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N. Mukhametgazy[1], I.Sh. Gussenov[1, 2], A.V. Shakhvorostov[2], S.E. Kudaibergenov*[1, 2]

[1]*Satbayev University, Almaty, Kazakhstan;*
[2]*Institute of Polymer Materials and Technology, Almaty, Kazakhstan*
(Corresponding author's e-mail: skudai@mail.ru)

Salt tolerant acrylamide-based quenched polyampholytes for polymer flooding

In our previous papers [1, 2] we considered the behavior of linear and crosslinked polyampholytes based on fully charged anionic monomer — 2-acrylamido-2-methyl-1-propanesulfonic acid sodium salt (AMPS) and cationic monomer — (3-acrylamidopropyl)trimethylammonium chloride (APTAC) in aqueous-salt solutions, swelling and mechanical properties. In the present paper we report the applicability of salt tolerant amphoteric terpolymers composed of AMPS, APTAC and acrylamide (AAm) in enhanced oil recovery (EOR). The amphoteric terpolymers of different compositions, particularly [AAm]:[AMPS]:[APTAC] = 50:25:25; 60:20:20; 70:15:15; 80:10:10 and 90:5:5 mol.% were prepared by free-radical polymerization, identified and their viscosifying ability with respect to reservoir saline water (salinity is 163 $g \cdot L^{-1}$) at 60 °C was tested. It was found that due to polyampholytic nature, the AAm-AMPS-APTAC terpolymers exhibited improved viscosifying behavior at high salinity water. As a result, the appropriate salt tolerant sample [AAm]:[AMPS]:[APTAC] = 80:10:10 mol.% was selected for polymer flooding experiments. Polymer flooding experiments on high permeable sand pack model demonstrated that only 0.5 % oil was recovered by amphoteric terpolymer. While injection of polyampholyte solution into preliminarily water flooded core sample resulted in the increase of oil recovery up to 4.8–5 %. These results show that under certain conditions the amphoteric terpolymers have a decent oil displacement ability.

Keywords: polyampholyte terpolymers, viscosifying ability, high permeability, homogeneity, porous media, oil viscosity, incremental oil recovery (IOR), reservoir heterogeneity conditions.

Introduction

Oil and gas sector remains one of the main components of the economy of Kazakhstan, and its development will determine the prospects for the state economy. The problem of enhanced oil recovery (EOR) is especially important for Kazakhstan to enter into the world's top five oil exporters.

Nowadays many water-soluble polymers have been intensively developed and explored as viscosity-enhancing and flocculating agents, food additives, etc. [3–5]. Polyacrylamide and its derivatives have been successfully employed in wastewater treatment, papermaking and oil industry due to their thickening ability, flocculation and rheological behaviors [6–8].

The most widely used synthetic EOR polymer is polyacrylamide (PAA) and its derivatives [9–12]. This polymer is usually used in hydrolyzed polyacrylamide (HPAM) form to achieve higher viscosity within a certain range of brine salinities [13]. Unfortunately, HPAM may undergo a severe hydrolysis and precipitate at high temperature and brine salinity [14]. In this context, designing suitable polymers for EOR from high-temperature and high-salinity reservoirs is a challenging task. In fact, relatively high oil viscosity and brine salinity are common phenomena for Kazakhstani oil reservoirs. For example, the viscosity of Karazhanbas field oil may be higher than 350 cp, while brine salinity of Zhetibay and Moldabek fields may exceed 150 $g \cdot L^{-1}$. In polymer flooding technology, stable oil displacement is realized through the reduction of

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*Corresponding author.

oil/water viscosity ratio by using HPAM and other polymers [15]. Unfortunately, the high salinity of make-up water requires higher polymer concentrations to provide a certain viscosity value [16], which may increase the production cost of recovered oil. Moreover, high water salinity may cause polymer precipitation in the porous media of a rock, which results in permeability damage [14] and polymer waste. In order to overcome these problems, we suggest to use as viscosifying agent (or water thickeners) the tailor made amphoteric terpolymers AAm-AMPS-APTAC that possess the thermal stability and are able to swell and increase the viscosity in saline water due to specific, so called *"antipolyelectrolyte"* effect [17, 18]. Previous studies [19–21] indicated that many polymers containing AAm and ionic monomers can be used for EOR process [22–31].

In this work a series of ternary polyampholytes with different molar concentrations of AAm, AMPS, and APTAC were synthesized and tested as oil recovery agent by using sand pack model and artificial high porosity core. The obtained results are perspective for development of novel salt- and temperature tolerant amphoteric terpolymers for EOR.

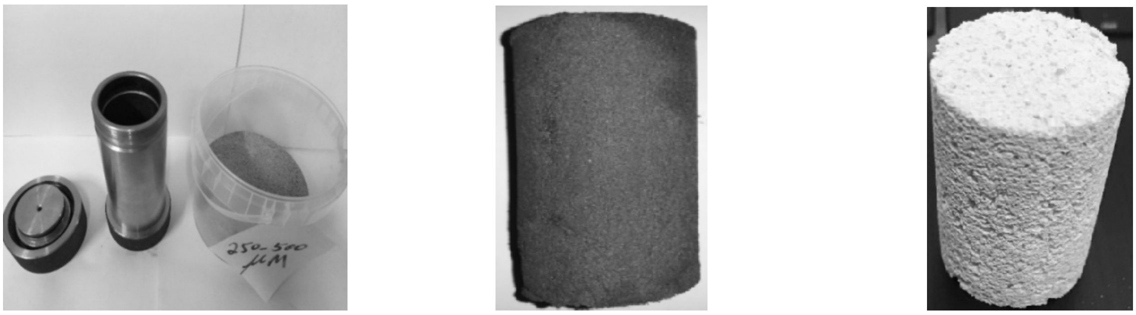
Experimental

Materials

Acrylamide (AAm, purity ≥98.0 %), 2-acrylamido-2-methylpropanesulfonic acid sodium salt (AMPS, 98 wt.% in water) and (3-acrylamidopropyl) trimethylammonium chloride (APTAC, 75 wt.% in water), and ammonium persulfate (APS, 99 % purity) were purchased from Sigma-Aldrich Chemical Co. and used without further purification.

To provide the sand pack flooding experiments, 250–500 μm sand grains were packed into 8.3 cm length and 4.3 cm diameter steel cylinder (Fig. 1*a*, *b*). The porosity and permeability of the sand pack were equal to 44 % and 16 Darcy, respectively. The model was saturated with 100 $g \cdot L^{-1}$ East Moldabek (well#2092 M-II) brine and East Moldabek oil (well# 2027 M-III-U-I). The viscosity and density of oil sample were equal to 138 cp and 0.8916 $g \cdot cm^{-3}$ at 25 °C. Brine water solution was injected into the sand pack model at the rate of injection 0.1 $cm^3 \cdot min^{-1}$.

The core flooding experiments were carried out with core sample of 4.4 cm length and 2.9 cm diameter with permeability 5 Darcy (Fig. 1*c*). The artificial high porosity core sample with pore volume 24.12 cm^3 (porosity is 83 %) and Karazhanbas oil (well# 1913) were used. Viscosity and density of selected oils were equal to 420 cp and 0.93 $g \cdot cm^{-3}$ at 30 °C and 64 cp and 0.907 $g \cdot cm^{-3}$ at 60 °C. Brine solution with concentration of 163 $g \cdot L^{-1}$ was used for core flooding test. 0.5 % polymer solution was injected into the core at the rate of 1 $cm^3 \cdot min^{-1}$.



a *b* *c*

Figure 1. Sand pack (*a*, *b*) and artificial high porous core (*c*)

Methods

^1H NMR spectra of AMPS-APTAC-AAm terpolymers in D_2O were registered on impulse Fourier NMR spectrometer Bruker 400 MHz (Bruker, Germany).

The sand pack and core flooding experiments were conducted with the help of special core flooding set up "УИК-С(2)" (Russia).

The viscosity of polymer solutions was determined by glass capillary viscometer with diameter 1.47 mm. Oil density and viscosity were determined by Stabinger viscometer.

The following procedures for sand pack and core flooding tests were used: 1 — vaccum saturation of the porous media with brine; 2 — brine displacement by oil (drainage); 3 — oil displacement by brine (imbibition); 4 — polymer injection.

The sand pack-flooding test was conducted at room temperature and injection rate 0.1 $cm^3 \cdot min^{-1}$while the core flooding was performed at 60 °C and injection rate 1 $cm^3 \cdot min^{-1}$.

Results and Discussion

Synthesis and characterization of AAm-AMPS-APTAC copolymers

Acrylamide-based quenched polyampholytes AAm-AMPS-APTAC with molecular weights $(5–6) \cdot 10^5$ Daltons were synthesized *via* conventional free radical (co)polymerization in the presence of APS at 60 °C during 4 h at various molar ratio of initial monomers [AAm]:[AMPS]:[APTAC] = 50:25:25, 60:20:20, 70:15:15, 80:10:10 and 90:5:5 mol.% (Fig. 2).

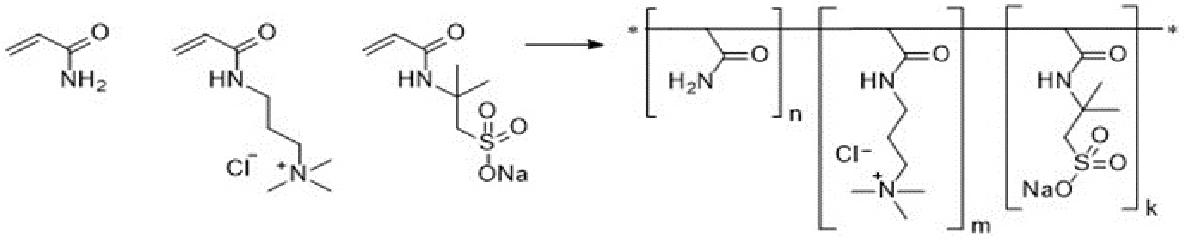
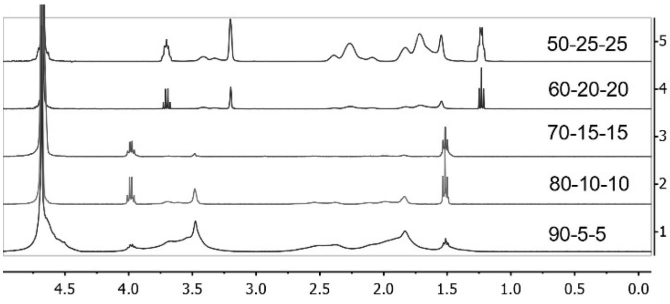


Figure 2. Preparation protocol of ternary polymers AAm-AMPS-APTAC

The results of ^{1}H NMR spectra of AAm-AMPS-APTAC registered in D_2O along with initial and final molar compositions of monomers are shown in Fig.3. The molar composition of AAm-AMPS-APTAC terpolymers was estimated from the integral peaks of methyl groups that belong to AMPS and APTAC monomer units. It is seen that experimentally found molar compositions of terpolymers deviate from the theoretically prescribed by 1–2 mol.% that are within the experimental error. Earlier the isoelectric points (IEP) of AMPS-APTAC copolymers were found around of pH 6.1 ± 0.1 [1]. Taking into account that acrylamide is nonionic (or neutral) monomer presenting in the composition of terpolymers, one can expect that the values of the IEP will be close to AMPS-APTAC copolymers.



Molar composition of [AAm]:[AMPS]:[APTAC], mol.%	
Theoretically prescribed	Experimentally found from ^{1}H NMR spectra
90:5:5	92:3:5
80:10:10	81:9:10
70:15:15	70:14:16
60:20:20	60:20:20
50:25:25	50:24:26

Figure 3. ^{1}H NMR spectra and molar composition of AAm-AMPS-APTAC terpolymers.

Sand pack flooding experiments

Figure 4 demonstrates mass of produced oil versus injected water volume and photos of effluents. The volume of each effluent sample is 10 cm^3, whereas the pore volume (PV) of the model is 55 cm^3. As seen from Figure 4 water breakthrough occurred before the injection of the first 10 cm^3 water (≈ 0.18 PV) and oil production started to decline after 0.18 PV of injected water into the model. Water flooding oil recovery factor (ORF) demonstrates that the water flooding resulted in displacement of approximately 25.7 % of oil initially in place (OIIP) (Fig. 5).

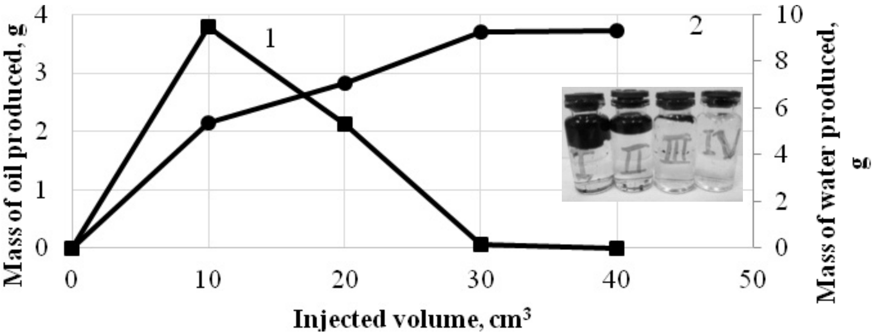


Figure 4. Dependence of mass of produced oil (curve 1) and water (curve 2) *vs* injected water volume. Insert is photos of each effluent containing 10 cm^3 of fluid

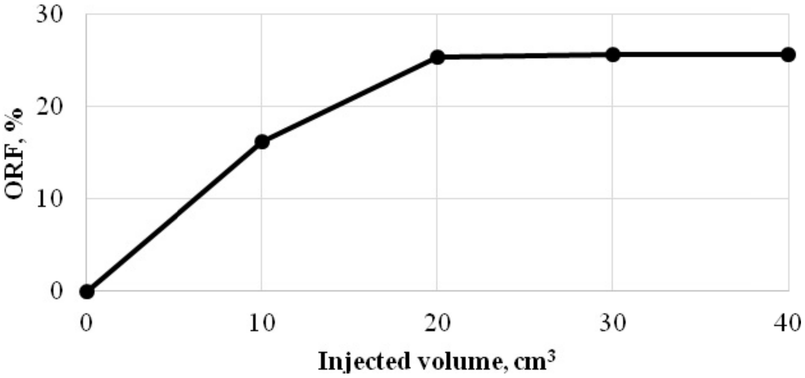
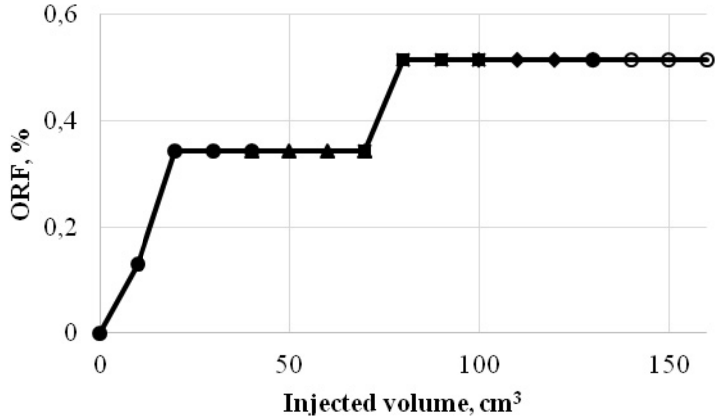


Figure 5. Dependence of ORF *vs* injected volume of water

Injection of 0.5 % brine solution of AAM-AMPS-APTAC terpolymers into high permeable sand pack model stepwise increases oil production (Fig.6). For instance, injection of AAM-AMPS-APTAC (50–25–25 and 60–20–20 mol.%) increases the oil recovery up to 0.35 %, while ORF is 0.51 % when terpolymers 70–15–15, 80–10–10 and 90–5–5 mol.% are injected. Thus, polymer flooding experiments on high permeable sand pack model demonstrated that only 0.51 % oil is recovered by amphoteric terpolymers in comparison with water flooding.



● — 50–25–25; ▲ — 60–20–20; ■ — 70–15–15; ♦ — 80–10–10; ○ — 90–5–5 mol. %

Figure 6. Dependence of ORF on injected volume of 0.5 % brine solution of AAm-AMPS-APTAC terpolymers

Core flooding experiments

In the next series of experiments 0.5 % solution of AAm-AMPS-APTAC (80–10–10 mol.%) in 163 g·L^{-1} brine was injected into preliminarily water-flooded core sample. The reason is that among the tested AAm-AMPS-APTAC terpolymers the highest viscosity in brine exhibited amphoteric terpolymer with composition of 80–10–10 mol.%. (Table 1).

Table 1

Dependence of the dynamic viscosity of AAm-AMPS-APTAC on composition of terpolymers

Molar composition of AAm-AMPS-APTAC, mol.%	90:5:5	80:10:10	70:15:15	60:20:20	50:25:25
Dynamic viscosity, mPa·s^{-1}	5.5	10.4	6.7	8.0	6.6

The injection of 0.5 % solution of AAm-AMPS-APTAC (80–10–10 mol.%) in brine into the pre-water flooded core sample resulted in a notable oil recovery increase. Figure 7 clearly shows the increasing of the mass of produced oil in the course of polymer flooding.

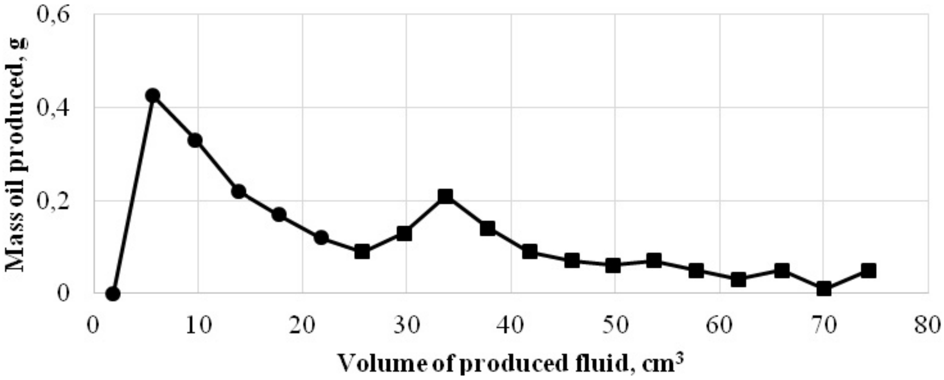


Figure 7. Mass of produced oil *vs* the produced volume of fluid in the course of water flood (●) and polymer flood (■). Injected polymer solution is 0.5 % AAm-AMPS-APTAC (80–10–10 mol.%) in 163g·L^{-1} brine. Karazhanbas oil 64 cp at 60 °C. The injection rate is 1 cm^3·min^{-1}

The increase of the pressure drop at the beginning of polymer flooding also indicates that the *in-situ* polymer solution viscosity is higher than that of water, and the higher viscosity results in more favorable oil mobility ratio between the displacing and the displaced fluids (Fig. 8).

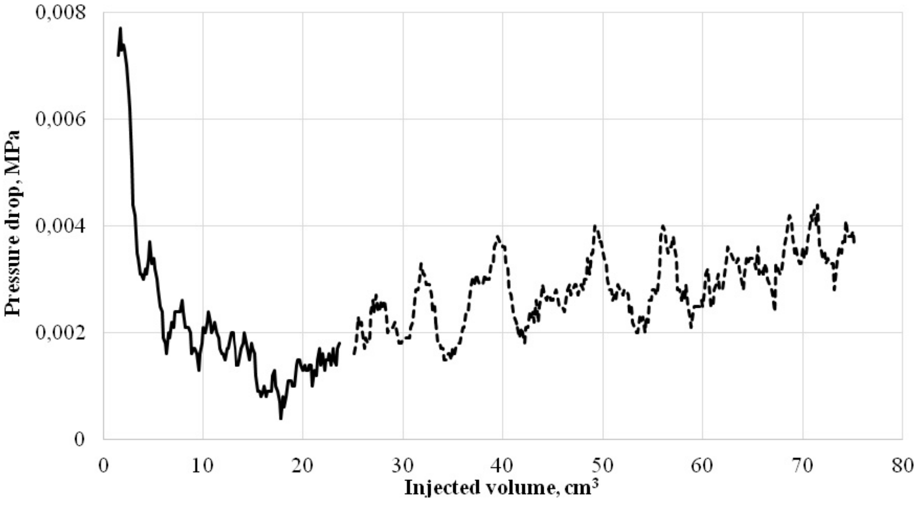


Figure 8. Change of pressure drop *vs* injected volume in the course of water flood (▬) and polymer flood (▪▪▪). Injected polymer solution is 0.5 % AAm-AMPS-APTAC (80–10–10 mol.%) in 163g·L^{-1} brine. Karazhanbas oil 64 cp at 60 °C. The injection rate is 1 cm^3·min^{-1}

As seen from Figure 9 the ORF increased by 4.8–5 % due to injection of 0.5 % polymer solution. This is explained by increasing of the viscosity of brine solution (or viscosifying) due to disruption of intra- and interionic contacts between oppositely charged AMPS and APTAC moieties demonstrating *antipolyelectrolyte* effect. In pure water, the electrostatic attraction between oppositely charge macroions leads to formation of compact structure and low viscosity. In saline water the anions and cations of salts screen the electrostatic attraction between positively and negatively charged macroions and the macromolecular chain expands. This phenomenon leads to increasing of the solution viscosity and consequently to viscosifying effect. In its turn the viscosification of brine solution leads to decreasing of the mobility ratio (M_r) which is defined as ratio of displacing phase mobility (water) to displaced phase mobility (oil). It is commonly accepted that in polymer flooding, favorable M_r is achieved by increasing the viscosity of water.

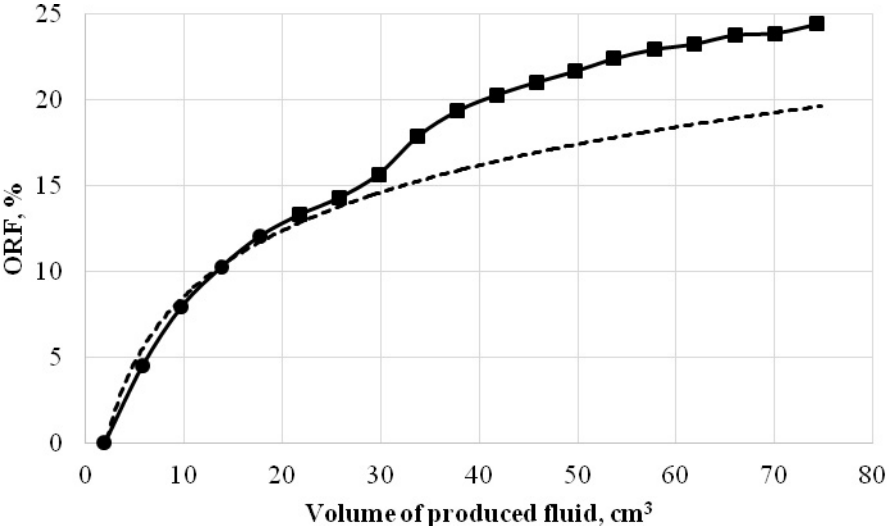


Figure 9. Dependence of ORF *vs* produced volume in the course of water flood (●) and polymer flood (■). Water flooding trend is shown by the dashed line (▪▪▪). Injected polymer solution is 0.5 % AAm-AMPS-APTAC (80–10–10 mol.%) in 163g·L^{-1} brine. 0.5 % solution of AAm-AMPS-APTAC (80–10–10 mol.%) in 163 g·L^{-1} brine. Karazhanbas oil 64 cp at 60 °C. The injection rate is 1 cm^3·min^{-1}

It is interesting to evaluate the state of core samples after polymer flooding experiment. For this purpose, the sand pack model was cut into two pieces and the interior walls along with injection and outlet faces were analyzed (Fig. 10). Interior walls of core sample contain some amount of adsorbed polymer solution while the injection face of the core is less oil saturated than the outlet face. This fact confirms the oil displacement by amphoteric terpolymer in the course of injection of polymer solution.

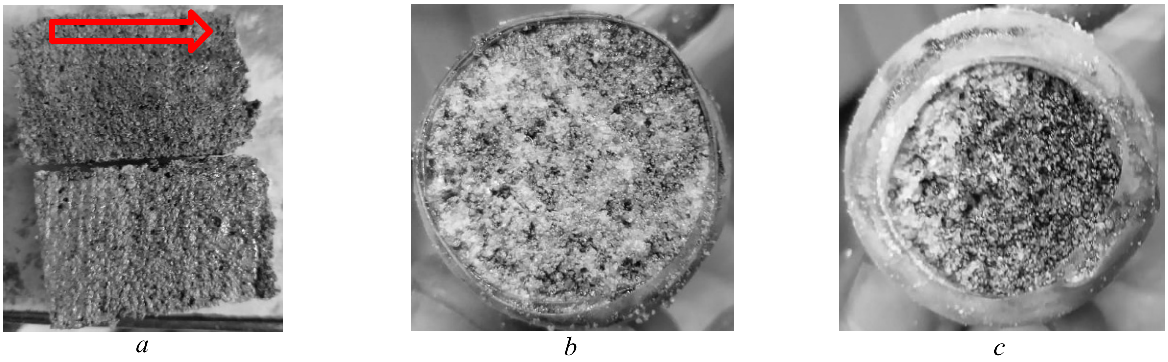

a *b* *c*

Figure 10. The view of core sample after the testing.
Core cut along the axis (*a*), injection (*b*) and outlet (*c*) faces of core sample

Conclusions

Salt tolerant acrylamide-based quenched polyampholytes were synthesized from acrylamide (AAm), anionic (AMPS) and cationic (APTAC) monomer units by free radical polymerization. The compositions of amphoteric terpolymers were established by 1H NMR spectroscopy. Amphoteric terpolymers showed a good solubility in oilfield water with the salinity 163 g·L^{-1}. Among the terpolymers the highest viscosity in brine exhibited amphoteric terpolymer AAm-AMPS-APTAC with composition 80–10–10 mol.%. Sand pack and core flooding experiments were carried out with 0.5 % of AAm-AMPS-APTAC (80–10–10 mol.%) solution in 163g·L^{-1} brine for Karazhanbas oil at 60 °C. In sand pack flooding experiments, the injection of different AAm-AMPS-APTAC recipes resulted in only 0.5 % ORF increase. However, in the core flooding tests the injection of AAm-AMPS-APTAC (80–10–10 mol.%) solution resulted in 4.8–5 % ORF increase. The increase of pressure drop during polymer flood indicated improvement the mobility ratio in comparison with water flooding.

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Н. Мұхаметғазы, И.Ш. Гусенов, А.В. Шахворостов, С.Е. Құдайбергенов

Тұзғатөзімді акриламид негізіндегі жоғары зарядталған полиамфолиттерді жер қыртысына айдау

Алдыңғы мақалаларда авторлар толығымен зарядталған анионды мономер — 2-акриламидо-2-метил-1-пропансульфон қышқылы натрий тұзы (АМПС) және катионды мономер — (3-акриламидопропил)триметиламмоний хлориді (АПТАХ) негізінде түзілген сызықты және айқаса байланысқан полиамфолиттердің сулы-тұзды ерітінділердегі ісінуі және механикалық қасиеттерін қарастырды. Осы мақалада мұнай өндіруді жақсартылған қалпына келтіру кезінде АМПС, АРТАХ және акриламидтен (ААм) тұратын, тұзғатөзімді амфотерлі терполимерлердің қолданылу мүмкіндігі туралы хабарланған. Әр түрлі құрамдағы амфотерлі терполимерлер, атап айтқанда [ААм]:[АМПС]:[АРТАХ] = 50:25:25; 60:20:20; 70:15:15; 80:10:10 және 90:5:5 мол.% бос радикалды полимерлеу әдісімен дайындалды, анықталды және олардың 60 °C температурада тұзды суға (тұздылығы 163 г·л$^{-1}$) қатысты тұтқырлық қабілеті тексерілді. Амфотерлі терполимерлер полиамфолиттік сипатта болғандықтан, ААм-АМПС-АРТАХ терполимерлері тұздылығы жоғары суларда жоғары тұтқырлық қасиеттерін көрсетті. Соның нәтижесінде, тұзғатөзімді [ААм]: [АМПС]:[АРТАХ] = 80:10:10 мол.% үлгісі полимерді айдау тәжірибесі үшін таңдап алынды. Полимер ерітіндісін айдау тәжірибесі жоғары өткізгіштігі бар құмды модельде амфотерлі терполимердің көмегімен тек 0,5 % мұнайдың алынған шығымын көрсетті. Алдын-ала сумен қаныққан керн үлгісі арқылы полиамфолит ерітіндісін айдау барысында мұнай өндірудің жоғарылауы 4,8–5,0 % дейін артты. Бұл нәтижелер амфотерлі терполимерлердің белгілі бір жағдайларда мұнайды ығыстыру қабілетіне ие екендігін көрсетеді.

Кілт сөздер: полиамфолит терполимерлері, тұтқырлық қабілеті, жоғары өткізгіштігі, біртектілігі, кеуекті орта, мұнай тұтқырлығы, мұнай өндірудің жоғарылауы, қабаттың біртектілігі.

Н. Мухаметгазы, И.Ш. Гусенов, А.В. Шахворостов, С.Е. Кудайбергенов

Солеустойчивые сильнозаряженные полиамфолиты на основе акриламида для полимерного заводнения

В предыдущих статьях авторами рассмотрены поведение линейных и сшитых полиамфолитов на основе сильнозаряженного анионного мономера — натриевой соли 2-акриламидо-2-метил-1-пропансульфоновой кислоты (АМПС) и катионного мономера — (3-акриламидопропил)триметиламмоний хлорида (АПТАХ) в водно-солевых растворах, набухание и механические свойства. В настоящей статье сообщается о применимости солестойких амфотерных терполимеров, состоящих из АМПС, АПТАХ и акриламида (ААм) для полимерного заводнения. Амфотерные терполимеры различного состава, а именно [ААм]:[АМПС]:[АПТАХ] = 50:25:25; 60:20:20; 70:15:15; 80:10:10 и 90:5:5 мол.%, были приготовлены свободнорадикальной полимеризацией, идентифицированы и их загущающая способность протестирована применительно к пластовой (соленой) воде нефтяного резервуара (соленость 163 г·л$^{-1}$) при 60 °С. Найдено, что благодаря полиамфолитной природе терполимеры ААм-АМПС-АПТАХ показали улучшенный загущающий эффект при высокой солености воды. В результате этого для экспериментов по полимерному заводнению выбран солестойкий образец [ААм]:[АМПС]:[АПТАХ]: = 80:10:10 мол.%. Полимерное заводнение на высокопроницаемой песчаной модели показало, что только 0,5 % нефти извлекается амфотерным терполимером. Тогда как закачка раствора полиамфолита в образец керна, предварительно насыщенного водой, приводит к увеличению извлечения нефти до 4,8–5,0 %. Эти результаты показывают, что при определенных условиях амфотерные терполимеры способны в достаточной степени извлекать нефть.

Ключевые слова: терполимеры полиамфолитов, загущающая способность, высокая проницаемость, однородность, пористая среда, вязкость нефти, увеличение нефтеотдачи, условия неоднородности пласта.

Information about authors

Mukhametgazy, Nurbatyr — PhD student, Satbayev University, Almaty, Kazakhstan; Satbayev street 22a, 050013. e-mail: nurbatyr.kaz@gmail.com; https://orcid.org/0000–0001–5957–0305; ORCID 0000–0001–5957–0305.

Gussenov, Iskander Shakhsavanovich — PhD, Satbayev University, Senior researcher of the Institute of Polymer Materials and Technology, Almaty, Kazakhstan; Satbayev street 22a, 050013. e-mail: iskander.gusenov@mail.ru; ORCID 0000–0002–9820–7952.

Shakhvorostov, Alexey Valeryevich — M.Sc., Institute of Polymer Materials and Technology, Almaty, Kazakhstan; Atyrau-1, 3/1, 050019; e-mail: alex.hv91@gmail.com; ORCID 0000–0003–3502–6123.

Kudaibergenov, Sarkyt Elekenovich — Dr. Chem. Sci., Professor, Director, Institute of Polymer Materials and Technology, Head of the Laboratory of Engineering Profile, Satbayev University, Almaty, Kazakhstan; Atyrau-1, 3/1, 050019. e-mail: skudai@mail.ru; ORCID 0000–0002–1166–7826.