Research of the relationship between the chain length of the dialkyl phosphate ester and the oil-based fracturing fluid viscosity

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Abstract: For the usage of dialkyl phosphate as a gelatinizer in oil-based fracturing fluids, two methods of preparation are involved. The improved ethyl phosphate method is used to control one of the ester chains by using a single alcohol substitute for mixed alcohols. A new method for polyphosphates is used to control both the ester chains. Different kinds of alcohols are used as the raw materials in a 3×3 orthogonal experiment (ratio of reactants, reaction temperature and reaction time) to obtain the optimum reaction conditions. Furthermore, 14 kinds of gelatinizers are produced in these experiments. The viscosity of the products is used as an evaluation index. The results based on these two methods showed that there is a positive correlation between the length of the dialkyl phosphate ester chain and the viscosity of the fracturing fluid with a certain number of carbon atoms. In other words, the viscosity of the fracturing fluid increased with an increasing length of the ester chain length. Then the growth slowed down. The viscosity of the products that were produced by the polyphosphate method was visibly improved by 7.72–45.35 % of the level of the other.

Keywords: gelatinizer; ethyl phosphate; phosphorus pentoxide; monoethyl phosphate; polyphosphate.

INTRODUCTION

In recent years, the yield of conventional oil and gas has shown a downward trend and hence, countries worldwide have shifted their focus on tight sandstone gas, shale gas and other unconventional oil and gas resources to increase oil and gas yields. Such reserves amount to about 1.9×10^{14} m^3 in China,[1] which is doubled the number of conventional reserves. Hydraulic fracturing is a necessary method to develop these reservoirs. The hydroxypropyl guar gum system is a

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common system for water-based fracturing fluids that has a good fracturing outcome because of its high viscosity. Due to the large quantity of residues after gel breaking, this fracturing fluid system has a poor effect under low porosity and low permeability conditions. An oil-based fracturing fluid overcomes the above shortcomings admirably with no residue after gel breaking. Meanwhile the characteristics of not water sensitive and not acid sensitive gives it obvious advantages during the development of unconventional oil and gas reserves.[2,3] In the study of oil-based fracturing fluids, some researchers[4,5] focused on the study of cross-linking agent, gelatinizer and solubilizer, *i.e.*, the usage of new Fe^{3+} cross--linking agents to improve the rate of Al^{3+} cross-linking, the usage of different alcohol mixes with benzene and toluene to prepare a gelatinizer with increased viscosity[6] and the usage of a Fe^{3+} complexing cross-linking agent to improve the cross-linking rate by increasing the dispersion of Fe^{3+}.[7] Gelatinizers play an important role in oil-based fracturing fluids, when gelatinizer bonds with the cross--linking agent, basing oil will be thickened and the viscosity will be increased. They can also play the role of a drag reducer in the processes of injection and backflow.[6–9]

This paper is focused on dialkyl phosphate. Using two methods, the ester chain type of the gelatinizer could be controlled and the length of the ester chain determined, and hence, the relationship between the ester chain length and the viscosity of the oil-based fracturing fluid could be found. The ethyl phosphate method is the current method. It was proposed by Huddleston[10] and used by Kong *et al.*,[6] Hou *et al.*,[7] Smith and Persinski,[8] Chu *et al.*,[11] Wang *et al.*[12] and Wang *et al.*[13] to prepare dialkyl phosphate in their studies of oil-based fracturing fluids. However, the use of mixed alcohols in this reaction has two limitations: first, the reaction temperature cannot be the optimal reaction temperature for all of the alcohols, which leads to incomplete reaction. Secondly, the product variety and the concentration of all kinds of products cannot be determined. Thus, the kinds of products that play a critical role in the final performance of fracturing fluids cannot be known either. Due to the above-mentioned limitations, the current method – an improved ethyl phosphate method – was remodified in the present study by controlling one of the dialkyl phosphate ester chains to determine the type of products. The polyphosphate method can control both of dialkyl phosphate ester chains to alleviate the shortcomings of the improved ethyl phosphate method.

EXPERIMENTAL

Improved ethyl phosphate method

Through the reaction between ethyl phosphate and phosphorus pentoxide, an intermediate monoethyl phosphate was produced. Through another reaction between this intermediate and an alcohol, dialkyl phosphate was synthesized. One ester chain of this dialkylphosphate

was ethyl, and the other ester chain was targeted. In another words, this method can only determine one of the dialkyl phosphate ester chains.
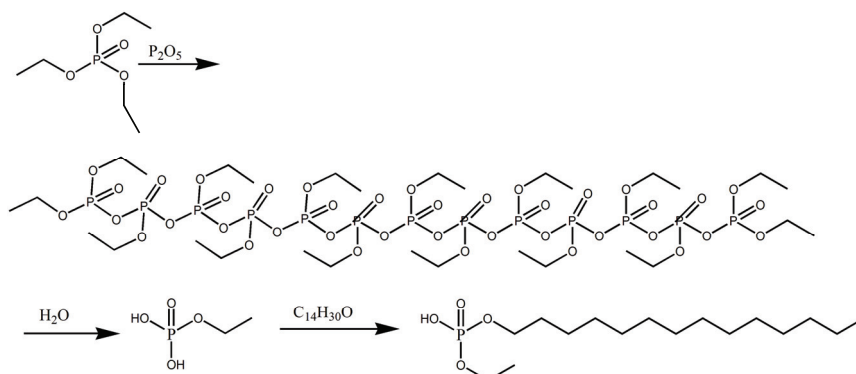
The molecular reaction mechanism is given in Fig. 1.

Fig. 1. The mechanism of the improved ethyl phosphate method (taking tetradecyl alcohol as an example).

Preparation and purification of monoethyl phosphate

Monoethyl phosphate was the intermediate for the synthesis of dialkyl phosphates. The 3×3 orthogonal test combined reaction rate, reaction temperature and reaction time to determine the optimum conditions. Then a purification process was used to improve the product quality. Viscosity is an important parameter for an evaluation of the performance of a fracturing fluid, and when other conditions are constant, the greater the viscosity, the greater the crack.[14] Therefore, viscosity was chosen to evaluate the experimental product.

Preparation of monoethyl phosphate. The reaction between $(CH_3CH_2O)_3PO$ and P_2O_5 was use to generate the polyphosphate, which could be hydrolyzed to a monoester. The conversion rate of polyphosphate would directly affect the quality of the subsequent reactions. After obtaining the optimum reaction conditions by the 3×3 orthogonal test, the mixed titration method was used to determine the percentages of the monoester and the diester.

Purification of monoethyl phosphate. The monoethyl phosphate product contains monoethyl phosphate, dialkyl phosphate, ethyl phosphate and alcohol, the ethyl phosphate and alcohol being impurities.[15] Due to the insolubility of monosodium phosphate in water and in organic solvents, the ethyl phosphate and alcohol could be removed by a solid–liquid separation procedure after neutralizing one of the hydroxyls from monoethyl phosphate using sodium hydroxide. Then by acidizing the solid obtained from the previous solid–liquid separation procedure, the phosphate ester was regained. *n*-Hexane was used to extract the phosphate ester. The purified phosphate ester could then be obtained by distillation. Thus, the operation steps were: first, to the products produced under the optimum reaction conditions in the flask, sufficient *n*-hexane was under stirring to fully dissolve the solids, *i.e.*, the original solution was obtained. The treatment solution was obtained by the addition of some amount of ethanol in 50 % NaOH under stirring. Secondly, the treatment solution was added slowly under stirring into the flask containing the original solution until no more white precipitate emerged. The sediment was obtained by suction filtration. Thirdly, excess dilute hydrochloric acid was added to the precipitate and the mixture was stirred for 2–3 h at a temperature of 50 °C. Sufficient hexane and a small amount of 2-propanol from the broken emulsion were used

to extract the phosphate ester and the distillation was used to remove the hexane and 2-propanol to obtain the purified product.

Synthesis of gelatinizer

The optimal reactant ratio, reaction temperature and reaction time for the reaction between alcohol (1-butanol, 1-hexanol, 1-octanol, 1-nonanol, 1-tridecanol, 1-tetradecanol or 1-octadecanol) and phosphorus pentoxide as the orthogonal factors were determined in an orthogonal experiment. The index of this experiment was the conversion rate of dialkyl phosphate (one kind of oil is choose as the base fluid (in this section, mineral oil was used) and the ratio of the viscosity of the cross-linked base fluid to the viscosity of the mineral oil was used to measure the conversion rate). This oil-based fracturing fluid was prepared by using mineral oil as the base fluid, dialkyl phosphate (self-made) as gelatinizer, $Al_2(SO_4)_3$ as cross-linking agent, NaOAc as the pH regulator and diethanolamine as the co-cross-linking agent (500 mL mineral oil + 2 % dialkyl phosphate + 0.6 % $Al_2(SO_4)_3$ + 1 % diethanolamine + 1 % NaOAc).

Polyphosphate method

The raw materials phosphoric acid and phosphoric anhydride were heated and stirred to synthesize polyphosphate intermediates in the anhydrous state. Then dialkyl phosphate was synthesized through the reaction between the intermediates and alcohols. In comparison with dialkyl phosphate prepared by the improved ethylphosphate method, the dialkyl phosphate made by the polyphosphate method is different because both ester chains of this dialkyl phosphate could be controlled.

Preparation of polyphosphate. Phosphorus pentoxide was charged into a 250 mL round-bottom flask fitted with a stirrer and condenser. The flask was placed in a 40 °C constant temperature water bath and the appropriate amount of phosphoric acid was added under stirring to avoid the phosphorus pentoxide becoming a pyrophosphate precipitate. The temperature was maintain at 40 °C for 0.5 h and then increased to 90 °C. Polyphosphate was obtained after 1 h at 90 °C.

Synthesis of dialkyl phosphate. The optimal reactant ratio, reaction temperature and reaction time, as the orthogonal factors, for the reaction between an alcohol (1-butanol, 1-hexanol, 1-octanol, 1-nonanol, 1-tridecanol, 1-tetradecanol, 1-octadecanol) and polyphosphate were determined in an orthogonal experiment. In general, to a certain amount of alcohol in a 250 mL round-bottom flask fitted with a stirrer and condenser, polyphosphate was add slowly in several batches, while maintaining the temperature at 40 °C. After complete addition of polyphosphate, the temperature was increased to the target temperature[18] and the required product was obtained after several hours.

The products obtained using the different alcohols under the optimum reaction conditions were purified by the same purification process as that used for monoethyl phosphate.

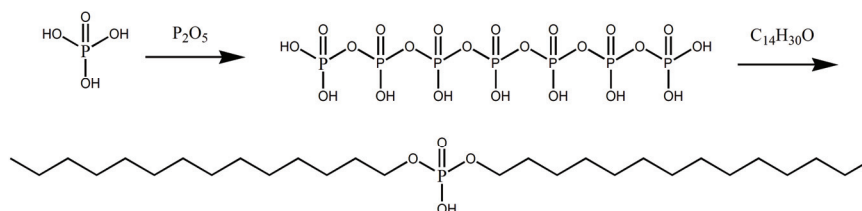The reaction scheme for the polyphosphate method is given in Fig. 2.

Fig. 2. Scheme of the polyphosphate method using *n*-tetradecanol as an example.

The purified products were then used as gelatinizers to prepare fracturing fluids, and the relationship between the ester chain length and the viscosity of the oil-based fracturing fluid was studied.

RESULTS AND DISCUSSION

Formation of monoethyl phosphate

Table I shows the orthogonal experimental factors for the preparation of monoethyl phosphate are given in Table I. The results of the orthogonal experiment are given in Table II.

TABLE I. Orthogonal experimental factors for the preparation of monoethylphosphate

Level	A, Reactant ratio[a]	B, t / °C	C, τ / h
1	1:1	70	4
2	1.25:1	80	5
3	1.5:1	90	6

[a]The reactant ratio is the ratio of ethylphosphate to phosphorus pentoxide

TABLE II. The result the orthogonal experiment. The factors and levels are from Table I

Experiment No.	A	B	C	Conversion rate, %
1	1	1	3	7.59
2	2	1	1	5.98
3	3	1	2	3.57
4	1	2	2	4.77
5	2	2	3	42.43
6	3	2	1	3.81
7	1	3	1	56.00
8	2	3	2	16.86
9	3	3	3	11.20
$K1$[a] / %	68.36	17.14	65.79	–
$K2$ / %	65.27	51.01	25.20	–
$K3$ / %	18.58	84.06	61.22	–
R[b] / %	49.78	66.92	40.59	–

[a]$K1$–$K3$ are the sums of the conversion rates under levels 1–3, respectively, [b]R is the difference between the largest number and smallest number of $K1$, $K2$ and $K3$ for each factor A–C, respectively

The results show that the main factor affecting this reaction is the reaction temperature (reaction temperature > reactant ratio > reaction time; Tables I and II). The optimum reaction conditions are $A_1B_3C_1$, namely, the mole ratio of $(CH_3CH_2O)_3PO$ to P_2O_5 is 1:1, reaction temperature is 90 °C and reaction time is 4 h.

Synthesis of dialkyl phosphate with 1-butanol by the improved ethyl phosphate method

The orthogonal experimental factors for the preparation of dialkylphosphate with 1-butanol are given in Table III. The conversion rates and dialkylphosphate ratios from the orthogonal experiment are given Table IV.

TABLE III. Orthogonal experimental factors for the preparation of dialkyl phosphate with 1-butanol

Level	A, Reactant ratio[a]	B, t / °C	C, τ / h
1	2:1	70	5
2	3:1	80	6
3	4:1	90	7

[a]The reactant ratio is the ratio of 1-butanol to phosphorus pentoxide

TABLE IV. The conversion rate and dialkyl phosphate ratio obtained in the orthogonal experiment. The levels and factors are defined in Table III

Experiment No.	A	B	C	Conversion rate, %	Dialkyl phosphate ratio, %
1	1	1	3	147.1	77.12
2	2	1	1	133.9	65.15
3	3	1	2	136.6	65.83
4	1	2	2	115.2	55.95
5	2	2	3	145.2	75.54
6	3	2	1	102.7	25.34
7	1	3	1	131.8	61.55
8	2	3	2	146.4	75.56
9	3	3	3	149.0	77.73
$K1$[a] / %	394.1	417.6	368.4	–	–
$K2$ / %	425.5	363.1	398.2	–	–
$K3$ / %	388.3	427.2	441.3	–	–
R[b] / %	37.2	64.1	72.9	–	–

[a]$K1$–$K3$ are the sums of the conversion rates under levels 1–3, respectively, [b]R is the difference between the largest number and smallest number of $K1$, $K2$ and $K3$ for each factor A–C, respectively

The results from Tables III and IV show that the main factor affecting this reaction is the reaction time (reaction time > reaction temperature > reactant ratio). The optimum reaction conditions are $A_2B_3C_3$, namely, the mole ratio of 1-butanol to P_2O_5 is 3:1, reaction temperature is 90 °C and reaction time is 7 h.

Experiment results of synthesis dialkyl phosphate with different alcohols by the improved ethyl phosphate method

According to the synthesis of dialkyl phosphate with 1-butanol, the same experimental procedure can be used to obtain the optimum reaction conditions for 1-hexanol, 1-octanol, 1-nonanol, 1-tridecanol, 1-tetradecanol and 1-octadecanol as the alcohol (shown in Table V) and the products produced under the optimum reaction conditions were used to prepare fracturing fluid. The relationship between the length of the ester chain and the viscosity of the oil-based fracturing fluid is given in Table VI.

The optimum synthesis conditions of monoethyl phosphate (shown in Table II) are mole ratio of triethyl phosphate to phosphorus pentoxide, 1:1, reaction temperature of 90 °C and reaction time of 4 h. Under these conditions, the yield

monoethyl phosphate was 56.00 %. Dialkyl phosphate was synthesized through this monoethyl phosphate. The optimum reaction conditions (shown in Table IV) were mole ratio of 1-butanol to phosphorus pentoxide, 3:1, reaction temperature 90 °C and reaction time of 7 h. The yield of dialkylphosphate reaches 83.25 %. After preparing these fracturing fluids, the viscosity could be measured and the results given in Table VI indicate that the viscosity of fracturing fluid increased with increasing length of dialkylphosphate ester chain. The addition of dialkyl phosphate increased the viscosity of fracturing fluid because the basing fluid was coated by a combination of the dehydrogenating dialkyl phosphate with Al^{3+} and then a three-dimensional network structure of the gel was formed. With increasing ester chain length, the viscosity increased maybe because the long ester chain could be wrapped with the coated base fluid molecules thereby making the coat more solid. The obtained result is in accordance with the results of Funkhouser.[16] It may also be because long ester chain is conducive for the formation of an overlap with another long ester chain from the coating molecule. It makes the gel group larger, which results in higher viscosity.[17]

TABLE V. Optimal reaction conditions for the synthesis of dialkyl phosphate with different alcohols

Syn-thesis	1-Butanol	1-Hexanol	1-Octanol	1-Nonanol	1-Tridec-anol	1-Tetradec-anol	1-Octadec-anol
A	3:1	2:1	4:1	4:1	4:1	3:1	3:1
B	90	90	90	90	90	90	90
C	7	6	7	6	7	7	7

TABLE VI. The viscosity of the oil-based fracturing fluid

Factor	1-Butanol	1-Hexanol	1-Octanol	1-Nonanol	1-Tridec-anol	1-Tetra-decanol	1-Octa-decanol
μ / mPa·s	33.7	41.2	65.7	72.1	134.4	147.9	150.7

Synthesis of dialkyl phosphate with 1-butanol by the polyphosphate method

The orthogonal experimental factors for the preparation of dialkyl phosphate from 1-butanol and polyphosphate are given in Table VII. Table 8 shows the results of the orthogonal experiment are presented in Table VIII.

TABLE VII. Orthogonal experimental factors for preparation of dialkyl phosphate from 1-butanol and polyphosphate

Level	A, Reactant ratio[a]	B, t / °C	C, τ / h
1	2:1	85	6
2	3:1	90	7
3	4:1	95	8

[a]The reactant ratio is the ratio of 1-butanol to phosphorus pentoxide

TABLE VIII. The results of the orthogonal experiment

Experiment No.	A	B	C	Conversion rate, %
1	1	1	3	11.36
2	2	1	1	25.05
3	3	1	2	36.89
4	1	2	2	38.62
5	2	2	3	45.10
6	3	2	1	52.77
7	1	3	1	17.25
8	2	3	2	34.44
9	3	3	3	43.13
$K1^a$ / %	67.23	73.30	95.07	–
$K2$ / %	104.59	136.49	109.95	–
$K3$ / %	132.79	94.82	99.59	–
R^b / %	65.56	63.19	14.88	–

[a]$K1$–$K3$ are the sums of the conversion rates under levels 1–3, respectively, [b]R is the difference between the largest number and smallest number of $K1$, $K2$ and $K3$ for each factor A–C, respectively

The results from Tables VII and VIII show that the main factor affecting this reaction was the reaction ratio (reactant ratio > reaction temperature > reaction time). The optimum reaction conditions was $A_3B_2C_2$, a mole ratio of 1-butanol to P_2O_5 of 4:1, a reaction temperature of 90 °C and a reaction time of 7 h.

Experimental results for the synthesis of dialkyl phosphates with different alcohols by the polyphosphate method

According to the results of the synthesis of dialkylphosphate with 1-butanol, the same experiment procedure was used to obtain the optimum reaction conditions for 1-hexanol, 1-octanol, 1-nonanol, 1-tridecanol, 1-tetradecanol and 1-octadecanol (shown in Table IX), and the purified products were used to prepare fracturing fluids. The relationship between the length of the ester chain and the viscosity of the oil-based fracturing fluid is presented in Table X.

As can be seen from Table VIII, the optimum synthesis conditions of dibutyl phosphate were a mole ratio of 1-butanol to phosphorus pentoxide of 4:1, a reaction temperature of 90 °C and a reaction time of 7 h. Under these conditions, the yield of the dibutyl phosphate reached 55.32 %. Moreover, the viscosity of the fracturing fluid increased with the increasing length of dialkyl phosphate ester chain. Furthermore the tendency of the polyphosphate methods was similar to that of the improved ethyl phosphate method, with the viscosity of the product improving by 7.72–45.35 % of the level of the improved ethyl phosphate method (Fig. 3).This may be due to the increase of ester chain length that not only enhanced the wrap with the coated base fluid molecules but also enabled overlapping with other molecules.

TABLE IX. Optimal reaction conditions for the synthesis of dialkyl phosphate with different alcohols

Synthesis	1-Butanol	1-Hexanol	1-Octanol	1-Nonanol	1-Tridec-anol	1-Tetradec-anol	1-Octadec-anol
A	4:1	4:1	4:1	4:1	4:1	4:1	4:1
B	90	85	85	85	90	85	90
C	7	8	8	8	7	7	8

TABLE X. The viscosity of the oil-based fracturing fluids

Factor	1-Butanol	1-Hexanol	1-Octanol	1-Nonanol	1-Tridec-anol	1-Tetra-decanol	1-Octa-decanol
μ / mPa·s	36.3	52.2	87.4	104.8	181.48	194.08	200.03
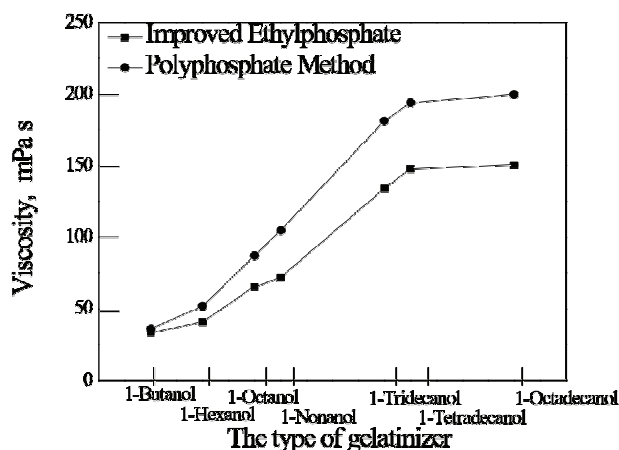
Fig. 3. The viscosity of the products from the two synthetic methods.

Infrared spectroscopy (IR) analysis

The IR spectrum of monoethyl phosphate is shown in Fig. 4. The band at 2993.9 cm^{-1} is typical for a carbon chain, the one at 2325.2 cm^{-1} is due to O=P–OH groups, while the bands at 1182.3 and 1103.7 cm^{-1} arise from the asymmetric and symmetric stretching vibrations of the P–O–C group, and 789.9 cm^{-1} is the characteristic peak of carbon chain –$(CH_2)_n$–.[19] The IR spectrum of C_{14} dialkyl phosphate, used as an example, is also shown in Fig. 4. In this spectrum, the bands at 2919.4 and 2986.9 cm^{-1} are characteristic peaks of the carbon chain; the band at 2352.1 cm^{-1} arises from the O=P–OH groups, the bands at 1210.1 and 1068.4 cm^{-1} are due to the asymmetric and symmetric stretching vibrations of the P–O–C groups, and that at 725.1 cm^{-1} is the characteristic for the carbon chains –$(CH_2)_n$–. These IR spectra show the different positions of the –$(CH_2)_n$– bands for different lengths of the –$(CH_2)_n$– group. It could be concluded that the former arises from –CH_2– and the latter from –$(CH_2)_n$– ($n > 4$).

Thus, it is obvious that the conversion of the monoethyl phosphate to dialkyl phosphate had occurred.
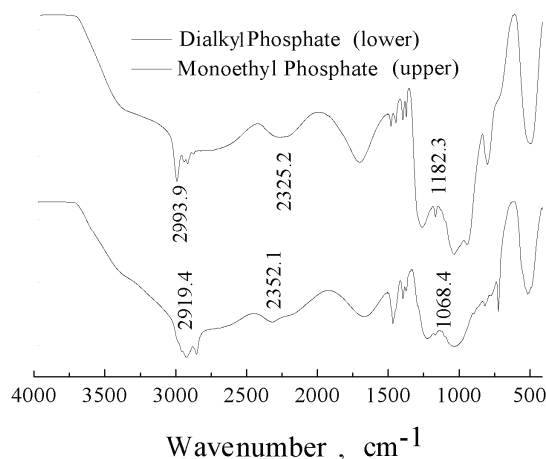

Fig. 4. IR spectra of monoethyl phosphate and C14 dialkyl phosphate.

CONCLUSIONS

The performance of fracturing fluid can be significantly improved through the polyphosphate method compared with the improved ethyl phosphate method while more alcohol and longer reaction times are needed for the former method. The viscosity of fracturing fluid increased with increasing length of the dialkyl phosphate ester chain.

И З В О Д
ИЗУЧАВАЊЕ УТИЦАЈА ДУЖИНЕ ЛАНАЦА ДИАЛКИЛ-ФОСФАТНИХ ЕСТАРА НА ВИСКОЗНОСТ ТЕЧНОСТИ ЗА ХИДРАУЛИЧНО РАЗБИЈАЊЕ („FRACKING")
НА БАЗИ УЉА

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За синтезу диалкил-фосфата, који се користе као угушћивачи течности на бази уља за хидраулично разбијање ("fracking"), примењена су два поступка. Први, побољшани етил-фосфатни поступак – омогућава контролу само једног естарског ланаца, заменом једне ОН групе у моноетил-фосфату, док други, нови полифосфатни поступак, омогућава контролу оба естарска ланца. Различите врсте алкохола су се користиле као сиро-

вине у 3×3 ортогоналном дизајну експеримента (однос реактаната, температура и време одигравања реакције) да би се установили оптимални услови извођења реакције. Као резултат ових експеримената, добијено је 14 различитих врста угушћивача, који су поређени на основу вискозности. Показано је да угушћивачи синтетисани на оба начина испољавају исти тренд, тј. да дужина ланаца диалкил-фосфатних естара утиче на пораст вискозности течности за хидрулично разбијање до одређеног броја угљеникових атома. Другим речима, вискозност флуида се повећава са порастом дужине естарских ланаца и достиже максимум на C_{13}, а након тога опада. Вискозност производа на бази диалкил--фосфатних естара добијених полифосфатним поступком је била 7,7–45,3 % већа у односу на аналогне производе добијене побољшаним етил-фосфатним поступком.

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