Nanosilica-Based Loss Circulation Composition to Cure Moderate to Severe Losses

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The loss circulation composition comprising a combination of a nanoparticle-based dispersion and a chemical activator has been designed to treat moderate to severe losses. The nanomaterial used is an environmentally friendly nanosilica-based dispersion. The composition is designed to give delayed gelling of the nanoparticle-based dispersion. A major advantage of this technology is its ability to place the composition into the target loss circulation zone before the nanoparticle-based dispersion gels up. Premature gelling of the nanoparticle-based dispersion would avoid premature setting of the treatment fluid before it reaches the target zone. The newly developed system can be used effectively up to 300 °F. In this paper, experiments have been performed with three different types of nanoparticles differing in their surface charges and particle sizes. Two negatively charged nanoparticle-based dispersions with a particle size of 5 nm and 17 nm, respectively, and one positively charged nanoparticle-based dispersion with a particle size greater than 17 nm have been evaluated as loss circulation materials. Two different types of chemical activators, one organic and the other inorganic, have been used in this study, and their effect on the gelling time has been evaluated. The gelling time experiments have been done at four different temperatures viz. 150 °F, 200 °F, 250 °F, and 300 °F. The effect of activator concentration and different shear rates on the gelling time of the three nanoparticle-based dispersions has been studied. Permeability plugging tests have been performed using 2 mm slotted disks to evaluate the effectiveness in controlling moderate to severe losses. [DOI: 10.1115/1.4049600]

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Introduction

Loss circulation is defined as partial or total loss of the circulating drilling fluid into the formation [1]. There are many reasons which can lead to loss of fluid to the formation. The drilling fluid is lost when it enters depleted zones, natural or induced fractures, low-pressure zones, etc. Uncontrolled or total loss of a drilling fluid into the formation is not desirable as it can lead to pressure control situation and loss of the well [2,3].

The severity of loss circulation depends on the extent to which a drilling fluid is lost to the formation. Depending on the extent to which a drilling fluid is lost to the formation, loss circulation can be classified into minor loss or seepage loss (less than 10 barrels/hour (bbl/h)), severe loss (for example, greater than 100 bbl/h), and higher or complete fluid loss [4]. Loss circulation can lead to a host of different problems which include well-control issues, stuck-pipe, reservoir damage due to plugging of pores and pore throats by mud particles, and poor hydrocarbon production. These problems eventually result in nonproductive time and severe economic losses.

Different Treatment Fluids to Control Loss Circulation. Different types of loss circulation treatments and methods involving the use of novel additives have been described in the literature to treat lost circulation. Most of the lost circulation treatments described in the literature are based on micro and macro-particles.

A novel shear-sensitive plugging fluid designed to gel rapidly has been described. This fluid which has been described as a "loose" invert emulsion gels rapidly after passing through the bottom-hole assembly in normal drilling operations thereby curing massive mud losses [5]. Eco-friendly loss circulation materials (LCMs) made from an edible herb have been described. These loss circulation materials form a bridge that helps to reduce the losses. These new loss circulation materials were then used to cure losses in different wells with good success [6]. Polyurethane grouting has been applied to a lost circulation zone in a geothermal well. The success of the grouting of the loss zone was attributed to packing off the hole and squeezing the grout, using sufficient polyurethane to sweep away the drilling mud and controlling the gel time [7]. A cross-linked polymer gel has been described with controlled gelation time and high gel strength. The polymer, prepared from an acrylamide monomer, crosslinking agent, and initiator by free radical polymerization, can be used at high temperatures. The polymer gel, which has been designed to withstand pressures up to 9.8 MPa at 150 °C, can effectively plug 3 mm-width slot [8]. A cross-linked polymeric gel comprising hydrolyzed polyacrylamide (PAM) and derivative of hydrolyzed polyacrylamide along with polyethyleneimine has been described. An optimum concentration of the additives involved in the gel was required to form a stable and rigid gel. The gel was shown to cure losses in a zone having 36% porosity and 300D permeability at room temperature and moderately high-pressure conditions [9]. The cross-linked organic polymer gellant system comprising polyacrylamide, phenol, and formaldehyde has also been studied. It was observed that the gel system was capable of preventing losses in porous media at room temperature and moderately high-pressure conditions [10]. Metal ions such as magnesium, manganese, and calcium when injected into the drilling fluid containing polymers such as polyacrylamide help to increase the viscosity of the drilling fluid and subsequently form a gel. This gel can be employed to seal a loss circulation zone [11]. A smart magnetorheological fluid based on a mixture of a cross-linkable polymer (PVA) solution and magnetic microparticles has been described as a loss circulation material to mitigate

loss circulation. Chromium (III) acetate was found to be the most efficient for the PVA crosslinking reaction carried out in the presence of iron particles and under magnetic field. The results showed that a mixture of cross-linkable polymers and magnetic particles has the potential to perform as loss circulation control [12]. Double absorbent water-soluble resins have been described as loss circulation materials that can be used at temperatures up to 150 °C for more than 30 days. The resin after being pumped into the formation fracture has been designed to expand 5–10 times its original volume, adsorb on the fracture surface, and form a compact sealing layer [13]. Engineered fiber-based loss circulation control pills, based on a specially engineered fiber system and particle size distribution (PSD) principle, were developed to control loss circulation. The pills were designed in order to cure losses in fractures that are up to 5 mm in width [14]. A new chemical-sealant-based loss circulation material was developed to cure severe-to-total losses in highly fractured formations. The new sealant, which is dispersed in a non-aqueous carrier fluid, quickly forms a highly malleable viscous mass upon exposure to an aqueous reactant fluid and then subsequently sets harder under a wide range of temperatures [15]. The use of appropriately sized lost circulation materials has been shown to change the particle size distribution of a drilling fluid. A combination of loss circulation materials and wellbore strengthening materials was effective in bridging off the pore throats and microfractures thereby preventing fracture growth and subsequent fluid loss. A number of experiments have been conducted whereby wellbore conditions have been simulated to study the effect of mud solids type and particle size distribution on fluid loss reduction [16–20].

Class G and special blended cement with various amount of local bentonite and sugar cane fiber has been invested as loss circulation material. A cement system with 9% local bentonite, 2% calcium chloride, and 0.5% sugar cane fiber resulted in a permeability reduction of about 10% and gave optimum loss circulation control [21]. The use of a foamed acid-soluble cement system to mitigate loss circulation has also been investigated. The acid solubility of such a system makes it advantageous to be used in potential producing formations [22]. A tunable cement-based lost circulation treatment solution has been developed to cure partial and total losses. The lost circulation treatment solution, which is thixotropic, can be used in a density range of 10–15 lb/gal and working at temperatures up to 250 °F. The treatment formulation has been designed to develop rapid static gel strength and an early compressive strength up to 500 psi [23]. Two different types of acid-soluble cement systems namely traditional Portland cement-based system with appropriate amount of $CaCO_3$ and magnesium oxychloride cement have been described. In both these systems, sized $CaCO_3$ has been used to enhance the bridging effect at highly permeable formations. In addition to bridging capabilities, these fluids can be foamed to enhance the capability of preventing severe lost circulation [24]. Another cement plug has been described which has been designed to remediate total losses in the depleted zone. A conventional Class G cement slurry with bentonite gel has been designed to give rapid gel-strength development. The slurry was designed at 24% solid volume fraction with engineered LCM, which is a combination of a flake-solids package and synthetic fibers [25]. A novel acid-soluble formulation in the form of an engineered composite LCM solution with a multi-modal PSD has been described to manage severe-to-total loss circulation situations in naturally fractured reservoir formations. The solution developed was shown to plug laboratory-simulated fracture sizes in the range of 5000 μm [26]. A cementitious composition admixed with synthetic fibrous LCM has been used to prevent losses. The fiber-based cementitious slurry allows setting in fracture zones and causes strong impermeable net to prevent losses. The cementitious formulation designed with ground granulated blast furnace slag and silica fume admixed with synthetic LCM formed a slurry which is activated with calcium hydroxide [27]. Ultra-low-density cement slurries less than 5.5 lb/gal have been described to cure loss circulation in extremely depleted zones. These low-density or foamed cement systems have been designed

with a special acid-soluble cement along with some polymers. The cement slurries have been designed to manage low-fracture gradient formations by reducing loss circulation challenges [28]. Cross-linked cement systems comprising a stoichiometric mixture of magnesium and calcium oxides, carbonates, and sulfates have been described. The cement system was mixed with carboxymethyl hydroxyethyl cellulose as a gellant and a zirconium cross linker. The cement system crosslinks and sets rapidly when placed in a lost circulation zone and is 97% acid soluble [29]. Cement squeezes have been performed through bottom-hole drilling assemblies and eliminate the dependence on correct sizing of bridging particles. Large volume of cement squeezes were proven effective to restore wellbore integrity after loss rates as high as 320–480 bbl/h and total volume of losses to the formation of up to 4000 bbl prior to treatment [30]. However, almost all of the loss circulation treatments show both advantages as well as certain disadvantages. The control and prevention of loss of circulation by micro and macro material-based treatment fluids, however, has shown limited success in the oil industry. This has spurred the research on the development and use of nanoparticle-based fluids as effective solutions to mitigate loss circulation.

Use of Nanoparticles in Curing Loss Circulation. A summary of different nanomaterials and their use in controlling losing circulation is given in Table 1.

A new cross-linked nanocomposite organic/inorganic gel-based pill containing swelling polymer and colloidal particles has been described to control severe losses. The formulation, which can be easily prepared on site, gave good properties in terms of ease of pumping through drill pipes and the ability to adjust the setting time of the nanocomposite pill. This gel can be pumped in naturally fractured formation in order to seal a wide range of width cracks. The cross-linked gel showed good stability during the degradation tests performed at 120 °C for 16 h [2]. The use of nanoparticles such as silicon nanoparticles to combat loss circulation has been described. The nanoparticles aid in the formation of a more continuous and integrated mud-cake. The uniform particle size distribution provided better compaction medium which restricted the flow of liquids from the drilling fluid into the formation. The integrated mud-cake having low permeability and porosity prevents the filtrate from entering the formation thereby preventing fluid loss [31]. The use of in-house developed calcium-based and iron-based nanoparticles were investigated as lost circulation materials in invert-emulsion drilling fluids in order to minimize formation damage. Test results showed that the drilling fluids formulated with these nanoparticles were able to reduce the fluid loss compared with the control sample [32,33]. Two nanoparticles which were developed in situ (within the oil-based drilling fluid) and ex situ (within an aqueous phase) were tested as loss circulation materials for drilling fluids. Under the low-pressure and low-temperature API test, more than 70% reduction in fluid loss was achieved in the presence of nanoparticles as compared with only 9% reduction in the presence of conventional LCMs [34]. Oil-based drilling fluids with calcium and iron-based nanoparticles were developed and tested for their fluid properties. It was shown that drilling fluids formulated with a combination of conventional LCMs with these nanoparticles could significantly reduce fluid loss and create a thinner filter cake, compared with fluids containing conventional LCM alone. Due to their superior properties, these nanoparticles were able to fill the gaps between the micron-sized particles, leading to lower permeability and decreased filtrate flux [35]. The optimum concentration of a blend of standard LCM (graphite) and in-house prepared nanoparticles (iron III hydroxide and calcium carbonate) was developed as an effective LCM for drilling fluids. The LCM blend was effective in increasing the fracture pressure by 70% over the unblended water-based drilling fluid [36]. An additive package comprising carbon black nanoparticles and polymers was designed for drilling fluids which was able to substantially reduce fluid loss [37]. Incorporation of iron (III) hydroxide nanoparticles in

Table 1 Summary of different nanoparticles used in controlling loss circulation

Type of nanoparticles	Type of LCM	References
Nanocomposite organic and inorganic gel	Sealing pill containing cross-linked polymer gel containing polymer and colloidal particles.	[2]
Silicon nanoparticles	Silicon nanoparticles used as additives in the drilling fluid to mitigate loss circulation and differential sticking.	[31]
Iron-based and calcium-based nanoparticles	Iron-based and calcium-based nanoparticles in combination with graphite were evaluated as conventional lost circulation material in oil-based muds.	[32,33]
In situ and ex situ in-house synthesized nanoparticles	Drilling fluids formulated with the in-house synthesized nanoparticles were able to reduce the fluid loss compared with the control sample.	[34]
Iron-based and calcium-based nanoparticles	Combination of nanoparticles and LCM could significantly reduce fluid loss and create a thinner filter cake, compared with fluids containing LCM alone.	[35]
Iron (III) hydroxide and calcium carbonate	Loss circulation material drilling fluid blend containing optimum concentration of in-house developed nanoparticles and graphite was developed.	[36]
Carbon black particles	Additive package comprising carbon black nanoparticles and polymers was substantially able to reduce fluid loss.	[37]
Iron (III) hydroxide	Incorporation of iron (III) hydroxide nanoparticles incorporated in invert-emulsion drilling fluids resulted in the substantial reduction of fluid loss.	[38]
Silica and iron oxide nanoparticles	Silica nanoparticles as LCM performed better than iron oxide nanoparticles in terms of reduction in fluid loss.	[39]
TiO_2/polyacrylamide nanocomposite	TiO_2/polyacrylamide nanocomposites resulted in enhanced the rheological properties of water-based drilling fluid and 64% reduction in fluid loss.	[40]

invert-emulsion drilling fluids resulted in a substantial reduction of fluid loss. Nanoparticles and nano-induced aggregates were thought to be able to fill the gaps between rock grains as well as larger LCMs to provide more resistant seal over the fracture openings [38]. A comparison of silica and iron oxide nanoparticles was done in terms of their capacity to reduce fluid losses. It was observed that silica nanoparticles when added as an LCM to the reference mud was able to reduce the fluid loss by 48.3% as compared with the iron oxide nanoparticles which reduced the fluid loss by only 37.9% [39]. TiO_2/PAM nanocomposites have been used as an additive in water-based drilling fluids. The nanocomposites enhanced the rheological properties and resulted in 64% reduction in fluid loss. This additive was obtained through the polymerization of acrylamide monomer in the presence of TiO_2 nanoparticles using the solution polymerization method [40].

A lot of research has been done on the development and use of nanomaterials in mitigating loss circulation. However, most of this research involves the use of these nanomaterials as loss circulation materials for wellbore strengthening or as additives used in conjunction with conventional LCMs like graphite in order to control partial fluid losses into the formation. The use of nanomaterials as a treatment fluid to control moderate to severe losses is limited. Thus, this paper tries to investigate and describe the use of nanomaterials as a treatment fluid to mitigate issues that can arise due to moderate to severe losses.

A new loss circulation composition has been described in this paper. The loss circulation composition comprising a nanosilica-based dispersion and a chemical activator has been evaluated in terms of its ability to gel and turn into gelled solid at a desired temperature and pressure. Two types of chemical activators, one organic and the other inorganic, have been used in this study. The paper describes the effect of these chemical activators on the gelling time of three different nanoparticle-based dispersions. Two positively charged and one negatively charged nanoparticle-based dispersions have been used in this study. One of the most important advantages of using this novel loss circulation composition is its initial low viscosity of less than 10 cP. Such a low-viscosity loss circulation pill would make it easier for the mud engineer to pump the pill into the wellbore. Another advantage of this novel loss circulation composition is that it can be designed to get a predictable and controllable gelling time under any temperature and pressure conditions. These dual advantages of low viscosity and controllable gelling time would make it easier for the mud engineer to design a pill which is not only pumpable but also be able to reach the desired target loss circulation zone at the desired time and temperature and subsequently seal it.

The study also includes the effect of concentration of these chemical activators on the gelling behavior of the nanoparticle-based dispersions under both static and dynamic conditions at temperatures ranging up to 300 °F. The paper also describes the effect of shear on the gelling times and the change in viscosity of the nanoparticle-based dispersions. The efficacy of the novel loss circulation composition to seal off fractures and thereby prevent moderate to severe losses has been evaluated by performing permeability plugging tests (PPTs).

Materials Used in the Study. Experiments were performed with three different nanosilica-based dispersions, herein described in the paper as nanoparticle-1, nanoparticle-2, and nanoparticle-3. All the nanosilica-based dispersions were obtained from Akzo Nobel. These nanoparticle-based dispersions differ in their particle sizes and the charge on them. The properties of nanoparticle-1, nanoparticle-2, and nanoparticle-3 are given in Table 2. Nanoparticle-1 and nanoparticle-2 based dispersions with a negative surface charge have an alkaline pH of 9.4 and 10, respectively. Nanoparticle-3 based dispersion has an acidic pH of 3.8 and a positive surface charge. All the three nanoparticle-based dispersions vary in their particle sizes in the order of nanoparticle-3 > nanoparticle-2 > nanoparticle-1.

Two different chemical activators, one organic and the other inorganic, were used in the study. The organic activator used in the study is a C6-C8 fatty acid obtained from SABIC. The inorganic activator used in the study is 20% w/w NaCl brine. Five different organic and inorganic activator concentrations viz. 1%, 2.5%, 5%, 7.5%, and 10% were used in the study.

Research and Discussion

The experimental study was divided into two parts.

Table 2 Properties of nanoparticle-1, nanoparticle-2, and nanoparticle-3 based dispersions

	Commercial name	Particle size	Surface charge	pH
Nanoparticle-1	CEMBINDER 50	5 nm	Negative	9.4
Nanoparticle-2	CEMBINDER 17	17 nm	Negative	10
Nanoparticle-3	Levasil 30-516P	>17 nm	Positive	3.8

Fig. 1 Inverted tube tests of nanoparticle-based dispersion with activator

- Determination of static and dynamic gelling time of the three nanoparticle-based dispersions using two different activators
- Evaluation of the nanoparticle-based dispersions as a loss circulation material using the particle plugging apparatus.

Determination of Static Gelling Time of Nanoparticle-Based Loss Circulation Composition Using Organic and Inorganic Activator. The inverted sealed-tube method was used to determine the gelling time of the three nanoparticle-based loss circulation compositions under static conditions [41].

In the inverted sealed-tube method, 25 ml of the novel loss circulation composition comprising the nanoparticle-based dispersion and the activator was taken in a test tube. The test tube was then sealed off and subsequently kept in a preheated oven. Experiments were conducted by setting the oven at the desired test temperature.

The gelling time of the loss circulation composition was checked by periodically checking the test tubes kept in the oven. The gelling time of the nanoparticle-based dispersion in the presence of a chemical activator is defined as the time required for the composition to build viscosity and reach a specific gel strength. It should be noted that the determination of gelling time depended on the visual assessment of the researcher.

Effect of Organic and Inorganic Activator Concentration on the Gelling Behavior of the Three Nanoparticle-Based Dispersions. The gelling behavior of the three nanosilica-based dispersions in the presence of organic and inorganic activator was studied. Five different concentrations of the organic and inorganic activator viz. 1%, 2.5%, 5%, 7.5%, and 10% were used in the tests, and their effect on the gelling behavior of the nanosilica-based dispersions was evaluated. The inverted sealed-tube tests were done at four different temperatures viz. 150 °F, 200 °F, 250 °F, and 300 °F to study the effect of temperature on the gelling behavior of the loss circulation composition. The samples were kept in an oven at the desired temperature, and the flowability of the samples was checked periodically to estimate the static gelling time of the nanoparticle-based dispersions. The gelling time of the loss circulation composition was taken as the time at which the samples did not flow when the test tubes were placed in an inverted position. Figure 1 gives a representation of the inverted tube tests performed at 150 °F. The loss circulation compositions were observed for a period of 24 h in order to observe their gelling behavior. Samples which did not gel within a period of 24 h were discarded.

The results of the inverted sealed-tube tests on the loss circulation composition comprising the nanoparticle-1 based dispersion and inorganic activator are given in Fig. 2. Nanosilica-based dispersion is stable due to the silica-particle repulsion resulting from surface ionization in aqueous solution. Increase in temperature and addition of the inorganic activator results in weakened repulsive interactions between the nanosilica and leads to the collision of these nanoparticles. The collision of nanoparticles in the dispersion mainly results in the aggregation of nanosilica in the dispersion. This aggregation results into long chain-like networks caused by the formation of siloxane (Si–O–Si) bonds [42,43] and subsequently results in the gelling of the nanosilica-based dispersion. As the temperature and concentration of the inorganic activator increases, the repulsive interaction between the nanosilica particles also decreases. Thus,
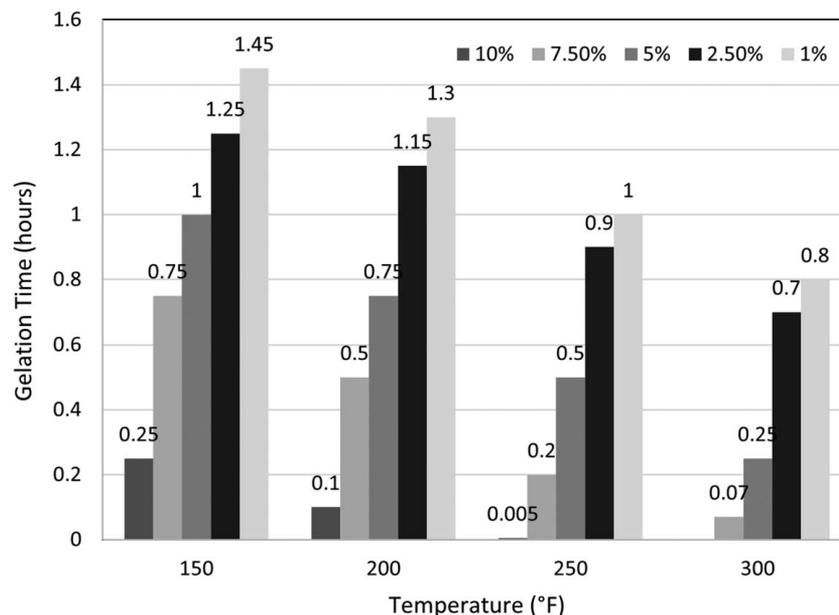

Fig. 2 Static gelling time of nanoparticle-1 based dispersion at four different temperatures
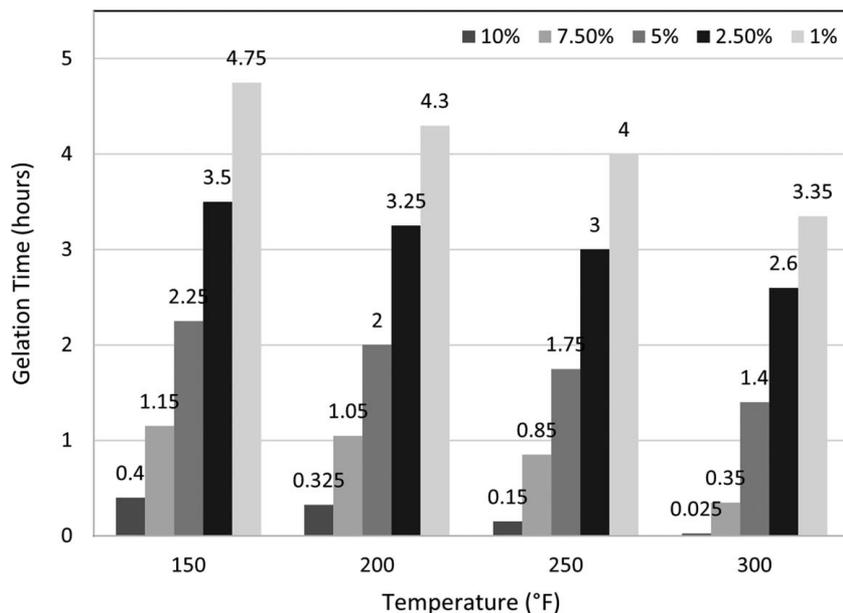
Fig. 3 Static gelling time of nanoparticle-2 based dispersion at four different temperatures

an increase in concentration of the inorganic activator results in decreased gelling time for the nanoparticle-based dispersion.

As observed from Fig. 2, the gelling time of the nanoparticle-1 based dispersions in the presence of the inorganic activator decreases with the increase in the inorganic activator concentration and temperature. Thus, the nanoparticle-1 based dispersion showed lower gelling times as the concentration of the activator and temperature was increased. Thus, for a loss circulation zone located at a certain depth and temperature, the loss circulation composition can be designed by optimizing the concentration of the inorganic activator and achieve a predictable gelling time.

It was observed that at lower concentrations of the inorganic activator, the strength of gelled solid obtained at the end of the experiment was too low for it to perform its role as a loss circulation material. Also, at higher concentrations of the inorganic activator, the nanoparticle-based dispersions gelled immediately. The usage of higher concentration of the inorganic activator can lead to prematurely gelling of the nanoparticle-based dispersion before it reaches the target loss circulation zone. Thus, the inorganic activator concentration must be optimized so that it not only gives good gelling time but should also result in a gelled solid having good strength.

The loss circulation composition comprising the nanoparticle-2 based dispersion and inorganic activator was also subjected to inverted sealed-tube tests. The testing was done at four different temperatures viz. 150 °F, 200 °F, 250 °F, and 300 °F. Figure 3 shows the gelling time for the nanoparticle-2 based dispersions in the presence of the inorganic activator. As observed with
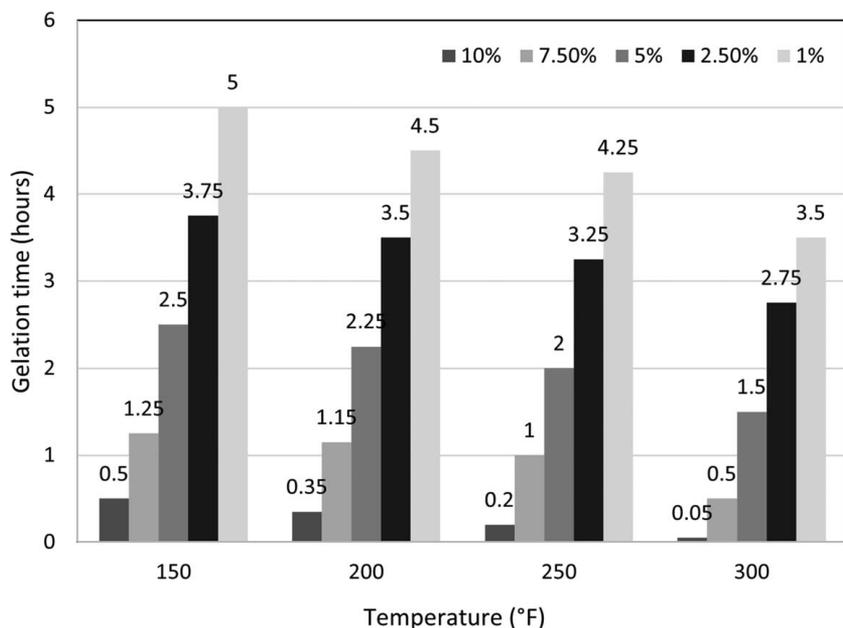

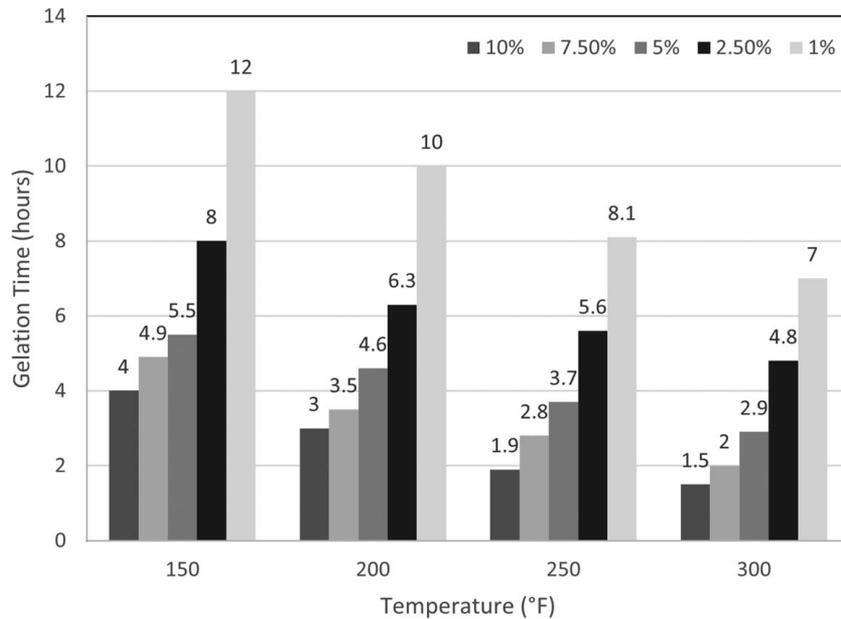**Fig. 4 Static gelling time of nanoparticle-3 based dispersion at four different temperatures**

Fig. 5 Effect of activator concentration on the static gelling time of nanoparticle-1 based dispersion using the organic activator

nanoparticle-1 based dispersions, the gelling time for the nanoparticle-2 based dispersion also decreased with increasing concentration of the inorganic activator. Also, for a particular inorganic activator concentration, the gelling time of the nanoparticle-2 based dispersion decreased with increasing temperature.

The nanoparticle-3 based dispersion was also subjected to inverted sealed-tube tests at four different temperatures using the inorganic activator. Similar to the nanoparticle-1 and nanoparticle-2 based dispersions, the gelling time for the nanoparticle-3 based dispersion also decreased with increasing concentration of the inorganic activator (Fig. 4). The nanoparticle-3 based dispersion in the presence of the inorganic activator showed the lowest gelling time with an activator concentration of 10% and the highest gelling time with an activator concentration of 1% for all the four different temperatures. These results showed that for all the three nanoparticle-based dispersions, higher inorganic activator concentration resulted in lowering of the gelling time at a particular temperature.

Inverted sealed-tube tests were further performed on the nanoparticle-based dispersions in the presence of an organic activator, and their gelling time was evaluated. The addition of the organic activator to the nanoparticle-3 based dispersion resulted in instantaneous gelling of the dispersion. This instantaneous gelling of nanoparticle-3 based dispersion was observed even with the addition of low amount of the organic activator. The nanoparticle-3 based dispersion which is in a colloidal state is stable under acidic pH (see Table 2). The pH of the organic activator is around 6. The colloidal state of the nanoparticle-3 based dispersion gets destabilized upon addition of the organic activator and gets subsequently gelled up even under ambient conditions. Such instantaneous gelling of a loss circulation composition would be disadvantageous as it would lead to premature gelling before it being placed in the loss circulation zone. Hence, inverted sealed-test tube tests were discontinued for the nanoparticle-3 based dispersion.

Nanopartilce-1 and nanoparticle-2 based dispersion which have an alkaline pH did not gel immediately upon addition of the organic activator under ambient conditions. Similar to the inorganic activator, the gelling time of the nanoparticle-based dispersions decreased with increasing concentration of the organic activator concentration. However, it was observed that the gelling times for the nanoparticle-1 and nanoparticle-2 based

dispersion when treated with the organic activator were higher as compared with the inorganic activator over the entire temperature range up to 300 °F. The gelling time of the nanoparticle-1 and nanoparticle-2 based dispersion in the presence of an organic activator is given in Figs. 5 and 6, respectively. Both the nanoparticle-1 and nanoparticle-2 based dispersions have a starting pH around 10. This pH of the nanoparticle-based dispersions decreases from 10 to about 8 upon addition of the organic activator. It was observed that the pH of the nanoparticle-based dispersions falls to about 4 as the viscosity of the dispersions start to increase. The nanoparticle-1 and nanoparticle-2 based dispersions are in a colloidal state under alkaline conditions due to particle–particle repulsion. This interparticle repulsion helps the dispersion to remain in a stable colloidal form. However, as the pH decreases and becomes acidic, the interparticle collision in the nanoparticle-based dispersion increases leading to aggregate formation. This aggregate formation then leads to a buildup in viscosity and eventually results in gelling of the nanoparticle-based dispersions. Nanoparticle-2 based dispersion in the presence of 1% organic activator did not gel even after 20 h at 150 °F and 200 °F. Hence, the experiment was stopped after 20 h of keeping the dispersion in the oven.

Effect of Size of Nanoparticles on the Gelling Behavior of the Three Nanoparticle-Based Dispersions. The gelling behavior of the three nanoparticle-based dispersions using the inorganic activator is shown in Figs. 2–4. One of the major conclusions that can be drawn from the inverted sealed-tube tests is that though the gelling behavior is same for all the three nanoparticle-based loss circulation compositions, the gelling time for a particular activator concentration and temperature varies for all the three nanoparticle-based dispersions. The gelling time as obtained from the inverted tube tests with a given concentration of the inorganic activator decreases in the order of nanoparticle-3>nanoparticle-2>nanoparticle-1. The effect on gelling time as a function of nanoparticle size at four different temperatures and for an inorganic activator concentration of 5% is given in Fig. 7.

The gelling behavior of the three loss circulation compositions is thus dependent on particle sizes of the nanoparticle-based dispersions. Nanoparticle-3 has the highest particle size of greater than 17 nm as compared with nanoparticle-1 and nanoparticle-2 which have a particle size of 5 nm and 17 nm, respectively. The surface
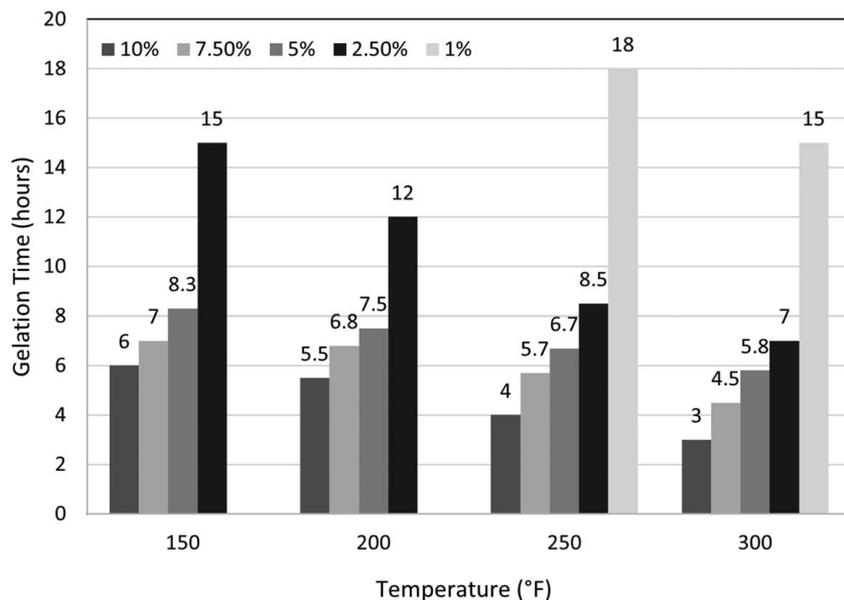
Fig. 6 Effect of activator concentration on the static gelling time of nanoparticle-2 based dispersion using the organic activator

area of a nanoparticle is dependent on the size of the nanoparticle. Greater the size of the nanoparticle smaller is its surface area. Thus, nanoparticle-3 which has the highest particle size of >17 nm has the smallest surface area as compared with the other two nanoparticles. Due to the higher particle size of the nanoparticle-3, the surface area available for the inorganic activator to react with the nanoparticle is also decreased. This results in higher gelling time for nanoparticle-3 based dispersions as compared with the other nanoparticle-based dispersions.

Determination of Dynamic Gelling Time of Nanoparticle-Based Loss Circulation Composition Using Organic and Inorganic Activator. The dynamic viscosity method was used to determine the dynamic gelling time of the three nanoparticle-based

loss circulation compositions under the conditions of shear. This method has been described in an earlier paper [11]. The dynamic gelling time of the three nanoparticle-based dispersions has been determined by using a Model 50-type HTHP rheometer. A No. 05X bob with a speed of 3 rpm was used in the experiments. The nanoparticle-based composition (44 ml) was initially taken in the HTHP viscometer cup and was subsequently subjected to the desired temperature and pressure. The loss circulation composition was periodically monitored for any change in viscosity at fixed intervals of time. The dynamic gelling time was taken as the time when the nanoparticle-based dispersion shows a continual increase in the viscosity in the plot of viscosity versus time as shown in Fig. 8 [44].

It is very important to determine the effect of shear on the gelling performance of the nanoparticle-based loss circulation
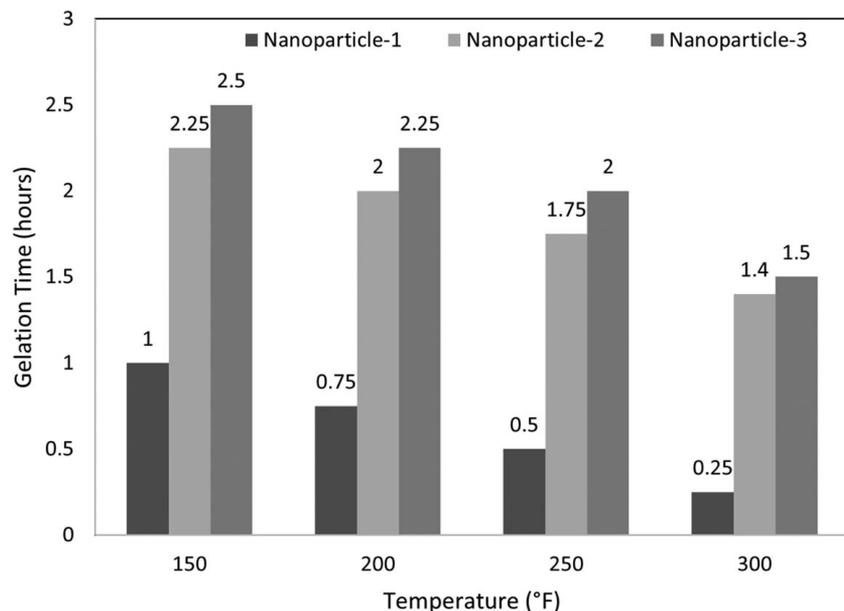

Fig. 7 Relationship between sizes of nanoparticles on the static gelling time using 5% inorganic activator concentration
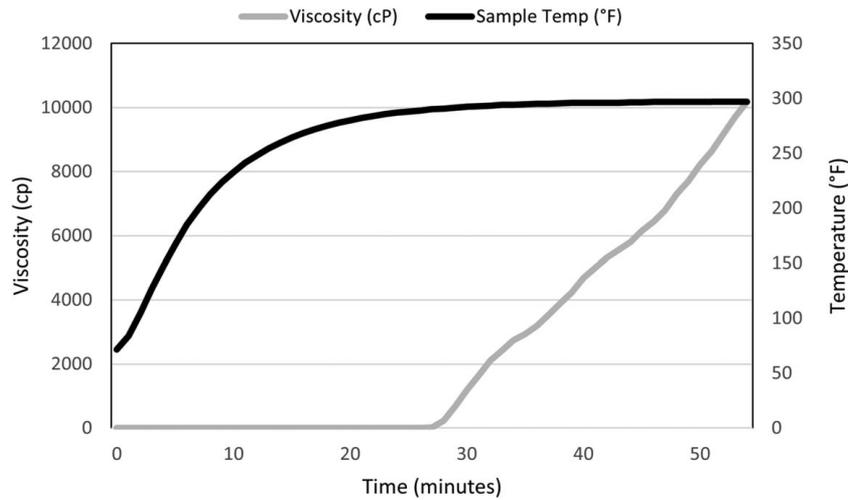
Fig. 8 Representative plot of the change in the viscosity of the nanoparticle-based loss circulation composition at 300 °F

compositions. Under the effect of shear, there is a chance that the nanoparticle-based loss circulation composition may gel up before it being placed in the loss circulation zone. Thus, to determine the shear effect on the gelation behavior of the loss circulation composition, dynamic viscosity tests were performed on nanoparticle-based dispersion in the presence of organic and inorganic activators.

In the dynamic viscosity tests, the loss circulation composition comprising the nanoparticle-based dispersion and the activator was first loaded in a sample cup at ambient conditions and was then gradually heated up to the desired test temperature. All the dynamic gelling tests were performed by gradually heating the samples up to 300 °F using a Model 50-type viscometer.

Figure 8 shows a representative plot of the change in the viscosity of the nanoparticle-based loss circulation composition when it was gradually heated from room temperature to 300 °F. As can be seen from the graph, the loss circulation composition initially showed low viscosity. However, this viscosity increases over a period of time during the test. This increase in the viscosity of the loss circulation composition at a certain point of time is a typical

representation of a liquid-to-solid transition. The time at which the viscosity of the loss circulation composition shows an abrupt increase is taken as its dynamic gelling time.

The dynamic gelling test results performed on nanoparticle-1, nanoparticle-2, and nanoparticle-3 based dispersions in the presence of the inorganic activator is shown in Fig. 9. Nanoparticle-1 when mixed with the inorganic activator having a concentration of 10% gelled instantaneously at room temperature. All the other nanoparticle-based loss circulation compositions in the presence of the inorganic activator showed delayed gelling. The dynamic gelling tests showed that the gelling time was related with the concentration of the inorganic activator. The dynamic gelling time for all the three nanoparticle-based dispersions showed a decrease with increasing concentration of the inorganic activator. The dynamic gelling time decreased in the order of nanoparticle-3 > nanoparticle-2 > nanoparticle-1. Such a gelling behavior of the three nanoparticle-based dispersions can be attributed to their decreasing particle sizes in the order of nanoparticle-3 > nanoparticle-2 > nanoparticle-1.
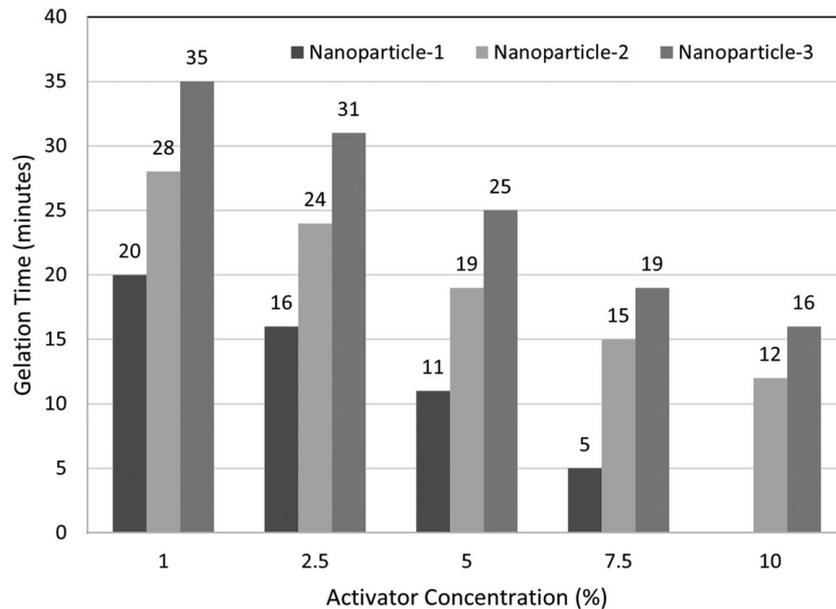

Fig. 9 Effect of inorganic activator concentration on the dynamic gelling time of nanoparticle-based dispersions at 300 °F
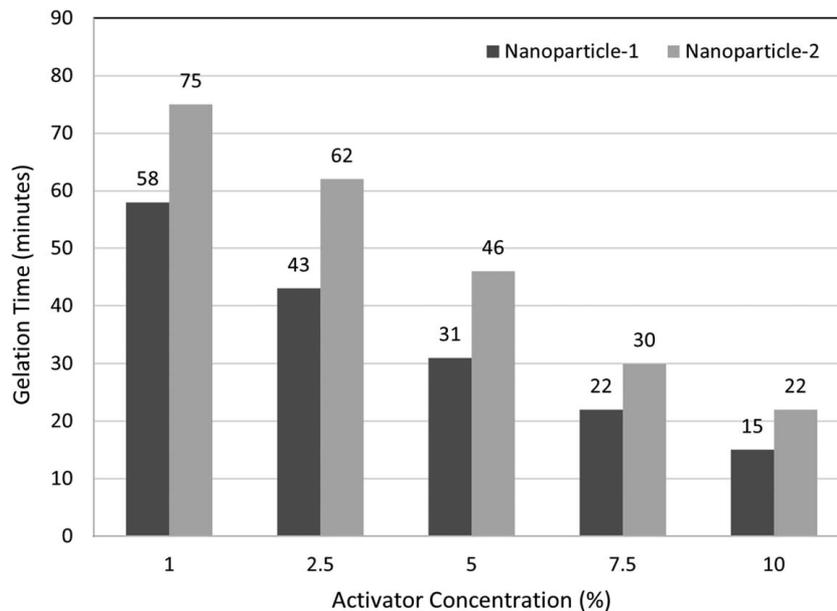
Fig. 10 Effect of organic activator concentration on the dynamic gelling time of nanoparticle-based dispersions at 300 °F

Further tests were performed on the loss circulation compositions using the organic activator at 300 °F. The results of the dynamic gelling tests using the organic activator are shown in Fig. 10. The dynamic gelling tests were performed on nanoparticle-1 and nanoparticle-2 based dispersions as the nanoparticle-3 based dispersion gelled almost instantaneously when mixed with the organic activator even at room temperature conditions. The results showed that the gelling time for the nanoparticle-based loss circulation composition using the organic activator was more as compared with the gelling time recorded for the nanoparticle-based dispersion using the inorganic activator. This type of gelling behavior was also observed during the inverted sealed-tube tests performed with the organic and inorganic activators. Like the results with the inorganic activator, the particle size effect was also observed when dynamic gelling tests were performed with the organic activator. The

dynamic gelling time decreased in the order of nanoparticle-2 > nanoparticle-1. Thus, it can be concluded that smaller the size of the nanoparticle, faster is the rate of the gelling process.

Effect of Shear Rate on the Gelling Behavior of the Three Nanoparticle-Based Dispersions. It was also especially important to study the effect of shear rate on the gelling behavior of the nanoparticle-based loss circulation composition. The loss circulation composition which must be pumped into the well needs to gel up after a specific time period and thereby seal the loss circulation zone. It is possible that the loss circulation composition is subjected to different shear rates when it is pumped into the well. The mud engineer would need to know the gelling behavior of the loss circulation composition at different shear rates so that it does not gel
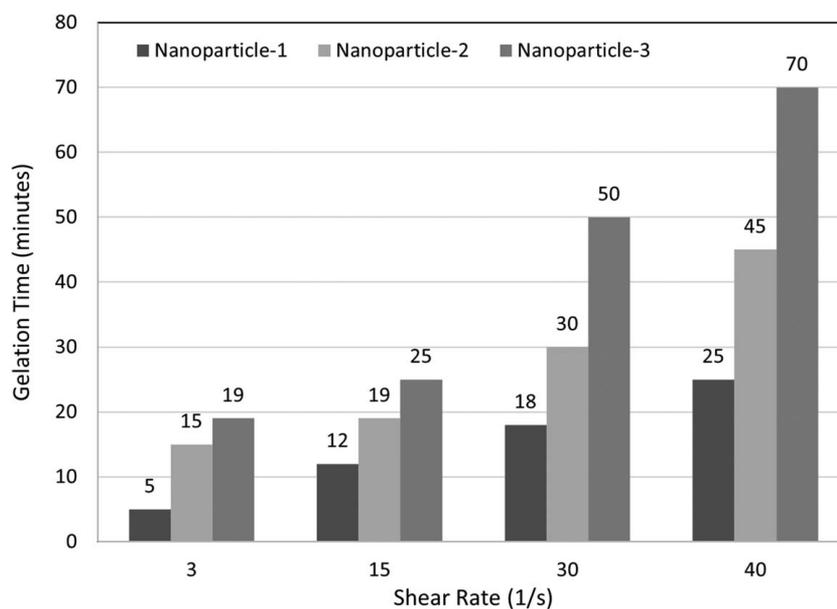

Fig. 11 Relationship between shear rate and dynamic gelling time of nanoparticle-based dispersions at 300 °F
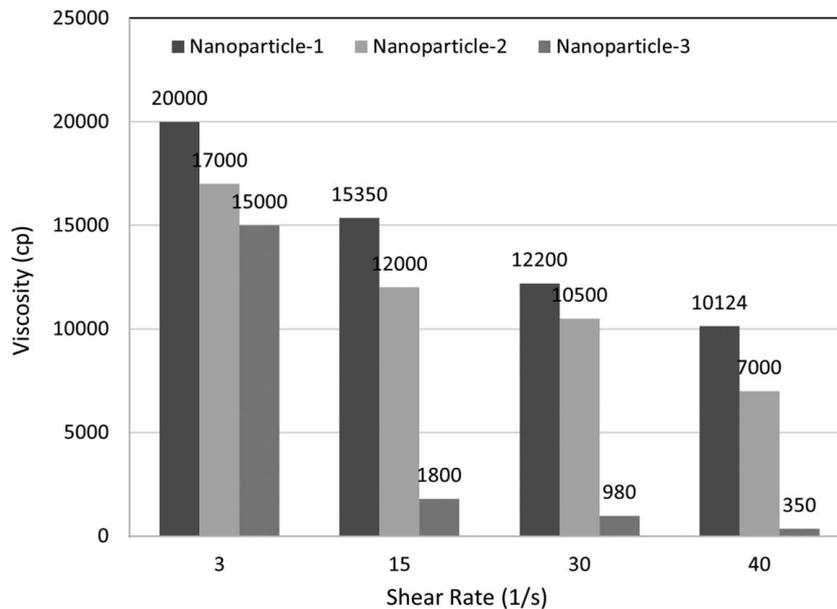
Fig. 12 Relationship between shear rate and the viscosity buildup of nanoparticle-based dispersions at 300 °F

up prematurely and block the drill pipe during pumping. Such a scenario would result in non-productive time and considerable loss of money.

Figure 11 shows the gelling behavior of the loss circulation composition when subjected to four different shear rates viz. 3 s^{-1}, 15 s^{-1}, 30 s^{-1}, and 40 s^{-1}. The gelling behavior of the three nanoparticle dispersions was studied at 300 °F. All the experiments were conducted with 7.5% inorganic activator concentration. From the results shown in Fig. 11, the gelling time of the loss circulation compositions decrease with the increase in shear rate. Nanoparticle-1 based dispersion which has the smallest particle size among the three nanoparticle dispersions used in the study shows lower gelling time as compared with the other two nanoparticle-based dispersions.

Under shear, there is a possibility that the time required by the loss circulation composition to get converted to a solid would be higher. Thus, to study the effect of shear on the change in the viscosity of the loss circulation composition, experiments were performed at 300 °F and the viscosity of the three loss circulation compositions was measured after a test time of 100 min. The viscosity measurements were performed on loss circulation composition comprising nanosilica dispersions and inorganic activator. The experiments were conducted using four different shear rates viz. 3 s^{-1}, 15 s^{-1}, 30 s^{-1}, and 40 s^{-1}. The effect of different shear rates on the viscosity of the loss circulation compositions is given in Fig. 12. It was observed that the viscosity buildup was inversely proportional to the shear rate, i.e., increase in shear rate resulted in a decrease in viscosity buildup. From these results, one can conclude that an increase in shear rate will result in a corresponding increase in the time required by the loss circulation composition to get converted into a solid.

Permeability Plugging Tests. To determine the efficiency of the nanoparticle-based compositions as loss circulation material, permeability plugging tests were performed. These tests were conducted using a specialized filtration-type apparatus called as the permeability plugging apparatus or PPA. This test is used to determine the effectiveness of additives in a drilling fluid or a composition to prevent fluid loss into a permeable medium.

A PPT is performed in a specialized filtration-type apparatus (particle plugging apparatus) to determine the effectiveness of additives to prevent fluid loss into a permeable medium. The novel nanoparticle-based composition was thus tested as a treatment fluid to prevent loss circulation using the PPT. The particle plugging apparatus for the test is shown in Fig. 13.

The following procedure was used during the test:

- set the temperature controller to the desired temperature,
- pour the sample into the cell and place the cell into the heating jacket in the same orientation,
- apply appropriate amount of pressure through the back-pressure regulator to the top of the cell,
- after opening the top valve, collect the fluid from the back-pressure collector in a measuring cylinder and record the amount. Ensure that all the fluid has been expelled. This will be reported as the Spurt volume (ml),
- keep collecting periodically over the next 30 min and record the total fluid collected over the 30 min period as total fluid (ml), and
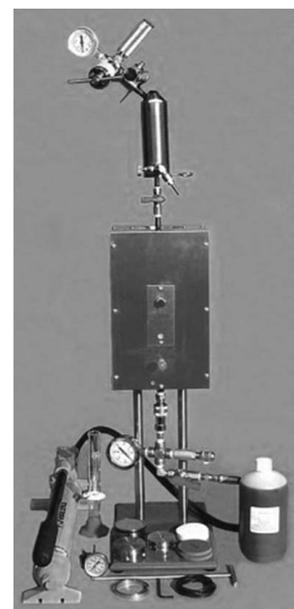

Fig. 13 Test assembly for the particle plugging test

Table 3 Results of the particle plugging test for the three nanoparticle-based dispersions using the inorganic activator at 300 °F

	Nanoparticle-1	Nanoparticle-2	Nanoparticle-3
Spurt loss (ml)	0.8	0.6	1
PPA total (ml)	2	1.8	1.8

Table 4 Results of the particle plugging test for the three nanoparticle-based dispersions using the organic activator at 300 °F

	Nanoparticle-1	Nanoparticle-2	Nanoparticle-3
Spurt loss (ml)	0.6	0.6	1.2
PPA total (ml)	1.8	2.0	2.4

- The PPA fluid loss (PPA total) is calculated as follows:

$$\text{Spurt loss} = [4V7.5 - 2V30] \qquad (1)$$

$$\text{Total loss} = 2V30 \qquad (2)$$

where V7.5 is the fluid loss at 7.5 min and V30 is the fluid loss at 30.0 min.

Permeability plugging tests were performed with the three nanoparticle-based dispersions using both the organic and inorganic activators. 2 mm slotted disks were used as a filter medium in this test. This was done in order to simulate fractures of 1–2 mm width in the loss circulation zone [11] and test the efficiency of the nanoparticle-based compositions to control moderate losses. The test was performed at 300 °F at a differential pressure of 1500 psi. Six different experiments wherein the three nanoparticle-based dispersions mixed with organic and inorganic activators, respectively, were kept static for a period of 3 h. After 3 h, the top valve of the permeability plugging apparatus was opened. An inorganic and organic activator concentration of 7.5% and 10%, respectively, was used for the particle plugging test study. The results of the tests are shown in Tables 3 and 4. The test results show that the three nanoparticle-based dispersions in the presence of the organic and inorganic activators gave very low spurt and total loss of the filtrate for the entire test period at 250 °F. This test shows that the nanoparticle-based dispersions in the presence of both the organic and inorganic activators would be able to control moderate to severe losses.

Field Placement Method for the Nanoparticle-Based Dispersions. The nanoparticle-based dispersions can be deployed in the field to control loss circulation as a

(a) one-component loss circulation material
(b) two-component loss circulation material

In the one-component method, both the nanoparticle-based dispersion and the chemical activator can be mixed on the surface and pumped downhole through an open-ended drill pipe. The concentration of the activator can be adjusted depending upon the depth of the loss circulation zone. Gelling time tests need to be done at the desired temperature of the loss circulation zone to decide upon the concentration of the activator. The activator concentration should be sufficient to cause gelling of the nanoparticle-based dispersion in the loss circulation zone and not result in premature gelling of the dispersion. In the two-component method, the first component, i.e., the nanoparticle-based dispersion can be pumped and placed in the loss circulation zone. The second component, i.e., the activator can be pumped through a coil tubing and mixed subsequently with the nanoparticle-based dispersion. In this method, an excess amount of the activator can be used to cause rapid gelling of the nanoparticle-based dispersion in the loss circulation zone. In this method, the risk of premature gelling of the nanoparticle-based dispersion can be avoided as both the components would be made to mix in the loss circulation zone. However, accurate placement of both the components in the loss zone would be needed to effectively control loss circulation.

Conclusions

- Three different nanosilica-based dispersions were tested for their potential as a loss circulation material in the presence of organic and inorganic chemical activators.
- The nanosilica-based dispersions showed good potential as a loss circulation material.
- Experiments performed to determine the effect of activator concentration on the gelling time of the loss circulation composition showed that the gelling time decreased with increasing concentration of the both the organic and inorganic activator.
- For a particular organic and inorganic activator concentration, the gelling time of the loss circulation composition decreased with increasing temperature.
- Dynamic viscosity experiments showed that the viscosity of the nanosilica-based loss circulation composition increased with temperature over a period of time.
- Experiments performed using both the organic and inorganic activators showed that by varying the activator concentration, the gelling time of the loss circulation composition can be controlled for a loss circulation zone at a certain depth and temperature.
- PPA tests demonstrated good loss circulation zone sealing efficiency for the three nanosilica-based dispersions using both the organic and inorganic activators.

Conflict of Interest

There are no conflicts of interest.

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