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# Spontaneous imbibition experiments for enhanced oil recovery with silica nanosols

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**Abstract:**
Experimental oil displacement as a result of spontaneous imbibition of silica nanosols has been carried out using two types of sandstone as the reservoir rock. The permeability of the cores ranged from 0.34 to 333 mD, while the porosity was 11% and 22%, respectively. During the research, the influence of the concentration and nanoparticle size, as well as the permeability of the rock, on the process of spontaneous imbibition, was studied. Silica nanosols were considered as an object of study. The nanoparticle size ranged from 10 to 35 nm. The mass concentration of nanoparticles varied from 0.01% to 0.25%. It was found that the use of silica nanosols significantly increases the rate of the spontaneous imbibition process. It was established that a silica nanosol with a nanoparticle size of 10 nm and a concentration of 0.25% allows to displace more than six times oil compared to the reservoir water model in the same time. As a result, it was shown that the oil displacement efficiency and the efficiency of spontaneous imbibition increase along with an increase in the nanoparticle concentration and a decrease in the nanoparticle size.

## 1. Introduction

The recovery factor during oil field development does not exceed 50% of geological reserves, whereas it ranges from 2% to 10% in fields with hard-to-recover reserves, such as low-pore and low-permeability reservoirs containing high-viscosity oils. The development and industrial application of modern methods of enhanced oil recovery, which are able to provide a synergistic effect in the development of new and already developed oil fields, is a priority for the growth of the global crude oil production. Modern methods of enhanced oil recovery (Fink, 2021; Kang et al., 2022) comprise thermal (in-situ combustion, hot water injection, steam injection) (Kovscek, 2012), chemical (polymers, surfactants & foams, nanoparticles) (Gbadamosi et al., 2022), gas (carbon dioxide, nitrogen, hydrocarbon gas) (Phukan and Saha, 2022) and other methods (microbial (Alkan et al., 2019), hybrid (Hassan et al., 2020), acoustic (Marfin et al., 2022), and ultrasonic (Hamidi et al., 2021) methods).

Capillary imbibition plays the most important role within the framework of oil and gas production activities as a method of oil displacement from reservoirs with heterogeneous porosity or fractured porous ones (Wang et al., 2020; Cai et al., 2021). Spontaneous imbibition is the process of spontaneous liquid or gas displacement from a porous medium by another immiscible liquid under the action of capillary force.

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Capillary force is caused by surface phenomena occurring at the interface between a liquid and another medium. Such phenomena owe their appearance to the curvature of the surface of this liquid as a result of the emerging surface tensions. The force of gravity compensates for the effect of surface tension; therefore, the occurrence of such an effect for large masses of liquid is observed mainly in narrow channels. This phenomenon is instrumental in the displacement of oil and gas from heterogeneous porous and fractured-porous reservoirs (Meng et al., 2022).

Spontaneous imbibition is based on the phenomenon of capillarity and constitutes one of the qualitative methods for determining wettability. In order to carry out this process, an oil-saturated natural core is placed in a solution in a graduated cylinder for measuring the volume and velocity of the displaced oil (Feldmann et al., 2020; Sakthivel and Kanj, 2021; Sircar et al., 2022). This method does not allow for the fact that it is not only the wettability on which the imbibition rate depends, but also the relative permeability, viscosity, and surface tension, pore structure, as well as the initial water and oil saturation (Guo et al., 2021; Sakthivel and Kanj, 2021). It can be used to analyze the relative effect of displacing fluids on capillary forces (Xu et al., 2019). In addition, the process of capillary imbibition in formation is used as part of laboratory research to determine the wettability index of rocks, i.e., phenomena occurring at the interface of two phases, one of which is a solid and the second is a liquid or a gas.

In recent years, nanotechnologies (nanofluid flooding) alone or in combination with traditional methods of enhanced oil recovery have shown promise (Minakov et al., 2021b; Yakasai et al., 2021; Pryazhnikov et al., 2022; Goharzadeh et al., 2023; Shao et al., 2023). Nanotechnology is a new direction, that includes the creation of substances, devices, and systems using nanosized materials (1-100 nm) as well as phenomena occurring at the nanoscale. The addition of certain nanoparticles to flooding fluids is known to change the wettability and reduce interfacial tension (Minakov et al., 2020a), which, in turn, can significantly affect the increase in oil recovery. Nanofluids represent a new class of flooding fluids that will be applied as alternative methods for enhanced oil recovery and solve problems with residual oil in reservoirs that have existed for the last decade.

Currently, there are several studies devoted to the study of capillary imbibition with nanosuspensions. The influence of the addition of carbon nanodots on the process of capillary imbibition was studied (Sakthivel and Kanj, 2021). It was shown that the addition of nanodots at very low concentrations of 2,000 ppm increases the imbibition rate by 25% compared to formation water, which can be very promising for their application. Spontaneous imbibition tests of a nanofluid with hydrophilic carbon nanoparticles with the addition of Tween-80 are presented (Zhao et al., 2020). It was found that the extraction of oil during spontaneous imbibition of carbon nanoparticles nanofluid can reach 24%, whereas using the NaCl solution leads to 11%. Nuclear magnetic resonance was used to reveal the mechanism of nanofluid migration in the pore space. Also, the use of nuclear magnetic resonance technologies for characterizing pore sizes in dense sandstones in the study of forced and spontaneous imbibition is reflected in Cao et al. (2022). The results of the study showed that the recovery of oil with surfactants was higher than with brine, which is primarily due to the increased content of oil in mesopores and macropores. Compared to spontaneous imbibition, forced imbibition could increase the infiltration of water into micropores but prevent the extraction of oil from mesopores and macropores. In turn, led to a higher contribution of micropores to oil recovery than mesopores. In the forced imbibition, the driving forces include capillary force, gravity forces, and displacement force. Most of the injected water flows through the fracture, while only a small portion of the injected water enters the cores to displace the oil owing to the extremely low permeability. In forced imbibition, there is greater resistance to the displacement force and less open end area, so that mesopores and macropores may be blocked and oil trapped in them (Cao et al., 2022).

Dai et al. (2017) presented the results on the use of silicon dioxide nanoparticles modified with surfactants (vinyltriethoxysilane and 2-mercaptobenzimidazole). Approximately 38% of oil was recovered from the low permeability core (with gas permeability of 54 mD and porosity of 20%) using 0.1 wt% nanofluid after approximately 10 days. The results showed that modified silica nanoparticles have little effect on interfacial tension, but they can change the wettability of a surface from oil-wet to water-wet. Zhou et al. (2019) performed experiments on the spontaneous imbibition of a nanofluid based on a brine with silicon oxide nanoparticles into a core. They found that 70.8-72.0 wt.% of the oil was distributed in the mesopores. The final oil recovery from cores immersed in a nanofluid with 0.1 wt.% nanoparticles was 32.5%. The authors in their study used nuclear magnetic resonance to visualize the distribution of nanofluid and oil in a core in a spontaneous impregnation experiment.

Experiments on the spontaneous imbibition of active nanofluids have shown that the presence of active surface groups can effectively increase the oil recovery of ultra-low permeability cores (Li et al., 2017). The difference between conventional nanofluids and active nanofluids is that active nanofluids contain active surface groups. The interfacial activity of nanoparticles in active nanofluids is achieved by changing the shape of the surface groups of active silica nanoparticles to minimize their energy at the interface. Nuclear magnetic resonance images of cores immersed in nanofluid and brine at different stages of impregnation were provided. They showed that there was good transport of kerosene using nanofluid compared to brine.

Active silica nanoparticles obtained by condensation of hexanedioic acid with the -OH group of silica were used in the experiments. An active silica-based nanofluid at a low concentration can exhibit the same enhanced oil recovery efficiency as a highly concentrated conventional nanofluid. Later, Song et al. (2021) compared the efficiency of nanofluids based on active silicon dioxide and silicon dioxide nanofluids with the surfactant dodecyl sulfobetaine. Oil recovery during spontaneous imbibition of a surfactant solution was worse than that of active nanofluids due to a smaller effect on the change in wettability. In another work (Zhong et al., 2022), active nanofluids consisting of hydroxypropyl sulfobetaine with different carbon chain lengths were studied. Increasing the carbon chain length of the surfactant showed an adverse effect on the performance of the nanoparticles due to particle aggregation and settling.

A study of the spontaneous imbibition of nanofluids based on salt solutions with nanoparticles of different materials (MgO, $\gamma$-$Al_2O_3$, and $TiO_2$) into carbonate reservoirs is presented in Nowrouzi et al. (2022). The size of all the nanoparticles was close and amounted to 20 nm. The highest oil recovery was observed during spontaneous imbibition of nanofluids based on a solution of $MgSO_4$ salts and $\gamma$-$Al_2O_3$ nanoparticles. Studies of the effect of nanoparticle size are extremely rare. So in another work of the same authors (Nowrouzi et al., 2019), the results of the influence of the size and concentration of nanofluid with nanoparticles of titanium oxide based on sea water of different salinities on the parameters of enhanced oil recovery as a result of spontaneous imbibition into carbonate rock are presented. Experimental results show that using a higher concentration and smaller $TiO_2$ particle size reduces the interfacial tension and contact angle. Therefore, imbibition experiments show that nanofluids containing smaller titanium oxide nanoparticles are more effective.

In the work from Sobhani and Ghasemi Dehkordi (2019), silicon oxide nanoparticles with a primary particle size of 7 nm were used. The influence of the nanoparticle concentration on the efficiency of oil displacement during spontaneous imbibition of a nanofluid are presented. The maximum oil displacement efficiency was shown to be obtained at 0.05 wt.% $SiO_2$ nanoparticles. The results of using the nanofluid as the second absorbable liquid after the brine are also presented. The total recovery was shown to increase to 40%, which was less than the oil recovery amounted of 53% when using the nanofluid as the first absorbed liquid.

In the works (Zhao et al., 2018; Ali, 2022; Zhao et al., 2022) are worth noting as they consider nanofluids with nanoparticles stabilized with various chemical agents. Various acids (Ali, 2022) (for example, oleic acid, polyacrylic acid) can be used as such agents, as well as surfactants (for example, TX-100 (Kuang et al., 2018), and alpha olefin sulfonate (Rezaei et al., 2020), natural powders of surfactants obtained green way (Towler et al., 2017; Zhao et al., 2022). Chemically stabilized nanofluids can have good thermal stability, salt resistance, and colloidal stability. The results of experiments (Zhang et al., 2022) showed that various surfactants (anionic-nonionic, anionic, nonionic, and amphoteric) differently affected the efficiency of imbibition of stable nanofluids. Due to the synergistic effect between nanoparticles and surfactants, nanofluids have a better ability to change wettability and separate the oil drop from the rock surface than pure surfactant solutions. The mechanism of spontaneous nanofluid imbibition can be considered a synergistic effect between silica nanoparticles and surfactant. However, in this case, it is difficult to distinguish the contribution of nanoparticles and surfactants to the efficiency of oil displacement.

In addition to experimental studies of the spontaneous imbibition of nanofluids to improve oil recovery, there are works that actively use mathematical modeling (Cai et al., 2014; Khosravi et al., 2021) and numerical simulations (Wang et al., 2017). A review of this topic is beyond the scope of the current study. To date, there has been progress in modeling the process of spontaneous imbibition, but there are also limitations (for example, the presence of various assumptions in the mathematical model).

Summing up, there are many works devoted to the study of nanofluids nowadays, in which chemical agents, including surfactants, and the concentration of nanoparticles are studied. This article presents the results of improved oil recovery from sandstone due to the spontaneous imbibition of silica nanosols without the addition of any surfactants. The aim of this research is to study the influence of the concentration and size of silicon oxide nanoparticles, as well as the properties of oil and core, on the spontaneous displacement of oil by silica nanosols. The effect of nanoparticle size was obtained from a capillary imbibition study.

## 2. Materials and methods

### 2.1 Silica sols

Silica nanosols obtained by diluting highly concentrated sols (RusSilica, Russia) were used in the work. Highly concentrated sols were obtained as follows. First, sodium silicate is dissolved in water to obtain sodium liquid glass. Next, ion exchange processes are carried out (to remove sodium ions from the reaction medium) with simultaneous sol formation and further thermal stabilization of silica sol. The stable silica sol is then filtered and concentrated to the desired concentration. Four types of silica sols (SL10, SL15, SL20, and SL35) were used, differing in primary particle size. The initial concentration of nanosols SL10, SL15 was 30 wt%, SL20, 40 wt%, and SL35, 50 wt%. Colloidal stability analysis was monitored using a Turbiscan analyzer. The finished sols remained stable for more than 2 months. A brine model (3 wt% sodium chloride solution) was used.

1) Microscopic analysis
Electron microscopic studies were carried out on a high-resolution scanning electron microscope FE-SEM Hitachi S-5500. Electron microphotos of nanoparticles are shown in Fig. 1. All electron photographs were taken in the secondary electron mode at an accelerating voltage of 3 kV, a beam current of 10 μA, and a focal length of 100 to 200 μm.
2) Size distribution of nanoparticles in liquid
Measurement of the size distribution of nanoparticles in nanosols (Fig. 2) was carried out using an acoustic spectrometer (Dukhin and Goetz, 2001). A summary of hydrodynamic size, dispersion, and geometric standard deviation (GSD) is given in Table 1. Nanosols SL10, SL15, and SL20 have a narrow particle size distribution. Suspensions with a geometric standard deviation greater than 1.2 are conventionally considered to be polydisperse (for example, SL35 nanosol).
3) Physical properties
Three weight concentrations of nanoparticles were con-

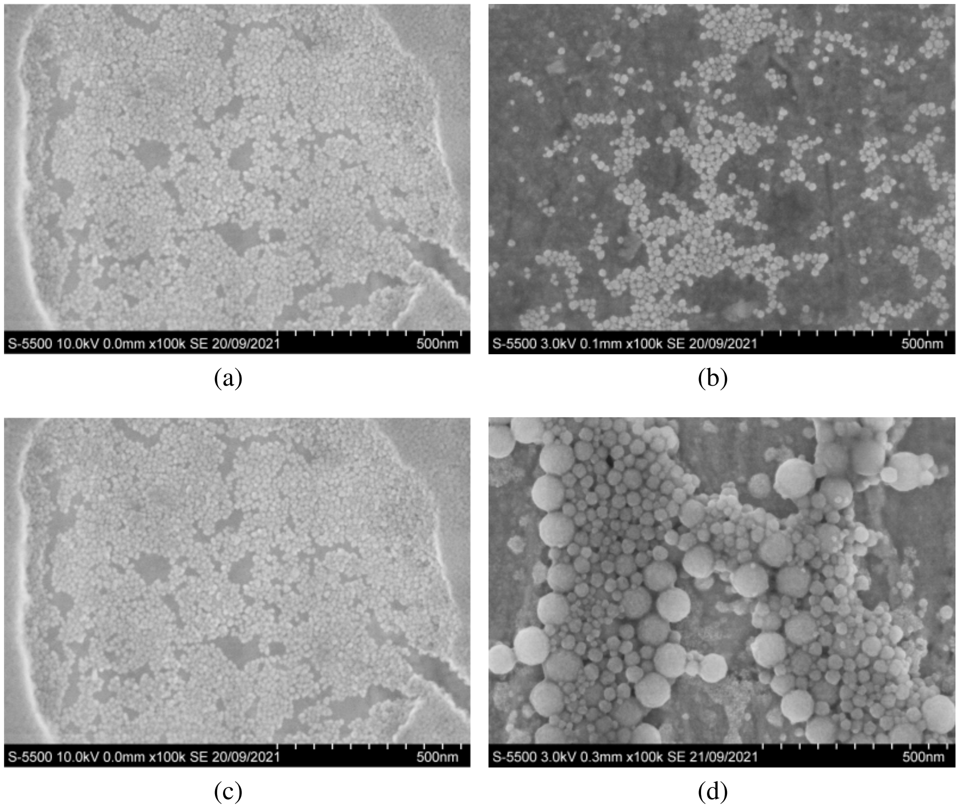



**Fig. 1**. Electron microphoto of nanoparticles, magnification x100k.(a) SL10, (b) SL15, (c) SL20 and (d) SL35.

sidered. Silica nanosols with a given concentration were obtained by diluting highly concentrated sols with distilled water. The density of nanosols $\rho_{sol}$ was estimated using the mixture rule (Pak and Cho, 1998):

$$\rho_{sol} = \rho_p \phi + \rho_f (1-\phi) \tag{1}$$

where $\rho_p$, $\rho_f$ are the density of particles and liquid respectively; $\phi$ is the volume fraction of particles in the suspension. In the calculation the particle densities of silica and alumina were 2.2 and 3.97 g/cm$^3$ respectively.

The volume fraction $\phi$ is expressed in terms of mass fraction $w$ as follows:

$$\phi = \frac{\rho_f w}{\rho_f w + \rho_p (1-w)} \tag{2}$$

The physical parameters of the displacement fluids used (water and nanosols) are shown in Table 2. The viscosity $\mu_{sol}$ was measured by the rotational method at a temperature of 25°C according to the procedure described in (Minakov et al., 2020b; Pryazhnikov and Minakov, 2020). Viscosity index data for the concentrations considered in the oil displacement experiments are also shown in Table 2.

### 2.2 Cores and crude oil

Cylindrical cores with a diameter of 30 mm, drilled from a block of Berea sandstone, were used in the work. The drilled cores were kept in an oven (100 °C) for their complete drying. Then the measurement of geometric dimensions (diameter $D$ and length $L$), mass, as well as the determination of porosity $\phi$ and gas permeability $k$ of the cores were carried out. After

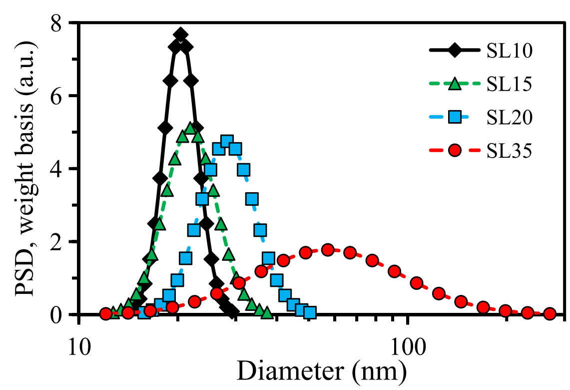



**Fig. 2**. Size distribution of nanoparticles in nanosols.

**Table 1**. The property of nanoparticles.

| Sample | Particle size (nm) | GSD | ln(GSD) |
|---|---|---|---|
| SL10 | 20.3 | 1.065 | 0.063 |
| SL15 | 21.9 | 1.050 | 0.049 |
| SL20 | 28.2 | 1.088 | 0.084 |
| SL35 | 57.2 | 1.252 | 0.225 |

**Table 2**. List of used displacement fluids.

| Name | Concentration (wt.%) | Particle size (nm) | Density (kg/m$^3$) | Viscosity (mPa·s) |
|---|---|---|---|---|
| Water | 0 | / | 997.0 | 0.8909 |
| 0.01% SL10 | 0.01 | 10 | 997.1 | 0.8913 |
| 0.1% SL10 | 0.1 | 10 | 997.5 | 0.8944 |
| 0.25% SL10 | 0.25 | 10 | 998.4 | 0.9024 |
| 0.1% SL15 | 0.1 | 15 | 997.5 | 0.8935 |
| 0.1% SL20 | 0.1 | 20 | 997.4 | 0.8930 |
| 0.1% SL35 | 0.1 | 35 | 997.6 | 0.8924 |
| 0.1% AL11 | 0.1 | 11 | 997.8 | 0.8917 |

**Table 3**. Physical characteristics of cores.

| Core plug | Diameter (mm) | Length (mm) | Permeability (mD) | Porosity (%) | Pore volume (ml) | Fluid |
|---|---|---|---|---|---|---|
| V18 | 28.49 | 55.08 | 333 | 22.3 | 7.8 | Water |
| V14 | 28.88 | 55.06 | 324 | 22.0 | 7.9 | 0.01% SL10 |
| V13 | 28.96 | 55.03 | 328 | 22.2 | 8.0 | 0.1% SL10 |
| V12 | 28.83 | 55.08 | 326 | 22.0 | 7.9 | 0.25% SL10 |
| V15 | 29.13 | 55.09 | 323 | 22.2 | 8.2 | 0.1% SL15 |
| V9 | 29.12 | 55.07 | 320 | 22.1 | 8.1 | 0.1% SL20 |
| V6 | 28.38 | 55.06 | 332 | 21.8 | 7.6 | 0.1% SL35 |
| V5 | 28.90 | 55.09 | 323 | 21.9 | 7.9 | 0.1% AL11 |
| S1 | 29.95 | 35.42 | 0.34 | 10.7 | 2.7 | Water |
| S4 | 29.90 | 35.34 | 0.34 | 11.6 | 2.9 | 0.1% SL10 |
| S5 | 29.98 | 35.57 | 0.35 | 11.3 | 2.8 | 0.25% SL10 |
| A22 | 29.52 | 36.78 | 250 | 20.4 | 5.1 | Water |
| A23 | 29.61 | 36.67 | 247 | 20.4 | 5.2 | 0.1% SL10 |
| A24 | 29.65 | 36.70 | 245 | 20.7 | 5.2 | 0.25% SL10 |

**Table 4**. Viscosity and density of the crude oil used.

| Crude oil | Viscosity (mPa·s) | Density (kg/m$^3$) |
|---|---|---|
| 1 | 79.3 | 901 |
| 2 | 28.9 | 861 |

that, the cores were saturated with crude oil in a saturator for two days. After saturation, the cores were reweighed to estimate the pore volume (PV). The physical characteristics of the cores are listed in Table 3.

Two samples of crude oil differing in density and viscosity coefficient were used in the experiments. The characteristics of the crude oils used are presented in Table 4. Series V cores were saturated with Oil 1, and series S (S1, S4 and S5) and series A (A22, A23, A24) cores were saturated with Oil 2.

## 2.3 Imbibition tests and experimental procedure

To study the effect of capillary forces on the process of oil displacement from the pore space of the core, a series of experiments was carried out. The experiment was as follows. The oil-saturated core was placed in a brine model or nanosuspension with a given concentration and size of nanoparticles. After some time, the replacement of oil by nanosuspension occurred due to the action of capillary forces (Fig. 3). The process of spontaneous imbibition was recorded on a camera, and an estimate of the volume of displaced oil was also made. The duration of the experiment was about 66 days. The temperature in the Ammot cell was maintained at room temperature (25 °C). The Amott cell was graduated with a resolution of 0.1 ml.

## 2.4 Contact angle and interfacial tension

The study of interfacial tension $\sigma$ and contact angle $\theta$ was carried out using the technique presented in Minakov et al. (2021a). Measurements were taken with an automatic ten-

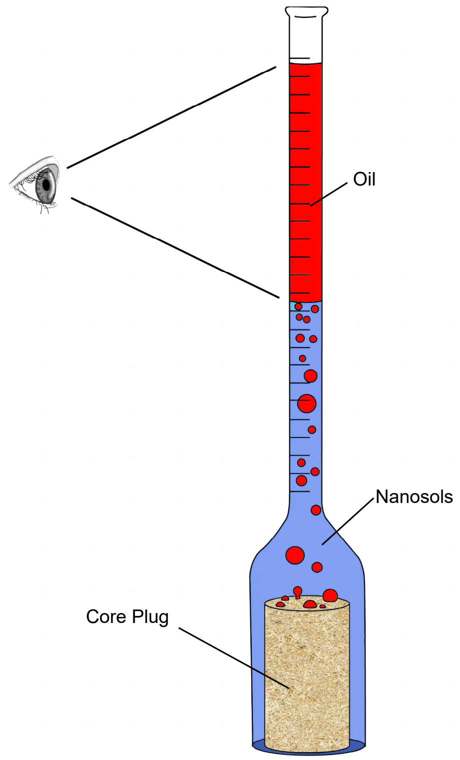



**Fig. 3**. Amott cell for the study of spontaneous imbibition of silicasols.

siometer (Fig. 4) based on the pendant drop method. DropImage Advanced software was used to determine nanosol-oil interfacial tension $\sigma$ and nanosol-oil-rock contact angle $\theta$. The interfacial tension was determined from the results of measurements of the parameters of a pendant oil drop in the nanosol. The contact angle was measured by the captive bubble method.

## 3. Results and discussion

### 3.1 Contact angle and interfacial tension measurements

The determination of interfacial tension in oil-nanosol systems has been carried out. Fig. 5 shows photographs of an oil drop in water and SL10 nanosols with different concentrations of nanoparticles. Table 5 shown that with an increase in the concentration of nanoparticles, the interfacial tension of oil-nanosol SL10 decreases. Thus, for the 0.25% SL10 nanosol, the interfacial tension decreased by 10% compared to water (36.3 mN/m).

It has been found that the addition of $SiO_2$ nanoparticles to water significantly affects the oil wettability characteristics of the rock. Analysis of Fig. 6 shows that the presence of nanoparticles in the liquid increases the contact angle, which also increases quite significantly, from 115° (brine) to 155° (nanosol 0.25% SL10). The addition of nanoparticles to the liquid significantly impairs the wettability of the rock by oil. In fact, the addition of even 0.1 wt.% nanoparticles to water makes the sandstone impervious to oil. This circumstance is extremely important, since, as shown in many studies, an increase in the contact angle is the main factor affecting the oil recovery factor during waterflooding.

**Table 5**. Interfacial tension of nanosol-oil and contact angle of nanosol-oil-core.

| Fluid | Interfacial tension (mN/m) | Contact angle (deg) |
|---|---|---|
| Water | 36.3 | 115 |
| 0.01% SL10 | 36.2 | 145 |
| 0.1% SL10 | 35.7 | 153 |
| 0.25% SL10 | 32.8 | 157 |
| 0.1% SL15 | 35.3 | 153 |
| 0.1% SL20 | 34.6 | 152 |
| 0.1% SL35 | 33.5 | 151 |
| 0.1% AL11 | 36.0 | 152 |

### 3.2 Effect of nanoparticle concentration on oil recovery

Initially, a study of capillary displacement of oil from a sandstone sample with an average permeability of 326 mD, saturated with an oil sample of oil 1, using SL10 nanosol with various concentrations of silica nanoparticles was carried out. The mass concentration of nanoparticles in the nanosuspension varied from 0 to 0.25%.

Photos of the process of spontaneous imbibition of brine are shown in Fig. 7. After 7 days from the start of the experiment, small drops of oil appear on the surface of the core immersed in brine. After 20 days, small oil droplets begin to collect into larger droplets on the surface of the core. After 30 days from the start of the experiment, a noticeable oil film is formed on the surface of the brine. The average duration of experiments on the study of capillary impregnation was about 66 days.

Similarly, studies on the spontaneous imbibition of nanosuspensions have been carried out. Photos of the process of spontaneous imbibition of nanosuspension 0.25% SL10 are shown in Fig. 8. It has been shown that the addition of nanoparticles to a liquid significantly affects the rate of capillary impregnation. Already after one day, rather large drops of displaced oil are visible on the surface of the core. It can be seen even with the naked eye that the process of capillary replacement of oil in the nanosuspension is much more intense. This is clearly seen from the comparison of photographs in Fig. 8. At the same time, it should be noted that in the case of capillary replacement with brine, rather small oil drops are formed, while when using a nanosuspension, oil drops are several times larger in size. This also indirectly confirms the data on a significant decrease in the interfacial tension of the nanosuspension-oil.

For comparison, Fig. 9 shows photos of the core after 7 days of spontaneous imbibition of the SL10 nanosuspension with different concentrations of particles. The process of oil displacement from the core when using a nanosuspension is much more intense compared to brine even at the initial stages

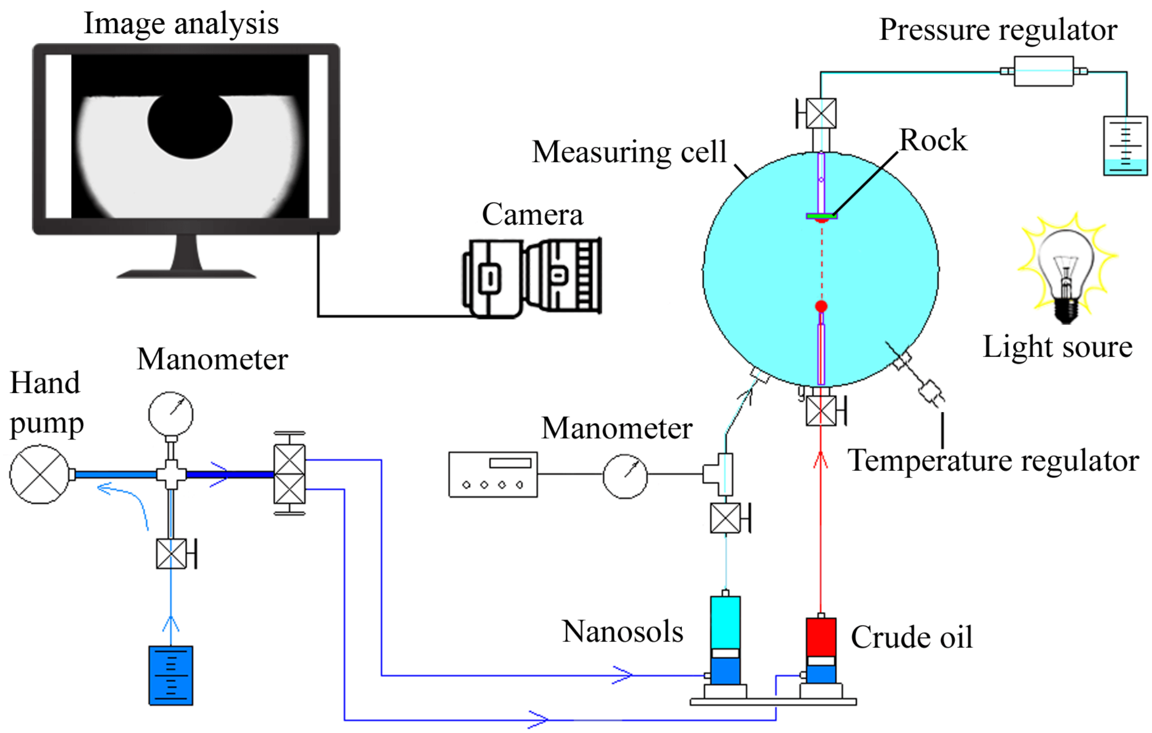



**Fig. 4**. Schematic diagram of the tensiometer.

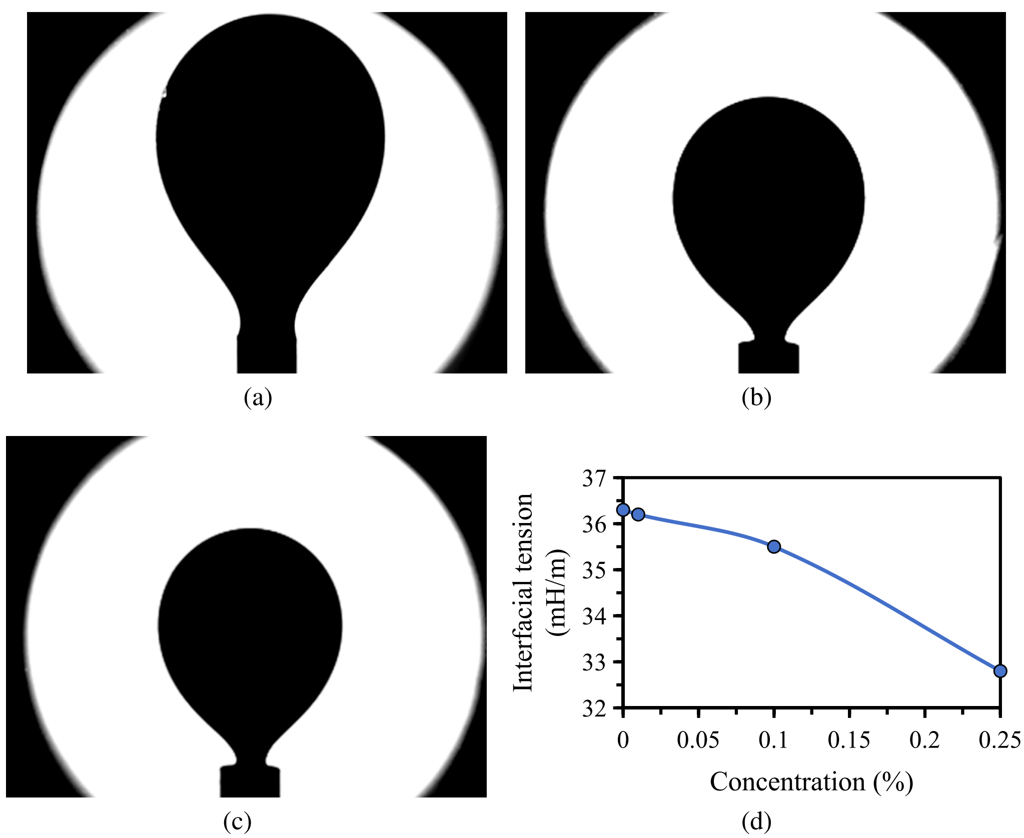



**Fig. 5**. Photo of a pendant oil drop (a) in brine, (b) in a 0.1% SL10 nanosol, (c) in a 0.25% SL10 nanosol and (d) interfacial tension versus the concentration of nanoparticles in water.

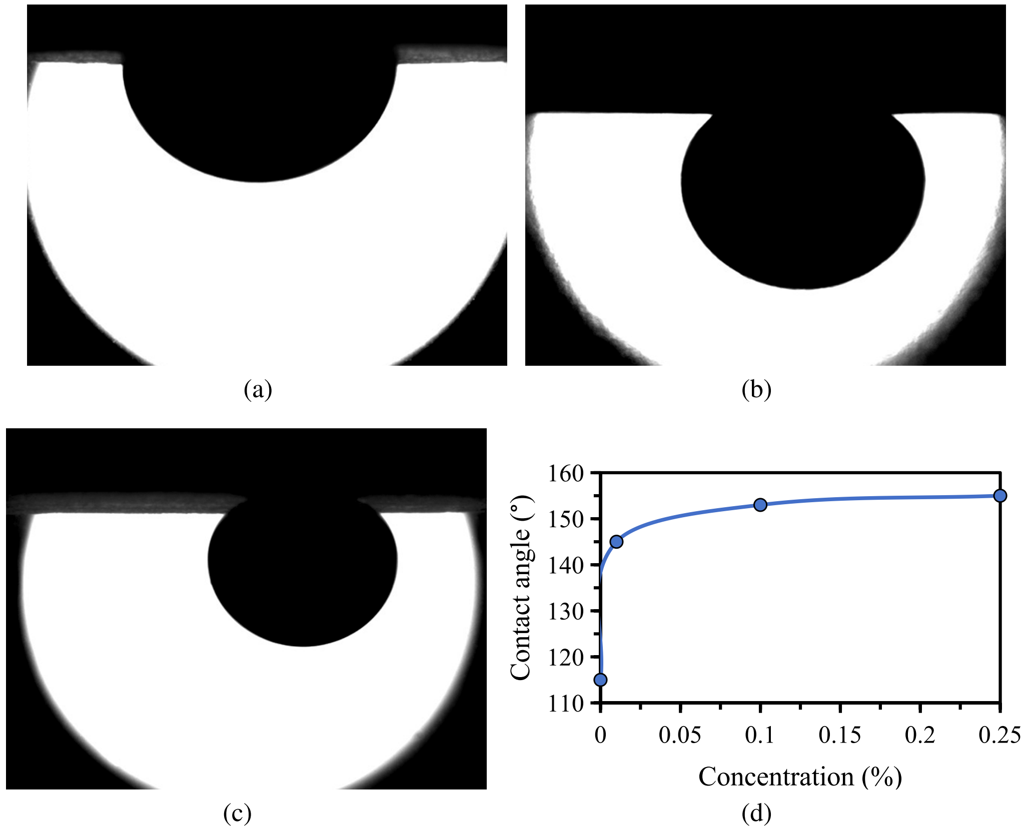



**Fig. 6**. Photo of a captive oil drop on the rock (a) in the brine, (b) in 0.1% SL10 nanosol, (c) in 0.25% SL10 nanosol and (d) contact angle on the concentration of nanoparticles in water.

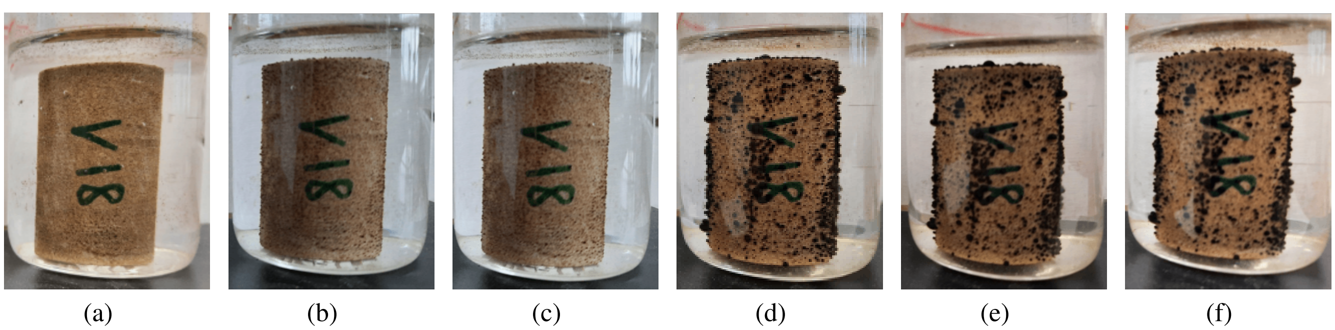



**Fig. 7**. Photos of the process of spontaneous imbibition in brine at various points in time. (a) Start of the experiment, (b) after 1 day, (c) after 7 days, (d) after 20 days, (e) after 28 days and (f) after 66 days.

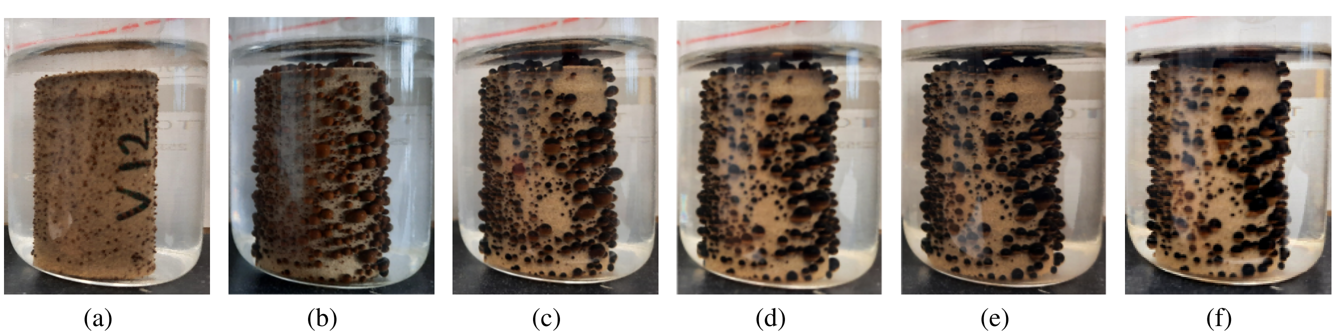
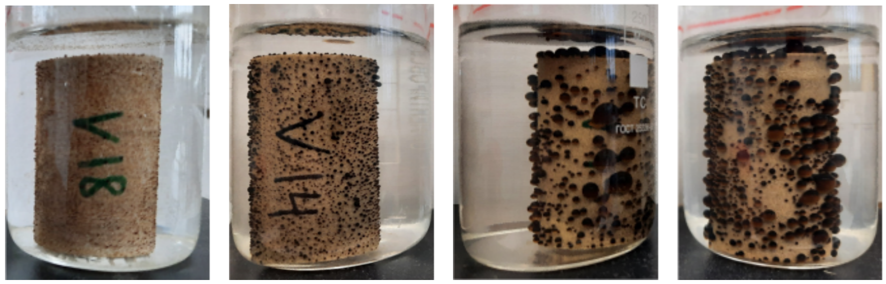



**Fig. 8**. Photos of the process of spontaneous imbibition of silica nanosol 0.25 wt% SL10 at different time points: (a) the beginning of the experiment, (b) a day later, (c) after 7 days, (d) after 20 days, (e) after 28 days and (f) after 66 days.



(a) (b) (c) (d)

**Fig. 9**. Photos of the process of oil displacement from the core during spontaneous imbibition after 7 days from the start of the experiment: (a) brine, (b) 0.01% SL10, (c) 0.1% SL10 and (d) 0.25% SL10.

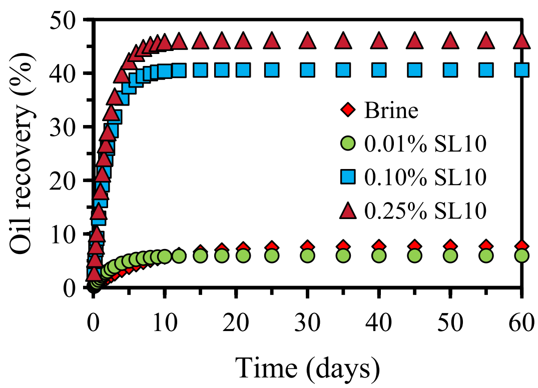



**Fig. 10**. Dynamics of oil recovery during spontaneous imbibition of silica sols with different concentrations SL10.

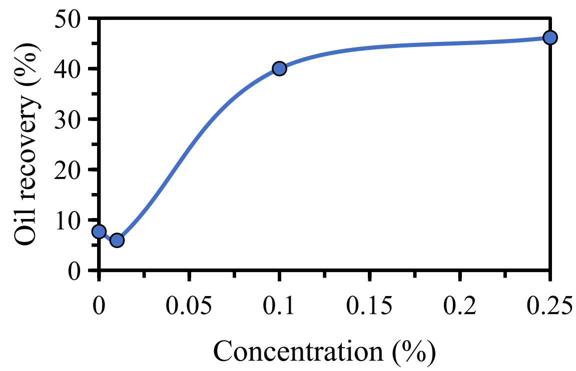



**Fig. 11**. Dependence of the oil recovery on the concentration of SL10 nanoparticles.

of this process. The volume of capillary displaced oil depends on the concentration of nanoparticles.

The dynamics of oil recovery (OR) is shown in Fig. 10. The use of 0.01% nanosol silica SL10 did not lead to an increase in oil recovery. However, it affected the rate of increase in oil displacement efficiency compared to brine. At concentrations of nanoparticles above 0.1%, there is a significant increase in the rate of change and value of OR.

The dependence of the oil recovery on the concentration of SL10 nanoparticles for 66 days is shown in Fig. 11. The volume of displaced oil increases with an increase in the concentration of silicon oxide nanoparticles. The maximum volume of oil is displaced when using a concentration of nanoparticles of 0.25%. The increase in the volume of oil here is 6 times compared to the volume of oil displaced by brine, this is a very significant increase.

This fact indicates that when the nanosuspension penetrates into the core, the capillary pressure significantly changes due to the wettability characteristics. The obtained results of the study of spontaneous imbibition are in good agreement with the data of measurements of the interfacial tension coefficient and the wetting angle. It was shown that in the presence of nanoparticles in a liquid, the contact angle increases, while also very significantly, from 115° to 155° (the surface becomes hydrophilic), Thus, for a 0.25% suspension, IFT decreased by 10.7% compared to water. The major driving force of spontaneous imbibition is capillary pressure $p = 2\sigma\cos\theta/R$, where $\sigma$ represents the interfacial tension of the << liquid/oil >> system, $\theta$ represents the contact angle, $R$ is the pore radius (Zhang et al., 2023). A change in interfacial tension, as already noted, could not affect the increase in capillary force. The capillary force includes the complex $\cos\theta$. This complex changes by a factor of 2.1 when comparing water and a 0.25% SL10 suspension. The wettability altering factor $(1-\cos\theta_{after})/(1-\cos\theta_{before})$ decreased from 0.313 to 0.138 with an increase in concentration from 0.01% to 0.25%, while changing by 2.1 times. This is the reason for the increase in the rate of capillary impregnation with an increase in the concentration of nanoparticles.

## 3.3 The effect of nanoparticle size on oil recovery

After studying the influence of the concentration of nanoparticles on the process of capillary displacement of oil, a study of the influence of the size and material of nanoparticles on the process of spontaneous imbibition was carried out. Analysis of the literature shows that such a study was performed for the first time in our work. The following nanosuspensions SL10, SL15, SL20, and SL35 were chosen as the object of study; their description is presented in Section 2.1. For comparison with nanosuspensions with silicon oxide particles, a nanosuspension with aluminum oxide particles with a size of 11 nm (manufactured by Bardakhanov LLC) was additionally considered. The mass concentration of nanoparticles in all nanosuspensions was 0.1%. The dynamics

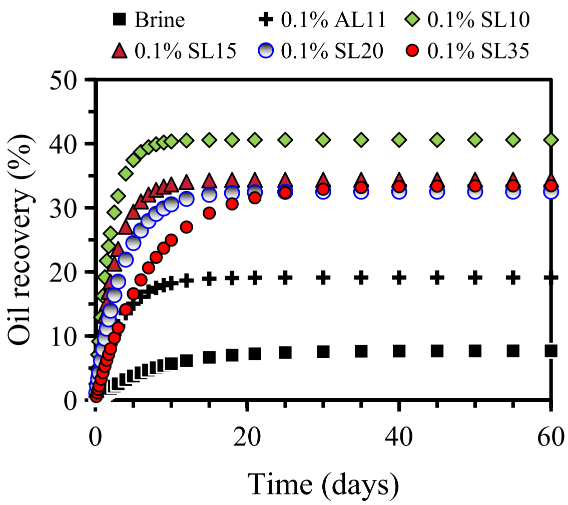



**Fig. 12**. Dynamics of oil recovery during spontaneous imbibition of silica sols with different nanoparticle sizes.

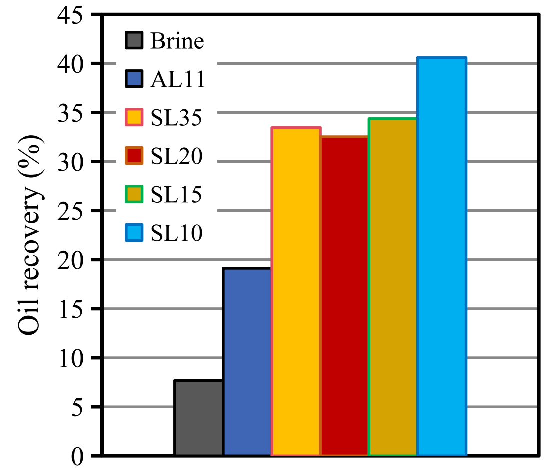



**Fig. 13**. Dependence of the oil recovery as a result of spontaneous imbibition of silica nanosols on the size of nanoparticles 0.1 wt%.

of the oil recovery during spontaneous absorption of silica nanosols of different sizes of nanoparticles from time to time is shown in Fig. 12.

As can be seen from Fig. 12, different nanosuspensions have different effects on the process of spontaneous imbibition. The suspension of aluminum oxide nanoparticles with an average nanoparticle size of 11 nm has the least effect on the process of capillary impregnation, and the suspension with silicon oxide particles with a particle size of 10 nm has the greatest effect. A noticeable precipitate formed in the AL11 suspension after 66 days. Some of the aluminum oxide nanoparticles are deposited during the experiment. At the same time, suspensions with silicon oxide nanoparticles are much more colloidally stable. This is extremely important for their practical use. This is due to the fact that the nanosuspension with aluminum oxide was prepared from the nanopowder using a standard two-step method with pretreatment with ultrasound. While the remaining suspensions were obtained by diluting concentrated silicasols. Quantitative data on the oil recovery various suspensions for 66 days are shown in Fig. 13.

Based on the results of studying the effect of the material and size of nanoparticles on the process of spontaneous imbibition, the following conclusions were drawn. The maximum value of the oil recovery coefficient is achieved when using a nanosuspension based on silica sol SL10, and the minimum value is obtained when using a suspension with 11 nm aluminum oxide nanoparticles. It was found that the oil displacement ratio for spontaneous water imbibition is 7.7%. The oil displacement efficiency when using suspensions is higher, so for a suspension of 0.1% AL11 it is 19.1%, and when using 0.1% SL10 it is 40.6%. It is possible that the reason for the different effect of the two types of nanoparticles on spontaneous displacement is their surface charge. When nanoparticles are present in a liquid, an electrical double layer is usually formed, consisting of ions in the liquid. This occurs because the surfaces of the particles typically carry a surface charge that attracts these ions. The structure of the electrical double layer determines not only the thermodynamic, but also the electrokinetic characteristics of the surface and colloidal systems. In this case, an important role is played by the layer boundary, at which the molecules of the liquid medium practically lose their hydrodynamic mobility. The plane separating the moving external medium from the stationary boundary layer is called the slip plane. In this plane, the charge potential matters. This quantity is called zeta potential. Ion adsorption affects the surface tension of the interphase boundary, since there is a repulsion of charged particles in the surface layer. The effect of charge on surface tension is an effect related to electrocapillary phenomena (surface phenomena involving charged particles). The zeta potential of 0.1% SL10 (suspensions of silicon oxide particles) is negative and equal to -35 mV, and 0.1% AL11 (suspensions of aluminum oxide particles) has a positive charge of +46 mV.

The use of silicasols SL15, SL20, SL35 gives approximately the same result and is approximately 32.5%-34.4%. Thus, it has been shown that silica nanoparticles in this case are more preferable than aluminum oxide particles. Another important fact is that the nanosuspension with alumina was prepared from a ready-made nanopowder, while the rest of the suspensions were obtained by diluting concentrated silica sols. From a practical point of view, the use of concentrated (up to 50 wt.%) suspensions in the field is much more preferable than the use of nanopowders, which have a very low bulk density and require special conditions for the preparation of nanosuspensions. The conducted studies on capillary impregnation allow us to draw a conclusion about the effect of the size of nanoparticles (see Fig. 14). As a result, it was found that the displacement efficiency during capillary impregnation increases with decreasing nanoparticle size.

The maximum oil recovery was recorded for the SL10 nanosol with an average nanoparticle size of 10 nm, the minimum value for the SL35 nanosol. These results are confirmed by the data on the effect of the addition of nanoparticles on the coefficient of interfacial tension of the oil-nanosuspension. It was shown that the coefficient of interfacial tension for the considered samples of oil and nanosuspension decreases the

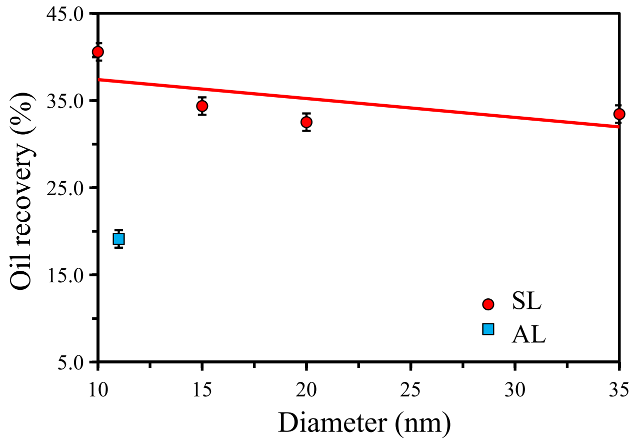



**Fig. 14**. Dependence of the oil recovery as a result of spontaneous imbibition of 0.1 wt% nanosuspensions on the size of nanoparticles. Symbols: circles–silicon oxide nanosols, square–aluminum oxide nanosuspension. Line: Linear approximation of SL data.

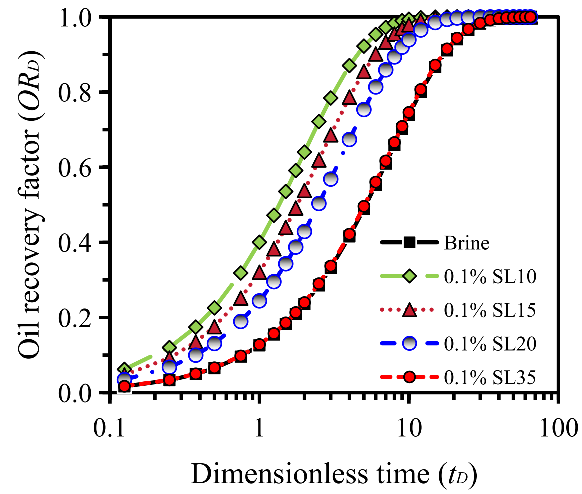



**Fig. 15**. Dimensionless oil recovery by spontaneous imbibition versus dimensionless time. Symbols–experiment, line–equation (4).

stronger, the smaller the size of the nanoparticles.

A dimensionless parameter was proposed (Mattax and Kyte, 1962) that allows scaling the behavior of oil recovery during spontaneous imbibition from highly water-wet:

$$t_D = 2174 \times \sqrt{\frac{k}{\phi}} \times \frac{\sigma}{\sqrt{\mu_o \mu_{df}} L^2} \tag{3}$$

where $t_D$ is the dimensionless time, $L$ is the characteristic length (mm), $\theta$ is the interfacial tension of oil-displaced fluid system (mN/m), $\mu_o$ is the oil viscosity (mPa s), $\mu_{df}$ is the displaced fluid viscosity (mPa s), $t$ is the time of imbibition (days).

A simplified form for estimating the oil recovery factor (Aronofsky et al., 1958):

$$R(R_D) = \frac{R}{R_{\max}} = (1 - e^{-\alpha t_D}) \tag{4}$$

where $R$ is the oil recovery during spontaneous imbibition, $R_{\max}$ is the marginal oil recovery during spontaneous imbibition, $\alpha$ is the oil production decline constant.

Fig. 15 shows the dimensionless oil recovery $R(R_D)$ versus dimensionless time. Dependences of dimensionless oil recovery on dimensionless time for brine and nanosols differing in nanoparticle size are given. It was found that the dimensionless oil recovery during spontaneous imbibition is higher when using nanosols with smaller nanoparticle sizes at the same nanoparticle concentration.

## 3.4 The effect of rock permeability

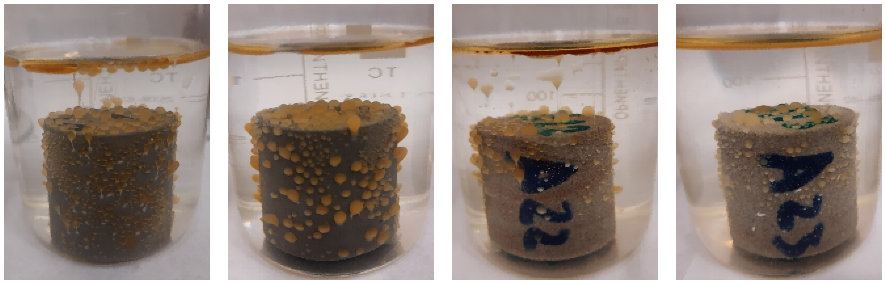
Experiments on capillary displacement of the oil 2 model from core samples with an average permeability of 0.34 and 240 mD were carried out. Suspensions of SL10 nanoparticles with 0.1% were used. Fig. 16 shows photos of cores with different permeability immersed in brine and nanosol at the end of the spontaneous imbibition experiment.

As a result of experiments on the effect of rock permeability on the process of capillary displacement, conclusions were drawn that the use of a nanosuspension leads to an increase in the volume of oil displaced from cores with an average permeability of 0.34 and 247 mD. For both core samples, with an increase in the concentration of nanoparticles, the oil displacement coefficient increases.

Fig. 17 compares the oil recovery factor for different rock permeability values. It has been established that the effect of the addition of nanoparticles on the efficiency of capillary impregnation is much more pronounced on cores with low permeability, for which the effect of capillary pressure is obviously much more significant.

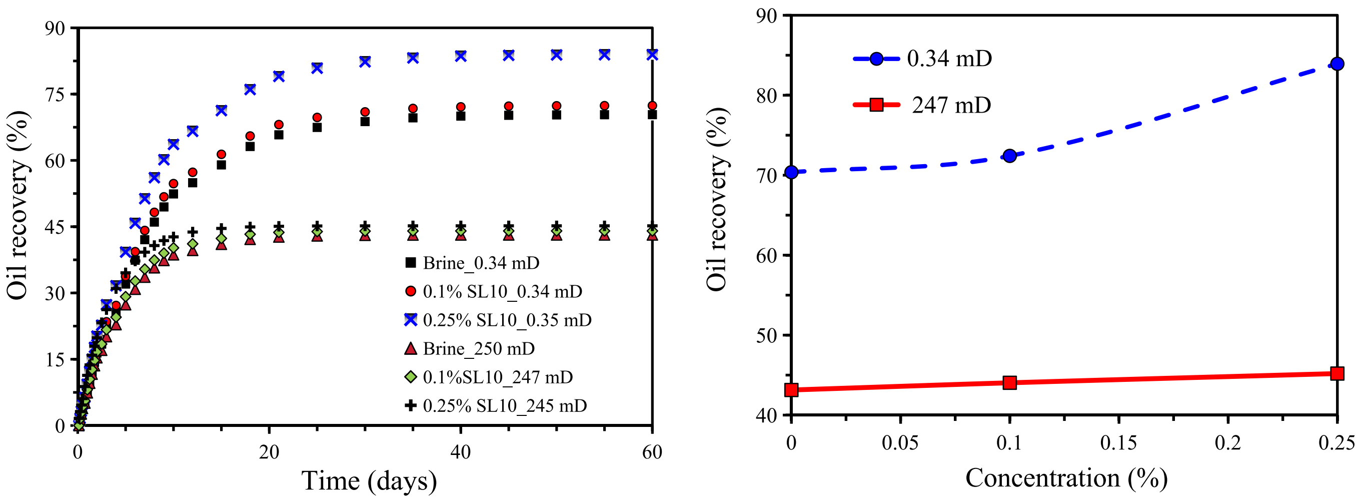
## 4. Conclusions

An experimental study of the spontaneous imbibition of various nanosols with silicon oxide nanoparticles into an oil-saturated core was carried out. Berea sandstone was used as the reservoir rock. Samples with different permeability and porosity are considered. Permeability and porosity ranged from 0.34 to 333 mD and from 10% to 22%, respectively. A study of the effect of concentration, size of nanoparticles, as well as rock permeability on the process of capillary impregnation was carried out. It has been shown that the addition of nanoparticles to a liquid significantly increases the rate of spontaneous imbibition. The study of the effect of particle concentration showed that with an increase in the concentration of nanoparticles, the volume of capillary displaced oil, other things being equal, increases. Thus, in particular, it was demonstrated that nanosuspension with SL10 silica at a particle concentration of 0.25% for the same time allows capillary displacement of more than six times more oil compared to brine. This fact indicates that nanosuspensions significantly reduce the capillary pressure due to the wettability characteristics. The results of the study of capillary impregnation are in good agreement with our measured data on the interfacial tension coefficient and the contact angle, which also indicate that with an increase in the concentration of nanoparticles, the interfacial tension decreases and the hydrophilic properties of



(a) (b) (c) (d)

**Fig. 16**. Photos of the core at the end of the spontaneous imbibition experiment. (a) 0.34mD core in brine, (b) 0.34 mD core in 0.1% SL10, (c) 247 mD core in brine and (d) 247 mD core in 0.1% SL10.




(a) (b)

**Fig. 17**. Displacement of oil from core with different permeability in the process of spontaneous imbibition of SL10. (a) Dynamics of oil recovery and (b) dependence of oil recovery on SL10 concentration.

the suspension increase. This is the reason for the increase in the rate of capillary impregnation with an increase in the concentration of nanoparticles. The studies carried out allow us to conclude that the size of nanoparticles has an effect. It was found that the spontaneous imbibition rate increases with decreasing nanoparticle size. These results are confirmed by data on the effect of the addition of nanoparticles on the oil/nanosuspension interfacial tension coefficient. It was shown that the coefficient of interfacial tension for the considered samples of oil and nanosuspension decreases the stronger, the smaller the size of the nanoparticles.

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## Conflict of interest

The authors declares that there is no conflict of interest regarding the publication of this article.



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