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Original article

Wettability, interfacial tension, and capillary imbibition of nanomaterial-modified cross-linked gels for hydraulic fracturing

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Abstract:
For the first time, systematic studies were conducted to investigate the effect of concentration, size, material, and shape of nanoadditives on the wettability, interfacial tension, and capillary imbibition rate of cross-linked gels for hydraulic fracturing. Guar gum biopolymer was used as the gelling agent and sodium tetraborate in glycerol was used as the crosslinker in the preparation of cross-linked gels. Spherical nanoparticles of silica and alumina, as well as single-walled carbon nanotubes, and alumina nanofibers were used as nanoadditives. The nanoparticles had an average size ranging from 11 to 216 nm, and their concentration in the gels ranged from 0.01 to 0.8 wt%. The study revealed a non-monotonic dependence of the contact angle, interfacial tension coefficient, and capillary imbibition rate of nanomodified cross-linked gels on the concentration and average size of nanoparticles. The gels exhibited maximum hydrophobic properties at a nanoparticle concentration of 0.2 wt% and an average size of 70-80 nm. At the same time, the addition of single-walled carbon nanotubes has the most significant effect on the wettability properties of the gels, reducing the capillary imbibition rate by three times. Thus, it has been shown that controlling the concentration, size, material, and morphology of the nanoadditives can significantly alter the wetting characteristics of hydraulic fracturing fluids. This provides an opportunity for more flexible control of the hydraulic fracturing process depending on the reservoir characteristics.

1. Introduction

Hydraulic fracturing is a highly daunting operation in the oil and gas production industry. In order for hydraulic fracturing to be successful, the fracturing fluid must possess specific physical and chemical properties. These properties include compatibility with the formation material, the ability to suspend proppant, the required viscosity characteristics to create fractures and transport proppant, ease of removal from the formation after treatment, high stability to maintain its properties throughout the treatment, and ease of preparation (Speight, 2016; Zhao et al., 2023). Wettability is another property that is of decisive importance in various fields such as micro-electromechanical systems (Liu et al., 2022), enhanced oil recovery (Goharzadeh et al., 2023), hydraulic fracturing (Lu et al., 2023; Guo and Wortman, 2024). Wettability alteration from oil-wet to water-wet affects enhanced oil recovery (Goharzadeh et al., 2023). Fracturing fluid wettability data can be used to estimate the imbibition depth into the rock matrix

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that occurs under the influence of capillary forces (Guo and Wortman, 2024). The work (Lu et al., 2023) shows that to predict the micro-fracture propagation pressure, it is necessary to take into account parameters such as contact angle, surface tension and microcrack width.

Therefore, developing new or improving existing fracturing fluids is a challenging problem that demands extensive fundamental research in mechanics, colloidal and physical chemistry. Currently, research is underway to discover effective additives that can improve the properties of hydraulic fracturing fluids. According to a literature review, the use of nanoparticle additives may be a promising technology for developing new high-performance fracturing fluids in the near future (Lafitte et al., 2012; Hurnaus and Plank, 2015; Zhao et al., 2017; Mao et al., 2022; Marsden et al., 2022).

Recent studies (Zhao et al., 2017; Mao et al., 2022; Marsden et al., 2022) have shown that nanoparticles can be effectively used to cross-link gels in fracturing fluids. One of the main advantages of using nanoparticles for fracturing fluids is their ability to modify fluid properties by modifying nanoparticle surfaces. Nanoparticles are highly surface-active, which can lead to cross-linking of nanoparticles and surfactant micelles.

Lafitte et al. (2012) conducted one of the pioneering studies in this area, using latex nanoparticles to further cross-link guar gum with boric acid. The latex nanoparticles had a size of 15 nm, which provided a high specific surface area for efficient cross-linking even at lower concentrations. The study has demonstrated that utilizing latex nanoparticles enabled a 20-fold decrease in boric acid concentration when compared to the traditional cross-linking technique.

In their study, Hurnaus and Plank (2015) investigated the mechanisms of guar gum cross-linking by metal oxide nanoparticles, specifically TiO_2 and ZrO_2. The study has found that the primary mechanism is the formation of hydrogen bonding between the hydroxyl group at the surface of the nanoparticles, resulting from hydrolysis, and the hydroxyl groups of the guar molecules. Moreover, the size of the nanoparticles is a crucial factor in the cross-linking process. This study has demonstrated that larger nanoparticles significantly worsen the cross-linking conditions of guar molecules. Subsequent research has revealed that the effect of nanoparticles on the process is not so clear. According to Zhao et al. (2017), the effective viscosity of nanomodified silica gel exhibits a non-monotonic dependence. At high concentrations of nanoparticles, the effective viscosity is lower than that of the base case. The authors hypothesized that the addition of nanoparticles leads to the growth of micelles at low particle concentrations, resulting in an increase in effective viscosity. However, when the concentration of nanoparticles is increased further, the initial network structure of polymer molecules is disrupted, leading to the formation of larger micelles that entangle with each other and prevent cross-linking. As a result, the viscosity of the gel decreases. Such non-monotonic behavior of effective viscosity depending on the concentration of nanomodified gels was also noted by the authors of the works (Zhang et al., 2017; Wu et al., 2018). Zhang et al. (2023a) have conducted a detailed study on the mechanisms of this behavior and have found a synergistic effect of hydrogen and covalent bonds between the nano-cross-linking agent and guar gum molecules. This has significantly improved the rheological characteristics of the nano-modified gel and enhanced its thermostability. Besides, in this work, the influence of nanoparticles on the wetting characteristics of nanomodified gel was investigated in addition to their effect on the rheological characteristics of gels. The results have shown that the addition of nanoparticles to gels increased the hydrophilic properties of the rock surface, which could aid in blocking water inflow.

Consequently, there is intense research on the effect of nanoparticles on the viscosity of hydraulic fracturing gels. It has been established that the interaction between nanoparticles and polymer solution micelles can significantly alter the rheological properties of fracturing fluids. The incorporation of nanoparticles into conventional fracturing fluids has been shown to greatly enhance their effective viscosity, as well as viscoplastic and viscoelastic properties (Hurnaus and Plank, 2015; Wang et al., 2019; Liu et al., 2020). Our findings on the impact of nanoparticles on the viscosity and rheology of clay-polymer drilling fluids indirectly support this conclusion. (Minakov et al., 2018, 2019). Moreover, recent studies have shown that nanoparticles can enhance the thermal stability of hydraulic fracturing fluid (Ma et al., 2023; Xin et al., 2023). Additionally, due to their high specific surface area, nanoparticles require orders of magnitude lower concentrations for cross-linking gels compared to typical crosslinkers. In addition, nanoparticles are not susceptible to thermal degradation.

Another property of nanoparticles when added into gels is their ability to affect the filtration loss of the gels during hydraulic fracturing operations. This can cause formation damage, reduced permeability, capillary pressure, and fluid loss. Hence, reducing the filtration loss of hydraulic fracturing fluid can improve the efficiency of the hydraulic fracturing operation. Barati (2015) has demonstrated that adding nanoparticles to gels reduced filtration loss by several times for low permeability core samples (less than 0.1 mD). A detailed study of this phenomenon is presented in a research thesis (Tangirala, 2019).

Along with the traditional nanoparticles, researchers are searching for new nanomaterials to enhance the properties of fracturing fluids. These include nanocellulose (Zhang et al., 2021), ZnO (Patel et al., 2022), graphene oxide (Wu et al., 2021), multi-wall carbon nanotubes (Tang et al., 2021), complex nanoemulsions (Wang et al., 2022), and others. Although this area is currently under intensive investigation, there are still many questions regarding the effect of nanoparticle concentration and the properties of the fracturing fluid. Therefore, further systematic studies are needed to investigate the effect of nanoparticle additives on the physicochemical properties of fracturing fluids. The main concern is the effect of the average nanoparticle size on the properties of fracturing gels. Solving this issue could lead to the development of novel hydraulic fracturing fluids that are more efficient and cost-effective.

Currently, the use of nanoparticles in hydraulic fracturing technologies is only being investigated fragmentarily. It is im-

Table 1. Nanomaterials used to modify hydraulic fracturing fluids.

Sample	Particle shape	Specific surface area (m^2/g)	Characteristic size (nm)	Manufacturer
SiO_2	Spherical	149	18	OOO Bardakhanov
SiO_2	Spherical	55	50	OOO Bardakhanov
SiO_2	Spherical	38	72	OOO Bardakhanov
SiO_2	Spherical	30	91	OOO Bardakhanov
SiO_2	Spherical	24	114	OOO Bardakhanov
Al_2O_3	Spherical	140	11	OOO Bardakhanov
Al_2O_3	Spherical	40	38	OOO Bardakhanov
Al_2O_3	Spherical	20	76	OOO Bardakhanov
Al_2O_3	Spherical	14	108	OOO Bardakhanov
Al_2O_3	Spherical	7	216	OOO Bardakhanov
Aluminan	Nanofibers	/	8.7*	OOO Nanosynthesis
SWCNTs	Nanotubes	510	1.6*	OOO OcSiAl.ru

Notes: *Diameter.

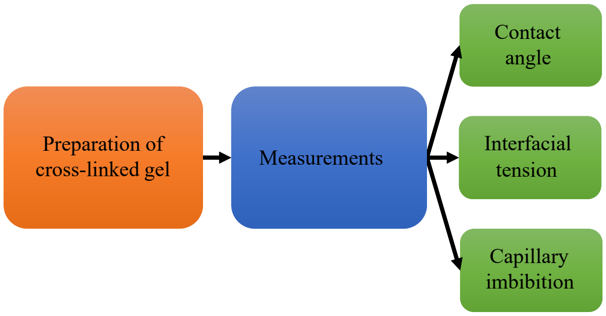


Fig. 1. Research chart.

portant to note that the effect of various chemical reagents on the wetting characteristics of rocks by fluids used for enhanced oil recovery during reservoir flooding has been extensively studied, unlike the wetting characteristics and interfacial tension of fluids used for hydraulic fracturing. Of particular interest are the results obtained in our work on the influence of nanofiber and carbon nanotube additives on the wetting characteristics of gels. There have been no similar studies previously.

At present, there are very few works in this direction; actually no data are available in literature on the effect of nanoparticles on the wettability characteristics of fracturing gels, although these characteristics have a key influence on the filtration characteristics of the bottom-hole zone after hydraulic fracturing operation. As already mentioned, here we can reference the research conducted by Zhang et al. (2023a) and the article by Tang et al. (2021), which demonstrated that the addition of nanoparticles in polymer fracturing fluid resulted in enhanced wettability. Surfactants were found to have a synergistic effect when added to the nanomodified gel. Currently, there are no systematic data available on the effect of nanoparticle addition on the wetting characteristics of hydraulic fracturing gels. In our work, systematic studies were conducted for the first time to investigate the effect of concentration, size, material, and shape of nanoadditives on the wettability, interfacial tension, and capillary imbibition rate of cross-linked gels for hydraulic fracturing.

2. Materials and methods

This section explains the preparation technique for the modified gels and the measurement of their properties (interfacial tension, contact angle, and capillary imbibition). Fig. 1 schematically illustrates the main study phases. The framework for our work included the following steps. At the first step, cross-linked gels were prepared (see Section 2.2) and modified with nanomaterials. The nanomaterials used are described in Section 2.1. The second step was related to the measurement of interfacial tension and contact angle, a detailed description is presented in Section 2.3. The final step was to analyze the experimental results and determine the effect of adding nanomaterials on the contact angle (Section 3.1), interfacial tension (Section 3.2), and imbibition rate (Section 3.3).

2.1 Materials for the preparation of cross-linked gels

Guar gum biopolymer (Altrafine Gums, India) was used as a gelatinizing agent, and sodium tetraborate in glycerol was used as a crosslinker when preparing cross-linked gels. Currently, a wide variety of nanomaterials are used to improve the properties of hydraulic fracturing fluids. In this work, the most common and available spherical particles, SiO_2 and Al_2O_3, were selected. Nanotubes were also used for the reason that they have fundamental differences from spherical nanoparticles and allow one to obtain unique characteristics of modified materials. A variety of nanomaterials (Table 1) were used to modify hydraulic fracturing fluids, including spherical nanoparticles of silica and alumina with average sizes ranging

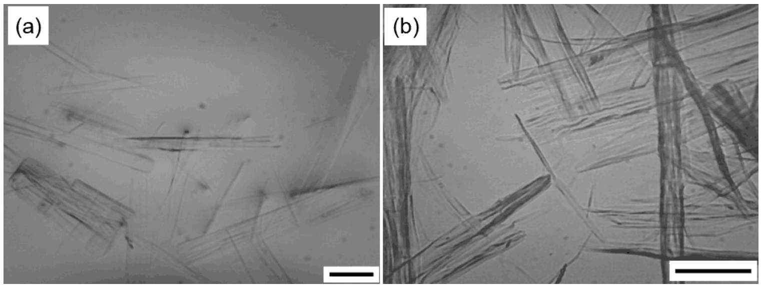


Fig. 2. Morphology of ANF bundles at (a) 4x (scale size of 100 µm) and (b) 10x (scale size of 50 µm) magnification.

from 11 to 216 nm. The concentration of nanoparticles in the gels ranged from 0.01 to 0.8 wt%.

Alumina nanofibers (with Aluminan trade mark, further designated as ANF) produced through a proprietary technology based on oxidation of aluminum melt in a controlled gas environment were used in this study. The resulting alumina nanofibers (γ-Al_2O_3) grow as an uniaxially oriented array with weak bonding between adjacent nanofibers (Fig. 2).

A detailed study of the fiber structure and morphology is provided in our previous works (Kuular et al., 2020). Transmission electron microscopy has revealed that the alumina nanofibers have a polycrystalline ribbed structure with an average diameter of 8.7 ± 2.4 nm. Therefore, the colloidal suspension of ANF used in this work is a mixture of short single ANFs 0.2-1 µm in length, and small bundles of 3-5 ANFs with a length of 1-5 µm. In addition to electron microscopy, the average effective size of ANFs was directly measured in the suspension using a DT1202 acoustic and electroacoustic spectrometer (Dispersion Technologies, USA). The analysis of the size distribution of ANF in suspension has revealed an average effective size of 525 nm, which is consistent with the electron microscopy data. The aspect ratio of Aluminan was $L/D \sim 58$ (Minakov et al., 2022). The concentration of nanofibers in the gels ranged from 0.01 to 0.4 wt%. Our work is the first to present the application of nanofibers to improve hydraulic fracturing gels.

In particular, the work includes the pioneering investigation of the effect of single-walled carbon nanotubes (SWCNTs) on the wettability characteristics of hydraulic fracturing gels. For this purpose, we used TUBALL SWCNTs (produced by OOO OCSiAl.ru, Russia). The nanotubes had an average diameter of 1.6 ± 0.4 nm and a specific surface area of 510 m^2/g according to BET analysis. Based on atomic force microscopy data, it has been found that the length of SWCNTs exceeded 4 µm. The results of SWCNT characterization by electron microscopy of their suspensions using electroacoustic spectrometry are presented in the work (Lysakova et al., 2023). The concentration of SWCNTs in the emulsions ranged from 0.01 to 0.1 wt%.

2.2 Preparation of cross-linked gels

The preparation of cross-linked gels involves several stages. First, an aqueous particle suspension is synthesized. The method of synthesizing suspensions with different nanomaterials varies. A detailed description of how to prepare suspensions of spherical nanoparticles, nanofibers, and nanotubes is provided below. After preparing the suspension, the required amount of guar gum biopolymer (Altrafine Gums, India) was added as the gelatinizing agent. Further, the suspension was stirred for 15 min. using a Prince Castle 152-18 high-speed stirrer (OFITE, USA) to prepare the linear gel. The cross-linking agent used was a solution of sodium tetraborate with 0.1 wt% glycerol. The crosslinker was added and stirred until the Weissenberg effect was obtained to create a cross-linked gel. The stirring mode remained unchanged from the time of pouring the biopolymer. Methodological studies have shown that this concentration of gelatinizing agent and crosslinker is optimal for obtaining the required rheological properties of gels as well as is economically feasible.

When using spherical nanoparticles with characteristic average size below 200 nm, suspensions were prepared using a two-step method commonly used in the synthesis of nanofluids (Chen et al., 2008). First, the required amount of nanoparticle powder was poured into a container with distilled water. Then the suspension was subjected to mechanical stirring for 15 min using a high-speed stirrer, followed by ultrasonic treatment for 10 min using ultrasonic device Volna UZTA-0.4/22-OM (22 kHz, 400 W, 50%). A similar technique was used when preparing suspensions with alumina nanofibers. A detailed study of the preparation of suspensions with nafen was carried out in (Minakov et al., 2022). In work (Minakov et al., 2022), a method for preparing colloidally stable suspensions with ANF concentrations of up to 2 wt% was developed.

The technique for dispersing stable CNT suspensions was developed by us earlier and is described in detail in (Rudyak et al., 2021, 2023). The main points are noted here. Preparing aqueous suspensions of SWCNTs is a challenging task due to the difficulty in achieving a homogeneous distribution and dispersion of nanotubes in liquid. Currently, there is no standard methodology for preparing suspensions with CNTs, however, there are several approaches that partially address this problem. One approach to separating CNT conglomerates is ultrasonic treatment, i.e., a mechanical method. This process separates CNTs that are combined into bundles and ropes. To prevent the CNTs from recombining into bundles, surfactants must be used. To prepare suspensions with SWCNTs, the required amount of SWCNT powder and surfactant were added to distilled water. Sodium lauryl sulfate was used as a surfactant to stabilize the suspension. The concentration of the surfactant remained same as the concentration of SWCNTs

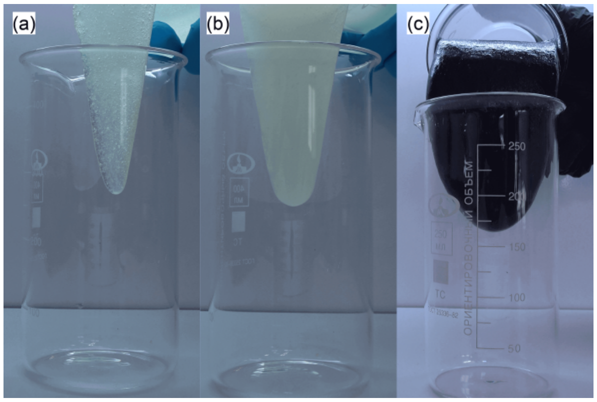


Fig. 3. Photographs of SWCNT-modified gels. (a) non-modified gel, (b) 0.1 wt% SiO_2-modified gels and (c) 0.1 wt% ANF-modified gels.

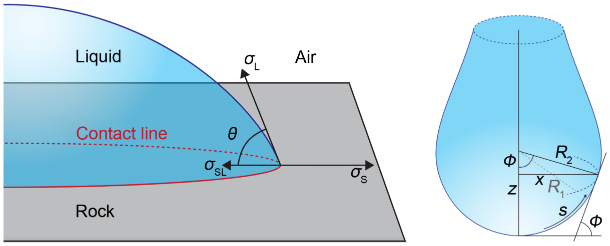


Fig. 4. Scheme for determining contact angle and interfacial tension.

in all cases. The SWCNT suspension was prepared by mechanically stirring it in an IKA high-speed stator-rotor disperser, followed by ultrasonic treatment. The preparation process involved multiple mixing stages using high-speed (20 min) and ultrasonic (20 min) dispersants. The suspension was subjected to ultrasonic treatment at maximum power for 120 min. The minimum energy to be transferred to the suspension had to be at least 2,000 W h l^{-1}.

Photographs of the prepared modified cross-linked gels are shown in Fig. 3. Visually, the nanomaterials are homogeneously distributed in the gel volume. The gels are colloidally stable. The formulation recipe of stable gels modified with nanomaterials was worked out through numerous laboratory studies.

2.3 Interfacial tension and contact angle

The interfacial tension σ and contact angle θ were investigated using previously employed methodology (Minakov et al., 2020). A DROPimage Advanced software-equipped automatic tensiometer was used to determine the gel-oil interfacial tension σ and gel-rock-air contact angle θ (Fig. 4). The interfacial tension σ was determined from measurements of pendant gel drop parameters, while the contact angle θ was measured by the captive bubble method.

The tensiometer operates on the principle of the pendant (or lying) drop method. This method determines the surface tension by measuring the dimensions of the liquid drop under study. Since the dimensions are constantly changing, the measurements and calculations of surface tension were carried out in real-time. The pendant drop method is widely employed due to its ability to work with small amounts of liquid and high temperatures, as well as chemically active materials. The accuracy of the determination can reach a few tenths of a percent when using an accurate optical instrument. The Interfacial Tension-820-P tensiometer is composed of a measuring unit with an optical cell, thermostat, liquid dosing system, pressure control system, and an interface for an external computer. It also includes DROPimage Advanced software, which enables the recording of drop images and the acquisition of all necessary characteristics.

The contact angle of the gel-rock system was measured on quartz glass plates and Berea Sandstone plates with an average porosity of 22% and permeability of 240 mD. The plate was prefabricated and secured in the holder at the bottom of the measuring cell. A gel delivery needle was placed at the top of the cell. The plate with the drop was exposed in the area of best visibility of the video camera. The light intensity was adjusted to obtain the required image contrast.

3. Research results and discussion

3.1 Contact angle

3.1.1 Effect of nanoparticle concentration on contact angle

At the beginning, the effect of spherical nanoparticle concentration on the contact angle of fracturing gels was investigated. Two types of gels were considered: Those modified with 50 nm spherical silica nanoparticles and those modified with 11 nm spherical alumina nanoparticles. Figs. 5 and 6 present typical photographs of the modified gel droplets on the substrates. The results suggest that the addition of nanoparticles has a significant effect on the contact angle.

Fig. 7 presents data on contact angle as a function of nanoparticle concentration. The results show that the addition of nanoparticles has a non-monotonic effect on the contact angle. At low concentrations of nanoparticles, the contact angle increases with increasing concentration, indicating a decrease in wettability. At concentrations above 0.2 wt%, the contact angle decreases as particle concentration increases, indicating improved wetting of the rock by the gel. The

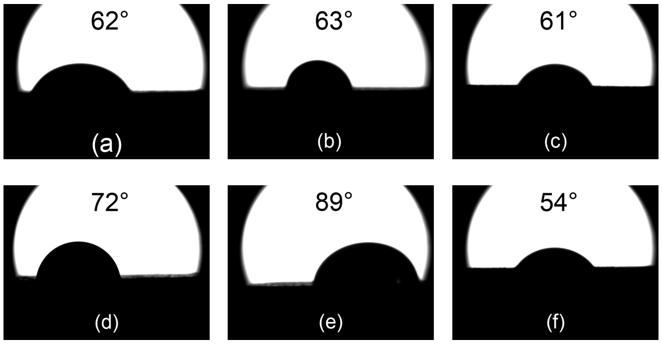


Fig. 5. Photographs of cross-linked gel droplets with different concentrations (wt%) of Al_2O_3 nanoparticles (11 nm) laying on sandstone: (a) 0, (b) 0.01, (c) 0.05, (d) 0.2, (e) 0.4 and (f) 0.8.

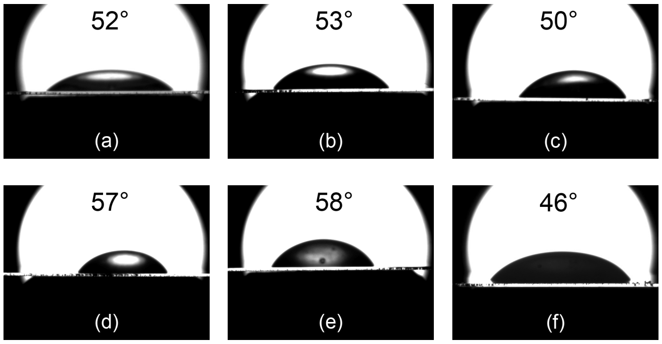


Fig. 6. Photographs of cross-linked gel droplets with different concentrations (wt%) of SiO_2 nanoparticles (50 nm) laying on glass: (a) 0, (b) 0.01, (c) 0.05, (d) 0.2, (e) 0.4 and (f) 0.8.

maximum contact angle was observed at nanoparticle concentrations of 0.1-0.2 wt%. The addition of nanoparticles can cause a significant variation in contact angle, up to 50°. It was observed that at low concentrations, nanoparticle addition worsens the wettability of fracturing gels when interacting with rock. However, at higher concentrations, it improves the wettability.

The reduction of permeability to oil in the case of joint filtration of oil and water can be caused by increased water saturation of the well bore zone in the area of fractures. Therefore, hydrophobization and deterioration of filtration conditions of hydraulic fracturing gels in the well bore zone may contribute to improvement of the oil inflow from the reservoir to the well. Sun et al. (2017) demonstrated that changing the wettability of the proppant pack can enhance oil production after hydraulic fracturing. If the formation has a high water cut, it is reasonable to use reagents that reduce contact angles to improve oil permeability. Therefore, the deterioration of wetting characteristics of hydraulic fracturing gels, found when adding small amounts of nanoparticles, is a positive fact. In this case, the optimum concentration of nanoparticles does not exceed 0.2 wt%. The results on the wettability of nanoparticle-modified gels on quartz glass plates are in agreement with the data obtained for sandstone. However, gels have better wettability on quartz glass (contact angle is lower by 15-20°).

Thus, a non-monotonic dependence on concentration was observed, with an extremum in the range of 0.1-0.2 wt%. In our opinion, such behavior is attributed to the additional cross-linking of guar molecules caused by nanoparticles at low concentrations. Currently, this mechanism has been studied just partially, for example, in Zhang et al. (2017). However, due to the presence of strong adsorption capacity of the negative surface charge of nanoparticles, electrostatic repulsion between micelles increases at high nanoparticle concentrations, which prevents cross-linking of guar molecules. Such behavior was systematically detected for the first time in our study. It is also interesting to compare the results on the effect of nanoparticles on the wettability of the obtained gels and Newtonian aqueous suspensions. At present, the effect of nanoparticles on the wettability characteristics of nanosuspensions is studied quite comprehensively. As a result, it has been shown that the hydrophilic behavior of rock monotonically increases with increasing nanoparticle concentration (Minakov et al., 2021). This enhances the washing of oil from rock and is the primary mechanism for improving oil recovery when adding nanoparticles. The behavior of wettability of gels is more complicated, characterized by non-monotonic dependence on the concentration of nanoparticles.

3.1.2 Effect of nanoparticle size and material on contact angle

The effect of nanoparticle size and material on the wetting properties of cross-linked hydraulic fracturing gels was inves-

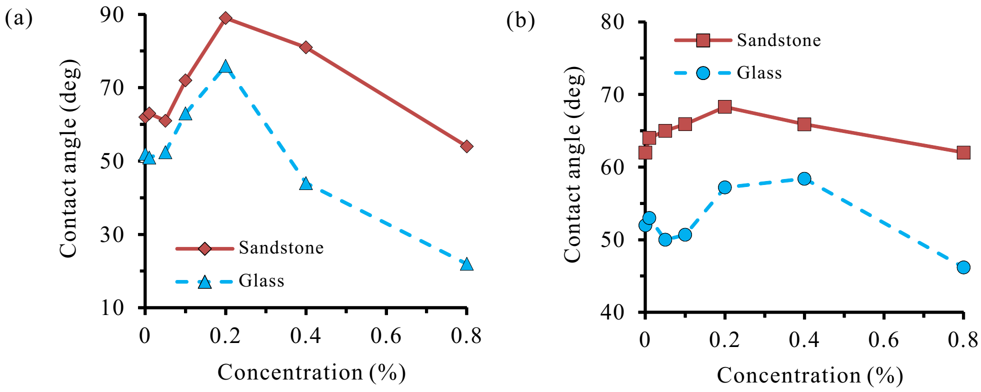


Fig. 7. Dependence of the contact angle in the system consisting of air, nanoparticle-modified gel, and rock on the nanoparticle concentration: (a) Al_2O_3, 11 nm and (b) SiO_2, 50 nm.

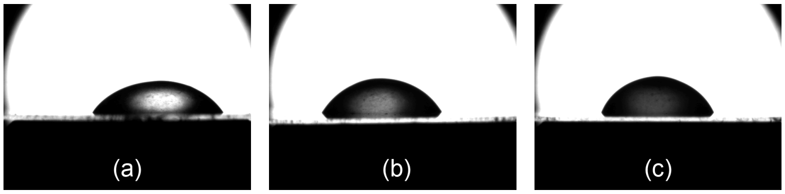


Fig. 8. Photographs of cross-linked gel droplets with 0.1 wt% Al_2O_3 nanoparticles with different average diameter laying on the glass: (a) 11 nm, (b) 38 nm and (c) 76 nm.

tigated. For this purpose, silica and aluminum oxide nanoparticles with sizes ranging from 11 to 216 nm (corresponding to specific surface areas from 7 to 150 m^2/g) were considered at the same weight concentration of 0.1%. The results showing the effect of size and material of spherical nanoparticles are shown in Figs. 8 and 9. The results of the study indicate that for both materials the dependence on the average nanoparticle size is also non-monotonic. Initially, the surface wettability of the hydraulic fracturing gels deteriorates with increasing average nanoparticle size. At a nanoparticle concentration of 0.1 wt%, changing the average size of the Al_2O_3 nanoparticles from 11 to 76 nm results in an increase in contact angle of approximately 15°. These data are consistent with our recent studies on the effect of nanoparticle additives on the wettability of fluids used for reservoir flooding (Minakov et al., 2020).

It was shown that as the size of the nanoparticles in the nanosuspension decreases, their hydrophilicity increases. However, with further increase in particle size, the dependence of wetting properties on nanoparticle concentration in nanosuspensions and nanoparticle-modified cross-linked gels differs significantly. In cross-linked gels the dependence of the contact angle on the nanoparticle size has an extreme. The maximum contact angle is observed at an average nanoparticle size close to 70-80 nm. With further increase in nanoparticle size, the hydrophilicity of the nano-modified gels increases.

Thus, there is an average nanoparticle size at which the hydrophobic properties of the gels are most pronounced. This behavior is reported for the first time in this work. On the one hand, as the average nanoparticle size decreases, the specific surface area of the nanoparticles increases significantly and the number of nanoparticles per unit volume increases. This increases the electrostatic repulsion between the micelles, which prevents the cross-linking of the guar molecules. On the other hand, if the nanoparticles are too large, the specific area and number of nanoparticles are greatly reduced, they become normal macroscopic particles and their effect on electrostatic repulsion is weakened. At the same time, however, their influence on the additional cross-linking of the guar molecules is reduced. Therefore, there is an optimum nanoparticle size at which these competing processes are compensated. Similar behavior was observed for Al_2O_3 and SiO_2 nanoparticles (Fig. 9). The sensitivity of the gels to the addition of silica nanoparticles is found to be higher. The results obtained on silica glass plates are in agreement with those obtained on sandstone plates. The effect of nanoparticle size is weaker on glass substrates.

3.1.3 Effect of particle shape on contact angle

In addition to the modification of hydraulic fracturing gels with spherical nanoparticles, the addition of non-spherical nanomaterials was investigated. For this purpose, the addition of alumina nanofibres and single-walled carbon nanotubes (SWCNTs) was considered. The effect of alumina nanofibres additives and SWCNTs on the properties of fracturing fluids is first investigated in the present paper. Typical results on the effect of the concentration of these nanoadditives are shown in Figs. 10 and 11. The study has found that the wettability of sandstone gels decreased as the concentration of SWCNTs increased. Meanwhile, the effect of SWCNT additives on the contact angle starts at low concentrations. The study demon-

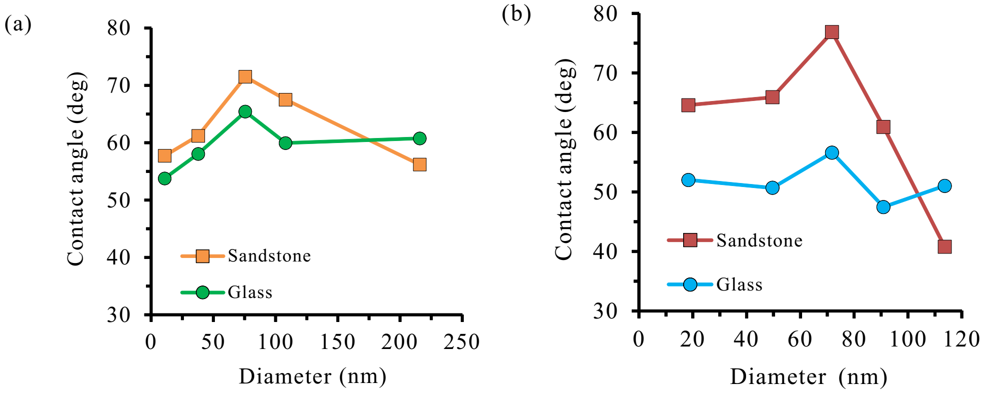


Fig. 9. Contact angle for gels with 0.1 wt% nanoparticle additive of different average size: (a) Al_2O_3 and (b) SiO_2.

Fig. 10. Photographs of cross-linked gel droplets on sandstone at different concentrations (wt%) of SWCNTs: (a) 0.01, (b) 0.025, (c) 0.05 and (d) 0.1.

strates that the optimal concentration of the additive is 0.05 wt% SWCNT. The primary interaction mechanism between nanotubes and guar gum molecules involves the formation of a robust cross-linked structure through the adsorption of micelles and the reinforcement of hydrogen bonds between nanotubes and the thickener. The analysis of the effect of adding alumina nanofibers (Fig. 11(b)) did not reveal a significant impact on the contact angle of the gels on either sandstone or glass.

Thus, it has been shown that controlling the concentration, size, material, and morphology of nanomaterials can significantly alter the contact angle of hydraulic fracturing fluids. Additives worsen the wetting characteristics of gels at low concentrations and large particle sizes, but improve them at relatively high concentrations and small particle sizes. This enables more flexible control of the fracturing process based on reservoir characteristics.

3.2 Interfacial tension

3.2.1 Effect of nanoparticle concentration on interfacial tension

A systematic study was conducted to investigate the impact of different nanomaterial additives on the interfacial tension coefficient of cross-linked gels used in hydraulic fracturing. The interfacial tension was measured based on droplet geometry. The results obtained are presented in Fig. 12. When measuring the interfacial tension of gels, it is important to consider that the change in interfacial tension occurs in two stages: During the droplet leveling process and during the detachment process. The high viscosity of gels can cause the droplet to have a rounded or elongated initial shape, which gradually changes to an equilibrium shape. When the initial droplet shape is rounded, the interfacial tension decreases, reaches a minimum (which is considered the true value) and then increases. On the other hand, when the initial droplet shape is elongated, the interfacial tension gradually increases approaching the true value.

The study has investigated the relationship between interfacial tension and the concentration of nanoadditives. The results of interfacial tension measurements in the gel-air system are presented in Fig. 13. The analysis of the measurement results has revealed that the interfacial tension coefficient increases with increasing concentration of oxide nanomaterial additives, reaching a maximum at a concentration of 0.1-0.2 wt%, and then monotonically decreases. This behavior is consistent with the dependence of the contact angle on concentration. The interfacial tension coefficient increased by approximately 20% with the addition of 0.1 wt% SiO_2 nanoparticles. A similar increase was observed for gels modified with alumina nanofibers. However, a more significant effect (approximately 30%) was observed for gels modified with 0.1 wt% SWCNTs. Here, the interfacial tension increased consistently with increasing the concentration of nanotubes. This increase in the interfacial tension coefficient of gels for hydraulic fracturing fluids could be beneficial as it would hinder their deep penetration into the reservoir during fracturing, thereby reducing formation contamination.

3.2.2 Effect of nanoparticle size on interfacial tension

Next, the effect of nanoparticle size on the surface tension coefficient of nanomodified hydraulic fracturing gels was investigated. For this purpose, additions of silica and alumina nanoparticles were considered at a fixed concentration of 0.1 wt%, other factors being equal. The average size of the nano-

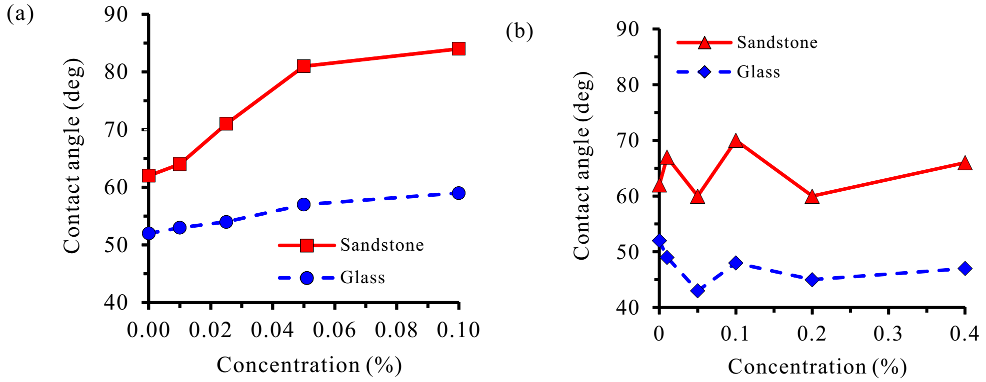


Fig. 11. Contact angle in the system “air-modified gel-surface”: (a) SWCNTs and (b) ANF.

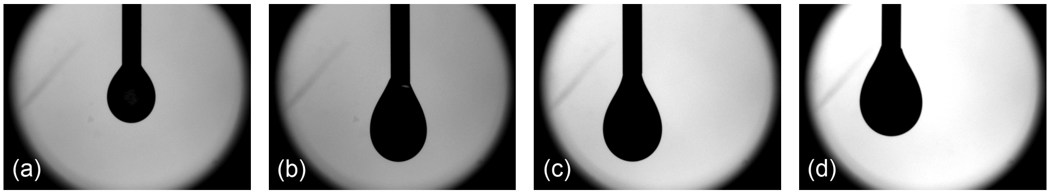


Fig. 12. Photographs of cross-linked gel droplets with added SWCNT. (a) 0.01 wt%, (b) 0.025 wt%, (c) 0.05 wt% and (d) 0.1 wt%.

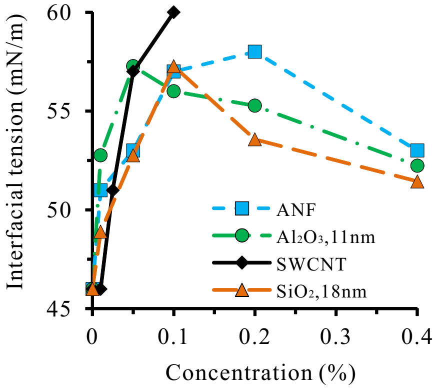


Fig. 13. Interfacial tension depending on particle concentration.

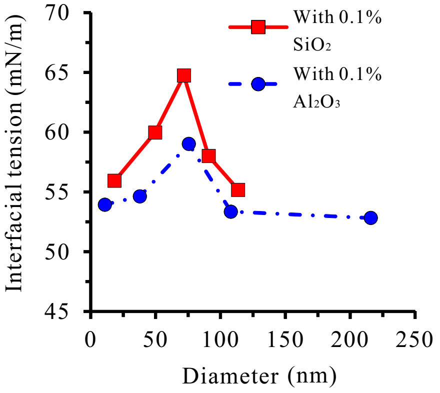


Fig. 14. Interfacial tension of gels modified with 0.1 wt% nanoparticles of different sizes.

particles varied in a wide range from 11 to 216 nm. The results of the measurements are shown in Fig. 14. For both nanoparticle materials, a non-monotonic behavior of the interfacial tension of the gels was found as a function of the average nanoparticle size. The interfacial tension of the gels reaches a maximum at an average nanoparticle size of about 50-70 nm. The interfacial tension of the gel varies by 30% as the particle size changes. Other factors being equal, silica nanoparticles have a greater effect on the interfacial tension coefficient than alumina nanoparticles. The presence of a non-monotonic dependence of gel interfacial tension on nanoparticle size allows for more flexible control of the hydraulic fracturing fluid properties.

3.3 Capillary imbibition of hydraulic fracturing gels

Capillary imbibition is the process of spontaneous displacement of a liquid or gas from a porous medium by another immiscible liquid under the action of capillary forces. Capillary forces are caused by surface phenomena that occur at the interface between a liquid and another medium. The occurrence of such phenomena is associated with the curvature of the liquid surface as a result of surface tension. This phenomenon plays an important role in the displacement of oil and gas from heterogeneous porous and fractured porous reservoirs.

Capillary imbibition is undoubtedly a complex process. The imbibition rate depends not only on the wettability, but also on the relative permeability, viscosity, surface tension of the fluids, the structure of the pore space, and the initial water and oil saturation. However, the results obtained in this work

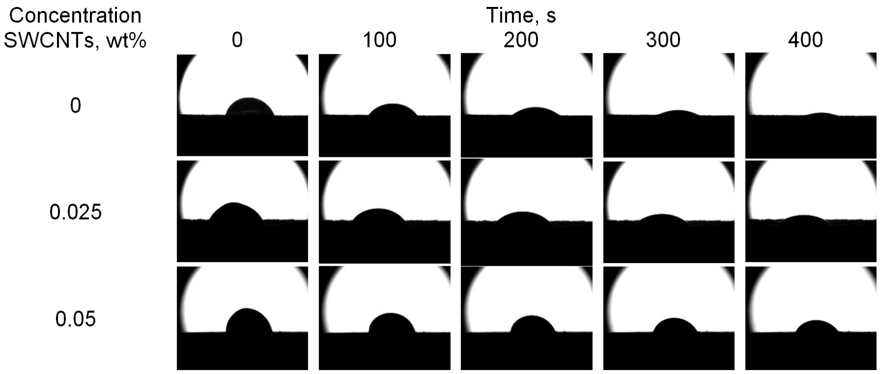


Fig. 15. Photographs of SWCNT-added gel drop on sandstone during the imbibition process.

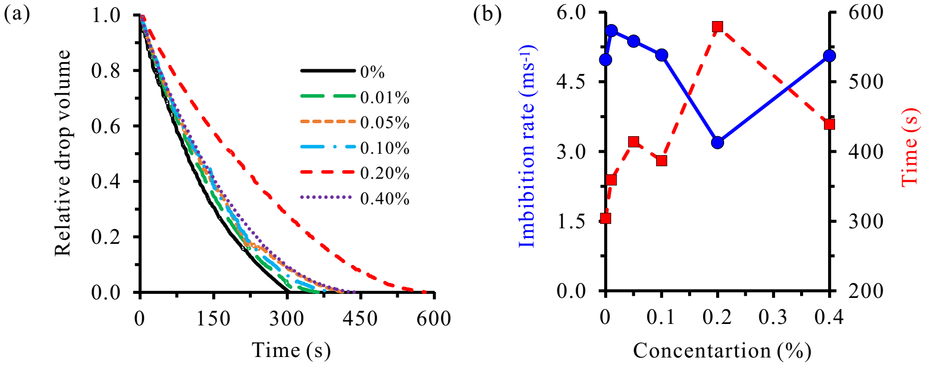


Fig. 16. Capillary imbibition of SiO_2-added gels (18 nm) in sandstone. (a) Variation in relative droplet volume and (b) Initial imbibition rate and Total imbibition time.

can be used to estimate the penetration rate of hydraulic fracturing gels into the rock. A study of the effect of nanoparticles on the capillary imbibition rate of nanosuspensions in oil-saturated rock is presented in our recent work (Pryazhnikov et al., 2024).

3.3.1 Effect of nanoparticle concentration on capillary imbibition of gels

A study of the capillary imbibition rate of sandstone with nanomodified hydraulic fracturing gels was carried out. The experiments were conducted as follows: A drop of gel was placed on a core plate cut from sandstone, and gradually absorbed into the sample (Fig. 15). The entire process was recorded by camera. The volume of the drop during the imbibition process was determined by software. The effect of nanoparticle concentration, size, and material on the variation of the relative drop volume was investigated.

Typical results of drop imbibition experiments on a sandstone substrate are shown in Figs. 16 and 17. Here, the plots show the change in relative volume of the drop, reduced to its initial volume during its imbibition, as well as the imbibition rate, and the time during which the drop was completely absorbed into the sample. Analysis of the experimental data as a function of nanoadditive concentration allowed identifying the following patterns. It was found that the imbibition rate of nanomodified gels depends significantly on the nanoparticle concentration. For example, the addition of 0.2 wt% silica nanoparticles (18 nm in diameter) reduces the gel imbibition rate by 1.8 times (Fig. 16), while the addition of 0.05 wt% SWCNTs reduces it by 3 times (Fig. 17). At the same time, the dependence of the imbibition rate on the concentration also has a non-monotonic character. The plots of imbibition rate and time as a function of concentration show extreme points. In general, the data show that the minimum imbibition rate is observed at the nanoadditive concentrations at which the maximum increase in contact angle and interfacial tension of the gels is observed. Reduced capillary imbibition time is a positive factor for hydraulic fracturing fluids because it prevents the formation of contaminants and the loss of the fluid itself.

To explain the mechanism of the dependence of the rate of capillary imbibition on the concentration of nanoadditives, an analysis of the intensity of the capillary imbibition process was performed based on the classical Lucas-Washburn model (Lucas, 1918; Washburn, 1921) According to the work (Lucas-Washburn), the height of the capillary rise of liquid h in the tube can be estimated by the formula:

$$h(t)=\sqrt{\frac{r\sigma\cos\theta}{2\mu}t} \tag{1}$$

where r is the radius of the capillary, σ is the interfacial tension, μ is the viscosity of the Newtonian fluid, θ is the contact angle, t is time.

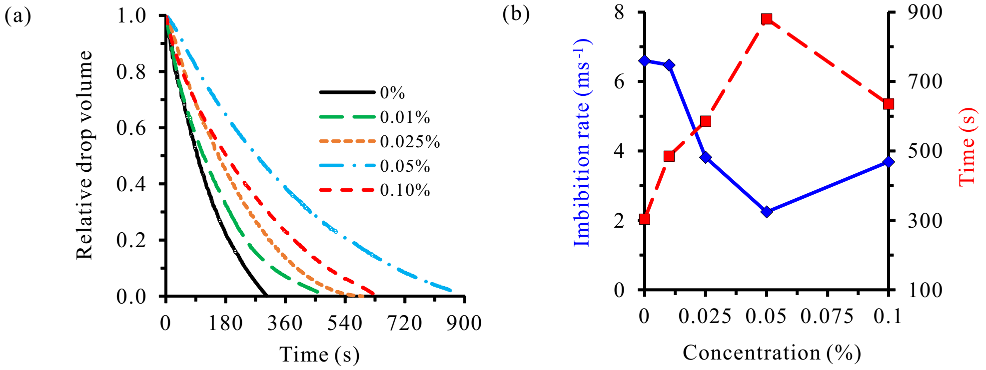


Fig. 17. Capillary imbibition of SWCNT-added gels in sandstone. (a) Variation in relative drop volume and (b) Initial imbibition rate and Total imbibition time.

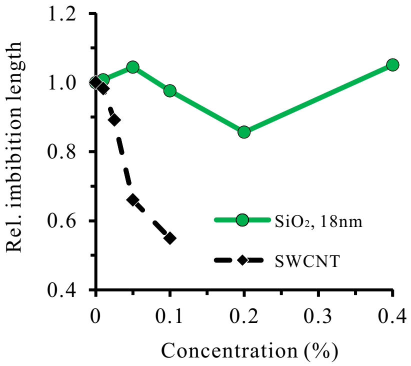


Fig. 18. The relative length of imbibition depends on the concentration of nanomaterials.

This expression is often used to estimate the length of capillary imbibition into a porous medium. In this case, the tube radius is replaced by the effective pore radius (Lundblad and Bergman, 1997). Unfortunately, it is impossible to use this expression in our case, since it describes the behavior of Newtonian fluids. In our case, the imbibition of gels with complex rheology with viscoelastic flow characteristics is considered. An expression h_{nm}/h_0 is used to determine the behavior of the relative length of capillary imbibition, which characterizes the ratio of the length of capillary imbibition of gels modified with nanomaterials compared to the base cross-linked gel (without nanomaterials):

$$\frac{h_{nm}}{h_0} \sim \sqrt{\frac{\sigma_{nm}\cos\theta_{nm}}{\sigma_0\cos\theta_0}} \tag{2}$$

where the index "nm" and "0" denote gels modified with nanomaterials and the base cross-linked gel, respectively.

Fig. 18 shows the dependence of the relative depth of imbibition on the concentration of aluminum oxide nanoparticles and carbon nanotubes. At low concentrations of nanomaterials, this value decreases several times. Qualitatively, this dependence on concentration is in good agreement with the experimentally measured dependences of the imbibition rate shown in Figs. 16 and 17. The experiment also observed a decrease in the imbibition rate by several times at low concentrations of nanoparticle additives. Thus, it can be concluded that the main mechanism behind this behavior is the changes in contact angle and interfacial tension upon addition of nanomaterials to hydraulic fracturing gels.

3.3.2 Effect of nanoparticle size on capillary imbibition of gels

Similar to the wetting and interfacial tension studies, the effect of average size on the capillary imbibition process of gels in sandstone was investigated. Investigation of the effect of nanoparticle size has shown that the imbibit ion rate of the nanomodified gel also depends on the average size of the nanoparticles (Fig. 19). The experimental data on imbibition rate and time of gels with 0.1 wt% Al_2O_3 and SiO_2 nanoparticles in sandstone are shown in Fig. 20. As can be seen, the dependencies on the average particle size also have an extremum in the neighborhood of 70-80 nm, which is close to the corresponding extremes for the contact angle and the interfacial tension. At these sizes, the capillary imbibition rate has a minimum. It was found that by controlling the nanoparticle size it is possible to change the capillary imbibition rate by a factor of about 1.5, all other factors being equal. In this case, the effect of the nanoparticle material is not so significant.

The capillary imbibition results obtained for the hydraulic fracturing gels are in qualitative agreement with the interfacial tension and contact angle measurements. The reason for this behavior can be explained analyzing the behavior of one of the main driving forces in filtration, which is the capillary pressure $p = 2\sigma\cos\theta/r$ (Zhang et al., 2023b). Other things being equal, a change in surface tension and contact angle as a function of nanoparticle concentration and size leads to a corresponding change in capillary force and hence in the imbibition rate of the nanomodified gel. At the same time, the contribution of the interfacial tension is not so significant (about 20%), and the main contribution to the capillary pressure is made by the $\cos\theta$.

4. Conclusions

A systematic study of the wettability, interfacial tension, and capillary imbibition properties of hydraulic fracturing

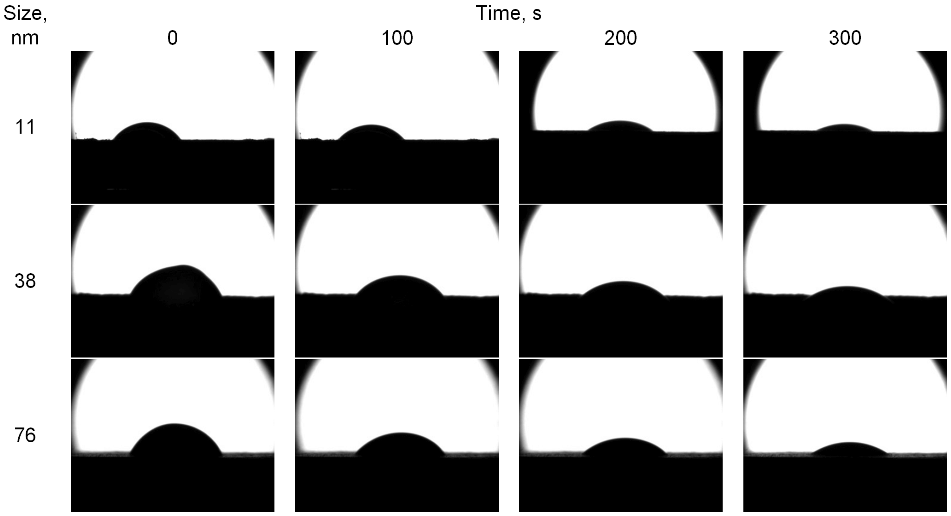
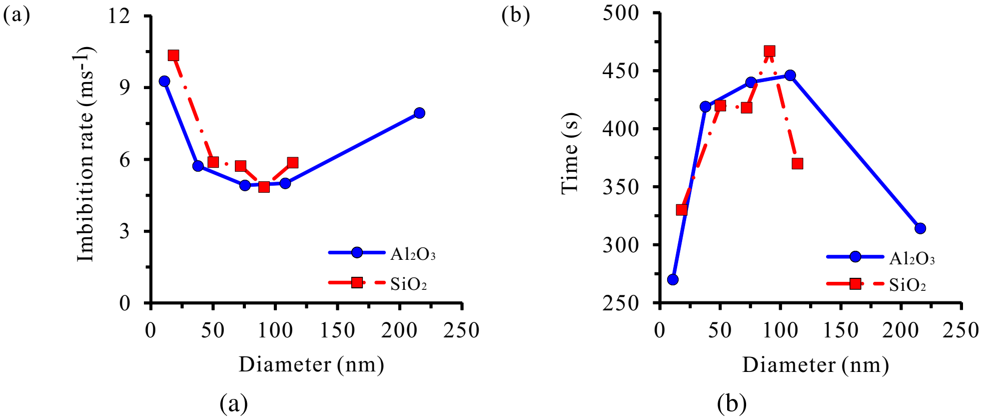


Fig. 19. Photographs of the gel drop with the addition of 0.1% Al_2O_3 nanoparticles of different sizes on sandstone during the imbibition.



(a) (b)

Fig. 20. (a) Initial rate of relative volume reduction and (b) total imbibition time and of a gel drop with 0.1 wt% Al_2O_3 and SiO_2 nanoparticles on sandstone as functions of nanoparticle diameter.

gels modified with different nanomaterials was performed. Spherical silica and alumina nanoparticles, SWCNTs, and alumina (Aluminan) nanofibers were used as nanoadditives. A systematic study of the effect of nanoadditive concentration, size, material, and shape on the studied properties of gels was performed for the first time.

The following conclusions were drawn in the course of the study.

1) Analysis of the results has shown that the addition of nanoparticles has a non-monotonic effect on the contact angle and interfacial tension of hydraulic fracturing gels. It was found that nanoparticle addition at low concentrations worsens the wettability of the gels when interacting with the rock, and increases the interfacial tension, while at higher concentrations it improves the wettability and decreases the surface tension. The maximum of contact angle and interfacial tension was observed at nanoparticle concentrations of 0.1-0.2 wt%.
2) For the first time it was shown that in cross-linked gels the dependence of the contact angle and the interfacial tension on the average size of the nanoparticles has an extreme. The maximum hydrophobicity in nanomodified gels was observed at an average nanoparticle size of about 70-80 nm. As the nanoparticle size decreases or increases with respect to this value, the hydrophilicity of nanomodified gels increases.
3) Other factors being equal, silica nanoparticles have a greater effect on the wettability characteristics and interfacial tension compared to alumina nanoparticles.
4) The effect of alumina nanofibers and SWCNT additives on the properties of gels was investigated for the first time. It was found that the addition of SWCNTs significantly affects the wettability (increasing the hydrophobic behavior) and increases the interfacial tension (by 30%) even at very low concentrations of 0.05-0.1 wt%. At the same time, the analysis of the effect caused by alumina nanofibers has shown no significant influence of this additive on the contact angle.

5) It is shown that the imbibition rate of nanomodified gels depends significantly on the nanoparticle concentration. For example, the addition of 0.2 wt% of silica nanoparticles (with diameter of 18 nm) reduces the gel imbibition rate by a factor of 1.8, while the addition of 0.05 wt% SWCNTs reduces it by a factor of 3. In the case of spherical nanoparticles, the dependence of the imbibition rate on concentration and average size is also non-monotonic. The minimum imbibition rate was observed at nanoadditive concentrations and sizes at which the maximum increase in contact angle and interfacial tension were observed.

It has been shown that by controlling the concentration, size and morphology of additives, it is possible to control the properties of hydraulic fracturing fluids. Deterioration of the wetting properties of hydraulic fracturing gels, increase in their interfacial tension and, as a consequence, increase in the capillary imbibition rate, observed with the addition of optimal concentration and size of nanoparticles, is a positive fact, because it will contribute to hydrophobization and deterioration of the filtration conditions of hydraulic fracturing gels in the wellbore region, which, in turn, can contribute to improvement of the oil inflow from the reservoir to the wellbore. At the same time, the optimal concentration of nanoadditives does not exceed 0.2 wt% for spherical nanoparticles, and 0.05 wt% for SWCNTs. This opens up the prospect of using nanoadditives to control the properties of hydraulic fracturing fluids. This can lead to more efficient management of the hydraulic fracturing operation.

Acknowledgements

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Conflict of interest

The authors declare no competing interest.

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