a temperature of 80°C, and 10 mL of 11 wt % potassium persulfate solution was added under continuous stirring to initiate the grafting polymerization of the acrylic acid-AM-bis(acrylamide) mixture [30, 31]. A mixture consisting of 79.8 mL of 98.5 wt % acrylic acid solution, 20.9 g of AM, 0.055 g of bis(acrylamide), and 85 mL of 75.7 wt % KOH solution was prepared in an ice bath, heated to 70°C, and poured into a solution of starch and potassium persulfate. The resulting solution was stirred at $60-70^{\circ}$ C until appearance of the gel effect (after 20-30 min), after which the heating was stopped. Copolymer I was dried in an air flow at a temperature of 50°C to a constant weight. The dried copolymer was ground and divided into fractions with a particle size of less than 0.25 mm (fraction I) and in the range of 0.25-0.50 mm (fraction II) by sequentially passing the copolymer powder through sieves with a mesh size of 0.25 and 0.50 mm. The weight ratio of components AM : acrylic acid : starch : crosslinker in the copolymer was approximately 14 : 83 : 3 : 0.036. The resulting copolymer was stored at a temperature of 5° C.

We also used the commercial copolymer of acrylamide and potassium acrylate with a grain size of 0.3-0.5 mm, copolymer II.

The content of carboxyl groups in copolymers I and II was determined potentiometrically. For this purpose, a suspension of swollen copolymers (0.1 wt %) in a 0.01 mol/L HCl aqueous solution with pH 2 was prepared and kept for a week at a constant temperature until the equilibrium state was reached. The resulting suspensions were titrated with a 0.1 mol/L NaOH solution on an Akvilon pH-420 pH meter (Russia). The content of carboxyl groups in copolymers I and II was 6.4×10^{-3} and 2.2×10^{-3} mol/g, respectively.

The equilibrium degree of free swelling of the copolymers α was determined at different pH values by the gravimetric method using a Shimadzu MOC-63U moisture analyzer (Japan); 0.05 g of the copolymer was placed in 100 mL of bidistilled water and kept for 3 days until complete swelling was attained. Then the pH of the hydrogel suspensions was adjusted to the desired value by adding 0.01 mol/L HCl solution or 0.01 mol/L NaOH solution, and the suspensions were kept under stirring for 3 days to reach the equilibrium state. The value of α was calculated from the ratio

$$\alpha = (G_{\rm w} - G)/G. \tag{1}$$

where G_w is the weight of the hydrogel equilibrium swollen in water at room temperature and G is the weight of the dry copolymer.

To assess the degree of swelling of copolymers after their binding to sand and to study the effect of copolymers on the ability of sand to retain moisture, samples were prepared as follows. A weighed portion of the copolymer was added to 20 g of sand and mixed thoroughly. The weight ratio of sand : polymer in the samples was varied in the range from 100 : 0.1 to 100 : 1. Sand-polymer mixtures were poured into plastic cups with small holes at the bottom and covered with filter paper; the cups with the samples were placed in a container filled with 0.001 mol/L phosphate buffer with pH 6.5 and kept for 3 days for equilibrium saturation of the samples with moisture. The moisture content of sand-copolymer mixtures at maximum moisture saturation was estimated by the formula

$$W = (M_{\rm w} - M_{\rm n} - G)/M_{\rm n},$$
 (2)

where M_w is the weight of the wet sand-copolymer mixture and M_n is the weight of the dry sand. The result obtained was compared with the moisture content of the initial sand at the maximum moisture saturation calculated as

$$W = (M_{\rm ws} - M_{\rm n})/M_{\rm n},$$
 (3)

where $M_{\rm ws}$ is the weight of wet sand.

The degree of swelling of the copolymers in sand (in the limited pore space of a substrate) was calculated using the formula

$$W = (M_{\rm w} - M_{\rm ws} - G)/G$$
 . (4)

The moisture content was measured gravimetrically on a Shimadzu MOC-63U moisture analyzer (Japan).

Water retention curves for the initial sand and sand-polymer mixture were measured by equilibrium centrifugation, as described in [32, 33]. The results were rearranged in coordinates log P-W (P is the external pressure created by rotation of the centrifuge), and the data were approximated using the van Genuchten model [34], which describes *S*-shaped dependences log P-W with one inflection and the minimum number of approximation parameters.

The viscosity of 1 wt % suspensions of hydrogel I was determined using VPZh-2 capillary viscometers (Ekroskhim, Russia) with capillary diameters of 1.77 and 2.37 mm.

Samples with a protective polymer-sand layer on the surface were prepared as follows. Plastic containers were filled with 60 g of quartz sand; the thickness of the sand layer was 5 cm, and the surface area of the sample was 16 cm². Using a spray gun, a 1 wt % suspension of hydrogel I in water with a flow rate of 2 L/m^2 was applied to the surface. The samples were dried to a constant weight in air. The strength of the polymer-sand crust was measured by the penetration method (cone immersion) using a Rehbinder conical plastometer [35].

For anti-erosion experiments, 80 g of quartz sand was added to a Petri dish; the thickness of the sand layer was 1 cm, and the surface area of the sample was 16 cm². The suspension of the polymer hydrogel (17.2 mL, 1 wt %) in water was evenly applied to the sand with a flow rate of 2 L/m^2 and dried in air to a constant weight. Control samples were prepared by a similar treatment of the sand surface with bidistilled water. The stability of the samples to wind erosion was studied by exposing them to an air flow generated with a BaBylissPRO BAB7000IE electric hair dryer (Babyliss, France); the air flow rate was set using a Testo 440 anemometer (Testo, Germany). The Petri dish with the sample was placed horizontally; the air flow was directed at an angle of 30° from a distance of 5 cm for 5 - 30 min.

To assess the water resistance of the sand before and after application of the polymer to it the samples were treated with water from a spray gun in a pulsed mode for 10 min. Petri dishes were placed at an angle of 45° , and water streams were directed at them horizontally; the total volume of the water applied was 500 mL. The samples were weighed after treatment with water and subsequent drying, and the weight loss of the sample was calculated.

The antimicrobial activity of hydrogel I was evaluated according to the standard method for determining the minimum inhibitory concentration (MIC) [36]. The test cultures were gram-negative bacteria Pseudomonas aeruginosa 4.8.1, gram-positive bacteria Staphylococcus aureus, and yeast Yarrowia lipolytica 367-2 from the collection of the Federal Research Center Fundamentals of Biotechnology, Russian Academy of Sciences. Microorganisms were grown on M9 medium of the following composition (g/L): Na₂HPO₄, 6; KH₂PO₄, 3; NaCl, 0.5; NH₄Cl, 0.2; MnSO₄, 0.0004; MgSO₄, 0.0025; CaCl₂, 0.0002; glucose, 10; the medium had pH 7.0. The cultivation was carried out in the presence of various concentrations of hydrogel I under continuous stirring at a temperature of 28°C for 2 days. The growth of the cultures was assessed visually from an increase in the turbidity of the solutions. The maximum concentration of the copolymer was 0.5 wt % or 3.2×10^{-3} mol/L of anionic groups.

All experiments were performed with three repetitions. Statistical data were obtained by processing the experimental data in Excel.

RESULTS AND DISCUSSION

When mixed with water, both network copolymers, copolymer I synthesized in this work and commercial copolymer II, swelled and formed transparent suspensions of the hydrogel. Photographs in Fig. 1 give a visual representation of the state of the particles of the synthesized and commercial copolymers after swelling. For better visualization, colorless hydrogel particles were stained with a red dye, rhodamine 6G, distributed in the volume of the particles. Swollen particles of copolymer I spread over the glass surface and formed droplets 2 mm high on it (Fig. 1a). In contrast, copolymer II after swelling formed gel particles 4 mm in height (Fig. 1b).

Figure 2 shows dependence of the degree of swelling of copolymers α on the pH of an aqueous solution at room temperature. It can be seen that the degree of swelling for both copolymers increases with an increase in the pH of the solution from 3 to 9, reflecting a progressive increase in the degree of dissociation of carboxyl groups in hydrogels [37, 38].

After the distribution of the copolymers in the sand, the granules found themselves inside the pores of a limited volume, which could have affected the swelling ability of the polymers. The volume fraction of pores in fine-grained quartz sand with a particle size of 0.1-0.2 mm (ORT-6, RF) was determined earlier in [39]. The pore diameters were in the range from 0.01 to 0.2 mm with a pronounced size distribution maxi-

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Fig. 1. Photographs of water-swollen particles of (a) copolymer I, fraction II and (b) copolymer II. The hydrogels were treated with a solution of red dye, rhodamine 6G.



Fig. 2. Dependence of the equilibrium degree of swelling α of (1) copolymer I and (2) copolymer II on the pH of the aqueous salt-free solution.

mum at 0.02 mm. To assess the swelling of copolymers in a limited pore space, an aqueous buffer solution with pH close to neutral was used. Figure 3 shows the degrees of swelling of copolymers I and II in the free state (α) and in sand (α_{lim}). The figure shows that the swelling of copolymer I in the sand ($\alpha_{lim} = 70$) is significantly lower than that in the free state ($\alpha = 440$), which is associated with spatial constraints of the copolymer in the pores of the substrate and resistance from sand particles. In contrast, the degree of swelling of copolymer II during transition from the free state to sand decreases by only 15%: from $\alpha = 206$ to $\alpha_{lim} =$ 175. Thus, in the absence of external restrictions, a higher degree of swelling was exhibited by copolymer I: 440 versus 206 for copolymer II; at the same time, in the limited pore space, copolymer II swelled better: $\alpha_{lim} = 175$ versus 70 for copolymer I. In other words, when the external environment was replaced, inversion in the swelling capacity of crosslinked polymers was observed.



Fig. 3. Degree of swelling of copolymer I (dark rectangles) and copolymer II (gray rectangles) in the free state and in the sand; the samples were saturated with 0.001 mol/L phosphate buffer with pH 6.5. The average values of α are indicated for a content of the hydrogel in the sand of 0.2–1.0 wt % in the case of copolymer I and 0.1–0.3% in the case of copolymer II.

Effect of Polymer Hydrogels on the Water-Retaining Capacity of Sand

Figure 4 shows S-shaped curves of water retention for the initial quartz sand and sand with polymer hydrogels approximated in accordance with the van Genuchten model [34]. They reflect the main hydrophysical characteristic of the soil, the relationship between the pressure of soil moisture log P and humidity W. The higher the indicator W for the same value of log P, the higher the ability of sand to store and retain water. Water retention curves for the initial sand (curves 1) contain three sections. At low values of centrifugation speed (i.e., at low values of moisture pressure), water retention remains at the initial level with W = 27% (region I), and an increase in the speed of centrifugation above a certain critical value (it corresponds to $\log P = 1.5 - 2.0$) leads to a sharp decrease in W (area II), after which W smoothly decreases with increasing $\log P$ (area III). The same profile is also typical of the curves of water retention of sand saturated with polymer hydrogels (curves 2-4) with the difference that the boundaries between the regions are blurred and S-shaped curves are smoothed out.

Of particular interest are the points of intersection of the water retention curve with the secants represented by equations $\log P = 2.17 + W/100$ and $\log P =$ 4.18. The intersection of the first secant with the water retention curve gives the lowest moisture capacity of the sample (LM), and the intersection of the second secant yields moisture that is not available for plants (the so-called wilting moisture, WM) [40]. The difference between these values determines one of the important soil-hydrophysical constants, the range of moisture available for plants RMA = LM – WM.

Introduction of copolymers I and II into sand had a noticeable impact on the profiles of water retention curves (Fig. 4). Both polymers displaced water retention curves to the right, which reflected the ability of polymer hydrogels to store and retain water. Note that a tangible amount of water is retained in the sandhydrogel mixture even at maximum log P (3.94), which corresponds to an applied pressure of 850 kg/cm². Copolymer II acted as a more efficient water absorbent: the same values of moisture content W were achieved at a lower content of this polymer compared with copolymer I (Figs. 4a, 4b).

The results of the analysis of water retention curves for the sand and polymer-sand mixtures are summarized in Table 1, which, in addition to the above-mentioned constants LM, WM, and RAM, gives the maximum moisture capacity of the samples W_s before centrifugation, that is, before applying external pressure. In the sand, at a sufficiently high maximum humidity $W_s = 27\%$, the lowest moisture content is not above 4%, and the wilting moisture is 1%. The range of moisture available to plants for the sand is only RAM = LM – WM = 3 wt %.

These indicators underwent significant changes upon addition of the swelling copolymers to the sand. With an increase in the proportion of the copolymer in the mixture with the sand there was an increase in all indicators, including the main characteristic of soil moisture retention, the range of available moisture. Earlier, the authors of [41] showed that aggregated fertile soils of loamy composition are characterized by a range of available moisture of about 16%. This criterion makes it possible to consider optimal polymersand mixtures with a copolymer I content of 0.8 wt % and a copolymer II content of 0.2 wt %.

Structural Properties of Polymer Hydrogels

Protective anti-erosion coatings on coarse-grained soils (soils) are formed by spraying aqueous polymer formulations over the treated surface; it is considered optimal to use 1-2 wt % solutions for linear polymers with a flow rate of 1-3 L/m² [16, 18, 19]. The viscosity of 1 wt % water suspension of copolymer I measured



Fig. 4. Water retention curves for the sand with different content (a) copolymer I and (b) copolymer II. (a) The content of copolymer I is (1) 0, (2) 0.2, (3) 0.5, and (4) 1.0 wt %; (b) the content of copolymer II is (1) 0, (2) 0.1, (3) 0.2, and (4) 0.3 wt %. Samples were saturated with 0.001 mol/L phosphate buffer at pH 6.5.

by flow viscometry was 32 mm²/s for fraction I with a dry particle size of less than 0.25 mm and 53 mm^2/s for fraction II with a dry particle size of 0.25–0.50 mm. Such suspensions easily passed through the viscometer capillary with a diameter of 2.37 mm; they could be applied to the surface by spraying, which was obviously due to low mechanical characteristics of the swollen particles of copolymer I which were easily deformed and even partially destroyed when passing through the small holes of the spray gun. On the contrary, the granules of copolymer II, both initial with a size of 0.3-0.5 mm and those obtained after grinding to a size of less than 0.25 mm, formed elastic hydrogel particles in water, the size, elastic modulus, and strength of which did not allow them to pass through a 2.37 mm capillary. Such a suspension did not pass through the holes of the spray gun, and it could not be applied to the surface of the sand by spraving.

Processing of the sand with 1 wt % water suspension of copolymer I (fraction I) at a flow rate of 2 L/m^2

Content of	Soil-hydrological constant, %			
copolymer in sand, wt %	Ws	LM	WM	RAM
0	27	4	1	3
Copolymer I				
0.2	43	16	12	4
0.5	62	29	19	10
1.0	91	52	30	21
Copolymer II				
0.1	44	15	6	9
0.2	64	27	10	17
0.3	80	35	16	19

 Table 1. Soil-hydrological constants of sand with different content of copolymers I and II

led to the formation of a continuous polymer-sand coating (crust); the crust thickness was 5-6 mm. A photograph of such a crust after its separation from the sandy base is shown in Fig. 5a. The water suspension of copolymer II was evenly distributed over the sand surface with a spatula, which was accompanied by adhesion of the initial swollen particles but did not lead to the formation of a continuous crust, as shown in Fig. 5b.

The mechanical strength of the coating formed on the sand after application of the aqueous formulation based on copolymer I was estimated by the penetration method on a Rehbinder plastometer [35] to be $106 \pm$ 22 mPa. The strength of the polymer-sand composition formed by copolymer II was minimal and comparable to the strength of unstructured sand.

The initial sand and the sand treated with 1 wt % water suspensions of both copolymers were tested for resistance to the action of air currents created by an electric hair dryer (Fig. 6). In the control experiment, the initial sand was applied to the Petri dish and the air flow was directed at it at a speed of 40 km/h. A sand loss of 100% was recorded 5 s after the start of blowing. The sand treated with copolymer II (Fig. 6b) was almost completely removed from the Petri dish after 5 min of exposure to the air flow at a speed of 40 km/h

(Fig. 6d). Expressed stability was shown by a crust of sand and copolymer I (Fig. 6a) which retained its integrity after the action of air flow at a speed of 76 km/h for 30 min (Fig. 6c).

In addition, the water resistance of the coatings formed on the sand surface by copolymers I and II was studied (Fig. 7). In the control experiment, it was shown that a 10-min treatment with water leads to 100% washing out of the sand from the Petri dish. The sand and copolymer I crust (Fig. 7a) when treated with water quickly swelled and turned into a jellylike coating which completely blocked the washout of the sand (Fig. 7c). In contrast, the sand treated with copolymer II (Fig. 7b) was easily washed out with water and flowed out of the Petri dish (Fig. 7d).

Effect of Copolymer I on the Viability of Microorganisms

The influence of copolymer I on the viability of microorganisms was tested using the standard method for determining the minimum inhibitory concentration [36]. For this purpose, test cultures, such as gramnegative bacteria *Pseudomonas aeruginosa 4.8.1*, grampositive bacteria *Staphylococcus aureus 209P*, and yeast *Yarrowia lipolytica 367-2*, were grown in the presence of different concentrations copolymer I. The maximum content of copolymer I in the medium was 0.5 wt %. In all cases, the growth of microorganisms comparable to the control was observed. This indicates that copolymer I in the investigated concentration range 0.1–0.5% has no antimicrobial properties.

CONCLUSIONS

The swelling capacity of the synthesized and commercial network polyelectrolytes in the free state (in air) and under conditions of limited volume (after distribution in fine sand) has been investigated. In air, the loosely crosslinked copolymer I exhibited a greater degree of swelling; in the limited pore space of sand, a greater degree of swelling was shown by copolymer II. Both copolymers at a flow rate of ~0.2–0.8 wt % increased the range of moisture available for plants in



Fig. 5. Photographs of composite polymer-sand structures formed on the sand surface after treatment with 1 wt % aqueous suspension of (a) copolymer I and (b) copolymer II.

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Fig. 6. Polymer-sand compositions based on (a) copolymer I and (b) copolymer II; (c) coating of sand and copolymer I after exposure to wind at a speed of 76 km/h; and (d) the sample of sand and copolymer II after exposure to wind at a speed of 40 km/h. See text for explanations.



Fig. 7. Polymer-sand compositions based on (a) copolymer I and (b) copolymer II, as well as (c, d) after their treatment with water, respectively. See text for explanations.

sand (sandy soil) to the level corresponding to wellaggregated fertile loamy soils.

In an aqueous medium, copolymer I formed easily deformable hydrogel particles; the resulting suspen-

sion was sprayed onto the sand surface. The formed protective coating had a high dry strength, and when treated with water, it swelled and formed a jellylike layer on the soil surface which was not washed out by water. Copolymer I did not inhibit the growth of bacteria and yeast cells. The obtained results are useful for the interpretation of the behavior of loosely crosslinked hydrogels in the limited pore space of the soil and for the development of environmentally friendly soil conditioners with a combined water-saving and structuring action.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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