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THE STUDY OF DEPENDENCE OF SILICATE-POLYMER SOLUTIONS ON COMPONENTS CONCENTRATION IN THE COMPOSITION

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Abstract

Purpose: The aim of this article is to study the possibility of xanthan usage as a polymer additive in silicate-gel solutions when creation compositions for enhancing oil recovery. The dependence of the time of gel formation of the silicate-polymer compositions and the pH of the gel-forming solution from various concentrations of xanthan and polyacrylamide was analyzed. **Research methods:** Direct and reverse titration, potentiometric titration, pH-metry, determination of the dynamic viscosity on a rotational viscometer, axial compression method to determine the elastic modulus were used to determine the possibility of xanthan usage as a polymer additive instead of polyacrylamide.

Results: Such investigations as determination the influence of polymer type and concentration on the elastic modulus and syneresis of compositions in the gel state have been performed to determine the possibility of xanthan usage as a polymer additive of gel-forming composition and influence of this polymer on the dynamic viscosity of gel-forming compositions, gel strength, stability in time, the possibility of gel destruction. **Discussion:** Xanthan investigation as an alternative polymer has shown the perspective of its usage in the oil recovery industry. The environmental friendliness of technologies developed with the use of xanthan, which is safe for the environment, does not lead to an irreversible change in the reservoir properties of the formation. The lower molecular weight of xanthan in comparison with synthetic polymers determines the improved filtration properties of xanthan solutions, especially in low permeability conditions, which also makes it possible to carry out deeper treatments of the formation.

Key words: silicate-polymer composition, polyacrylamide, xanthan, time of gel formation.

1. Introduction

Enhanced oil recovery (EOR) is carried out due to the greater efficiency of water isolation in heterogeneous formations; creation of a durable waterproof screen and connection to the development of oil-saturated, previously unused zones by subsequent flooding; improving the rheological properties of the gel forming composition; increasing the strength of the gel based on it; increasing the stability in time; reduction of the time of gel formation. One of the most important problems in the modern oil industry is the development of modern gel-forming compositions satisfying technological, economic and environmental requirements.

2. Analysis of the latest investigations and publications, problem statement

One of the tasks of oil extraction is implementation of technologies that allow increasing the oil recovery of existing oil wells, especially in the case of the

recovery of reservoirs with heavy and high-viscosity oils. One of the methods to improve the efficiency of oil recovery is a method using chemical reagents, based on flooding of injection wells with surfactants, polymer compositions, emulsions [1]. Technological practice shows that the greatest effect when regulating the filtration permeability of water-containing layers in reservoirs is achieved when using gel-like compositions. Solutions of sodium silicate and hydrochloric acid were used as components of the gel-forming solution [2].

The concentration of sodium silicate and the pH of the medium are the determining parameters of the gel-forming compositions. However, their influence on the time of gel formation largely depends on other physical-chemical factors, primarily on the mineralization of the medium and temperature [3]. Temperature dependence can be described by a simple mathematical dependence, and the influence of concentration, pH and mineralization do not have simple mathematical description and can be presented as tables and series of empirical

mathematical dependences. The research methods of the mentioned three factors are very simple and foresee the preparation of a gel-forming solution of the required composition and fixing the time of loss of fluidity from the moment of components mixing [4].

High thickening ability of biopolymers is the main property that determines the use of biopolymers in oil recovery technologies, while, biopolymers have higher rheological properties than chemically synthesized polymers. Besides, many biopolymers are capable to form gel structures with high water-insulating ability [5]. Ratio of the viscosity of oil and injected solutions causes a great influence on the process of increasing oil recovery during water flooding. Introduction of polymers to the solutions causes the viscosity increase, which leads to alignment the oil displacement front.

Chemically synthesized polymer polyacrylamide is used nowadays and has a long history of use. Partially hydrolyzed polyacrylamide was widely used in almost all of the original polymer flooding projects [5], no matter that its high molecular weight causes significant increase of viscosity and anion repulsions between polymer molecules and segments of one molecule lead to stretching and adhesion of molecules, which also causes movability decrease of the [6]. However, toxic compounds can be formed when polyacrylamide production and commercial form of the polymer may contain substances hazardous to human health. Therefore, the use of natural polymers obtained by the biotechnological method (polysaccharides) helps to reduce the anthropogenic load on the environment due to the absence of pollutants during their natural decay [7,8].

Xanthan is more resistant to mechanical and salt destruction, and is also less adsorbed on the reservoir rock than polyacrylamide. Moreover liquid glass provides chemical interaction with biopolymer molecules, bio-surfactants and salts of multivalent metals of layer mineralized water, which ultimately leads to the formation of a strong reinforcing mesh, which effectively reduces water permeability of the washed zones and increases the coverage of the layer by flooding and significantly improves the oil displacement process [9,10].

3. Purposes and problems of a research

The purpose of this work is to study the possibility of using xanthan as a polymer additive in silicate-gel solutions when developing compositions for

enhanced oil extraction. The objective of this study is to analyze the dependence of the time of gel formation of silicate-polymer mixtures and the pH of the gel-forming solution at various concentrations of xanthan and polyacrylamide.

4. Materials and methods of a research

Pre-prepared and titrated aqueous solutions of sodium silicate (10-12%), hydrochloric acid (8-10%), and sodium chloride (20%) were used as initial solutions for preparing compositions.

The following polymers were investigated as polymeric additives to the gel-forming composition: the biopolymer xanthan and, for comparison, polyacrylamide as rather widely used.

During the research we used direct and reverse titration, potentiometric titration, pH-metry, determination of the dynamic viscosity on a rotational viscometer, axial compression method to determine the elastic modulus.

5. Research results

The solution of sodium silicate was poured into a pre-mixed solution of hydrochloric acid and sodium chloride when preparing a composition with low pH values (acidic region). The pH value of the resulting composition was measured immediately after mixing the solutions. The time of gel formation was determined from the moment of mixing the components to the moment of loss of fluidity.

Experimental values were averaged and mathematically processed. The obtained S-shaped curve describes dependence of time of gel formation on pH of medium. The maximum time of gel formation is in the range of pH 1-2 and in the range of more than 10 (Table 1).

Dependence of gel formation on the content of sodium silicate is shown on Fig. 1.

The time of gel formation at different pH values changes (in semi-logarithmic coordinates) almost linearly with small angle of a slope if sodium silicate concentration is 5% and higher. A steeper dependence is observed if concentration less than 5%. It should be mentioned that the strength of the resulting gel decreases significantly decreases with decreasing sodium silicate concentration in this concentration range.

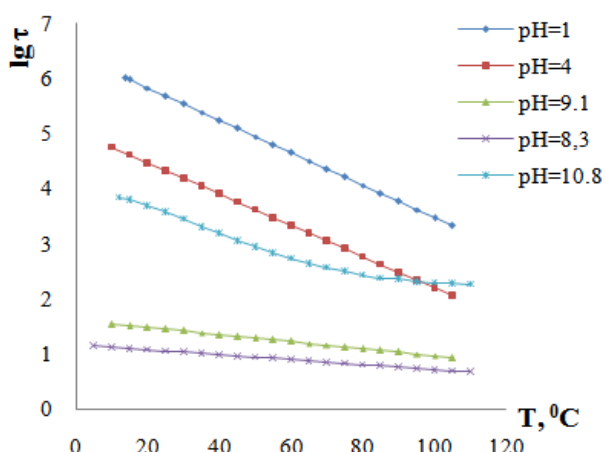


Fig. 1. Dependence of the logarithm of time of gel formation on temperature at different pH values

Additional mineralization of the gel-forming composition by the addition of sodium chloride leads to a decrease the time of gel formation. This dependence is very close to linear in semi-logarithm coordinates in the range of pH values below 10. The slope of the graph decreases up to an inverse dependence at pH more than 11.1 in the region of higher pH values (Fig. 2).

However, it should be mentioned that the time of gel formation is very big in this pH range and it is rather difficult to accurately fix the moment of mobility loss of the composition. Therefore, the probability of occurrence of random deviations is

very high in this range. However, the decrease in the dependence of the time of gel formation on mineralization is fixed quite reliably with the pH of the medium increase. Investigations of the effect of the concentrations of hydrochloric acid and of the polymer on the rheological properties of the silicate-polymer composition have been conducted in order to obtain a silicate gel with a long time of the gel formation beginning.

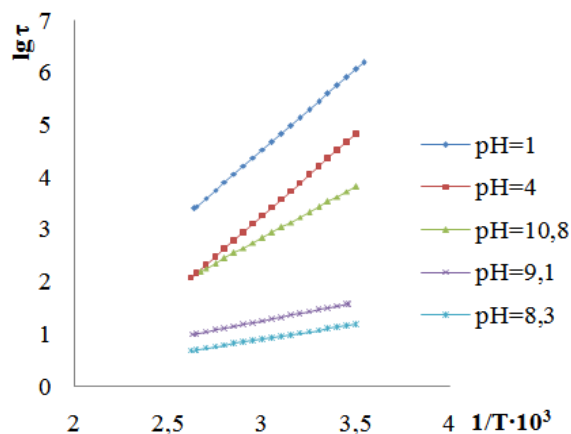


Fig. 2. Dependence of the logarithm of time of gel formation on reverse temperature at different pH values

Times of gel formation of silicate-polymer compositions are shown in the Table 1 at different polymer concentrations and pH of gel-forming solution.

Table 1

Times of gel formation of silicate-polymer compositions at different polymer concentrations and pH of gel-forming solution

pH of the solution	Time of gel formation			
	Without polymer	Xanthan		Polyacrylamide
		0,01%	0,1%	0,01%
11,15	4 days	3 days	2 days 18 h	2 days 22 h
11,05	20 h 32 m	19 h 01 m	18 h 22 m	18 h 50 m
10,9	7 h 12 m	4 h 55 m	4 h	4 h 45 m
10,7	2 h 11 m	1 h 48 m	1 h 46 m	1 h 50 m
10,55	47 m	20 m	17 m	30 m
10,3	13 m 15 s	11 m 40 s	10 m	10 m 11 s
9,8	2 m 20 s	1 m 35 s	50 s	1 m 10 s
9,0	30 s	22 s	20 s	20 s
8,05	20 s	16 s	15 s	15 s
7,05	10 s	4 s	3 s	3 s
4,6	1 h 06 m	55 s	45 m	1 h 52 m
1,9	11 days	9 days 7 h	8 days	12 days
1,7	12 days	11 days 3 h	10 days 18 h	13 days
0,7	4 days 5 h	3 days 5 h	3 days 6 h	5 days 10 h
0,4	2 days	1 days 10 h	1 days 15 h	2 days 19 h

Various concentrations of components of gel-forming compositions were used to study the effect of the type and concentration of the polymer on the dynamic viscosity of gel-forming compositions. Table 2 shows the results of a study of the effect of

the polymer type and concentration on the dynamic viscosity for two systems with sodium silicate and hydrochloric acid contents of 3 % and 0.65 % and 6 % and 0.9 %, respectively. Experiments were carried out at a temperature of 20 °C.

Table 2

Influence of polymer type and concentration on the dynamic viscosity (mPa·s)

Components		Polymer	Polymer concentration, mass %					
Name	mass %		0	0.001	0.005	0.01	0.015	0.02
Sodium silicate	3	Xanthan Polyacrylamide	0,99	0,97	1,10	1,20	1,28	1,35
Hydrochloric acid	0.65		0,99	0,75	1,06	1,15	1,22	1,28
Sodium silicate	6	Xanthan Polyacrylamide	1.15	1.16	1.26	1.33	1.42	1.47
Hydrochloric acid	0.9		1.15	1.13	1.20	1.28	1.34	1.42

The sphere of use of gel-forming compositions can be determined by the time of gel formation at the preparation temperature, the time of gel formation at the layer temperature, the gel strength, the gel stability in mineralized water, stability over

time, the possibility of gel destruction. That is why the influence of the polymer type and concentration on the elastic modulus and syneresis of compositions have been investigated (Tables 3, 4).

Table 3

Influence of the polymer type and concentration on the gel elastic modulus (Pa) at 20 °C

Components		Polymer	Polymer concentration, mass %				
Name	mass %		0	0.001	0.005	0.01	0.015
Sodium silicate	3	Xanthan Polyacrylamide	2.0	2.6	3.5	3.5	3.5
Hydrochloric acid	0.65		2.0	2.8	3.7	3.7	3.7
Sodium silicate	6	Xanthan Polyacrylamide	7.0	7.5	8.9	8.9	8.9
Hydrochloric acid	0.9		7.0	8.0	10.1	10.1	10.1

Table 4

Influence of the polymer type and concentration on the value of gel syneresis (g/100 g of gel) at 20 °C

Components		Polymer	Polymer concentration, mass %				
Name	mass %		0	0.001	0.005	0.01	0.015
Sodium silicate	3	Xanthan Polyacrylamide	4.0	3.90	3.55	3.15	3.00
Hydrochloric acid	0.65		4.0	3.88	3.40	3.00	2.82
Sodium silicate	6	Xanthan Polyacrylamide	1.82	1.80	1.78	1.65	1.60
Hydrochloric acid	0.9		1.82	1.79	1.70	1.60	1.50

The process of gel formation depends on temperature highly. Components of the gel-forming solution were mixed in a certain sequence in strictly defined amounts, thermostated at different temperatures in the range of 20-100 °C to determine time of gel formation. The control over the gel formation process was carried out visually after 1-10

minutes in order to determine the fluidity loss of the solution. The time of gel formation was defined as the time elapsed from the moment the last component of the composition was added until the complete loss of fluidity occurred. The experimental results are presented in Table 5.

Table 5

Dependence of time of gel formation on the temperature of gel-forming solution containing 6 % of sodium silicate and 0.01 % of xanthan

HCl content, mass %	pH of the gel	Time of gel formation, s			
		20 °C	40 °C	60 °C	80 °C
0.45	11.40	2160000	864000	259200	8040
0.5	11.20	1036800	432000	10080	5580
0.6	11.15	295200	86400	6600	2820
0.7	11.10	70800	46200	3600	2100
0.8	10.9	19380	11580	900	720
1.0	10.7	5623	2239	795	316
1.1	10.3	720	420	120	20
1.5	9.1	36	25	18	14
1.6	8.3	16	11	9	8
1.7	7.05	5	Instantly	Instantly	Instantly
1.8	4.60	3960	1200	960	600
	4.00	31622	7079	1584	446
2.0	1.70	1004400	363600	92340	16200
	1.00	602559	199526	42658	11220
3.0	0.65	295200	104400	73200	10080

As can be seen from Table 5, at pH 8-9, the gelation time changes slightly with temperature change, which makes it possible to predict the moment of gel formation regardless of the formation temperatures.

The experimental data was processed by the least squares method. Thus, experimental dependences $lg\tau - T$ and $lg\tau - 1/T$ are described by linear equations with coefficients close to 1 (Table 6).

Table 6

Equations of the experimental dependences $lg\tau - T$ and $lg\tau - 1/T$

pH	$lg\tau=aT+b$	Correlation coefficient	$lg\tau=a\cdot 1/T+b$	Correlation coefficient
1	$lg\tau = -0,029T + 14,3$	0,9990	$lg\tau = 3160 \cdot 1/T - 4,9$	0,9960
4	$lg\tau = -0,029T + 14,3$	0,9930	$lg\tau = 3060 \cdot 1/T - 5,96$	0,9978
8,3	$lg\tau = -0,029T + 14,3$	0,9874	$lg\tau = 545 \cdot 1/T - 0,67$	0,9913
9,1	$lg\tau = -0,029T + 14,3$	0,9979	$lg\tau = 740 \cdot 1/T - 0,97$	0,9973
10,8	$lg\tau = -0,029T + 14,3$	0,9957	$lg\tau = 2115 \cdot 1/T - 3,5$	0,9982

Equation $lg\tau = a \cdot 1/T + b$ is of high interest for description kinetics of gel formation since the rate of gel formation V is proportional to the rate constant of gel formation k and is inversely proportional to the time of gel formation τ :

$$V \sim k \sim 1/\tau$$

If Arrhenius equation can be used for the description the process of gel formation in this temperature range, than following dependence must be fulfilled:

$$lnk = -E_A/RT + Co,$$

where E_A – is activation energy of gel formation at this pH, kJ/mole;

R – 8,314 kJ/(mole·K), gas constant;
 T – temperature, K;
 Co – constant.

It can be obtained following equation using decimal logarithm:

$$lnk = -E_A/2,3 R \cdot 1/T + C.$$

Since $k \sim 1/\tau$, we can say that $lgk \sim -lg\tau$ and we get:

$$-lg\tau = -E_A/2,3 R \cdot 1/T + C.$$

Activation energy of the process can be calculated using equation $lg\tau = a \cdot 1/T + b$ for corresponding pH values:

$$E_A/2,3 R = a \cdot \quad E_A = a \cdot 2,3 R.$$

Table 7 shows activation energies of gel formation reactions for various pH values of the gel forming solution.

Silicic acid is released in the result of interaction between sodium silicate and acidic agents forming a sol, which turns into a gel over time. A silicate gel

has a spatial structure in which silicic acid molecules are linked by valence and hydrogen bonds. This determines the gel strength its formation in the entire volume of the working solution and prevents the precipitation of silicic acid as a separate phase.

Table 7

Activation energies for various pH values

pH	1	4	8.3	9.1	10.9
E _A , kJ/mole	60500	58570	10420	14150	40443

6. Discussion of results

The use of gel-forming compositions with exopolysaccharides has a number of disadvantages, the main of which is that the exopolysaccharide in the form of a culture liquid has low viscosity characteristics, and completely loses its viscosity properties when contact with waste water (mineralization 100 g / l and higher) due to precipitation. The most promising is the use of biopolymers, especially at the objects with carbonate and terrigenous reservoirs. A diverse range of geological and technical conditions requires the development of gel-forming compositions operating in a wide range of reservoir conditions.

Solutions and gel-forming compositions on the base of polyacrylamide are characterized by resistance to the action of mineral waters (up to 280 g/dm³), biodegradation and shear deformation.

The study of xanthan as an alternative polymer has shown the perspective of its use in the oil recovery industry. Xanthan is safe for the environment and the use of ecofriendly technologies on the base of xanthan does not lead to irreversible change in the reservoir properties of the formation. The lower molecular mass of xanthan compared to synthetic polymers determines the improved filtration properties of xanthan solutions, especially in low permeability conditions, which also makes it possible to carry out deeper treatments of the formation. Besides, the lower molecular weight of xanthan leads to low filtration resistance when moving in a porous medium until the moment of gel formation. Sufficiently high syneresis values at low concentrations of xanthan will significantly increase the filtration resistance of the insulated interlayer after gel formation is completed.

Energy activation values of the reaction of gel formation shows that the hardest structure formation proceeds at acidic and basic medium and the easiest structure formation proceeds in the neutral medium.

7. Conclusions

1. Mineralization of the gel-forming composition using sodium chloride leads to a decrease in the time of gel formation.

2. Xanthan with a concentration 0.1% can compete with the currently used PAA, which is more toxic in its effect on human health both during its production and use due to the content of the commercial form of substances hazardous to human health. In addition, the use of xanthan, obtained by a biotechnological method, reduces the technogenic load on the environment, since no polluting reagents are formed during its decay.

3. Successful structuring of gel-forming compositions using xanthan proceeds most easily in a neutral medium.

4. It has been established when studying influence the deposit temperatures on the moment of gel formation that optimal pH values are 8 – 9 when there is a slight change in gel formation time with temperature change.

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Вивчення залежності силікатно-полімерних розчинів від концентрації компонентів композиції

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Мета: Метою даної статті є дослідження можливості застосування ксантану в якості полімерної добавки до силікатно-гелевих розчинів при розробці композицій для підвищення нафтовіддачі пластів. Проаналізована залежність часу гелеутворення силікатно-полімерних композицій і рН гелеутворюючого розчину при різних концентраціях ксантану і поліакриламід. **Методи дослідження:** Для визначення можливості використання ксантану у вигляді полімерної добавки замість поліакриламід використовували пряме і обернене титрування, потенціометричне титрування, рН-метрію, визначення динамічної в'язкості на ротаційному віскозиметрі, метод одновісного стиснення для визначення модуля пружності. **Результати:** Для визначення можливості використання ксантану в якості полімерної добавки гелеутворюючої композиції і впливу цього полімеру на динамічну в'язкість гелеутворюючих композицій, міцність гелю, стабільність у часі, можливість руйнування гелю, проводились дослідження по визначенню впливу типу і концентрації полімеру на модуль пружності і синерезис композицій у стані гелю. **Обговорення:** Дослідження ксантану в якості альтернативного полімеру показало перспективність його використання у нафтовидобувній промисловості. Екологічність технологій розроблених з використанням ксантану, який безпечний для довкілля, не призводить до незворотньої зміни колекторських властивостей пласта. Менша молекулярна маса ксантану у порівнянні з синтетичними полімерами визначають покращені фільтраційні властивості розчинів ксантану, особливо в умовах низької проникності, що також дає можливість проводити більш глибокі обробки пласта.

Ключові слова: силікатно-полімерна композиція, поліакриламід, ксантан, час гелеутворення.

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Изучение зависимости силикатно-полимерных растворов от концентрации компонентов композиции

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Цель: Целью данной статьи является исследование возможности применения ксантана в качестве полимерной добавки в силикатно-гелевых растворах при разработке композиций для повышения нефтеотдачи пластов. Проанализировано зависимость времен гелеобразования силикатно-полимерных композиций и pH гелеобразующего раствора при различных концентрациях ксантана и полиакриламида. **Методы исследования:** Для определения возможности использования ксантана в виде полимерной добавки вместо полиакриламида использовали прямое и обратное титрование, потенциометрическое титрование, pH-метрию, определение динамической вязкости на ротационном вискозиметре, метод одноосного сжатия для определения модуля упругости. **Результаты:** Для определения возможности использования ксантана в качестве полимерной добавки гелеобразующей композиции и влияния этого полимера на динамическую вязкость гелеобразующих композиций, прочность геля, стабильность во времени, возможность разрушения геля, проводились исследования по определению влияния типа и концентрации полимера на модуль упругости и синергизм композиций в состоянии геля. **Обсуждение:** Исследование ксантана в качестве альтернативного полимера показало перспективность его использования в нефтедобывающей промышленности. Экологичность технологий разработанных с применением ксантана, безопасного для окружающей среды, не приводит к необратимому изменению коллекторских свойств пласта. Меньшая молекулярная масса ксантана по сравнению с синтетическими полимерами определяют улучшенные фильтрационные свойства растворов ксантана, особенно в условиях низкой проницаемости, что также дает возможность проводить более глубокие обработки пласта.

Ключевые слова: силикатно-полимерная композиция, полиакриламид, ксантан, время гелеобразования

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