

# COMPREHENSIVE MODIFICATION OF GELS POLYACRYLAMIDE

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**Abstract:** For successful use of flocculants it is necessary to give them new features artificially, among which the most important are rheological properties – shear stress at different strain rates. They can be changed while using methods of chemical and physical modifications and the most promising among them are physical methods of modification. Comparative rheological properties of polyacrylamides aqueous solutions without and with modification of the bifunctional connections of different chemical nature (glycols, amino acids) were given. The strengthening of the modification effect was achieved by the additional impact of physico-chemical factors: ultrasonic and microwaves with a frequency of 2.45 GHz for 10 s with a power of 700 watts. The thorough analysis of the rheological studies data for each flocculant was made. With modifying PG initial yield stress ( $\Theta_f$ ) increased 1.1–1.5 times, while with the modification by glycine, this value changed to 1.1–1.7 times. The optimal concentration of PE to obtain gels with high strength comprises: a modified GHG – 1.0%, modified by glycine with the influence of ULTRASONIC – 1.0% modified by glycine – 0.7%, modified by glycine with the impact of MVO – 0.3%, indicating high efficiency of the use of glycine and MVO. It was found out that  $\Theta_f$  in the case of using PG – 1.5 times higher than that of the original (if  $c_{\text{orr}} = 1.0\%$ ); when using the glycine – to 1.7 ( $c_{\text{orr}} = 0.7\%$ ) higher than that of the original; in the using of glycine together with ULTRASONIC – in 1.5 times (when  $c_{\text{orr}} = 0.1\%$ ) higher than that of the original, when using glycine in conjunction with MVO – in 1.9 times (if  $c_{\text{orr}} = 0.3\%$ ).

**Keywords:** polyelectrolytes, polyacrylamide, flocculants, modification, microwaves, shear stress, flow curves, rheological study

## INTRODUCTION

The synthetic high molecular weight polyelectrolytes (PE) on the basis of polyacrylamide (PAA) are widely used as flocculants in modern technological processes for deposition of the dispersed phase from solutions. The Range of polyelectrolytes (flocculants) used in colloidal systems for the separation (flocculation) of the solid phase from the liquid is wide enough. But in spite of this, now there are no samples of these technical substances with special technological features, because more frequently the chemical activity of industrial samples of flocculants is not enough for complete clarification of industrial suspensions. By the direct synthesis it is not always possible to obtain the desired polymers. That is why there is a problem of "finishing" or changing the properties of known flocculants to the other, not peculiar previously. In addition, the flocculants obtained by different synthetic methods, according to the known objective reasons are limited by their molecular weight. Its value determines the main quantitative and qualitative indicators of separation processes of microheterogeneous systems [14]. With the aim of changing their selectivity with respect to various specific industrial slurries, the most radical method of properties directed change of polyelectrolytes on the basis of the original macromolecular matrix is the chemical or physical

modification that allows to change the structure of macromolecules and to introduce the necessary functional groups [1].

The importance of solving such problems was confirmed in resolutions of the international conference "Time of polymers and composites", held in Italy, where the obtaining of new polymeric materials, studies of properties and search of new ways to use them is an actual problem of modern fundamental and applied Sciences were noticed [13].

The effectiveness of flocculation depends on the molecular weight of PAA macromolecules and on their structure [1]. Because of the wide variety of cleared suspensions and colloidal solutions there is a manufacturing need to additionally change the macromolecular design standard PAA and increase its molecular mass (MM). The main idea of this article is the establishment of block polymers with increased molecular weight due to the development of new methods of changing the molecular structure of flocculants' macromolecules. This effect can be achieved by chemical compounding of linear structures, i.e. by modifying. Modification of flocculants is one of the most productive ways of obtaining new forms of nanomaterials. It is known that the nanomaterials include polymeric compositions and in particular polyelectrolytes (flocculants) [8].

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The choice of modifier is determined by the main problem of the experiment. In this case, there are three choices:

- obtaining fringed macromolecules due to the interaction of single-function modifier with one functional group of PAA;
- obtaining parallel seldom cross-linked macromolecules due to the interaction of two functional groups of the modifier with the two macromolecules of PAA;
- obtaining a complex block structure due to the triple interactions of macromolecules with the modifier having at least three functional groups.

It follows that the type of macromolecular structures modified by flocculants can be managed by the proper selection of the modifier. Therefore, at the planning stage of the experiment with pre-defined performance properties of the modified polyelectrolyte, mono-, bi - or multifunctional organic compounds with reactive groups relative to the functional groups selected flocculants should be chosen accordingly. For the separation of suspensions the most effective are flocculants, which were modified by the bifunctional modifiers. Among the well-known chemically active bifunctional organic compounds in relation to the PAA are chosen the most available.

In the process of intermolecular installation of block structures obtaining of chemical compounds was assumed due to specific (with the formation of covalent or hydrogen bonding) interactions between the functional groups of the modifier and the polyelectrolyte, and with the additional influence of physical factors of ultrasonic and microwave irradiation (MVO).

This work presents results of rheological studies of PAA solutions obtained by chemical processing with specially selected amino acid polymer cross-linking agents (modifiers) under the influence of microwave and ultrasonic influence. Modification allows you to change character of the flow of PE gels with the help of deformation.

#### MATERIALS AND METHODS

Anionic polyelectrolyte based on PAA brand "Magnaflok" (M 919), which has a degree of anionic

70% and MW according to manufacturer, equal to 30 million was selected as a flocculant. Currently, it can effectively carry out flocculation processes for the purification of many industrial micro-heterogeneous systems [9]. The additional chemical processing of standard solutions by PAA different modifiers is widely used to improve the speed and quality of suspensions purification by flocculation. Modifiers, as noted previously, are specially selected individual compounds having in its structure more reactive functional groups capable of chemically linking PE macromolecules [3-5]. In the present studies such amino acids were deliberately used as a modifier: glycine (NH<sub>2</sub>-CH<sub>2</sub>-COOH), examination serine (BUT-CH<sub>2</sub>-CH(NH<sub>2</sub>)-COOH) and propylene glycol (PG). This choice is due to the fact that the use of amino acids and glycol as modifiers allows organizing the effective "crosslinking" of the polymer chains and increasing the chemical affinity PAA to proteins and oxygen-containing organic compounds.

Obtaining with the help of the suggested method, crosslinked (modified) flocculants were successfully used to clean dairy wastewater from protein compounds [10].

The micronization process – the solutions after chemical modification of microwaves (CVM) with a frequency of 2.45 GHz for 10 seconds at a power of 700 watts, was used as the physical impact. This process is used in chemical engineering [11].

Initial polymer gels were prepared by accurately weighed portion: PAA weight of 0.3 to 2.0 g was poured by 100 ml of distilled water and allowed to stand overnight at a temperature of 200 0C. Gels of modified flocculants were prepared by making a batch of gels obtained starting amino flocculant previously selected weight 0.2 g (10% of the sample weight PAA). Modification was held with stirring gels with modifiers for 40 minutes on a magnetic stirrer at a speed of 60 rev / min.

The general scheme of the initial PAA gels preparation without MVO is presented in Fig. 1.

The scheme of obtaining solutions PAA with extra micronization is presented in Fig. 2.

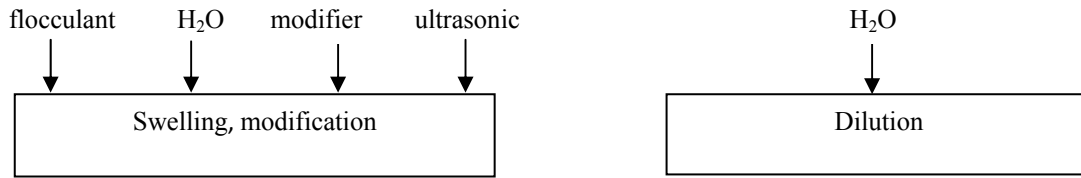
Modification of PAA with the influence of ultrasonic is presented in Fig. 3.



Fig. 1. Preparation the initial gels PAA without MVO.



Fig. 2. Obtaining the PAA solutions with additional micronization.



**Fig. 3.** Obtaining the PAA solutions with the influence of Ultrasonic.

The obtained solutions of original and modified flocculants were investigated by the rheological methods with the flow curves (rheogram) that graphically, as is known, reflect the laws of the system behavior when it is imposed on a changing deformation [7].

Shear stress depending on the strain rate of gelatinous flocculant solutions were measured in a rotary viscometer “Reotest-2” by known methods according to the instrument instructions”. The cylindrical measuring system with a ratio of diameters of cylinders 0.94 was used in this experiment. The strain rate varied from 1.5 to 1312 s<sup>-1</sup>. A designated amount of gel (25 cm<sup>3</sup>) was placed in a cylindrical cell, which continuously injected cylinder, and cylinder and box were connected with a measuring device. The gel was initially deformed at maximum speed (1312 s<sup>-1</sup>), and then we set the desired speed, started with the lowest. Counting was carried out after 10 minutes after turning on the appropriate rate. Rheological parameters were recorded at the same way during the return velocity. Checking of the method after overnight exposure of samples in the cell showed low (0.5–1.0%) the deviation of the measured parameters from the mean values.

**RESULTS AND DISCUSSION**

On the basis of obtained measurements we define the limits of strength: the minimum yield strength ( $\Theta_f$ ), Bingham yield strength ( $\Theta_v$ ) and maximum yield strength ( $\Theta_{max}$ ). The values of yield strength are presented in Fig. 4.

Flow curves of hydrogels of initial flocculant M 919 are presented in Fig. 5.

Fig. 5 shows that by increasing concentrations of flocculant in solution grows the strength characteristics of all of the investigated system,

indicating the raise in intermolecular contacts within the gel structure.

Rheograms of gels, modified by flocculants glycine, presented in Fig. 6, indicate that the shear stress at all strain rates in the investigated concentration range for PAA gels modified with glycine, higher than for the original. Hardening of the modified polymer is marked with increasing concentration of flocculant. Maximum hardening of these gels is achieved with polymer concentrations from 1 to 2%.

According to the known data [4], similar forces between macromolecules PAA containing carboxyl and amino groups, and an amino acid having similar functional groups may arise during the flow process of acid-base interaction (Fig. 7).

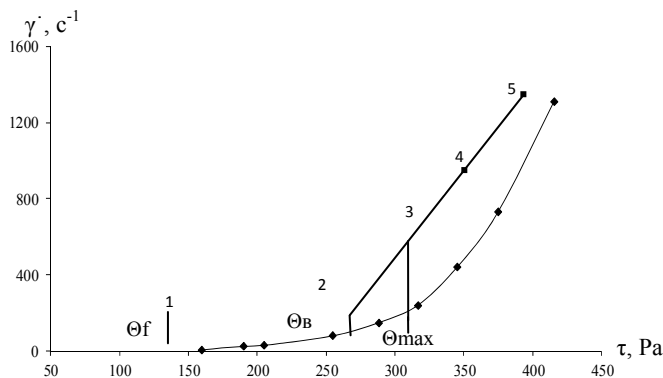
Flow curves of hydrogels flocculant M 919 modified by PG are presented in Fig. 8.

The rheological curves are shown in Fig. 9–10 modified forms of PAA with the help of glycine and with ultrasonic impact and MVO.

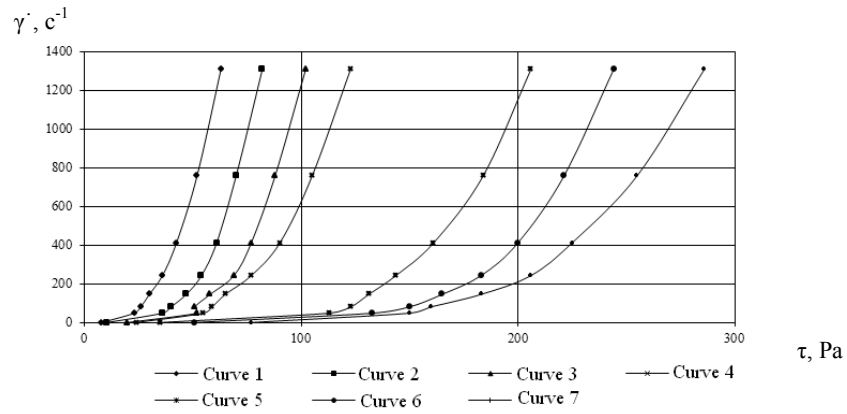
Some polymers cannot be obtained by the direct synthesis. Directional polymer degradation is needed to control the molecular weight.

Centers of destruction with the formation of a free radicals complex (R•) are the weak links in the chain, which have an abnormal structure due to monomers, connected by chance during the synthesis "head to head". Chain process develops with destruction of the skeletal bonds C-C under the influence of the radical [10].

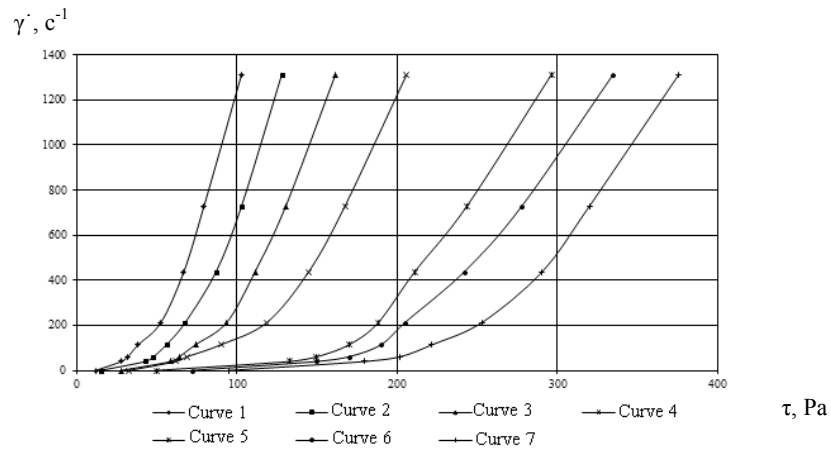
The speed of destruction and formation of new structures depends on the degree of external conditions influence. Obtained during the degradation, radicals are effectively “building” material used for producing new, more complex and branched structures in the process of physical modification.



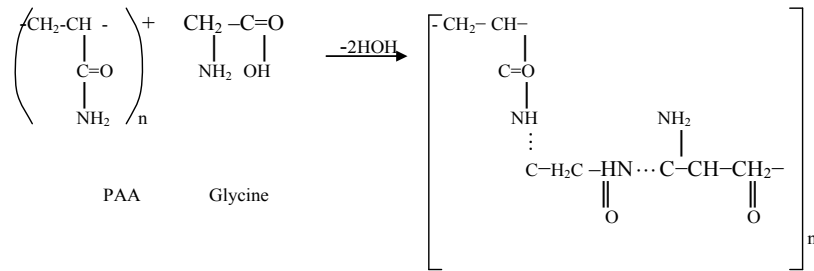
**Fig. 4.** Defining the limits of strength of PAA gels: 0–1 – zone of elastic deformation; 1–2 – zone of plastic flow with the highest viscosity; 2–3 – zone of avalanche breakdown of the structure; 3–4 – zone of viscous-plastic flow; 4–5 – zone of Newtonian flow with extremely destroyed structure.



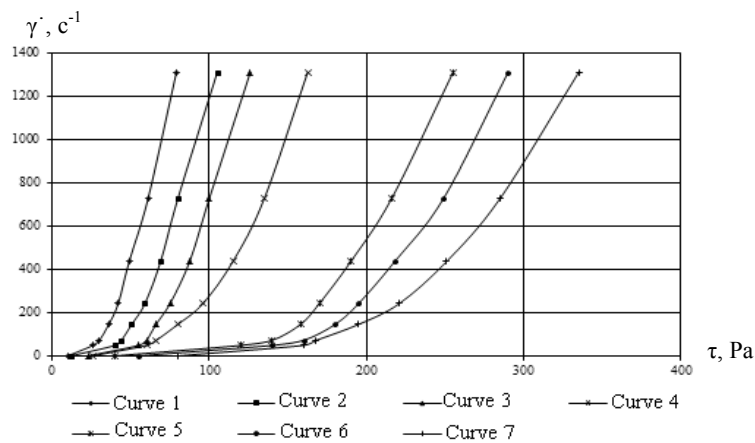
**Fig. 5.** Flow curves of hydrogels of initial flocculant M 919, with strength, (%): 1 – 0.3; 2 – 0.5; 3 – 0.7; 4 – 1.0; 5 – 1.2; 6 – 1.7; 7 – 2.0.



**Fig. 6.** Flow curves of hydrogels flocculant M 919 modified by glycine with strength, (%): 1 – 0.3; 2 – 0.5; 3 – 0.7; 4 – 1.0; 5 – 1.2; 6 – 1.7; 7 – 2.0.



**Fig. 7.** Modified PAA.



**Fig. 8.** Flow curves of hydrogels flocculant M 919 modified by PG, with strength, (%): 1 – 0.3; 2 – 0.5; 3 – 0.7; 4 – 1.0; 5 – 1.2; 6 – 1.7; 7 – 2.0.

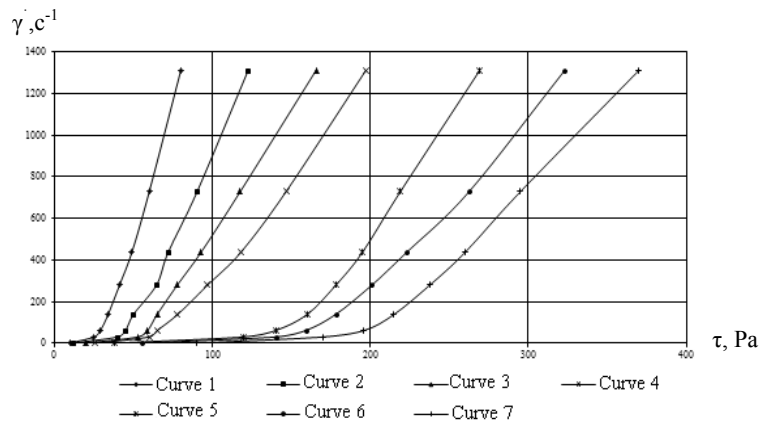
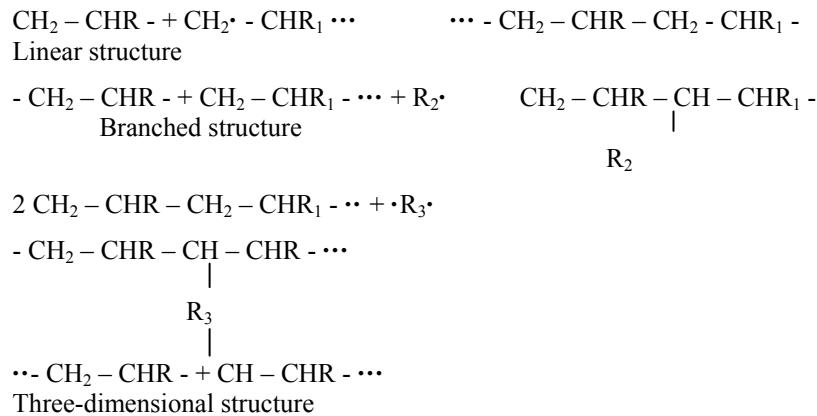
Radicals recombination can proceed with the formation of linear, branched and three-dimensional polymers.

This fact of new macromolecular formation exists because derived radicals in gel structures can be stuck and long time (about one month) stored therein before the recombination required conditions.

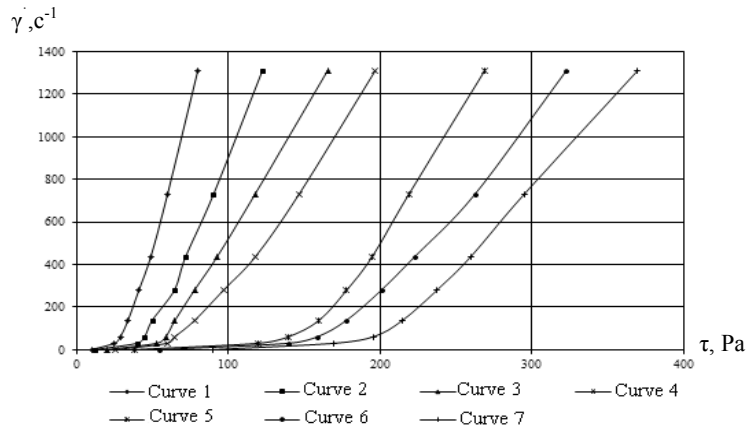
With increasing concentration of flocculants and with a constant concentration of the crosslinking agent, hardening of the structure of gels modified flocculants is marked. This significant increase is evidenced in the values of shear stress needed to break the modified system in comparison with the original. Maximum hardening of crosslinked gels, i.e. the maximum difference between the strength characteristics of the

modified PE and initial sample is obtained within the concentration range for all polymers in the range of 0.7–1.7%. However, for flocculants modified by glycine with the influence of MVO, this difference is the greatest. It can be assumed that in this range of flocculants modified by glycine, the most stable complexes are formed (PAA – crosslinking agent – PAA).

This fact can be explained by the geometric correspondence between the length of the modifier molecule and the distance between the macromolecules (they close in magnitude) and the specific impact of MVO, with results in partial degradation of the polymer and the formation of new macro-radicals and of new, more lasting space structures.



**Fig. 9.** Flow curves of hydrogels flocculant M 919 modified by glycine with the influence of ultrasonic, with strength, (%): 1 – 0.3; 2 – 0.5; 3 – 0.7; 4 – 1.0; 5 – 1.2; 6 – 1.7; 7 – 2.0.



**Fig. 10.** Flow curves of hydrogels flocculant M 919 modified by glycine with MVO, with strength, (%): 1 – 0.3; 2 – 0.5; 3 – 0.7; 4 – 1.0; 5 – 1.2; 6 – 1.7; 7 – 2.0.

Furthermore, in analyze of rheological curves it was observed that gels behave as Newtonian fluids (the viscous flow is present) at low concentrations of polyelectrolyte, but with the concentration increasing, flocculants behavior as the original and modified samples behavior as that were observed in the systems of Ostwald-de-Valais. These systems are always thixotropic. According to published data it can be assumed that in hydrogels modified by glycine with the impact of MVO, we observe the phenomenon of rheopexy, the main cause of which may be the formation of supramolecular structures between the molecules of the polymers and in result interchain interaction is enhanced. In the investigation of gels modified and unmodified flocculants of varying concentrations there were determined a minimum yield strength  $\Theta_f$ , corresponding to the beginning of flow (top destruction of the structure), yield stress – Bingham  $\Theta_B$  and maximum limits of flow (complete destruction of the system)  $\Theta_{max}$ . The results of calculations for the original and modified flocculants are presented in Table 1–5.

For each flocculant the thorough analysis of the rheological research data were made. While modifying flocculant M 919, with the help of modifiers, which were structurally different, but related in some chemical characteristics (functional groups) the

difference of (greater) impact on the strength of the associates was found. So an initial yield point PG ( $\Theta_f$ ) increased in 1.1–1.5 times in the modification, whereas by glycine this value changed in 1.1–1.7 times. The difference in the values indicates stronger macromolecules by the modifier, which contains amino and carboxyl groups. Optimal concentrations of PE for obtaining gels with high strength are: modified PG – 1.0% modified by glycine with the impact ultrasound – 1.0% modified by glycine – 0.7% modified by glycine with the impact MVO – 0.3%, this indicates a high efficiency of glycine and MVO. It was found that  $\Theta_f$  in the case of using PG – 1.5 times higher than the original have ( $c_{opt} = 1.0\%$ ), using glycine – 1.7 times ( $c_{opt} = 0.7\%$ ), using glycine with ultrasonic – 1.5 times higher ( $c_{opt} = 0.1\%$ ), using glycine with MVO – 1.9 times ( $c_{opt} = 0.3\%$ ).

According to the data obtained (Tables 1–5), yield stress have higher values for all concentrations of modified PAA. Moreover, with increasing concentration gel structures of modified samples become stronger compared to unmodified. The difference of strength characteristics is greatest for flocculants modified by glycine with the use of MVO, so the most durable mesh supramolecular structures are formed by the crosslinking of the flocculant with glycine and with the use of MVO.

**Table 1.** The dependence of the yield stress from the flocculant M 919 initial concentration (W)

Concentration W, %	$\Theta_f$	$\Theta_B$	$\Theta_{max}$
0.3	6.7	35.6	51.1
0.5	11.1	55.6	73.3
0.7	20.0	68.9	91.1
1.0	24.4	82.2	109.0
1.2	37.8	155.6	186.7
1.7	51.1	188.9	227.8
2.0	77.8	215.6	244.4

**Table 2.** The dependence of the yield stress from the flocculant M 919 concentration, modified by glycine \*

Concentration W, %	$\Theta_f/d_1$	$\Theta_B/d_2$	$\Theta_{max}/d_3$
0.3	11/1.6	50/1.4	80/1.4
0.5	15.6/1.4	72/1.3	108/1.5
0.7	28.12/1.4	90.6/1.3	128/1.7
1.0	37.5/1.6	115.6/1.4	166/1.6
1.2	50/1.3	178.1/1.15	247/1.3
1.7	73.4/1.4	208/1.1	273/1.2
2.0	93.75/1.2	253/1.2	328/1.3

Note. \*  $d_1, d_2, d_3$ - the degrees of increase of yield corresponding modified polymer gels in comparison with the similar outside the yield gels initial PE.

**Table 3.** The dependence of the yield stress from the flocculant M 919 concentration, modified by glycine with the influence of ultrasonic

Concentration W, %	$\Theta_f/d_1$	$\Theta_B/d_2$	$\Theta_{max}/d_3$
0.3	9.03/1.3	35.9/1	75.2/1.5
0.5	12.1/1.1	60/1.06	90.6/1.2
0.7	27.7/1.4	75.6/1.1	115.6/1.3
1.0	39.5/1.5	89.1/1.1	150/1.4
1.2	47.6/1.3	156.25/1	223/1.2
1.7	57.6/1.13	204.4/1.1	268.8/1.2
2.0	89.8/1.15	228.5/1.1	325/1.3

**Table 4.** The dependence of the yield stress from the flocculant M 919 concentration, modified by glycine with MVO

Concentration W, %	$\Theta_f/d_1$	$\Theta_B/d_2$	$\Theta_{max}/d_3$
0.3	12/1.8	53/1.5	97/1.9
0.5	17.6/1.6	75.5/1.4	120.6/1.7
0.7	29.4/1.5	112/1.6	164/1.5
1.0	35/1.5	135.3/1.6	194/1.7
1.2	50/1.4	197/1.3	250/1.4
1.7	73.5/1.4	226/1.2	288/1.3
2.0	94.75/1.2	276.5/1.3	338/1.4

**Table 5.** The dependence of the yield stress from the flocculant M 919 concentration, modified by PG

Concentration W, %	$\Theta_f/d_1$	$\Theta_B/d_2$	$\Theta_{max}/d_3$
0.3	7.8/1.2	39.4/1.1	68.2/1.2
0.5	12.5/1.13	58.6/1.1	81.8/1.1
0.7	21.9/1.1	78.5/1.14	106.1/1.2
1.0	37.5/1.5	100.5/1.2	139.4/1.3
1.2	43.8/1.2	166.7/1.1	219.7/1.2
1.7	54.7/1.1	199.3/1.1	257.6/1.1
2.0	84.4/1.1	225.8/1.1	307.6/1.3

The analysis of rheological characteristics allows us to consider that the yield stress, which was mentioned above, increases with the growth in the frequency of crosslinks between the molecules of polymers. According to [195], it is known that the higher the yield, the slower the ruined structure reconstructs. It follows that the greatest ability to relaxation of stresses (reconstruction of destroyed structures) has hydrogels, crosslinked by glycine at medium and high concentrations.

Rheological investigations of PAA gels with the using of serine modifier (hydroxy-amino acid) are presented in Fig. 11.

Experimental data show that the nature of flow curves for solutions of flocculants, modified by serine, very different from the character of its current solutions of unmodified samples.

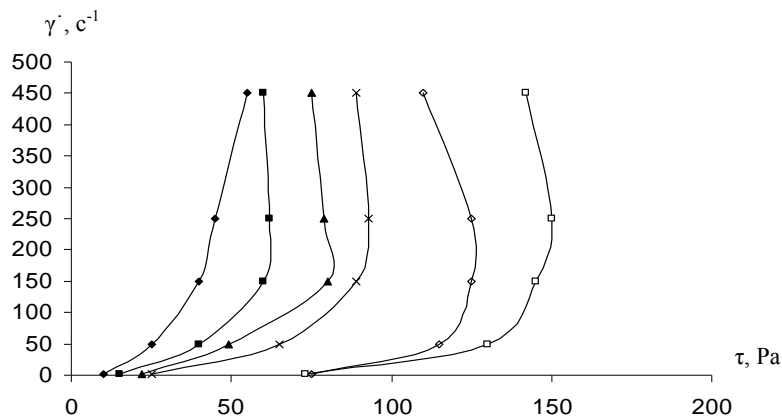
Such changes of the rheological properties indicate coagulation of modified PAA complexes in compact structure. Similar phenomena are observed in the transition of fibrillar structure of polymers

macromolecules in globular structure. Globules - are structures, consist of several coiled macromolecules. In the globular structures grid spatial are formed and also gels with the high content within the base polymer. The main reasons for this are the attraction between segments of a single polymer coil. The theoretical description of such a process was suggested in [8].

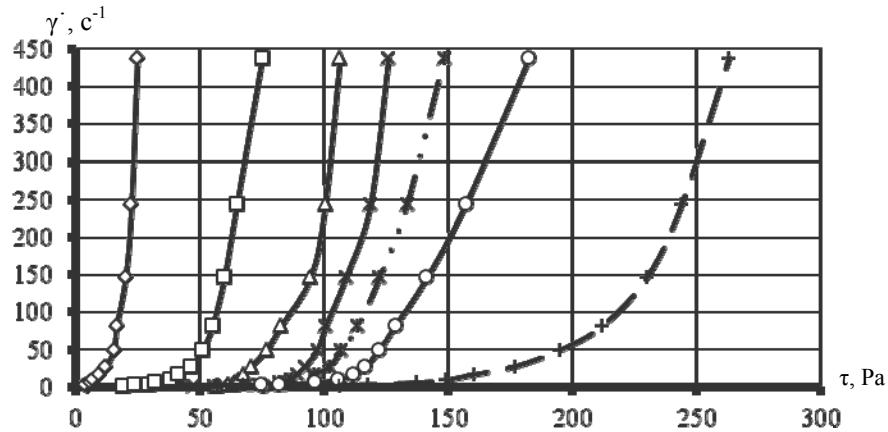
The most typical flow curves for flocculant M 919 modified by serine, are presented in Fig. 12, which have a different view than the curves in Fig. 11.

In solutions with the concentration more than 1.2% of PAA the inflection is observed on curves. This indicates the particular impact of MVO: in irradiation of PAA macromolecules radicals, which under applied shear stress and complicate further modify flocculant structure, arise [12].

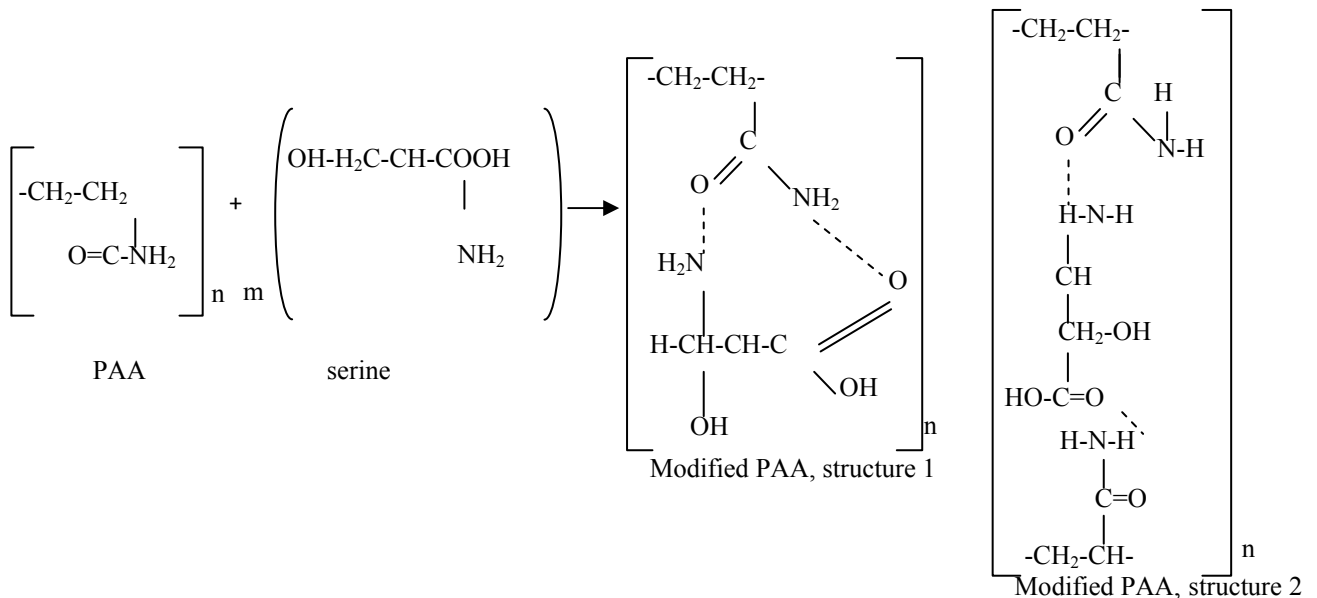
According to known data [6], between macromolecules of PAA containing amino and carboxyl groups, and an amino acid may occur such reaction of acid-base interactions, as in the case of glycine, with formation an additional hydrogen bond grid.



**Fig. 11.** Flow curves of hydrogels flocculant modified by serin, with strength, (%): 1 – 0.3; 2 – 0.5; 3 – 0.7; 4 – 1.0; 5 – 1.2; 6 – 1.7; 7 – 2.0.



**Fig. 12.** Flow curves of hydrogels flocculant M-919, modified by serine with MVO, with strength, (%): 1 – 0.3; 2 – 0.5; 3 – 0.7; 4 – 1.0; 5 – 1.2; 6 – 1.7; 7 – 2.0.



Shear stress was defined for the gels modified by serine flocculants with the influence of MVO, the values of which are presented in Tables 6–7.

Analysis of the data from tables 6-7 allow to make conclusions: the yield stress has higher values for all concentrations of PAA modified with MVO. Furthermore, modified samples with increasing concentration become stronger than unmodified. The yield of modified PAA increases with growing frequency of crosslinks between polymer molecules.

Fibrillar and globular structures of polymers can also be presented by electronic photographs in Fig. 13 and 14.

Analysis of the Fig. 11–14 allow to make following conclusions:

– there was observed the reduce of the strength characteristics of gels of the investigated PE, modified by hydroxy-amino acid, compared with the initial PAA

gels and gels modified by glycine;

- the different curves of flow for all samples of gels were noticed, but not for one of them (0.3%);
- the anomalous behavior of the rheological curves were recorded with concentrations of PAA 0.5% and more, it was characterized by a decrease in shear stress with increasing strain rate;
- the effect of macromolecular collapse with the transition linear macromolecules fibrillar structure in globular is established with concentrations of polymer gels 0.5% or more;
- the presence of macromolecular collapse on the basis of modern theories is explained by twisting the polymer macromolecules due to the formation of additional hydrogen grids between polar groups of the modifier (hydroxyl-amino acid) and the molecular chains of PAA, which is now considered as the simplified model of protein [9].

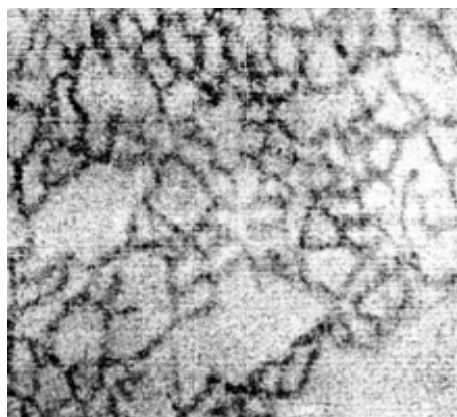


**Table 6.** Flow curves of hydrogels flocculant M-919, modified by serine

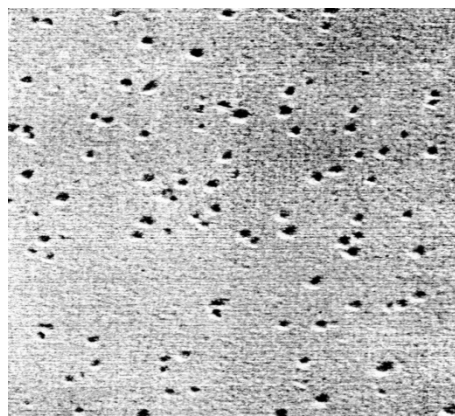
Concentration W, %	$\Theta_f/d_1$	$\Theta_B/d_2$	$\Theta_{max}/d_3$
0.3	8/1.1	40.1/1.1	90/1.7
0.5	10/0.9	65/1.1	115/1.5
0.7	21/1.0	78/1.1	130.5/1.4
1.0	25/1.1	105.5/1.2	145.5/1.3
1.2	38.7/1.0	185/1.1	203/1.0
1.7	63.2/1.2	208.1/1.1	250/1.0
2.0	79.4/1.0	250/1.2	325/1.3

**Table 7.** Flow curves of hydrogels flocculant M-919, modified by serine with MVO

Concentration W, %	$\Theta_f/d_1$	$\Theta_B/d_2$	$\Theta_{max}/d_3$
0.3	9/1.3	36/1.0	80/1.5
0.5	12.5/1.1	60/1.0	102/1.3
0.7	28/1.4	75/1.0	130/1.4
1.0	40/1.6	128/1.5	170/1.5
1.2	60/1.5	180/1.1	245/1.3
1.7	90/1.7	214/1.1	270/1.1
2.0	130/1.6	325/1.5	370/1.5



**Fig. 13.** Fibrils of polyacrylamide macromolecular[8]



**Fig. 14.** Globules of polyacrylamide macromolecular[8]

### CONCLUSIONS AND RECOMMENDATIONS

1. The possibility of changing the rheological properties of gels and modified amino acids of polyelectrolytes under deformation was noticed.

2. Strengthening of PAA gels is possible in the case of using amino modifiers without hydroxyl groups in its structure.

3. The phenomenon of macromolecular collapse is observed in the modification of polymers by hydroxyl-amino acid.

4. Physical modification with MVO, which is based

on the partial destruction of PAA macromolecules with simultaneous formation of radicals of different structures, supports the production of branched polymer structures. It is necessary to choose the optimal speed of deformation for the successful physical modification of PAA due to mechanic-chemical process.

5. This information from experiments can be used for modifying the features of flocculants solutions, which are based on PAA and for increasing their effectiveness in many industrial technological processes.

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