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RESEARCH ARTICLE

Synthesis of New Fixings of Mobile Sands

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ABSTRACT

The issues of the synthesis of a water-soluble polymer preparation, which can find application in agriculture as a structure-forming agent of soils and mobile sands to prevent water, wind, mechanical erosion, increase fertility, moisture absorption, moisture retention, consolidation of soils, dumps, and mobile sands to eliminate negative effects on the environment. Maleic acid and acrylamide were chosen as monomers for the copolymerization reaction, and potassium persulfate was chosen as the initiator. As it turned out, an increase in the concentration of the initiator from 0.01 to 0.05% (by weight of monomers) promotes an increase in the rate of the polymerization process, maintaining its value for a longer time, reducing the time of this process from 7.0-6.5 to 5, 5-6 hours. In this case, the yield of the polymerization reaction increased exactly from 81.2 to 96.0% for the reaction with the ratio of starting materials 1: 5. When a small amount of alkali is introduced into the reaction mixture, high molecular weight polymers can be obtained. In this case, the yield of the process increases, and the reaction time is reduced by 2-3 hours. Analysis of the kinetics of fixing processes using synthesized and various other reagents, as well as changes in the plastic strength of sands, showed the dependence of the conditions of penetration of the fixer with the formation of a free flow in space under the influence of gravitational or capillary forces on the type of binding agent and on the composition of the sand itself.

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Introduction

At present time water-soluble polymers (VSP) and polyelectrolytes (PE) are wide used in different ranges of national economy as additives to dispersion systems for soil structuring preventing of water and wind erosion, fixing of soils and mobile sands, stabilization of soil boring solutions, such as pulps, natural muddy and industrial sewage [1-6].

Effectivity of using VSP and PF has depended on their composition, presence in macromolecules of different by nature active hydrophobical functional groups and their ratio, degree of their ionization [3,4,7,8].

From theoretical and practical points of view for regulation of properties of dispersion systems by PE, obtained polymerization of acrylamide (AA) and acrylonitrile (AN); copolymerization of α - and ß-unsaturated dicarbonic acids including methacrylic acid (MA) and fumaric acid (FA) with AA and AN has a considerable importance [9]. In result of saponify cation of some PE the particle conversion of amidal and some others groups of polymers in carboxylate groups (to 30%) and increasing of dimensions of macromolecules balls and viscosity of polymer solutions, in result of electrostatical repulsion of the same charges in chains what has cause increasing of structure-forming, floculational, thicking and some others properties of PE and VSP [10].

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From above-stated it obviosly that PE containing carboxylic, amide and some others functional groups can be obtained by different methods such as polymerisation, copolymerisation, polymetanological transformations on the base of different monomers (comonomers) and polymers. Depending on conditions of synthesis (concentration, temperature) it is possible to synthesize VSP containing in organic and inorganic groups.

Using of PE is connected with properties of their water solutions, specifical conformational changings of their macromolecules in these solutions in dependence on concentration of PE, pH-medium, ionic strength of solution, temperature which have influenced on their hydrotation and ionization of polar groups of their macromolecule and also on interaction with others monomers and particles of dispersional systems. Structure-forming effect of PE has depended on composition, concentration, ionic strength and presence of active functional groups in polymer chains, molecular mass, nature and composition of opposeions and especially of state of macromolecules of PE in solution [8,11,12-14, 24].

Degree of influence PE on soil, sands and dispersions also has depended on their type and concentration in solution [3,20-27,39]. Possibility to formation on the surface of particles of dispersion systems swelling, strong, low-soluble in water films is very important property of VSP and PE because these films have learned agronomical structures in soils, fixing of grounds and mobile sands.

Such as follow from literature data the most effective PE possessing by strong flocculating and structure forming action on different dispersion systems are PE with ionizating active groups such as amid-, amine-groups; termary ammonium bases, carboxylic and carboxilate; PE containing such functional groups have adsorbed on the surface of solid particles under action of forces of chemical, hydrogen and some others nature on them and modified these surface owing to such processes as (neutralization, hydrophilization and bridge-formation), owing to which thes also has promote formation of particle aggregates (secondary structures) which have changed in considerable degree physico-chemical parameters of dispersional systems such as sedimentational, filtrational, porour formation and some others.

In literature there are data by synthesis and properties of copolymers of MA and FA, but literature data by their influence on properties of dispersional systems are very limited.

The aim this of investigating is problems of synthesis of water-soluble polymer preparates which can be used in agriculture as structure-forming agents of soils and mobile sands for averting of water, wind and mechanical erosion, increasing of fertility, mosture-absorbtion, moisture-keeping, fixing of grounds, and mobile sands with aim of removal of negative influence of this factors on environment.

Experimental Part

MA and FA were used as monomers for copolymerization which was carried out in water and $H_2S_2O_8$ was used as instiator. For regulation of pH medium KOH and NaOH, K_2HPO_4 (mark of pure for analysis), HCI(d=1,0251 g/sm^3) and some others reagents with corresponding characteristics were used. Influence of reaction duration, temperature and concentration of used compounds on reaction and also some colloid-chemical properties of obtained copolymers has been investigated. Extraction of copolymers from reaction medium was carried out by addition acetone before their full precipitation. Sedimat has been from liquid medium, was washed by acetone and then has dried been in vacuum before constant mass. Then preliminary experiments have been carried out by determination structure-forming ability of obtained copolymers according to selected samples of sands.

Sequence of synthesis of copolymers has included following stages: in three throatflask 200ml solution, containing MA and FA in mole ratios 1:1-1:10 were added than initiator $K_2S_2O_8$ in quantity 0,01-0,1% from comonomers mass was also added. The flask was supplied by system of avtomatical flowing by nitrogen in dependence of pressure changing in system and also by thermometer, mixing and refrigerator. After finish of flask load of reactor, the mixture of components has been heated for initiation of copolymerization. The system was endured in thermostat before reaction finishing, and then yield of reaction was calculated. After this neutralization and hydrolysis of obtained copolymers were carried out.

Identification of functional groups of copolymers has been carried out by IR spectrometry with using spectrometer Avatar FT-IR vicold-5,50. Thermo Ficher Scientific with Furje transformation in range of frequencies 400-500sm.$^{-1}$

Viscosital characteristics of PE were investigated by using rotary viscosity VSN-2M and also viscosity meter VBB-2

Kinetics of structure-formation has been characterized by value of plastical strength (P_m), which was measured on consistometer Ceppler [40].

For this mixture in quantity 40-50g was mixed during 2-3min and placed in aluminium boxe by high 20mm and inner diameter 50mm which was placed in exicator over saturated solution of line. Thichened sand has been impregnated by liquids containing in their composition fixings in quantity 100-200ml on 1kg of sand.

Results and Discussion

In process of copolymerization increasing copolymers concentration and viscosity of system has been observed owing to which mobility of macromolecules and correspondently rate of reactional stage of termination of growing chains were decreased and also decreasing of molecular mass of forming copolymers. For avoid of such negative phenomenous it is necessary to maintain optimal concentration of initiator and constant temperature to ending of copolymerization process. Rightly approached concentration of initiator has provided generation of free radicals and supporting their concentration on level during

out copolymerization expense on the yield of reaction copolymerization has been investigated results of which are presented on fig 1.

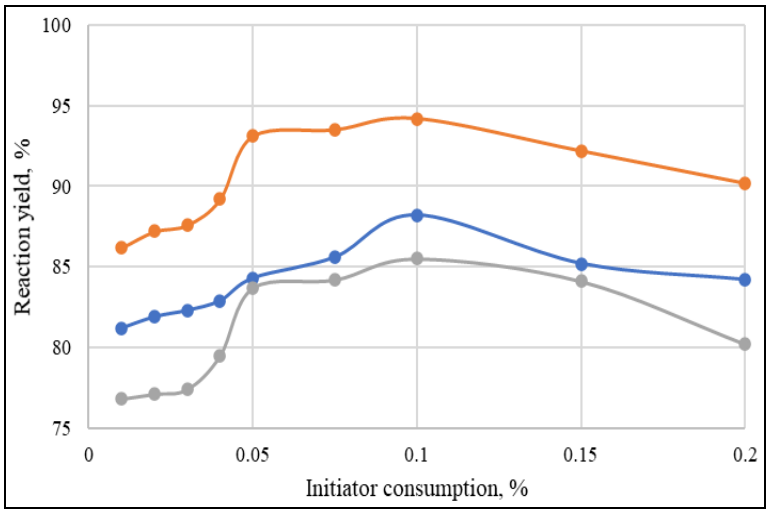

Fig. 1. Dependence of yield of copolymerization reaction of MA and AA from initiator expense at their mole rations: 1) 1:5; 2) 1:8; 3) 1:10.

Carrying out laboratory tests have shown, that changing of inhibitor concentration in considerable degree has influenced on reaction yield. Initiation of process is a rupture of baods -O-O-, energy of which consist 100-200 kDj. [41] and initiation is carried out of 30^0C. Incensing of initiator concentration from 0,01 to 0,05%. (from mass of conomers) has promoted to increasing rate of copolymerization, decreasing of duration of this reaction from 7,0-6,5 to 5,5-6,0 h and at this yield of copolymerization reaction has increased from 81,2 to 96,0% at initial ratio of comonomers 1:5. Approximately results were obtained at others conditions of carrying out copolymerization of MA and AA. As shown from fig.1 increasing of $K_2S_2O_8$ cconcentration higher 0,1% from conomers mass on the contrary has carried out to decreasing of yield of copolymers and their molecular mass and especially this effect was observed at using high concentrations of AA (1:10) in reaction mixture what obviously is connected with increasing of concentration of initial active enters and free radicals what cases recombination of growing chains. It was determining that optimal concentration of initiator was equaled 0,05- from conomers mass.

Influence of concentration of water-soluble initiator on duration of copolymerization reaction of MA and AA at their different ratio and temperatures has presented in table 1.

Table 1. Influence of initiator quantity on copolymerization reaction

Mole ratio of MA and AA	Expense of $K_2S_2O_8$	Time of copolymerization, h	Molecular mass of copolymer, g/mole,	Yield, %
1:5	0,05	7	$2,862 \bullet 10^6$	84,3
	0,1	5		88,5
	0,2	5		84,2
1:8	0,05	5	$2,8625 \cdot 10^6$	93,1
	0,1	4,5		94,2
	0,2	4		90,2
1:10	0,05	6,5	$2,8414 \cdot 10^6$	83,7
	0,1	6		85,5
	0,2	6,5		80,2

Temperature-48^0C, pH limit 1,2:1,8

In all experiment's formation of turbid and viscous solutions of obtained copolymers with their containing 9,10-9,55% was observed after 15h what witnessed about formation of colloidal solution of copolymers.

It is known that difference in activities of comonomers has caused to considerable decreasing of participation of second component in copolymerization. However, by reaction ability investigated comonomers (MA and AA) are very similar by what cause to support of high rate of reaction without that additional introduction of more active component. As a role values of activity of radicals and comonomers in reactions of copolymerization are characterized by reverse dependence. AA has polymerized with high rate owing to displacement of electronic density π-bond and it`s polarization and correspondently it`s radical has characterized by low activity. Also it is necessary to note sterically factor of functional group of MA frining to screening of double bond, what also negative by has influenced on rate and yield of copolymerization reaction. Probably by these causes yield of reaction at molar ratio AA:MA 5:1was lower in comparison with others systems.

At carrying out copolymerization in solutions in and emulsions a great importance has viscosity of medium. In investigation reaction formation of turbidity in systems carried increasing of their viscosity what caused decreasing degree of mobility of systems components and as a rule termination of growing chains was carried out. By this reason investigation was carried out by decreasing of viscosity by increasing of water-solubility of obtained copolymers by introduction in system 10% solution of NaOH or KOH to enchainment of pH-medium 7,5-8,5. At this from MA under action of KOH it`s salt was formed (KMA). In result of copolymerization of KMA and AA thick, transportation comogeneous mass was formed. Characteristics of copolymerization of KMa with AA are presented in table 2. (The best results are obtained at mass ratio KMA and AA 1:8).

Table 2. Characteristics of copolymerization reaction KMA with AA at their mass ratio 1:8

Expense of $K_2S_2O_8$	Temperature of reaction, ° C	Duration of reaction, h	Molecular mass of copolymers, g/mole	yield, %
0,05	40	5	$2,8625 \bullet 10^6$	96,1
	50	5		99,2
0,1	40	4,5	$2,8625 \bullet 10^6$	98,3
	50	4,3		99,6

Obtained results have shown that introduction in reaction mixture the small quantity of alkalies high molecular copolymers can be obtain with high yield and at this duration of reaction has been increased on 2-3 h.

Obtained copolymers were undergone to hydrolysis under action of alkalies (NaOH or KOH) or calcinated soda. For obtaine of PE with necessary degree of hydrolysis reaction was carried out at addition in initial solution NaOH or KOH in molar ratios with AA in limits 1:0, 2-1:1 and at this duration of hydrolysis was equaled 5-3h at temperature 36-98^{0}C.

Investigation of viscosity of PE solutions has allowed to estimate changings of their macromolecules conformations in solution. Structure-formation in their solutions was coming at achievement of some PE concentration that is when macromolecules can interact with each other and it is possible formation of associates– spatial net which has prevented to flow of liquid with increasing of viscosity.

Table 3. Specifically viscosity and pH solutions of copolymers and their hydrolyzed forms

Sample	Hydrolyzing agent	η_{red} /C	Concentration of agent, %
1*	NaOH	34.23	1
	KOH	37.36	1
	Na_2CO_3	23.31	1
2**	NaOH	20.15	1
	KOH	23.28	1
	Na_2CO_3	20.04	1

As shown from data of table 4. specific viscosity of solutions was increased with simultaneously at increasing of PE conversion and such dependence was characteristically for all obtained PE. Increasing of specifically viscosity was carried out owing to changing of volume part of solvented copolymer and intermolecular interaction of its macromolecules. As a role the more volume of viscosity of PE solutions the more their forming ability of on different dispersion systems. As shown from table 3 that more high values of 9sp/c were observed for forms PE, obtained by their hydrolysis in the presence NaOH. By this reason it is possible that structure forming and stabilizating ability namely of these PE on dispersions of soil, sand clayey minerals has differed by high degree. Results by investigation of plastically strength of sands treated by PE solutions are presented lower, In work [41] analysis of kinetics of processes with using of synthesized and some others reagents and also changing of plastical strength of sands has shown dependence of penetration of with formation in space of free under action of gravitational or capillary strengths from type of binging agent and composition of used sands.

Table 4.

Simple	Depth of immersion, mm											Thickness of crust, mm
	Minutes								Twenty-four hours			
	0	**1**	**3**	**6**	**10**	**20**	**30**	**120**	**1**	**5**	**10**	
1*+KOH	4.15	4.10	4.00	3.35	2.80	2.70	2.65	2.40	2.30	2.71	1.50	2
1+NaOH	5.90	5.80	5.15	4.30	3.20	3.00	2.70	2.40	2.30	1.76	1.33	3
1+Na_2CO_3	4.90	4.85	3.70	2.95	2.40	2.35	2.15	1.60	0.90	3.00	2.20	2
2**+KOH	3.15	3.10	3.00	2.85	2.80	2.30	1.80	1.45	1.30	5.20	3.30	1
2+NaOH	4.30	4.25	3.90	3.80	3.55	2.95	2.65	2.45	2.35	3.60	3.80	2
2+Na_2CO_3	4.35	4.25	4.15	3.90	3.45	3.20	2.85	2.60	2.45	5.30	4.10	2
HPAN	5,60	5,27	5,45	5,35	4,20	4,40	4,35	4,60	5,75	1,47		2
CMC	7,40	6,25	6,00	5,83	5,50	4,0	5,4	5,7	5,46	2,10		2

*1-MA+AA *2-MA+AA

Treatment of sands by samples 1+KOH, 1+NaOH HPAN has brought to increasing of strength, but using solution CMC has caused it`s decreasing. Changing of strength in time is characterized by changing values. Moisture (24h) of layers of sands, treatment by solution 1+NaOH in quantity 6 ml on 50 g of sand was equaled 5,6 %, what is on 1,23-1,27 times larger than at impregnation by solutions of 1+KOH and HPAN Impregnation of sand by these solutions has depended from composition and quantity of introduced reagent. Results of changing of immersion of cone of consistometer (10smt) have witnessed about high structure forming ability of copolymer obtained in acid medium without neutralization of carboxylic groups of AA by hydrolysis in comparison with obtained copolymers in NaOH solution. Porosity of sands in this case has secondary role about what values of saturation coefficient impregnating sands which are larger for samples 1+KOH; 2+KOH; 2+NaOH HPAN and CMC in 1,12, 1,34, 1,47, 1,12 and 1,4 b times correspondently what has witnessed about surplus of used solution as finding (5-12%). Obviously such divergences in values of impregnation are caused by high ability of copolymer macromolecules to sorbate on hydrated lagers of surface of sand particles decreasing dimensions surface [41]

Conclusions

Such on the base of experimental data method of synthesis of WSP with using initiator $K_2S_2O_6$ and NaOH, KOH and salt as hydrolyzed agents has been carried out. Investigations of kinetics of copolymerization of MA with AA at regulating quantitates of initiator have shown effectivity of elaborated method and at this time of reaction was decreasing on 15-25% and also yield of copolymers obtained at mass ratio MA and AA 1:8 and pH of medium 1,5-1,6 the was formed with higher characteristics structure forming properties. By this reason materials and copolymers synthesizes on the base of MA and AA can be used as fixators for defenses soil and natural resource and also in fight with collisional processes of soil and mobile sands.

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