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Removal of Calcium Carbonate Water-Based Filter Cake Using a Green Biodegradable Acid

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Abstract: The filter cake is a very thin layer of solid particles deposited from the drilling fluid onto the surface of the drilled formation. This is essential during the drilling process as it reduces fluid filtrate invasion, contributing for the well's integrity. When the drilling processes are over, this formation must be removed efficiently to allow for the primary cement jobs to be conducted efficiently, and for hydrocarbons to be produced without any restrictions. The removal process of the filter cake is challenging and requires specific techniques, depending on several factors including the filter cake homogeneity, drilling fluid components, weight of materials used, and the continuous phase of the drilling fluid (i.e., water or oil). This study evaluates the use of green biodegradable removal acid (GBRA) to dissolve calcium carbonate water-based filter cake. The removal efficiency of this new acid was analyzed using filtration and removal tests, conducted on a ceramic filter disc and real core samples at 212 °F. Then, the new formulation was subjected to physical properties measurements and biodegradation and corrosion tests. Furthermore, the performance of the new acid formulation was compared with 10 wt.% hydrochloric acid (HCl) to ensure its efficiency. The obtained results showed that the new acid formulation was effective at removing the filter cake, with a removal efficiency greater than 90%. The retained permeability was 100% and 94% with the ceramic filter disc and the real limestone core sample, respectively, confirming the good removal performance of the new acid formulation. The corrosion rate of GBRA (50 wt.%) at 212 °F was 0.032 lb/ft², which was within the acceptable range according to oil industry practices (i.e., less than 0.05 lb/ft² if the temperature is less than 250 °F), while HCl (10 wt.%) yielded a high corrosion rate of 0.68 lb/ft².

Keywords: biodegradable acid; filter cake removal; calcium carbonate; water-based; drilling fluid

1. Introduction

In drilling operations, the drilling fluid is introduced to achieve many objectives, such as lubricating and cooling the drill bit and the drill string [1], transferring the drilled cuttings to the surface while drilling, and suspending the drilled cuttings when the drilling operation is stopped [2]. Another main function of drilling fluid is to control the well by applying hydrostatic pressure to prevent formation fluids from invading the wellbore. Moreover, the drilling fluid is used to ensure wellbore stability by forming a thin and low permeability film on the wall of the well under an overbalance condition called filter cake [3–6].

For the drilling mud to prevent blowout and suppress the formation pressure, the drilling mud formulation usually contains a weighting agent such as barite, hematite, manganese tetroxide, or calcium carbonate [7–14]. These chemicals are added to the drilling fluid to attain the desired density [15]. Consequently, these high-weight additives are the main source of formation damage-related problems, particularly in the producing zones, and have a considerable effect on the formed filter cake structure.

The filter cake is a thin impermeable layer with permeability ranging from 0.01 to 0.0001 mD [16], which is formed on the face of the permeable formation [17], as shown in Figure 1. Filter cake formation is required to stabilize the drilled formations, minimize the drilling fluid filtrate into the drilled formation [15,18,19], and reduce solid particle invasion into the oil-bearing formations [20]. However, after the drilling operations, the drilling fluids and the filter cake film must be removed from the wellbore to enable a successful primary cementing job. Also, filter cake removal is mandatory before the start of production operations to avoid impeding the flow capacity at the wellbore [21].

Figure 1. The filter cake formation on the surface of the drilled formation.

The preflush fluids are designed to be injected after the drilling operation and before injecting the cement slurry to thoroughly wipe the remaining mud in the annular space [22]. These fluids are designed to be efficient in cleaning the drilling mud and chemically compatible with drilling mud and cement slurry. Additionally, they should be able to prevent sludge or emulsion formation and modify the formation rock wettability in some cases [23].

Filter cake removal is a complicated task, as the filter cake layer has different structural characteristics for different drilling fluid types (i.e., water- or oil-based drilling fluid) and different weighting additives [15]. The weighting material constitutes 70 to 80 wt.% of the filter cake layer [19,24,25]. Therefore, the removal efficiency of the filter cake mainly depends on the solubility of the weighting material of the treatment chemical. Another factor that determines the required number of stages to dissolve the filter cake is the compatibility of the removal fluid with the polymer breaker.

Rostami and Nasr-El-Din [26] developed a new self-destructing calcium carbonate-weighted water-based drilling fluid. The new formulation is based on the addition of polylactic acid (PLA), which is an ester that liberates lactic acid and alcohol when dissolved in water. The PLA is mainly used to dissolve the calcium carbonate from the filter cake body after hydrolysis with water. The cleaning solution was able to remove 75 to 78 wt.% of the filter cake formed by the new self-destructing drilling mud, which was higher than the normal drilling fluid.

Mahapatra and Kosztin [27] examined the possibility of using magnesium peroxide (MgO_2) as an internal breaker for the calcium carbonate-based filter cake on laboratory and field scales. By soaking the filter cakes formed by different drilling fluids with different concentrations of MgO_2 particles into 15 wt.% hydrochloric acid (HCl) solution for 2 h, the 10 wt.% MgO_2 particles were found to best enhance the filter cake removal efficiency. The optimized drilling mud formulation in an Omani field showed good results in two out of the three wells, where the actual oil production rate from the two wells exceeded the expected rate.

Elkakatny and Nasr-El-Din [28] proposed a low pH formulation based on tetrasodium glutamate diacetate (GLDA) to dissolve the calcium carbonate filter cake in a two-stage process. The two-stage process was recommended because of the incompatibility between the GLDA and the polymer breaker. During the first stage, 10 wt.% of the amylase enzyme was used to break the polymer, while GLDA with a pH of 3.3 was used as a treatment solution in the second stage. Ba geri et al. [19] studied the impact of sandstone cuttings on the removal efficiency of calcium carbonate-filter cake during drilling horizontal laterals in sandstone reservoirs. It was concluded that the dissolution rate of the calcium carbonate in GLDA, at a pH of 3.8, decreased from 100 to 80 wt.% when the filter cake layer contained 20 wt.% sandstone cuttings.

Elkakatny and Mahmoud [29] investigated the enzyme compatibility with 20 wt.% chelating agent solutions at different pH values for the removal of calcium carbonate filter cake. Three different chelating agents, i.e., ethylene diamine tetra-acetic acid (EDTA), diethylenetriamine penta-acetic acid (DTPA), and tetrasodium glutamate diacetate (GLDA), with different pH values were used. It was found that using chelating agent solutions alone was not able to remove the starch after soaking for 12 h. This indicated that the use of an enzyme to break down the polymer is necessary. The high-throughput assays enzyme (HTA) broke the starch completely after 6 h of reaction. The compatibility study confirmed that all the chelating agents considered in this work were not compatible with the HTA enzyme, indicating that the treatment should be conducted in two stages. The two-stage process was able to remove the filter cake with 100 wt.% removal efficiency.

Mohamed et al. [30] and Elkakatny [31] introduced a new acid formulation to dissolve a calcium carbonate oil-based filter cake. Their formulation contained 50 vol.% biodegradable acid to dissolve the filter cake, 5 vol.% mutual solvent to remove the oil film surrounding the filter cake to allow acid-filter cake reaction, and 45 vol.% water. Their formulation was effective in removing the filter cake, with 100 wt.% removal efficiency, and retained permeability. A further stimulation was observed on limestone core samples after the removal process because of the acid-rock interaction.

Although many new fluid formulations have been introduced to remove calcium carbonate-based filter cake in water-based fluids, the need for a cheap, compatible, environmentally-friendly, and efficient removal fluid still prevails. In this study, a new fluid formulation was introduced to dissolve calcium carbonate filter cake in water-based drilling fluids. The new formulation comprises 50 wt.% biodegradable acid; the remaining 50 wt.% is water. The new acid formulation was able to efficiently remove the filter cake, minimizing the treatment cost by reducing the number of stages and the duration of the acid treatment. This formulation also mitigated the impact of removal fluid on the environment. First, the materials used in this work are described and the experimental procedures are explained; then, our findings are discussed and summarized.

2. Material

A water-based drilling fluid was prepared using the fluid formulation described in Table 1. The drilling fluid consisted of xanthan polymer as a viscosifier, starch as a fluid loss control additive, potassium chloride as a clay stabilizer, potassium hydroxide to increase the pH of the drilling fluid, sodium sulfite as an oxygen scavenger, and calcium carbonate (with an average particle size, $D_{50} = 50 \mu\text{m}$) as a weighting material and bridging agent. The drilling fluid additives were added in order and mixed for a specific time. Twenty-five-micron ceramic filter discs and Indiana limestone core samples (with an average permeability of $k = 25 \text{ mD}$, diameter of $d = 2.5''$, and thickness $h = 1.7''$) were used as a filtration medium.

Table 1. Drilling fluid formulation on a lab-scale.

Additive	Amount
Water	308 g
Defoamer	0.33 cm ³
Xanthan gum	1.5 g
Starch	6 g
Potassium chloride	80 g
Potassium hydroxide	0.3 g
Sodium sulfite	0.25 g
Calcium carbonate (50 µm)	30 g

A green biodegradable acid (GBRA) was used as a removal fluid. The GBRA was an acid solution formulated with a combination of a catalyst and a reagent added to HCl, creating a biodegradable, nonbioaccumulating alternative to inhibited and potentially hazardous HCl. During its synthesis, water was added to HCl to quench the exothermic reaction created, thereby significantly increasing the strength of the acid. Simultaneously, a cathodic stimulation was created to reduce the corrosiveness by a factor of 50 at ambient temperatures [30,31]. Steel coupons were used in the corrosion test to measure the corrosion rate at different acid concentrations.

3. Experimental Work

3.1. GBRA Properties and Biodegradation Test

The acid formulation was analyzed by measuring its physical properties to optimize the treatment operation. These properties were viscosity, density, and surface tension. The density was measured using a graduated cylinder. A total of 100 cm³ of the fluid was poured into the graduated cylinder. The weight of the cylinder was recorded before and after pouring the fluid using a sensitive weight balance. Then, the difference in the weight was divided by the volume of the fluid to obtain its density. The viscosity and surface tension were measured using a kinematic viscosity tester and a tensiometer, respectively. All the measurements were performed at ambient temperature and under atmospheric pressure.

The acid formulation was sent to an accredited laboratory to measure the acid biodegradability using the OECD 301B biodegradation test. This is an aerobic biodegradation test where a material is introduced to an inoculum in a closed environment and the biodegradability of the acid is measured by carbon dioxide (CO₂) evolution 28 days later [32–34].

3.2. HPHT Filtration Tests

Filtration experiments were carried out to evaluate the filter cake removal efficiency with the new acid formulation. The experiments were performed in two stages: firstly, the filtration part formed the filter cake and secondly, the removal part evaluated the removal efficiency after soaking the filtration medium in the acid formulation for 6 h. Both steps were conducted at 212 °F, and 300 psi. Twenty-five-micron ceramic filter discs and real Indiana limestone core samples were used as a filtration medium. Two hundred milliliters of the removal fluid that was used consisted of 50 wt.% biodegradable acid; the remaining 50 wt.% was water. The weight and thickness of the filtration medium were measured before and after each step to characterize the filter cake and to calculate the removal efficiency. Moreover, the initial and final permeabilities of the filtration medium were measured to evaluate the retained permeability after the treatment. The removal efficiency and retained permeability were calculated using Equations (1) and (2):

$$\text{Removal efficiency, wt.\%} = \frac{\text{Removed filter cake weight}}{\text{Initial filter cake weight}} \times 100 \quad (1)$$

$$\text{Retained permeability, \%} = \frac{\text{Final permeability}}{\text{Initial permeability}} \times 100 \dots \quad (2)$$

3.3. Corrosion Test

The corrosion test was performed to evaluate the corrosion rate of the new acid formulation, to ensure its applicability in the oil industry. The test was conducted at 212 °F under 300 psi using an aging cell. One hundred milliliters of the removal fluid was poured into the aging cell, and a steel coupon was immersed in the fluid. Then, the cell was closed, pressurized, and heated. The test was run for 6 h, as per oil industry practices. Afterwards, the coupon was removed, washed, dried, and weighed, and the corrosion rate was calculated with Equation (3), using the weight difference and the surface area of the coupon. As per oil industry standards, an acceptable corrosion rate is ≤ 0.05 lb/ft². The corrosion rate of GBRA was measured for two concentrations, i.e., 25 and 50 wt.% HCl, and then compared with 10 wt.% HCl.

$$\text{Corrosion rate, } \frac{\text{lb}}{\text{ft}^2} = \frac{W_{\text{before}} - W_{\text{after}}}{\text{Surface Area}} \quad (3)$$

4. Results and Discussions

4.1. GBRA Properties and Biodegradability

The properties of the new acid formulation were measured to optimize the treatment job in the field. GBRA had a viscosity of $\mu = 1.58$ cP, density of $\rho = 1.1$ g/cm³, surface tension of $\sigma = 31.4$ mN/m, and a pH of 0.02. These properties were measured at room temperature and under atmospheric pressure. GBRA biodegradability was measured using the OECD 301B biodegradation test by the theoretical evaluation of carbon dioxide (ThCO₂). The GBRA achieved a biodegradation rate of 65% by day 28, exceeding the requirements for ready biodegradability (60% by day 28) based on OECD test criteria [33]. Consequently, a plateau of 81% was achieved by day 40 (Figure 2). With growing environmental concerns, this biodegradation rate would make GBRA a good candidate to remove calcium carbonate-filter cake from oil and gas wells.

Figure 2. Acid biodegradation by CO₂ evolution using OECD 301 B test.

4.2. Filter Cake Removal and Retained Permeability

Filtration experiments were carried out to form the filter cake on the filtration medium and to perform the removal process. Figure 3 shows the filtration performance of the drilling fluid using a 25-micron ceramic filter disc and Indiana limestone core sample at 212 °F under 300 psi. With a

ceramic disc, the drilling fluid yielded a total filtrate volume of 9.9 cm³ after the standard test time, i.e., 30 min. The filter cake weight and thickness were 5.57 g and 1.3 mm, respectively. The filtration performance on the core sample was similar to that on the ceramic disc, with a slightly higher fluid filtrate, i.e., 10.5 cm³. The removal process was performed on ceramic filter discs and real core samples under the same conditions. The filtration medium was soaked in 200 cm³ of 50 wt.% GBRA for 20 h. Figures 4 and 5 show the ceramic disc and core sample before and after the filtration and removal tests. From the clean surface of the filtration mediums, GBRA was effective in removing the filter cake, as its calculated removal efficiency was greater than 95 wt.% in both cases.

Figure 3. Filtration performance of the water-based drilling fluid using different filtration mediums.

Figure 4. Ceramic filter disc: (a) before filtration, (b) after filtration, and (c) after removal with 50 wt.% green biodegradable removal acid (GBRA).

Figure 5. Indiana limestone core sample: (a) before filtration, (b) after filtration, and (c) after removal with 50 wt.% GBRA.

Furthermore, the initial and final permeability of the core sample were measured to evaluate the removal of the internal damage caused by filtrate and solids invasion. For this purpose, real Indiana

limestone cores, with a 1.7" length and 2.5" diameter, were used to perform the filtration and removal tests. The initial core permeability was 25 mD measured using distilled water. After 20 h soaking in 50 wt.% GBRA, 94% of the initial permeability was restored, and the final permeability was 23.5 mD. This confirmed that the new acid system could be used to remove the internal damage caused by the fine particles invasion during the filtration process.

4.3. Corrosion Test

The corrosion test was conducted for the new acid formulation at two different concentrations of GBRA, i.e., 25 wt.% and 50 wt.%, at 212 °F. As shown in Figure 6, GBRA yielded a very low corrosion rate with both concentrations. The corrosion rate at 25 wt.% was 0.025 lb/ft² while, when the acid concentration increased to 50 wt.%, it was 0.032 lb/ft². Both corrosion rates were within the acceptable range, i.e., <0.05 lb/ft², as per the oil industry practices. Figure 7 shows that no noticeable damage was observed on the surface of the coupon after the corrosion test with 50 wt.% GBRA. GBRA was titrated using the titration test to determine the acid concentration in the solution. It was found that GBRA contained 20 wt.% of converted HCl, thus 50 wt.% GBRA consists of only 10 wt.% HCl. Therefore, the corrosion rate of the new formulation was compared with 10 wt.% HCl at the same conditions. Ten percent HCl was found to be very corrosive; the corrosion rate was 0.68 lb/ft², which is much higher than that of 50 wt.% GBRA (Figure 6).

Figure 6. The corrosion rate of GBRA and hydrochloric acid (HCl).

Figure 7. Steel coupon: (a) before and (b) after the corrosion test using 50 wt.% GBRA at 212 °F.

5. Summary and Conclusions

In summary, a new acid formulation was introduced to remove calcium carbonate water-based filter cake. Based on the obtained results, the following conclusions can be drawn:

1. The new acid formulation was efficient in removing the calcium carbonate water-based filter cake after 20 h soaking time with a removal efficiency and retained permeability of greater than 90%.
2. GBRA (50 wt.%) yielded a corrosion rate of 0.032 lb/ft² at 212 °F without adding any corrosion inhibitor that is below the standard corrosion rate approved by the oil and gas industry (<0.05 lb/ft² at a temperature less than 250 °F), while the corrosion rate of HCl (10 wt.%) was 0.68 lb/ft².
3. GBRA (50 wt.%) has a viscosity of $\mu = 1.58$ cP, density of $\rho = 1.1$ g/cm³, pH of 0.02, and surface tension of $\sigma = 31.4$ mN/m. These properties were measured at room temperature.
4. The new acid formulation achieved a biodegradation rate of 65% by day 28 using ThCO₂ measurement, exceeding the requirements for ready biodegradability; a plateau of 81% was achieved by day 40.
5. The high removal efficiency, ready biodegradability, and low corrosion rate make this new acid formulation a good candidate for calcium carbonate-filter cake removal in oil and gas wells.

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