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ИЗВЕСТИЯ

РОО «НАЦИОНАЛЬНОЙ
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ЧФ «Халық»

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В 2016 году для развития и улучшения качества жизни казахстанцев был создан частный Благотворительный фонд «Халык». За годы своей деятельности на реализацию благотворительных проектов в областях образования и науки, социальной защиты, культуры, здравоохранения и спорта, Фонд выделил более 45 миллиардов тенге.

Особое внимание Благотворительный фонд «Халык» уделяет образовательным программам, считая это направление одним из ключевых в своей деятельности. Оказывая поддержку отечественному образованию, Фонд вносит свой посильный вклад в развитие качественного образования в Казахстане. Тем самым способствуя росту числа людей, способных менять жизнь в стране к лучшему – профессионалов в различных сферах, потенциальных лидеров и «великих умов». Одной из значимых инициатив фонда «Халык» в образовательной сфере стал проект *Ozgeris powered by Halyk Fund* – первый в стране бизнес-инкубатор для учащихся 9-11 классов, который помогает развивать необходимые в современном мире предпринимательские навыки. Так, на содействие малому бизнесу школьников было выделено более 200 грантов. Для поддержки талантливых и мотивированных детей Фонд неоднократно выделял гранты на обучение в Международной школе «Мирас» и в Astana IT University, а также помог казахстанским школьникам принять участие в престижном конкурсе «USTEM Robotics» в США. Авторские работы в рамках проекта «Тәлімгер», которому Фонд оказал поддержку, легли в основу учебной программы, учебников и учебно-методических книг по предмету «Основы предпринимательства и бизнеса», преподаваемого в 10-11 классах казахстанских школ и колледжей.

Помимо помощи школьникам, учащимся колледжей и студентам Фонд считает важным внести свой вклад в повышение квалификации педагогов, совершенствование их знаний и навыков, поскольку именно они являются проводниками знаний будущих поколений казахстанцев. При поддержке Фонда «Халык» в южной столице был организован ежегодный городской конкурс педагогов «Almaty Digital Ustaz».

Важной инициативой стал реализуемый проект по обучению основам финансовой грамотности преподавателей из восьми областей Казахстана, что должно оказать существенное влияние на воспитание финансовой грамотности и предпринимательского мышления у нового поколения граждан страны.

Необходимую помощь Фонд «Халык» оказывает и тем, кто особенно остро в ней нуждается. В рамках социальной защиты населения активно проводится

работа по поддержке детей, оставшихся без родителей, детей и взрослых из социально уязвимых слоев населения, людей с ограниченными возможностями, а также обеспечению нуждающихся социальным жильем, строительству социально важных объектов, таких как детские сады, детские площадки и физкультурно-оздоровительные комплексы.

В копилку добрых дел Фонда «Халык» можно добавить оказание помощи детскому спорту, куда относится поддержка в развитии детского футбола и карате в нашей стране. Жизненно важную помощь Благотворительный фонд «Халык» оказал нашим соотечественникам во время недавней пандемии COVID-19. Тогда, в разгар тяжелой борьбы с коронавирусной инфекцией Фонд выделил свыше 11 миллиардов тенге на приобретение необходимого медицинского оборудования и дорогостоящих медицинских препаратов, автомобилей скорой медицинской помощи и средств защиты, адресную материальную помощь социально уязвимым слоям населения и денежные выплаты медицинским работникам.

В 2023 году наряду с другими проектами, нацеленными на повышение благосостояния казахстанских граждан Фонд решил уделить особое внимание науке, поскольку она является частью общественной культуры, а уровень ее развития определяет уровень развития государства.

Поддержка Фондом выпуска журналов Национальной Академии наук Республики Казахстан, которые входят в международные фонды Scopus и WoS и в которых публикуются статьи отечественных ученых, докторантов и магистрантов, а также научных сотрудников высших учебных заведений и научно-исследовательских институтов нашей страны является не менее значимым вкладом Фонда в развитие казахстанского общества.

**С уважением,
Благотворительный Фонд «Халык»!**

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SYNTHESIS AND CHARACTERIZATION OF PREFORMED PARTICLE GELS (PPG) TO INCREASE OIL RECOVERY

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Abstract. Excessive water generation is a major challenge in oil and gas production. This problem can lead to reduced production efficiency and increased operating costs. To solve this problem, the oil industry employs several techniques. One of these is the use of preformed particle gels (PPG). Many hydrogels have limited mechanical strength and tend to degrade under reservoir conditions characterized by high salinity, temperature and pH. This article presents experimental results on the application of composite hydrogels composed of acrylamide (AAm), sodium acrylate (SA), and bentonite as a capping agent. The polymer composition was prepared by radical polymerization in the presence of a crosslinking agent, N,N-methylenebisacrylamide (MBAA). Consequently, a series of PPGs with different compositions were prepared. These samples were characterised

by FTIR, mechanical analysis and thermogravimetric analysis. The swelling ability of the hydrogels was investigated both in water and in brine with different salinity. The effect of AAm/SA concentration on the mechanical properties and degree of swelling of the hydrogels was investigated. The results indicate that the degree of swelling in the hydrogels increases with higher sodium acrylate content. Moreover, the hydrogel particles show favorable swelling capacity when used in saline solutions. The diffusion mechanism was studied using the model proposed by Yavari and Azizyan, and it was found that the relaxation process predominates. These hydrogels show both thermal and mechanical stability with elastic modulus ranging from 62 to 85 Pa. The results confirm that PPGs based on AAm/SA/MBAA can serve as effective plugs that are temperature and salt resistant and exhibit high mechanical strength.

Keywords: hydrogels, PPG, oil recovery, water cut, sodium acrylate, acrylamide

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МҰНАЙ ӨНДІРУДІ ЖОҒАРЛАТУ ҮШІН ГИДРОГЕЛЬ БӨЛШЕКТЕРІН (PPG) СИНТЕЗДЕУ ЖӘНЕ ЗЕРТТЕУ

Аннотация. Мұнай және газды өндіру барысындағы, судың артық мөлшерде түзілуі өзекті мәселелердің бірі болып табылады. Осы жағдай өндірістің қарқындылығын түсіріп, керісінше операциялық шығындарды арттырады. Бұл мәселені мұнай өндіру саласында шешу үшін әр түрлі әдістерді қолданады, соның ішінде алдын ала қалыптасқан гидрогель бөлшектерін (PPG) қолдану әдісі де бар. Көптеген гидрогельдер әлсіз механикалық қасиеттерге ие, сонымен қоса су қоймасы жағдайында (жоғары тұздылық, жоғары температура және рН) жойылады. Бұл мақалада акриламид (AAm), натрий акрилаты (SA) және бентонит негізіндегі құрама гидрогельдерді жабу агенттері ретінде пайдалану бойынша тәжірибелік мәліметтер келтіріледі. Полимерлік композиция айкастырғыш агент ретінде — N,N-метиленбисакриламида (MBAA) колданып, бос радикалды сополимерлену жолымен алынған. Үлгілер FTIR, механикалық және термогравиметриялық талдау арқылы сипатталды. Гидрогельдердің ісіну қасиетін суда және әртүрлі тұздылықтағы тұзды ерітінділерде анықтады. Сонымен қоса гидрогельдердің ісіну дәрежесі температураның өзгергенде зерттелді. AAm/SA концентрациясының гидрогельдердің механикалық қасиеттері мен ісіну дәрежесіне әсері зерттелді. Алынған нәтижелер гидрогельдердің ісіну дәрежесі натрий акрилатының

мөлшерінің өсуімен артатынын көрсетті. Сонымен қоса, гидрогель бөлшектер тұзды ерітінділерде қанағаттанарлық ісіну қабілетін көрсетті. Явари және Азизян ұсынған модель көмегімен диффузия механизмі зерттелді. Релаксация процесі басым екені анықталды. Гидрогельдер механикалық және термиялық тұрақты. Юнг модульы 62 мен 85 Па арасында өзгереді. Алынған нәтижелер ААm/SA/МВАА негізіндегі РРG термо және тұзды тұрақты, сонымен қоса жоғары механикалық берік жабу агенті ретінде пайдалануға болатынын көрсетті.

Түйін сөздер: гидрогельдер, РРG, мұнай өндіру, сулану, натрий акрилаты, акриламид

Қаржыландыру: Бұл жұмыс Қазақстан Республикасы Ғылым және жоғары білім министрлігі Ғылым комитетінің қаржылық қолдауымен орындалды (грант №AP13068286).

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СИНТЕЗ И ИССЛЕДОВАНИЕ ГИДРОГЕЛЕВЫХ ЧАСТИЦ (PPG) ДЛЯ ПОВЫШЕНИЯ НЕФТЕОТДАЧИ ПЛАСТОВ

Аннотация. Образование избыточного количества воды является серьезной проблемой в процессе добычи нефти и газа. Это может снижать эффективность добычи и увеличивать операционные расходы. Для решения этой проблемы в нефтедобывающей отрасли используют различные методы, включающие применение предварительно сформованных гидрогелевых частиц (PPG). Многие гидрогели обладают слабыми механическими свойствами, а также разрушаются в пластовых условиях (высокая соленость, повышенная температура и рН). В этой статье приводятся экспериментальные данные по использованию композитных гидрогелей на основе акриламида (ААm), акрилата натрия (SA) и бентонита, в качестве закупоривающих агентов. Полимерная композиция была получена путем свободнорадикальной сополимеризации в присутствии сшивающего агента – N,N-метиленбисакриламида (МВАА). В результате была получена серия РРG с различным составом. Образцы были охарактеризованы с помощью FTIR, механического и термогравиметрического анализа. Набухающую способность гидрогелей определяли в воде и рассоле с различной соленостью, также была изучена степень набухания гидрогелей при изменении температуры. Исследовано влияние концентрации ААm/SA на механические свойства и степень набухания гидрогелей. Результаты показали, что степень набухания гидрогелей повышается с повышением содержания акрилата натрия. Также гидрогелевые частицы показали

удовлетворительные набухающие способности при использовании в соленых растворах. Исследован механизм диффузии с помощью модели, предложенной Явари и Азизьяном. Установлено, что преобладает процесс релаксации. Гидрогели обладают термической и механической устойчивостью. Модуль Юнга варьирует от 62 до 85 Па. Полученные результаты продемонстрировали, что PPG на основе AAm/SA/MBAA может быть использован в качестве закупоривающего агента, обладающего термо-, солеустойчивостью и высокой механической прочностью.

Ключевые слова: гидрогели, PPG, нефтеотдача, обводненность, акрилат натрия, акриламид

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Introduction

Polymer gels consist of interconnected polymer chains that swell greatly in a solvent but do not dissolve in it. Their ability to significantly change their volume depending on environmental variables such as pH, ionic strength, temperature, and other factors offers promising opportunities for their application in various fields, including medicine, agriculture, pharmacology, ecology, and catalysis (Abilova, 2020). For more than four decades, polymer flooding technology has been widely used in methods for enhanced oil recovery (Liu et al., 2010; Qiu et al., 2017). In the context of polymer flooding, polymers improve the cleanup efficiency, i.e., the fraction of the reservoir volume between the injection and production wells that comes into contact with the injected fluids. This is achieved by increasing the viscosity of the fluids, which ultimately leads to an increase in oil production (Xua et al., 2019). However, there is a problem that the injected polymer solutions seek preferential pathways and pass through layers with high permeability, which contributes to high water content (Amaral CNR et al., 2019). Therefore, the primary goal is to address the problem of channel flooding, as it leads to problems such as channel corrosion and a reduction in oil production, among others (Sth'efany et al., 2022). Most hydrogel systems used to enhance oil recovery are limited in their application to high temperature, acidity, and salinity (Pereira Kaio et al., 2020). Currently, hydrogel composites are used with various fillers, including silica, clay, and coal fly ash (Bai et al., 2015). In-situ gels using microgels and PPG are also commonly used. (Sun L. et al., 2019; Mehrabianfar et al., 2020).

PPG are particularly noteworthy for their role in controlling compliance. In the preparation of PPG, a gel is first synthesized from a polymer and a crosslinking agent. Then, this gel is broken down into particles of specific sizes. These gel particles are customizable in both size and strength. They are insoluble but easily dispersible in water, exhibit remarkable swelling ability, are environmentally benign, provide long-term stability, and have excellent plugging properties (Yu et al., 2018).

This article presents data on the synthesis and characterization of composite hydrogels based on AAm/SA/MBAA, which can be used as promising materials for enhanced oil recovery.

Materials

The following monomers were used to synthesize the hydrogel particles: Acrylamide (AAm) and sodium acrylate (SA). N,N'-methylenebisacrylamide (MBAA) served as a crosslinking agent. All reagents are produced by Sigma Aldrich and used without additional purification. Analytical grade ammonium persulfate (APPS) and sodium metabisulfite (SMB) from Reachim (Russia) were used as polymerization initiators. The bentonite clay (Zhejiang Qinghong New Material Co., Ltd.) was incorporated to improve the mechanical properties of the hydrogel particles.

Synthesis of hydrogels based on AAm/SA/MBAA

Hydrogel samples were synthesized by free radical polymerization with different starting molar ratios of monomers. The primary outline of the polymerization process is illustrated in Figure 1. The content of reagents used for the synthesis of hydrogels is listed in Table 1.

Table 1 – Monomer masses for synthesis of PPG

Aam/SA ratio, %	95/5	90/10	85/15	80/20	75/25
Composition of initial monomer compound	Mass, g				
Bentonite	0,5	0,5	0,5	0,5	0,5
Water	9	9	9	9	9
MBAA	0,0107	0,0107	0,0107	0,0107	0,0107
AAm	0,4645	0,4151	0,4068	0,3903	0,2430
SA	0,0248	0,0742	0,0825	0,0990	0,2464

Briefly, to prepare a hydrogel sample with a molar ratio of [AAm/SA] of [95/5] 0.0107 g of MBAA were added to 9 g of water and stirred until completely dissolved. Next, 0.4645 g of AAm were added to the mixture and thoroughly mixed into the solution. After that, 0.0248 g of SA were added and mixed for 2–3 minutes. Following, 0.5 g of dry bentonite were added into the mixture. The resulting solution was stirred on a magnetic plate for 2 hours to ensure complete mixing and to allow the bentonite to swell. The solution was then poured into cylindrical syringes with a 10 mL volume and purged with argon for 10 minutes to create an inert atmosphere. Subsequently, 0.1 mL of the first initiator solution containing 0.1 g/mL of APS was added and mixed using a metal spatula. Then, 0.1 mL of the second initiator solution containing 0.01 g/mL of SMB was added and mixed again. The syringe was sealed with a plastic cap and placed in a thermostat at a temperature of 60 °C for 2 hours for polymerization. Hydrogels of other compositions were prepared in a similar manner using the mass ratios of the monomers given in Table 1.

Investigation of AAm/SA/MBAA hydrogel properties

FTIR-spectroscopy

The functional groups of the hydrogels were analyzed using Cary 660 FTIR spectroscopy, manufactured by Agilent in the USA, and equipped with a pike MIRacle ATR accessory operating in Attenuated Total Reflection mode. To obtain the FTIR spectra, the hydrogels were immersed in 10 mL of deionized water and then freeze-dried for 24 hours to remove any residual moisture. The FTIR spectra were recorded at room temperature, covering the range of wavenumbers from 700 cm⁻¹ to 4000 cm⁻¹.

Swelling kinetics study

Determination of the kinetics of hydrogel swelling based on AAm/SA/MBAA was carried out by the gravimetric method, in which the mass of the hydrogel is measured before and after swelling.

The swelling kinetics was carried out by the gravimetric method (Gussenov et al., 2023). A dried hydrogel sample was placed in a glass of water with a volume of 100 mL. After 5 minutes, it was removed, the excess liquid was removed and weighed. Then the sample was re-placed in the same glass. The mass of the sample was measured through 5, 10, 15, 30, 60, 120, 180, 1440, 2880 min., etc. until the constant mass of the sample is reached. The kinetics of swelling was calculated by the formula:

$$SD = \frac{m - m_0}{m_0} \quad (1)$$

where

SD is the degree of swelling, g/g

m is the mass of the swollen hydrogel at a time, g

m_0 is the mass of the dry hydrogel, g

The degree of swelling of hydrogels was also carried out by gravimetric method. The dried hydrogel sample was placed in a glass of water for a day. A day later, the sample was extracted, the excess liquid was removed and it was weighed. The degree of swelling of the hydrogels was assessed under various conditions, including:

- Ambient Temperature: Measurements were taken at different temperature, specifically 20 °C, 40 °C, 60 °C, and 80 °C.
- Salt Concentrations in Solution: Swelling experiments were conducted in solutions with varying salt concentrations, ranging from 1, 10, 25, 50, 75, 100, to 150 g/L.

Mechanical properties of hydrogels

Mechanical analysis was conducted using a TEXT plus computer analyzer, which is a product of Stable Microsystems based in the UK. For the analysis, cylindrical hydrogel particles were used, with dimensions of a height of 10 mm and a contact area of 78 mm². These samples were positioned on a test table and subjected to compression using an upper loading cylinder probe (P/10) at a speed of 0.1 mm/s in compression mode. The power target mode was consistently applied throughout all the experiments.

Young's modulus, a measure of stiffness, was determined by analyzing the initial sections of the stress/strain curves. The magnitude of stress, denoting the force per unit area, was calculated based on the maximum force value recorded during the tests (2).

$$Stress = \frac{Force (g)}{Area(mm^2)} \quad (2)$$

Thermal analysis of hydrogels

The thermal stability of hydrogels was evaluated using the ThermoGravimetric Analyzer TGA1250 (FPI inc. China). Heating was carried out in the temperature range from room temperature to 600 °C with a heating rate of 10 °C/min in a nitrogen atmosphere.

Results and discussion

The copolymerization of AAm and SA proceeds in accordance with Figure 1.

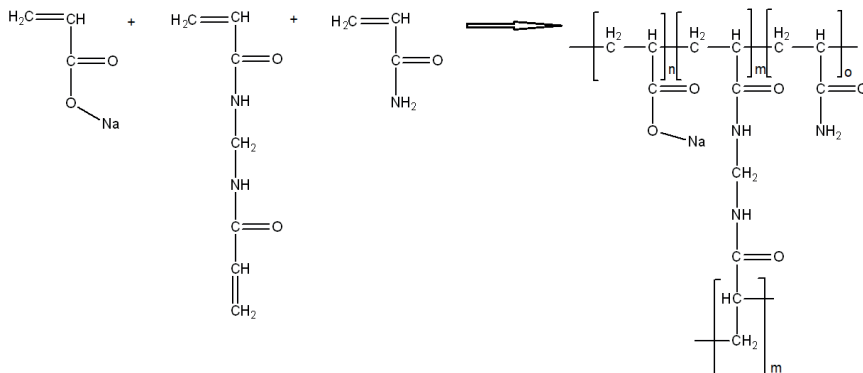


Figure1 - Scheme for synthesis PPG based on AAm/SA/MBAA

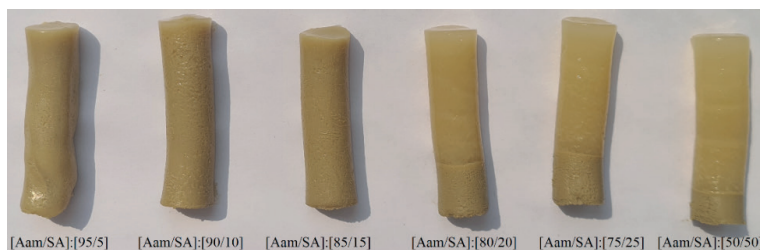


Figure 2 - Samples of synthesized hydrogels based on AAm/SA/MBAA

Figure 2 shows samples of synthesized hydrogel particles based on AAm/SA/MBAA. Due to the heterogeneity of the samples [AAm/SA]:[80/20], [AAm/SA]:[75/25], [AAm/SA]:[50/50], only the samples [AAm/SA]:[95/5], [AAm/SA]:[90/10], [AAm/SA]:[85/15] were used for the experiments.

The hydrogels obtained were studied by IR spectroscopy, thermogravimetry and mechanical analysis.

Figure 3 shows the IR spectra of the hydrogel composition.

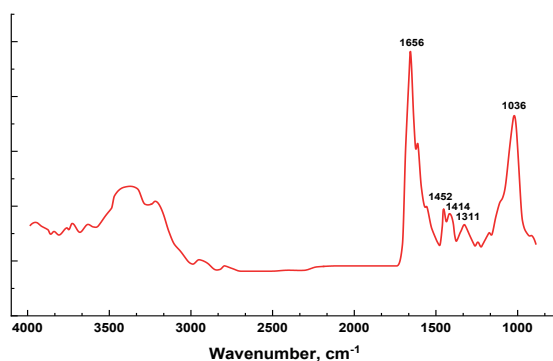


Figure 3 - IR spectra of AAm/SA/MBAA hydrogel

The IR spectra of the samples of synthesized hydrogels show several characteristic peaks. The absorption bands in the range 2800–3000 cm^{-1} correspond to the asymmetric and symmetric vibrations of the CH groups. The absorption bands at 1656 and 1590 cm^{-1} belong to the oscillations of the N-substituted groups, which are the groups of amide I and amide II, respectively. The absorption band at 1452 cm^{-1} is characteristic of the bending oscillations of the C-H groups of hydrogels. The absorption band at 14 cm^{-1} corresponds to the fluctuations of the C-N groups. Thus, the IR spectra indicate the formation of a composite hydrogel based on AAm/ SA.

The swelling ability and rate of hydrogels are influenced by a range of parameters owing to the capacity to retain water within their three-dimensional (3D) polymer mesh. These parameters include ionic strength, temperature, the degree of crosslinking in the gel structure, the type of monomer groups, and pH, among others. In efforts to describe the kinetics of swelling in gels, several kinetic models are employed, but many of these primarily focus on either diffusion or the relaxation of the polymer as the predominant process governing the swelling rate.

Here, a novel model, as proposed by Yavari and Azizyan (Yavari et al., 2022), has been explored. This innovative mixed model is designed to comprehensively describe the swelling process across the entire spectrum of swelling degrees and to determine whether the dominant mechanism is diffusion or polymer relaxation. The model is based on a straight forward Equation 3, which considers the kinetics of swelling and provides the means to assess the influences of both diffusion and polymer relaxation on the process.

$$S_t = S_e \left[1 - e^{(-k_1 t - k_2 t^{1/2})} \right] \quad (3)$$

S_t is the swelling of the hydrogel (g/g) at time t ,

S_e is the equilibrium swelling of the hydrogel (g/g),

k_1 and k_2 are the constants of the swelling rate.

According to this model, the relaxation of polymer chains is the most important factor in the swelling rate of gels when $k_1 \gg k_2$. On the other hand, when $k_1 \ll k_2$, the swelling rate is mainly influenced by diffusion. In cases where the values of k_1 and k_2 are relatively close, both the diffusion and relaxation modes contribute to the determination of the swelling rate. The kinetic curves of hydrogel swelling based on AAm/SA/MBAA are shown in Figure 4, and the data are listed in Table 2.

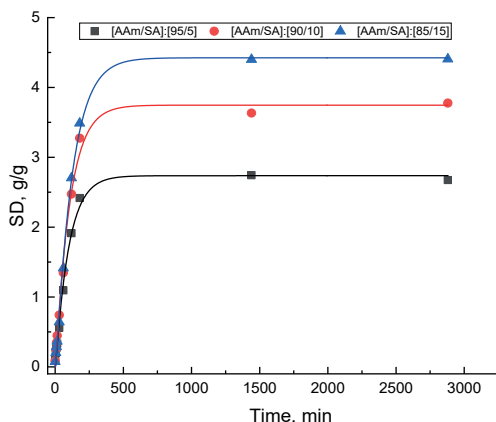


Figure 4 - Dependence of hydrogels SD on time

Table 2 - Data on the swelling of hydrogels

Sample	S_e	k_1	k_2	R^2	Main process
[AAm]/[SA]:[95]/[5]	4,42	0,0091	-0,0152	0,99	Relaxation
[AAm]/[SA]:[90]/[10]	3,47	0,0119	-0,0150	0,97	Relaxation
[AAm]/[SA]:[85]/[15]	2,74	0,0109	-0,0104	0,99	Relaxation

As can be seen from Figure 4 and Table 2, the points on the curve, the model developed by Yavari and Azizyan, describe the experimental data very well, as evidenced by the high value of the correlation coefficient ($R^2=0.99$). According to the obtained results, the swelling of hydrogels based on AAm/ SA is determined by the relaxation of the polymer chains.

As can be seen from Figure 4, equilibrium swelling of the hydrogel is reached on the second day. Also, in his research, the author (Wanli at al., 2019) chooses the swelling time of hydrogel particles during the subsequent oil displacement test equal to 2 days.

Moreover, the degree of swelling itself varies significantly depending on the composition of the monomer. With increasing sodium acrylate content, an increase in the degree of swelling of the hydrogel particles is observed (Figure 5)

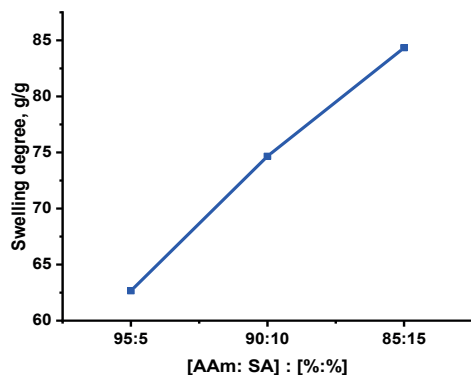


Figure 5 – Dependence of SD on the composition of the hydrogels

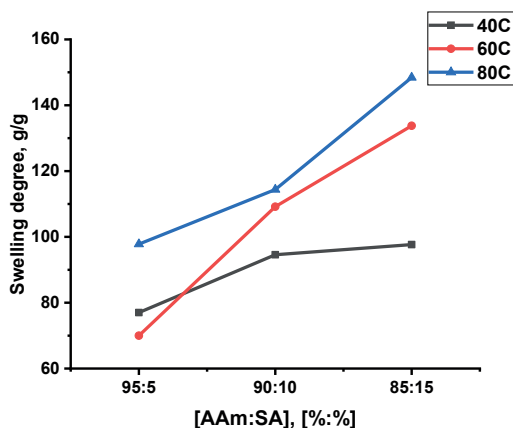


Figure 6 – Dependence of SD of hydrogels on the monomers content during temperature changes

Figure 6 shows the effect of temperature on the swelling of PPG. At higher temperatures, the amide group ($-\text{CONH}_2$) of acrylamide is hydrolyzed to the carboxyl group ($-\text{COOH}$). The ability to swell hydrogels increases with increasing temperature. A significant increase in swelling ability occurs at a temperature of 80°C . A similar effect was observed by the author (Suleimenov et al., 2020) in his work.

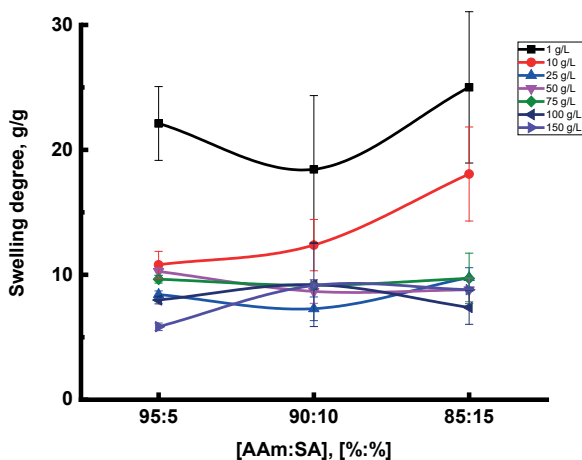


Figure 7 - The SD of hydrogels as a function of the ionic strength

The investigation of hydrogel swelling in saline water was conducted with the objective of assessing the degree of swelling under realistic conditions, as oil field reservoir waters often have elevated salinity levels. In Figure 7, it can be noted that hydrogels show a high degree of swelling at low concentrations of salt, at high concentrations there is a weak swelling, while the degree of swelling does not change significantly. This indicates the possibility of using these hydrogels in reservoir conditions. Similar results were obtained by Mehrabianfar P. (Mehrabianfar et al., 2020).

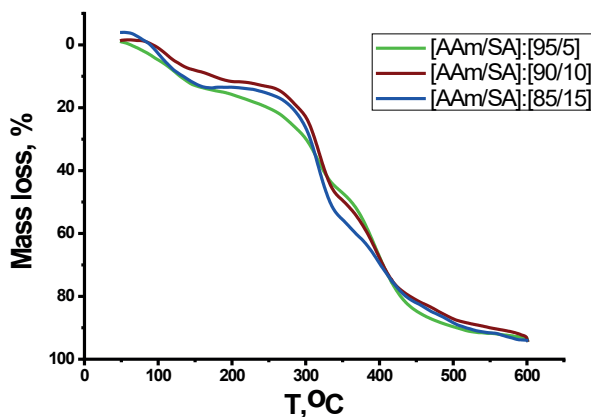


Figure 8 - TG curves of hydrogels

Thermogravimetric analysis showed that all samples exhibited remarkable stability in the temperature range from 25°C to 200°C, as shown in Figure 8. A significant loss of mass of the hydrogel particles was only observed at temperatures above 250 °C. This degree of temperature stability of the samples is more than sufficient for injection into oil-bearing formations, since temperatures in these formations do not usually exceed 80 °C.

The mechanical properties of hydrogels do not differ significantly in their strength (Figure 9, Table 3).

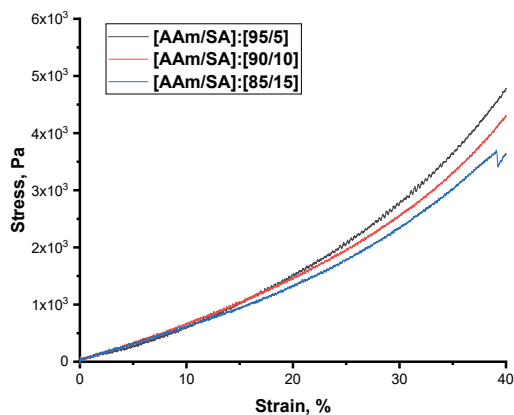


Figure 9 -Strain-Stress curves of hydrogels

Table3 - Dependence of the Young's modulus of hydrogels on the composition

Hydrogel composition [AAm/SA]	[95/5]	[90/10]	[85/15]
Young's modul, Pa	85,13 ± 0,36	82,58 ± 0,25	68,58 ± 0,19

As the concentration of sodium acrylate increases, the mechanical properties of the hydrogels tend to deteriorate. Conversely, an increase in the concentration of acrylamide results in an elevation of Young's modulus, indicating improved mechanical strength.

Conclusion

A series of hydrogels consisting of AAm/SA/MBAA with different component ratios were successfully synthesized. The interaction between these monomers in the hydrogel synthesis process is confirmed by the FTIR results. The resulting hydrogels exhibit favorable mechanical properties and a considerable degree of swelling. Moreover, they show remarkable swelling ability even at high temperatures and high salinity. The results of the thermogravimetric analysis indicate that these hydrogels are suitable for use in reservoirs with elevated temperatures.

Overall, the results indicate that these hydrogel particles have great potential to serve as an effective material for PPG, with the goal of enhancing oil recovery in oil fields.

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CONTENTS

A. Abdullin, N. Zhanikulov, B. Taimasov, E. Potopova, A. Raisova
INVESTIGATION OF THE MICROSTRUCTURE OF SYNTHESIZED
ZINC-PHOSPHATE CEMENT CLINKER.....7

G.F. Sagitova, N.B. Ainabekov, Yu.A. Nifontov, N.M. Daurenbek
SELECTION OF RAW MATERIALS FOR THE PRODUCTION OF BITUMEN
MATERIALS BASED ON LOCAL RESOURCES.....19

Kh. Akimzhanova, A. Sabitova, Zh. Kairbekov, B. Mussabayeva, B. Bayahmetova
CHEMICAL CHARACTERISTIC OF THE BLACK AND WHITE MUD
OF THE SHOSHKALY LAKE.....31

**A.S. Auyezkhanova, D.E. Zhanuzak, A.I. Jumekeyeva, Zh.K. Korganbaeva,
A.A. Naizabayev**
CHITOSAN-STABILIZED CATALYSTS FOR CYCLOHEXANE OXIDATION
TO KA-OIL.....44

Ya.A. Vissurkhanova, L.K. Abulyaissova, N.M. Ivanova, B.F. Minaev
MOLECULAR SIMULATION OF THE INTERACTION OF POLYVINYL
ALCOHOL WITH POTENTIAL ACTIVE CENTERS OF COPPER (II)
OXIDE SURFACE.....54

E.A. Gabrilyants, R.S. Alibekov, G.E. Orymbetova
DEVELOPMENT OF CAMEL MILK CHEESE TECHNOLOGY
AND RESEARCH OF QUALITATIVE CHARACTERISTICS.....69

**G.T. Yelemessova, L.K. Orazzhanova, A.N. Klivenko, N.N. Nurgaliyev, A.Ye.
Ayazbayeva, A.V. Shakhvorostov**
SYNTHESIS AND CHARACTERIZATION OF PREFORMED PARTICLE
GELS (PPG) TO INCREASE OIL RECOVERY.....79

E.A. Zhakmanova, G.Zh. Seytenova, R.M. Dyusova
REVIEW OF THE CURRENT STATE OF APPLICATION OF MATHEMATICAL
MODELING METHODS FOR THE PURPOSE OF OPTIMIZING REFINERIES
IN KAZAKHSTAN AND ABROAD.....92

**M. Zhumabek, K. Kassymkhan, R.O. Sarsenova, Zh. Tynybek, S.A. Tungatarova,
Z.T. Zheksenbaeva**
INVESTIGATION OF CATALYSTS OF THE CATALYTIC PROCESSING
OF NATURAL GAS METHANE INTO SYNTHESIS GAS VIA
TEMPERATURE-PROGRAMMED DESORPTION.....103

M. Ibrayeva, N. Duzbayeva, Zh. Mukazhanova, K. Kabdysalym, Achyut Adhikari ISOLATION OF FLAVONOIDS BY HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY FROM PLANT OF GENUS THYMUS SERPYLLUM L.	116
B. Imangaliyeva, B. Dossanova, G. Rakhmetova, A. Apendina, I. Nurlybaev FEATURES AND CHEMICAL PROPERTIES OF ANTHOCYANINS.....	124
B.Zh. Iskendirov, G.F. Sagitova, S.B. Kurbanova, G.F. Aitimbetova, A.S. Sadyrbayeva DEVELOPMENT OF TECHNOLOGY FOR PROCESSING RESIDUES FROM THE DISTILLATION OF A MIXTURE OF OILS AND GAS CONDENSATES.....	144
X.A. Leontyeva, D.S. Puzikova, G.M. Khussurova, P.V. Panchenko, A.K. Galeyeva ELECTROCHEMICAL DEPOSITION OF BISMUTH SULFIDE THIN FILMS.....	158
M.M. Mataev, M.A. Nurbekova, B. Keskin, Z.B. Sarsenbayeva SYNTHESIS AND PHYSICO-CHEMICAL PROPERTIES OF POLYCRYSTAL $\text{FeMnO}_3\text{-Ho}_3\text{Fe}_5\text{O}_{12}$	173
R. Safarov, Zh. Shomanova, E. Kopishev, Yu. Nossenko, Zh. Bexeitova, R. Kamatov SPATIAL DISTRIBUTION OF PM2.5 AND PM10 POLLUTANTS IN RESIDENTIAL AREA OF PAVLODAR, KAZAKHSTAN.....	181

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