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ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
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Қазақстан Республикасы Ұлттық ғылым академиясы «ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы» ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бұл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруда. Webof Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді геология және техникалық ғылымдар бойынша контентке адалдығымызды білдіреді.

НАН РК сообщает, что научный журнал «Известия НАН РК. Серия геологии и технических наук» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science. Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Social Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество и глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК. Серия геологии и технических наук в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по геологии и техническим наукам для нашего сообщества.

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RESULTS OF LABORATORY STUDIES OF ACID TREATMENT OF LOW-PERMEABILITY ROCK CORES

Abstract. To improve production in oil and gas wells, methods of stimulating the bottomhole zone of the well have been used for a long time. One of these methods is an acid stimulation, which is currently the most widely used in the petroleum industry. This study is conducted to investigate the dissolution patterns during passing of hydrochloric acid through the core samples oflow permeability reservoir of Kazakhstani field. This paper presents the results of a series of acid treatment tests performed on 1.5-inches core samples, a description of laboratory methods, procedures and concluding remarks regarding the experiments. Six different carbonate core samples were tested. The test program consisted of several pumping experiments with hydrochloric acid solution. The initial porosity of the tested samples varied from 1.5% to 19.2%, the initial gas permeability varied from 0.009 to 85.7 m D.Experiments have determined that acid treatment was ineffective for too low permeability core samples. At low permeability values, the acid could not penetrate into the core and form "wormholes", therefore the injection rate parameter was changed and in some cases the so-called "face" of the sample dissolution effect appeared, in which the acid affected only the surface of the core sample.From this it was concluded that the acid injection rate strongly affects the wormhole formation process.

Key words: oil production stimulation, acid treatment, carbonate rock, core sample, low permeability, injection rate, wormhole formation.

Introduction. Acid treatment is currently the most widely used method of oil production stimulation in the petroleum industry [1-8]. Acid treatment is carried out by injecting acid solutions into the formation to dissolve rock minerals in order to expand existing and create new fractures, increase the overall permeability, and therefore the productivity of the well. Several types and combinations of acids can be used for acidizing, but the most commonly used acids are hydrochloric acid (HCl) and hydrofluoric acid (HF) [9-12]. HCl reacts with carbonate rocks such as dolomite, calcite and siderite. HF is used to dissolve clastic rocks and reacts with clay minerals, feldspars and quartz.

According to authors of [13-14] oil and gas companies operating carbonate oil and gas condensate fields in Kazakhstan have been carrying out acid stimulation activities leading to a substantial increase in hydrocarbon production. A comprehensive review of historical treatments on several fields located in West-Kazakhstan region was performed to identify areas to improve post-stimulation well performance. Historical treatments in the oil field typically used straight hydrochloric acid as the main acid, polymer-gelled (self-diverting) acid as the chemical diverter, and linear guar gel for displacement, and diagnostic tests. Positive results of the laboratory studies, treatment modeling, and field trials were validated by the increasing normalized post-stimulation PI (productivity index) with each optimization step.

A modified acidizing system including acid jetting is proposed by the authors of [15-16]. Laboratory linear core flooding experiments and acid jetting experiments were conducted to study the wormhole efficiency with the new acid systems. The experimental results showed clear advantages of the modified acid systems. The modified acids have similar or better wormhole efficiency parameters compared with HCl having comparable dissolving power. When combined with acid jetting, further improvement in wormhole growth in low permeability limestone was achieved.

Two methods thatconsidered advantages and disadvantages of chemical treatment of a formation and the method of perforation with the use of the thermo-gas-cumulative effect are analyzed by [17].

Near wellbore formation damages have a great impact on productivity of the damaged formation. Combined skin factor approach, which takes into account the complexity of near wellbore damage zone and its modification during acid stimulation is presented by authors of [18-21]. Combined skin factor is introduced that takes into account the present damage subzones with different permeability, perforation channels, wormholes, relative permeability and viscosities of pumped fluids and their changes during acidizing process. In those works acidizing is a stimulation method to remove the effect of near wellbore damage through reacting with damaging materials or the formation rocks (carbonate or sandstone) to restore or improve permeability around the wellbore. Captured pores before treatment and captured pores after thermal-HCL acid treatment have demonstrated that image processing of the actual acidized rock data can select the optimized recipe concentration of acid that will increase permeability, hence recovery.

The volume of acid composition required to create a highly conductive channel in a core sample is a parameter which considered by [22]. A number of factors affects it. To make the most of the oil recovery enhancement, the integrated indicator including lithological, mineralogical and flow properties of the reservoir, as well as technological parameters of acid injection, shall be taken into account.

This study is conducted to investigate the dissolution patterns during passing of hydrochloric acid through the core samples from tight reservoirs of Kazakhstani field. This paper presents the results of acid treatment tests performed on 1.5-inches core samples, a description of laboratory methods, procedures and concluding remarks regarding the experiments.

Core Sample and Injection Fluid Preparation. Core samples were taken based on visual integrity and petrophysical properties. Broken and fractured core samples were removed from the collection.

Selected samples were washed under the influence of solvents in Soxhlet extractors to extract oil, water and salt. Toluene was used to recover hydrocarbons. Cleaning was carried out until the solvent ceased to change color. Then toluene was replaced with methanol to extract salts from the samples. This sequence was then repeated with a chloroform-methanol and methanol azeotrope. After cleaning with methanol, silver nitrate was added to the effluent to check for salt content. The purification was considered complete after no salts were detected by the addition of the silver nitrate solution. Then, the samples were dried to constant weight in a drying oven at the temperature of 105°C. Each sample was then cooled to outside temperature in a glass desiccant prior to analysis.

The gas permeability of standard 1.5-inches samples was measured by a calibrated permeameter with dry nitrogen fluid under steady state conditions. The gas flow and the pressure drop in the direction of flow in the core sample were measured under steady-state conditions. The overburden pressure used during measurements was 30 bar.

The measurement of the grain volume of the core samples was carried out using a calibrated beaker, in which the volumetric expansion of helium occurs. Before measuring each successive batch (maximum 20 samples), the porosimeter was checked for leaks and quality control was carried out on standard samples. The apparatus was calibrated using five stainless steel discs of known volumes and calculating the linear relationship between pressure and volume. A calibration value of 0.9999 is considered acceptable (1 = normal).

As a part of the standard core analysis, gas permeability, porosity and grain density were measured for all samples. The main properties of the core samples are presented in Table 1 and Figure 1.

Table 1.Description of the core samples

Sample #	Length, cm	Diameter, cm	Porosity, %	Permeability by air, mD	Permeability by water, mD
23	4.549	3.799	6.724	0.015	0.0021
25	4.611	3.798	11.863	16.784	0.7225
28A	4.695	3.691	12.651	8.955	0.8516
44	5.141	3.803	1.522	0.014	0.0016
54ds	5.136	3.796	19.249	85.713	39.6
55	4.928	3.803	4.375	0.009	0.0015

The mineralogical composition of the rock samples was determined using XRD analysis.The results are shown in the Table 2.

Table 2. Mineral composition of the rock

Sample #	**Depth, m**	**Shale**	**Feldspar**		**Carbonates**		**Other minerals**
		Hydromica (Illite)	**Potassic feldspar**	**Plagioclase (Albite)**	**Calcite**	**Dolomite**	**Quartz**
23	3383.09	0.00%	0.00%	2.00%	0.00%	84.90%	13.10%
25	3385.99	0.40%	0.00%	1.10%	0.00%	96.00%	2.50%
28A	3386.81	0.00%	0.00%	0.00%	0.00%	98.30%	1.70%
44	3321.79	0.00%	0.00%	0.00%	83.50%	7.40%	9.10%
54ds	3384.35	0.00%	0.00%	0.00%	2.40%	92.80%	4.80%
55	3384.98	0.00%	0.00%	0.00%	73.30%	25.70%	1.00%

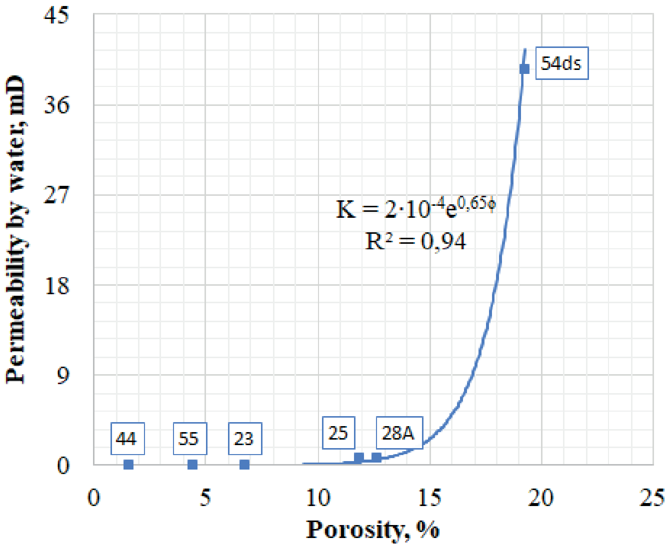


Figure 1.Permeability vs. porosity for considered core samples

During the test, the following fluids were selected:

a) Produced water. A 2% aqueous solution of KCl was filtered through a 0.45 μm filter and used for initial saturation of the core samples.

b) 15% HCl solution.

The density and viscosity of all injected fluids were measured under test conditions using a Cambridge viscosity electromagnetic viscometer and an Anton-Paar density meter.

A Teledyne ISCO 260D pump was used during pumping (Figure 2). This piston pump has a wide range of chemical pumping applications requiring flow rates up to 107 ml/min at pressures up to 7500 psi (517 bar). The 266 ml cylinder capacity allows accurate delivery at 1 ml/min for over 4 hours with a single filling.

Produced water and acid displacement experiments were carried out using 1000 ml cylindrical accumulators to prevent damage to the pump. They are made of corrosion-resistant steel Hastelloy C276 and can be used at high pressures of 1000 bar and temperatures up to 300°C (Figure 2).

The accumulator is equipped with a special piston that can move freely inside the cylinder and does not allow the injected fluid to mix with the pumped fluid. There are 1/8” inlet and outlet ports with needle valves and additional ports for connecting pressure gauges. In addition, a dedicated mixing ball is available to help homogenize and mix the injected fluid during testing.

During the test, the core holders are placed in a special oven to maintain a high temperature. The oven is protected from heat leaks and can operate at different temperatures up to 400°C.

Experimental procedure. The experiments are monitored in real time and all process parameters are recorded: fluid flow rate, temperature and pressure drop along the sample.

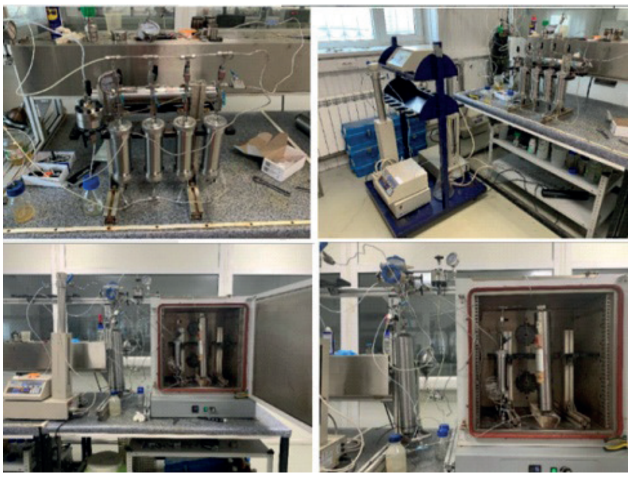


Figure 2. Flooding unit

Acid Treatment Experiment Procedure:

1. Horizontal core samples with a diameter of 1.5-inches are cleaned from hydrocarbons in a hot Soxhlet with toluene and methanol to remove salts. Next, the samples are dried in an oven at 105 °C until stable weight. After drying is complete, porosity and gas permeability are measured.
2. Core samples are completely saturated with filtered 2% KCl solution using vacuum. The core samples and the solution are evacuated in separate chambers for several hours, and then the solution is pumped into the core samples. After saturation, the samples are weighed and stored in the brine in sealed containers.
3. The core samples are loaded into hydrostatic core holders and placed in a furnace at a overburden pressure of 350 bar and water permeability is measured at a 4 different flow rates.
4. The core holders are placed in a furnace at 127°C and wait until the temperature stabilizes for at least 4 hours while maintaining a constant pore pressure.
5. After equilibration of the temperature, 1 PV of hot brine is pumped into the core sample at 10 bar back pressure.
6. After equilibration of pressure, acid is pumped into the core sample at a different flow rate. Acid pumping continues up to an allowable pressure of 200 bar or if the pumping time exceeds 1 hour. Differential pressure and flow rates are recorded during the experiment.
7. The acid is displaced with hot brine and the permeability is measured.
8. The sequence of fluid injection and permeability measurements is repeated for each cycle.
9. At the end of the cycle, the furnace is turned off and the samples are cooled down at the pore pressure of 10 bar.
10. Water permeability is measured at 4 different flow rates at room temperature.
11. Samples are unloaded from core holders and photographed after the experiment.
12. Core samples are cleaned from hydrocarbons in hot Soxhlet with toluene and methanol to remove salts. Next, the samples are dried in a furnace at the temperature of 105°C until stable weight.

Results and discussions. Six different carbonate core samples were selected. The test program consisted of several injection experiments with hydrochloric acid solution. The initial porosity of the tested samples varied from 1.5% to 19.2%, the initial Klinkenberg gas permeability varied from 0.009 to 85.7 mD.

Sample #23. Based on the low porosity (6.7%) and permeability (0.015 mD), it was assumed that acid injection into the sample would be difficult, and it was decided to pump a 15% HCl solution to dissolve the carbonates, and possibly to form wormholes.

Injection of a 15% HCl solution at high temperature (127°C) and pressure did not improve the permeability of the formation as the pressure reached its maximum (120 bar), but no production was observed. The pressure was maintained at this level for at least 4 hours, after which the test was completed (Figure 3).

Sample #25. During visual inspection, it was noted that the sample contains very small cavities.

The initial porosity and initial gas permeability of the sample are 11.863% and 16.784 mD, respectively. Water permeability is significantly lower (0.7225 mD) due to the low overburden pressure during gas permeability measurements (30 bar from 350 bar).

Injection of a 15% HCl solution at high temperature (127°C) and pressure did not improve the permeability of the formation as the pressure reached its maximum (120 bar), but no production was observed. The pressure was maintained at this level for at least 4 hours, after which the test was completed (Figure 3).

Sample #28A. During visual inspection, it was noted that the sample contains very small cavities.The initial porosity and initial gas permeability of the sample are 12.651% and 8.955 mD, respectively. The water permeability is significantly lower (0.8516 mD) due to the low overburden pressure during the gas permeability measurement (30 bar from 350 bar).

Sample #28A was tested twice. The first time, 15% HCl acid was pumped at the rate of 0.5 ml/min, but after almost 4 hours of testing, the acid did not enter the sample and the test had to be completed. A plaque was found on the inlet end of the sample, which presumably prevented acid from entering the sample.

The second time the acid was pumped at the rate of 5 ml/min, and after 4 minutes a breakthrough occurred (Figure 3).

Sample #44. During visual inspection, it was noted that the sample does not darken visible cavities, which is confirmed by its low porosity.

The initial porosity and initial gas permeability of the sample are 1.5% and 0.014 mD, respectively. The water permeability is significantly lower (0.0016 mD) due to the low overburden pressure during gas permeability measurements (30 bar from 350 bar).

Injection of a 15% HCl solution at high temperature (127°C) and pressure did not improve the permeability of the formation as the pressure reached its maximum (120 bar), but no production was observed. The pressure was maintained at this level for at least 4 hours, after which the test was completed (Figure 3).

Sample #54ds. During visual inspection, it was noted that the sample contains medium-sized cavities.The initial porosity and initial gas permeability of the sample are 19.2% and 85.7 mD, respectively. The water permeability is slightly lower (39.6 mD) due to the low overburden pressure during the gas permeability measurement (30 bar from 350 bar).

On this sample, it was decided to pump a 15% HCl solution. The acid was pumped at 127°C and the flow rate of 2 ml/min. After 40 minutes of the test, the pressure dropped sharply which could indicate of breakthrough occuring and wormhole propagation, and after another 20 minutes the test was completed (Figure 3).

Sample #55. During visual inspection, it was noted that the sample had no visible cavities, which was confirmed by its low porosity.

The initial porosity and initial gas permeability of the sample are 4.4% and 0.009 mD, respectively. The water permeability is significantly lower (0.0015 mD) due to the low overburden pressure during the gas permeability measurement (30 bar from 350 bar).

Injection of a 15% HCl solution at high temperature (127°C) and pressure did not improve the permeability of the formation as the pressure reached its maximum (120 bar), but no breakthrough was observed. The pressure was maintained at this level for at least 4 hours, after which the test was completed (Figure 3).

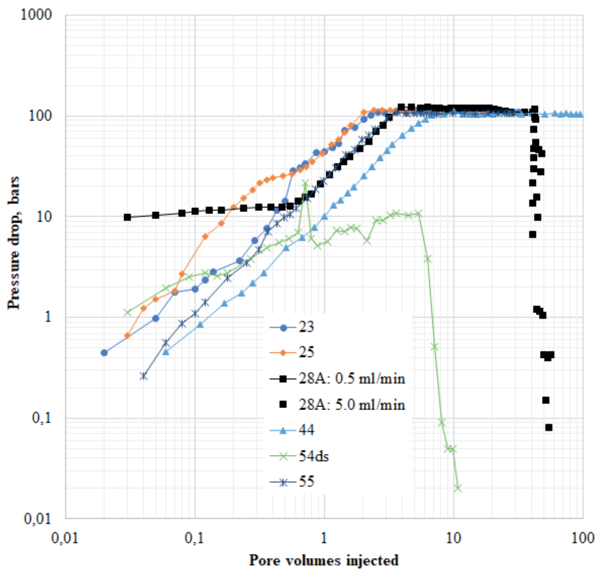


Figure 3.Pressure drop for all test cases

Dissolution pattern analysis

Sample #23 was unloaded and examined, no wormholes were found, which corresponds to its very low permeability after the treatment (Figure 4).

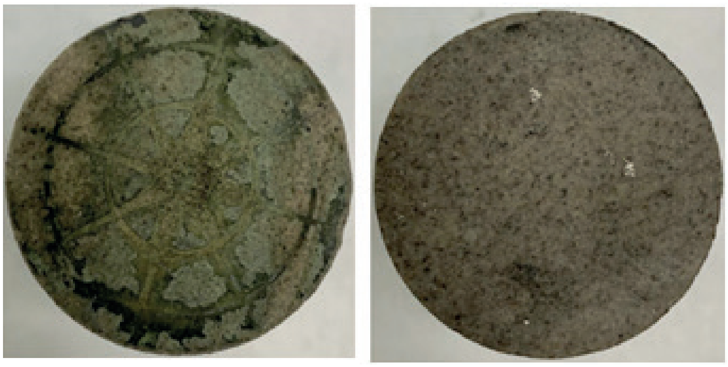

(a) (b)

Figure 4. Core sample #23 after acid injectiontest: (a) inlet; (b) outlet

Sample #25 was unloaded and examined, no wormholes were found, which corresponds to its very low permeability after the treatment. A plaque was found on the inlet end of the sample, which presumably prevented acid from entering the sample (Figure 5).

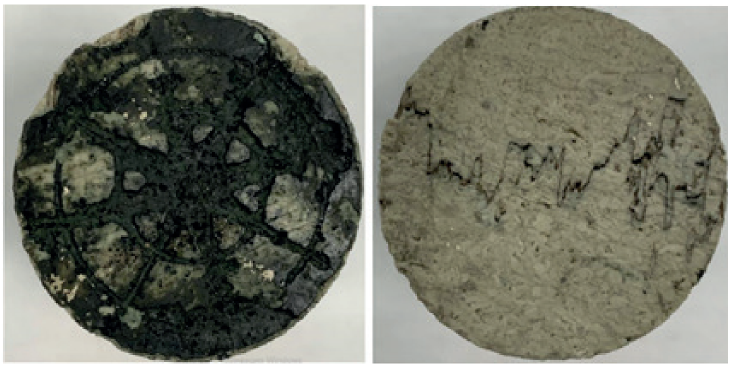

(a) (b)

Figure 5. Core sample #25 after acid injectiontest: (a) inlet; (b) outlet

After unloading the sample, a visual examination of the sample was performed, which confirmed the presence and propagation of wormholes, as well as partial face dissolution of the core (Figure 6).

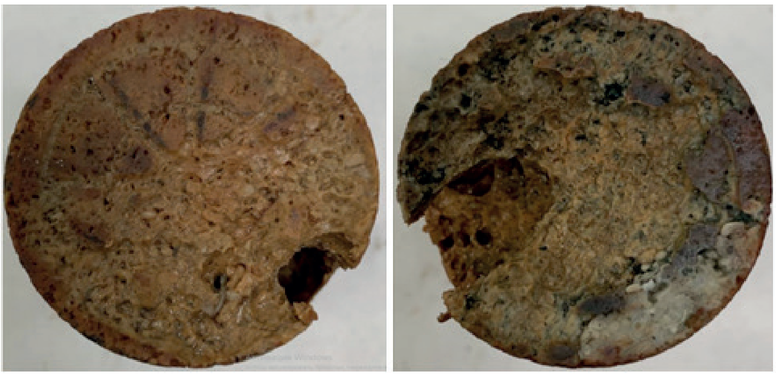

(a) (b)

Figure 6. Core sample #28A after acid injectiontest: (a) inlet; (b) outlet

Sample #44 was unloaded and examined, no wormholes were found, which corresponds to its very low permeability after treatment (Figure 7).

(a) (b)

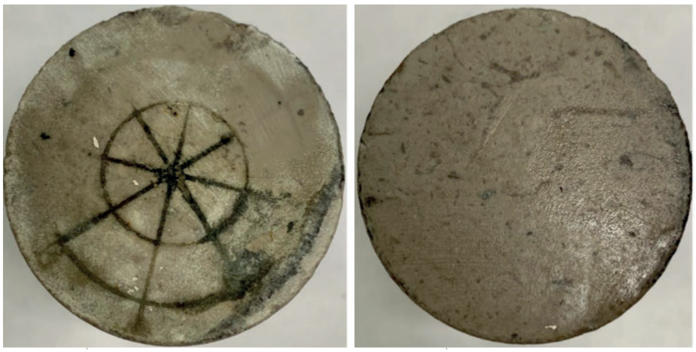
Figure 7. Core sample #44 after acid injectiontest: (a) inlet; (b) outlet

After pumping 15% HCl, it was impossible to measure the permeability due to deformation. After visual inspection of the state of the sample, the presence of wormholes and the dissolution of the end side of the sample were revealed (Figure 8).

(a) (b)

Figure 8. Core sample #54ds after acid injectiontest: (a) inlet; (b) outlet

Sample #55 was unloaded and examined, no wormholes were found, which corresponds to its very low permeability after treatment (Figure 9).



(a) (b)

Figure 9. Core sample #55 after acid injection test: (a) inlet; (b) outlet

Conclusion. The acid treatment experiments resulted in the following conclusions:

1. Acid treatment was effective for samples #28A, #54ds using 15% HCl acid. For core sample #28A, increasing the acid injection rate to 5 ml/min resulted in acid breakthrough. After that, the formation of

wormholes and partial dissolution of the core face were observed. Core sample #54ds had a relatively high gas permeability (85.713 mD), so acid breakthrough was expected.Hence, it can be concluded that the property of gas permeability is significant and crucial when selecting an object for acid treatment.

2. Since the core samples had low permeability (0.0009-16.784 mD), acidizing experiments was ineffective for samples #23, #25, #44 and #55. So the permeability of the samples was very low for fluid penetration into the sample and efficient matrix dissolution.

3. Samples #44 and #55 predominantly consisted of calcite, the reaction rate of which with hydrochloric acid is much higher compared to dolomite. But this did not affect the outcome of the experiment - hydrochloric acid was ineffective, due to low permeability of core samples. The low permeability does not allow the acid to enter the sample.The acid was unable to "pierce" the sample and make wormholes, but the so-called "face dissolution" effect appeared, in which the acid affected only the face of the core sample.

4. Samples #25 and #28A were predominantly composed of dolomite, the reaction rate of which is much slower compared to calcite, these samples were taken from close depths and represent the same lithology. These samples were tested twice. For the first time, acid was pumped at the rate of 0.5 ml/min into sample #25 and at the rate of 1 ml/min into sample #28A. In the process of acid injection, the face of the core was clogged with reaction products, for this reason the acid could not propagate further and form wormholes. The second time, it was decided to change the injection parameters for sample #28A (the injection rate was increased to 5 ml/min) and leave the injection rate the same for sample #25. During the test of sample #25, a blockage occurred on the face, and when testing sample #28A, a breakthrough and wormhole formation occured. At the low flow rate, the reaction products of the acid with carbonate do not have time to be carried out with the flow and clog the face of the sample. From this it was concluded that the pumping rate influenced the wormhole formation process.

5. Core sample #54ds consisted predominantly of dolomite and had very good porosity and permeability. Sample permeability to 2% KCl solution before acid injection was 39.6 mD. Acid injection was carried out at the flow of 2 ml/min and the breakdown occurred after 30 minutes. After the test, a detailed examination of the sample was carried out, which showed that a through thickness wormhole had formed in the core, and also the effect of "end dissolution" appeared and the acid were dissolved part of the core face of the sample.

6. As a result of acid treatment studies, it can be concluded that the acid treatment efficiency largely depends on the injection rate, rock permeability, rock lithology and other factors. These results were obtained with 15% HCl injection.

7. For acid treatment studies, the core samples should be carefully selected by characteristics such as porosity, permeability, and mineral composition.

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ӨТКІЗГІШТІГІ ТӨМЕН ТАУ ЖЫНЫС ӨЗЕГІНДЕ ҚЫШҚЫЛМЕН ӨҢДЕУДІҢ ЗЕРТХАНАЛЫҚ ТӘЖІРИБЕЛЕР НӘТИЖЕЛЕРІ

Аннотация. Мұнай өндіруді арттыру үшін мұнай және газ ұңғымаларында ұңғыманың түп аймағына әсер ету әдістері көптен қолданылады. Осындай әдістердің бірі қышқылмен өңдеу, қазіргі кезде мұнай өндірісінде кеңінен қолданылып жүр. Бұл жұмыс қазақстандық кен орындағы төмен өткізгіштікті коллектордың жынысөзек үлгісі арқылы тұз қышқылының өтуі бойынша тау жынысының еру заңдылықтарын зерттеу үшін орындалған. Бұл мақалада 1,5-дюйм жынысөзек үлгісі арқылы қышқылмен өңдеудің тәжірибе нәтижелері, процедурасы, тәжірибе бойынша қорытынды нәтижелері ұсынылған. Сынақтар алты карбонатты жынысөзек үлгісінде орындалған. Тәжірибе процедурасы тұз қышқылы ерітіндісін айдаудың бірнеше тәсілдерінен тұрады. Зерттелетін жынысөзек үлгілерінің бастапқы кеуектілігі 1,5% -дан 19,2% -ке дейін, бастапқы газ өткізгіштігі - 0,009-дан 85,7 мД-ге дейін өзгереді. Тәжірибелер нәтижесінде өткізгіштігі төмен жынысөзек үлгілері үшін қышқылмен өңдеу тиімсіз екені анықталды. Өткізгіштіктің төмен мәндері үшін қышқыл жынысөзектің ішіне ене алмайды да, саңылаулар құра алмайды, сондықтан айдау жылдамдығының параметрі өзгертілді және кейбір жағдайларда қышқыл тек қана әрекет ететін жынысөзекүлгісінің «беттік» әсері пайда болды, бұл кезде қышқыл тек жынысөзек үлгісінің бетіне әсер етті.Осыдан қышқылды айдау жылдамдығы саңылаулардың қалыптастыру процесіне зор әсер етеді деген қорытындыжасауға болады.

Түйінді сөздер: мұнай өндіруді арттыру,қышқылмен өңдеу, карбонатты тау жынысы,жынысөзек үлгісі, төмен өткізгіштікті, айдау жылдамдығы, саңылаулардың түзілуі.

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РЕЗУЛЬТАТЫ ЛАБОРАТОРНЫХ ИССЛЕДОВАНИЙ КИСЛОТНОЙ ОБРАБОТКИ НА КЕРНАХ НИЗКОПРОНИЦАЕМЫХ ПОРОД

Аннотация. Методы воздействия на призабойную зону скважины уже давно применяются в нефтяных и газовых скважинах для увеличения добычи. Одним из таких методов является кислотная обработка, которая в настоящее время наиболее широко используется в нефтяной промышленности. Данное исследование проводилось для изучения закономерностей растворения породы при прохождении соляной кислоты через образцы керна низкопроницаемого коллектора казахстанского месторождения. В данной статье представлены результаты серии экспериментов по кислотной обработке, выполненные на 1,5-дюймовых образцах керна, описание лабораторных методов, процедур и заключительные замечания по экспериментам. Эксперименты проводились на шести различных образцах карбонатного керна. Процедура экспериментов состояла из нескольких подходов по закачке раствора соляной кислоты. Начальная пористость исследуемых образцов керна варьировала от 1,5% до 19,2%, начальная газопроницаемость – от 0,009 до 85,7 мД. В результате экспериментов было определено, что кислотная обработка неэффективна для образцов керна с низкой проницаемостью. При низких показателях проницаемости кислота не могла проникнутьв керн и образовать «червоточины», поэтому был изменен параметр скорости закачки и в некоторых случаях появился так называемый эффект растворения «лицевой части» образца, при котором кислота воздействовала только на поверхность образца керна. Отсюда был сделан вывод, что скорость закачки кислоты сильно влияет на процесс образования червоточин.

Ключевые слова: интенсификация добычи нефти,кислотная обработка, карбонатная порода, образец керна, низкая проницаемость, скорость закачки, образование червоточин.

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