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SYNTHESIS AND PREPARATION OF POLYACRYLONITRILE AND VINYL SULFONIC ACID IN THE PRESENCE OF GOSSYPOL RESIN FOR DRILLING FLUIDS

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ABSTRACT

This paper discusses the synthesis and study of the physicochemical properties of modified polyacrylonitrile and vinyl sulfonic acid in the presence of gossypol resin. This polymer reagent will be used to increase the heat resistance and salt resistance of drilling fluids in harsh geological conditions. It was revealed that the modified polymer reagent has a suitably satisfactory stability at high (more than 200 °C) temperatures and salinity of reservoir deposits. The prospect of increasing the number of drilling fluids due to using a water-soluble reagent is aimed at solving the problem of the greatest use of drilling fluids. This research is aimed at deciding the problems facing compolyacrylonitrileies engaged in oil drilling in harsh geological conditions.

Keywords: Drilling Fluids, Gossypol Resin, Polymer Reagent, Polyacrylonitrile, Vinyl Sulfonic, Stability

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INTRODUCTION

Today, the development and creation of technology of high-quality multifunctional polymer chemical reagents and regulation of their properties working in difficult geological conditions is an impossible task without conducting in-depth studies of the physic-chemical interactions of organ mineral ingredients based on local and secondary raw materials, revealing patterns of influence of their structure, nature, type, content, and ratio of physic-chemical properties of reagents and drilling fluids based on them. In this regard, conducting research on the influence of the nature, type, content, and ratio of organ mineral ingredients on the physic-chemical properties of detergents that allow obtaining highly effective multifunctional polymer compositions based on local raw materials and industrial waste is an urgent task.[1-5]This paper discusses the synthesis and study of the physicochemical properties of modified polyacrylonitrile and vinyl sulfonic acid in the presence of gossypol resin. This polymer reagent will be used to increase the heat resistance and salt resistance of drilling fluids in harsh geological conditions. It was revealed that the modified polymer reagent has a suitably satisfactory stability at high (more than 200 °C) temperatures and salinity of reservoir deposits.

EXPERIMENTAL

In the experimental part, the following reagents were used as starting materials for the preparation of composite polymer materials for drilling fluids: polyacrylonitrile $(-CH_2-CH(CN)-)_n$,vinylsulfonic$CH_2=CHSO_3H$, gossypol resin. The gossypol resin of the Shymkent oil and fat processing plant was used in the work.[8-11]To implement the method of obtaining a composite reagent, we used a high-pressure desktop reactor of the TGYF-C brand in laboratory conditions. The production of fatty acids of gossypol resin was carried out by polymerization in an alkaline middling at a temperature of 80-90^{0}C for 2-2.5 hours. In this case, polymerization and saponification of naphthenic hydrocarbons and gossypol resin fall out together. The resulting semi-finished product consists of 60-70% natrium salts, primarily unsaturated fatty acids with a predominant fraction of C_{11}-C_{17}, i.e. (R-SOON).[7,8] IR spectra were using a ShimadzuYRPrestige-21 IR Fourier spectrometer to determine the spectral characteristics of the modified polymer reagent. A Jeol JSM-6490lV electron microscope was used to detect the structural element composition. The viscosity of water-soluble polymer solutions was measured in aUbellode

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capillary viscometer. The synthesized modified water-soluble polymer based on polyacrylonitrile in the existence of gossypol resin was tested on core samples using the UIK-S(2) core control facility. The review of the properties of polymer stabilizers was carried out using the texture/mechanical properties analyzer TAXT plus Stable Micro Systems. The analysis was carried out by puncture testing of the obtained dry films of the samples. An HDP/FSR nozzle was used, a small piece of the sample was placed and a puncture was carried out at a speed of 2 m/sec. The analysis of each sample was carried out with a repetition of three times, to derive the average values.

RESULTS AND DISCUSSION

The technology of obtaining modified polyacrylonitrile derivatives by hydrolysis of polyacrylonitrile with a solution of sodium hydroxide in the presence of formalin, sulfuric acid sodium has been developed. The resulting mixture was further modified with fatty acids of gossypol resin to obtain composite polymer stabilizers, providing an increase in the efficiency of the composition and a reduction in the cost of the process. The preparation of a composite reagent for drilling fluids, including a modified copolymer based on polyacrylonitrile (polyacrylonitrile, by hydrolysis in the presence of a mixture of sodium hydroxide) and vinyl sulfonic acid (vinyl sulfonic acid) was carried out with the following ratio of components, mass.%: 40-:60-:60:40.The resulting copolymer is saponified with an aqueous solution of sodium hydroxide or a mixture with sulfuric acid for 30-40 minutes, then gossypol resin is added – the cubic residue of the distillation process of fatty acids of cotton soapstock. The resulting composite reagent is thermally stable to polyvalent cations, reduces filtration, and improves the anti-wear properties of clay suspensions. The technology of drilling mud processing is simplified by eliminating the introduction of additional reagents.

The copolymerization process is carried out at 30-50°C, for 2-2.5 hours. Copolymerization is accompanied polyacrylonitrile by the let-out of a heat wave; the further course of the copolymerization reaction occurs due to the heat released and the temperature of the reaction mixture reaching up to 60 °C. The slowing down of the temperature rise and the decrease in the concentration of polyacrylonitrile to a minimum value and vinyl sulfate (0.2%) in the system indicates the end of the copolymerization process. The copolymerized product obtained by us is an aqueous suspension that is well separated from the dispersion medium by filtration or centrifugation methods.

A feature of the method for obtaining dispersion of water-soluble polymer surfactants (surfactants) is their colloidal-chemical behavior, depending on the nature of the particle surface, i.e., the degree of hydrophilicity and solubility of particles regulated by the ratio of active functional groups of different polarity.[12-13]The most extensive possibilities for regulating the hydrophobicity of the polymer phase are represented by the method of heterophase saponification in the presence of modifying agents – formalin, sulfuric acid sodium, fatty acids of gossypol resin, etc. allowing obtaining a polymer in a more hydrophilic visco-fluid state and a less hydrophilic granular form. The production process of sulfo-methylated derivatives of water-soluble polymer surfactants is carried out at a pH of 10-12 because, in an acidic and neutral medium, the functional groups of macromolecules are cyclized due to formalin and take a globular water-insoluble form. Thus, the proposed composite reagent-modified copolymer based on acrylonitrile and vinyl sulfonic acid (SANVSA) does not show high sensitivity to mineralization and water hardness due to the content of sulfur and imide groups of macromolecules, and the use of waste oil and fat industry waste in the process of copolymer modification bring to a decrease in the cost of the reagent.To determine the spectral characteristics of the modified polymer on the IR spectrum of the studied template of the modified polymer (Fig.-1), an intense band of NH monomers is observed at 3371 cm^{-1}. Crystallization water: the band is at 3371 cm^{-1}, but not as strong and somewhat narrower, in addition, there are weak bands at 1639 cm^{-1} (deformation fluctuations H – O – H).Basically, the NH "II amide band" shifts to the high-frequency region during association. In the solid-state $–CONH_2$ has two strong bands at 1650-1640 cm^{-1}, but the "I band" is stronger. Four bands of free and bound groups may appear in concentrated solutions. The group $–CH_2–$, connected by the electron acceptor ion N+, is also

shifted lower. If, when the amine is converted into a salt, new bands appear in this region at 1400- 480 cm^{-1}, then there is a group $–CH_2$ –N-.Asymmetric valent oscillations manifest themselves like other carbonyl groups = C = O, so the band appears at 1250 cm^{-1}.The intensity of the C–C vinyl esters is increasing. At 1087-1026 cm^{-1}, symmetrical valence oscillations are weaker than bands of 1250 cm^{-1}.In solid samples, the band at 675 cm^{-1} is often split into several bands. Pairing and the nature of the cycle do not affect the bands.[15]

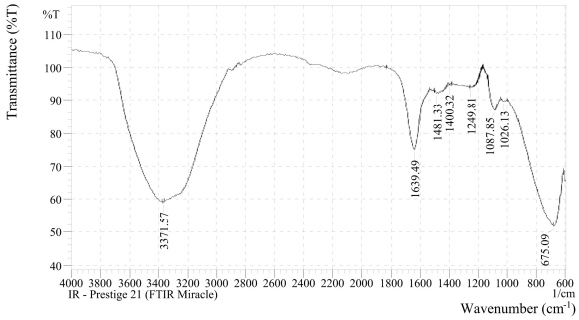


Fig.-1: IR Absorption Spectrum of a Sample of a Modified Copolymer Derivative of Acrylonitrile and Vinylsulfonic Acid

The appearance of carboxyl and amide groups has been established, and an enhancement in their composition of densely arranged carboxyl groups along the macromolecule chain should also be noted as a result of studies of the IR spectra of synthesized polymers. Therefore, the analysis of Inspectors demonstrated that the resulting modified reagent based on acrylonitrile and vinyl sulfoxylate contains functional groups:

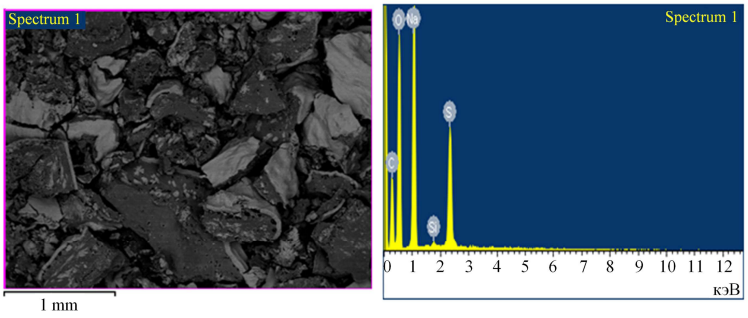


Fig.-2: Elemental and Mineralogical Composition of the Polymer of Acrylonitrile and Vinyl Sulfonic Acid

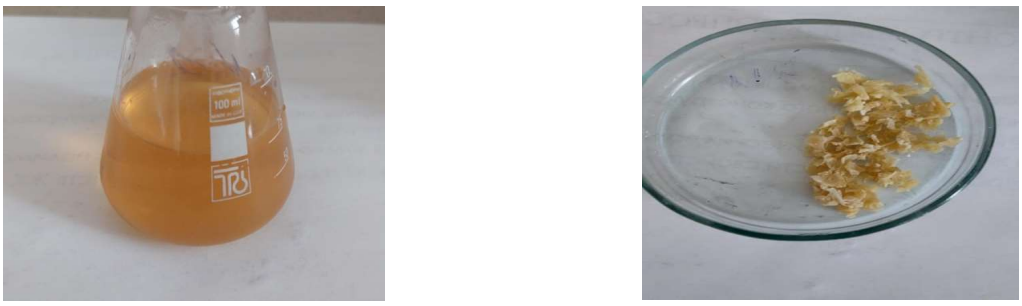
Wherefore, the analysis of IR spectra showed that the resulting modified polymer reagent based on polyacrylonitrile and vinyl sulfonic acid in the presence of fatty acids of gossypol resin contains functional groups: $-COO^-$; SN_3; NH^{2-}; $-SO^{3-}$. The appearance of the following elements is shown: C – 31.82%, O – 43.42%, Na – 20.68%, Si – 0.23%, S – 6.51%. As a result of the elemental and mineralogical composition of the resulting polymer reagent (Fig.-2). The optionof the ratio of monomers and modification conditions providea high conversion of monomers, which improves the give up of the latter product. The synthesized polymer has an amphiphilic structure throughout the structure, macromolecules that contain a hydrophobic group and a hydrophilic part are able to adsorb and reduce free energy at the phase interface.[5,6] The foundation of the balance speed in the adsorption seam depends on the flexiblenessof the chain of macromolecules, their structure, and functional structure and is caused by the diffusion of macromolecules to the interface of phases, or by the processes of dehydration and repose that spring up in the adsorbed seam itself, in particular, the opportunity of the united action of these factors is not excluded.[7] The decreased viscosity of polymer solutions was resolved depending on the concentration, temperature, pH of the medial, and electrical conductivity. To establish the optimal reaction time and temperature, the viscosity of the sampled polymer reagent was measured. The selection of the ratio of monomers and modification conditions ensures a high conversion of monomers, which increases the yield of the final product. The synthesized polymer of acrylonitrile and vinyl sulfonicin the

appearance of fatty acids of gossypol resin has a diphilic structure throughout the structure, whose macromoleculeshave a hydrophobic group and a hydrophilic part.[9-11]Tables-1 and 2 show the results of laboratory tests for the stabilization of dilute suspensions from hydroslude-montmorillonite clay of the Darbazinsky deposit in a wide range of polyelectrolyte concentrations.[12-15] Stabilization of clay suspensions occurs as for other composite polymer stabilizers at a certain concentration of composite polymer stabilizers in the system (Table-1).

Table-1: Change in Filtration Properties of a 15% Suspension of Hydroslude-Montmorillonite Clay of the Darbaza Deposit Depending on the Concentration of the Composite Polymer Stabilizer SANVSA

Concentration of PE, %	-		0.05	0.10	0.25	0.50	1.00
Conditional viscosity, (T^{200}_{100}), s	4.5		8.0	11.5	14.0	16.0	27.8
Specific gravity, $kg/m^3 \cdot 10^{-3}$	1.05		1.27	1.27	1.26	1.26	1.24
Daily sucks, %	4.0		13.5	11.0	5.0	1.0	0.0
Water output, $m^3 \cdot 10^{-3}$	33		40	40	9	5	4
Static shear stress, Pa	1 min	37.4	63.0	42.6	40.1	38.6	60.8
	10 min	39.2	61.1	45.3	41.3	40.7	61.2
Crust thickness, mm	4.0		5.5	5.0	5.5	1.0	0.5
pH	6.8		7.7	8.3	8.8	9.0	10.0

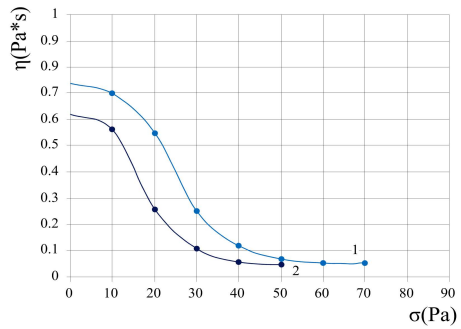
This is due to the difference in the crystal chemical structure, degree of dispersion, and hydrophilicity of clay mineral particles. Another growth in the concentration of composite polymer stabilizers in the clay-water system leads to a decrease in these indicators to a minimum, but at the same time the conditional viscosity increases due to an excess of viscous polyelectrolyte, which has a plasticizing effect that reduces the shear strength of clay suspensions (SNC, 10-4kg/ 10-2m^3), i.e., there is some dilution systems and loss of thixotropic properties of clay suspensions. The increased value of the conditional viscosity of clay suspensions with a polyelectrolyte content of 0.75-1% is probably due to their presence in excess and the formation of a combined structure of clay particles and supramolecular polyelectrolyte formations. An excess polyelectrolyte also has some structure-forming effect, which leads to an increase in the value of SNC (static shear stress). 0.5% of composite polymer stabilizers – synthesized acrylonitrile and vinyl sulfonic acid (SANVSA) - can be considered the optimal additive that causes stabilization of suspensions of hydroslude-montmorillonite clay of the Darbaza deposit in the system.[15-17] With an increase in the concentration of polyelectrolytes in the system, an adsorption-solvate layer with lyophilizing properties is formed on the surface of clay particles, blocking the surface of clay particles from compact coagulation and contributing to spatial coagulation structure formation. Smaller additives of SANVSA (above 0.25%) become stable, and the technological parameters meet the criteria of stable thixotropic washing liquids with a low solid phase content used when drilling small-diameter wells. At the same time, polyelectrolyte dramatically increases the viscosity of the system, apparently due to the formation of a combined structure of clay particles and supramolecular formations of polyelectrolyte.[18-19] According to the change in technological characteristics for the studied suspensions of hydrosulfide-montmorillonite clay of the Darbaza deposit, optimal polyelectrolyte additives equal to 0.5% SANVSA were determined. Photos of the obtained copolymer based on acrylonitrile and vinyl sulfonic acid and the dried polymer at a temperature of 80-90°C are shown in Fig.-3.



(a) (b)

Fig.-3: (a) Copolymer Based on Acrylonitrile and Vinyl Sulfonic Acid; (b) Dried Polymer

Thus, the results of complex studies of the technological deformation properties of stabilized clay suspensions demonstrate the possibility of directional regulation of the processes of coagulation structure formation and increasing the aggregate stability of clay mineral dispersions in the presence of composite polymer stabilizers SANVSA. To establish the consanguinity between the functional composition and the presence of carboxylate groups in the arrangement and the rheological properties of hydrous solutions of the synthesized water-soluble polymer, the dependence of the effective viscosity on the shear stress was studied (Fig.-4). To appraise the intensity of intermolecular interaction of macromolecules in the fluid of synthesized modified polymers, the temperature coefficient of viscosity of undisturbed structures is exploited. The value is determined by both the intensity of intermolecular interaction and the mobility of macromolecular chains in an aqueous solution. The smaller reserve of activation energy of the viscous flow of solutions of non-hydrolyzed forms synthesized in EPR can be explained by the greater mobility of polymer chains and the relatively low intensity of intermolecular interaction. An increase in the concentration of the emulsifier-stabilizer in the composition of emulsions causes an increase in the viscosity parameters of emulsion solutions. However, an increase in the effective viscosity of emulsion solvents occurs only at small concentrations of the emulsifier-stabilizer until the viscosity limits for this system are reached. With a subsequent increase in the emulsifier content, stabilization or decrease in the viscosity parameters of emulsion solvents is observed. Therefore, it should be borne in mind that the greater the surface activity of the emulsifier, the lower the contents, and the stabilization or reduction of structural and mechanical properties of reverse emulsions stabilized by these emulsifiers occurs.



designations: 1- hydrolyzed polyacrylonitrile in the presence of formalin and gossypol resin.
2-hydrolyzed polyacrylonitrile in the presence of formalin followed by sulfonation and gossypol resin
Fig.-4: Addiction of the Effective Viscosity η(Pa·s) of Hydrous Solutions (4%) of the Studied Water-Soluble Polymer on the Shear Stress τ(Pa)

This phenomenon can be explained by a decrease in interfacial tension with an increase in the content of emulsifiers and, as a consequence, an increase in the total interface of the phases in the emulsion. At the minimum interfacial tension for this interface, the maximum saturation of the adsorption layer with emulsifier molecules occurs and an equal hydrodynamic interaction between the globules of the aqueous phase is established. The higher the volumetric water content in emulsions, the greater the influence of the emulsifier content on their effective viscosity. Obviously, this is caused by both an increase in the total interface in the system and a more significant immobilization of thinning layers of the emulsion dispersion medium and an excessive number of emulsifier molecules in it. Rheological studies have made it possible to establish the structuring of hydrous solutions of synthesized modified water-soluble polymers. The results of physic- and colloidal-chemical properties show that the resulting high-molecular polymer surfactant belongs to amphoteric polyelectrolytes. The influence of the concentration of composite water-soluble polymers on the kinematic viscosity of Darbaza clay has been studied. The concentration of composite water-soluble polymers varied from 0.05 to 0.5 wt.%. It was found that composite water-soluble polymers based on polyacrylonitrile obtained by hydrolysis (sodium hydroxide solution), sulfonation (in the presence of formalin with sulfuric acid sodium) and subsequent modification with fatty acids of gossypol resin with a ratio (1:0.3-0.5) at a concentration in oil of 0.05 wt.% provides a reduction in the kinematic viscosity of oil by 5-9%. The determination of the kinematic viscosity of oil was carried out in accordance with GOST 33-2000.

Table-2: Dependence of the Change in the Kinematic Viscosity of Oil on the Content of Polymer Reagents at a Temperature of 25ºC

No.	Composition of water-soluble polymers	Kinematic viscosity of oil, mm^2/c by 25ºC					
		Concentration of water-soluble polymers, % mass.					
		-	0.05	0.1	0.20	0.30	0.5
1	Oil	23.211	-	-	-	-	-
2	Polyacrylonitrile+NaOH+C_{11}-C_{17}–COOH	-	14.672	14.873	14.788	16.128	17.162
3	Polyacrylonitrile+NaOH+CH_2O+ $Na_2S_2O_3$+C_{11}-C_{17}–COOH	-					

This way, with a minimum concentration of composite water-soluble polymer obtained by hydrolysis of polyacrylonitrile with a solution of sodium hydroxide or sulfonation with sulfuric acid sodium in the presence of formalin, followed by modification with fatty acids of gossypol resin, provides maximum reduction in oil viscosity, which characterizes the activity of a water-soluble polymer. Thus, the resolution of complex research of the technological deformation estates of stabilized clay suspensions demonstrates the possibility of directional regulation of the processes of coagulation structure development and increasing the aggregate stability of clay mineral dispersions in the presence of composite polymer stabilizers of acrylonitrile and vinylsulfonic acid.

CONCLUSION

Therefore, the conditions for obtaining a polymer reagent based on modified polyacrylonitrileand vinyl sulfonic acid in the presence of gossypol resin for use with drilling fluid were resolute. Optimal conditions for the process of receiving a water-soluble polymer reagent based on polyacrylonitrile and vinyl sulfonic acid in the presence of gossypol resin have been determined. The effect of temperature, concentration, pH of the medium, and the degree of water mineralization on the process of receiving a water-soluble polymer in composite dispersed systems has been studied. It was found that the synthesized water-soluble polymer reagent based on modified polyacrylonitrileand vinyl sulfonic acid in the presence of gossypol resin has a suitably respectable stability at high (more than 200 °C) temperatures and mineral content of formation waters. The resulting water-soluble polymer reagent during the useful capacity of oil from the Barboza field gives the best results. Optimal conditions for the process of receiving awater-soluble polymer reagent based on polyacrylonitrile in the presence of gossypol resin and vinyl sulfonic acid have been determined. Based on the results of rheological measurements, the compositions of drilling fluids capable of carrying rock particles to the surface during well drilling were selected. Formulations of new drilling fluids have been developed in a wide range of density variations, consisting of polyacrylonitrile modified with gossypol resin and vinyl sulfonic acid, which are recommended to prevent suction and opening of productive layers of oil fields with low reservoir pressure, as well as for opening productive layers of horizontal and obliquely directed wells, represented by carbonate and terrigenous reservoirs. Drilling mud formulations are protected by patents of the Republic of Kazakhstan.[20-21]

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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