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Research Article

Removal of Hematite Water-Based Filter Cake Using Hydrochloric Acid

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The filter cake layer, which is essential to reduce the fluid invasion to the drilled formation, has been reported to be composed mainly of the weighting materials. The complete removal of this layer is recommended to restore the rock permeability. Hematite is one material that is used to create the filter cake. However, the cleanup of hematite-based filter cake has not been studied thoroughly. The objective of the work presented in this paper is to investigate the removal of hematite water-based filter cake using hydrochloric acid. Numerous solubility tests were conducted to study the different factors affecting the treatment of hematite such as temperature and particle size. Based on these results, the treatment duration and acid concentration were selected. The high-pressure high-temperature filtration cell was used to build the filter cake and to soak it in the treatment solution at 100°C. Even though the solubility is increasing with acid concentration, however, no significant improvement was noticed above 12.5 vol.% of HCl. The particle size distribution has a considerable effect on the treatment, and the solubility was found to be 86 wt.% for particles finer than 25 microns and 47 wt.% for particle sizes above 75 microns, all in 10 vol.% HCl. The temperature has a significant influence as the solubility doubled when the temperature increased from 75°C to 100°C. The solubility rate decreased with time and reached the maximum in 16 hours which has been taken as treatment duration. Using 12.5 vol.%, 91 wt.% of the filter cake was successfully removed. In the literature comprehensive study on the cleanup of hematite-based filter cakes is lacking. This research provides new insights into hematite removal and address the different factors that impact the treatment efficiency.

1. Introduction

Drilling fluids are used to facilitate the drilling operations by performing several tasks such as lubricating downhole tools, suspending rock cuttings, and transporting them to the surface [1, 2]. Additionally, the drilling fluids must maintain a sufficient hydrostatic pressure to overbalance the formation pressure, which is essential to prevent formation flow into the wellbore and control the wells. However, the extra pressure causes an invasion of the drilling fluid to the penetrated formation, potentially causing formation plugging, permeability alteration, and as a result increased costs [3]. Drilling fluid invasion, in addition to plugging rock pores, can also plug the gravel packs and the completion screens, which significantly reduces the well productivity/injectivity significantly [4–6].

A filter cake (FC) is formed when the solid particles of the drilling fluids are precipitated in the interface of permeable rock and usually have a very low permeability [7, 8]. The drilling fluid filtration invasion into the penetrated formation is reduced by the filter cake [9]. The filter cake formation and properties are thus regarded to be important features of drilling mud [10–12]. The best filter cakes are described to be thin, impermeable, quickly formed, and easy to clean [13].

Even though the FC is critical throughout the drilling process to prevent drilling fluid from entering the drilled

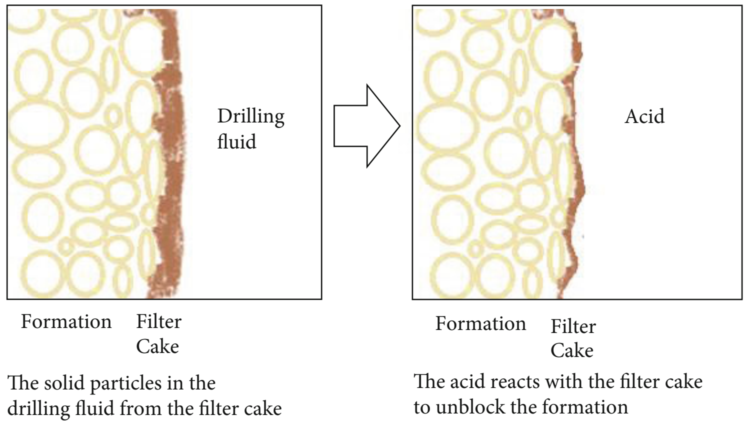

Figure 1: Sketch of the studied problem.

zones, effective FC removal after the drilling is required to enable efficient cementation and to regain the well productivity or injectivity [1, 14–16].

The filter cakes are characterized by several properties such as thickness, porosity, permeability, and particle size distribution; these properties affect the amount of mud filtration [17, 18]. Several factors affect the FC properties, namely, the type of weighting agents and polymers, salinity, clay, and sand content in the drilling fluid [19, 20].

Several studies demonstrated that the weighting material is the dominant element of the FCs and it composes around 70% of the filter cake layer by weight [21]. Weighting materials are added to the drilling muds to raise their density and, hence, to provide a sufficient hydrostatic pressure needed during the drilling operation to overcome the formation pressure. This required mud weight should be achieved while preserving adequate rheological properties and low solids settling [22, 23].

Iron oxide, also called hematite, has been introduced as one of the alternatives to barite as a weighting agent [24]. Hematite has slightly higher specific gravity than barite, and the hematite-based drilling fluids have close rheological properties the barite-based [25]. Two main drawbacks of hematite are its magnetic susceptibility and the high abrasiveness potential [24, 26, 27]. It also has a sagging tendency, but it can be minimized by micronizing the hematite particles; however, this can increase fluid loss [27].

Many factors influence filter cake treatment effectiveness, including the sizes of the drilling fluid particles and porosity of the drilled rock [28], the drilling fluid formula, particularly the weighting material, and the drilling fluid's liquid phase [29]. According to Hamzaoui et al. [30], the filter cake thickness and heterogeneity will continue to increase over time; therefore, any delay may complicate the treatment.

Since the weighting material is the dominant component of the filter cakes, the cleaning efficiency is largely determined by the weighting material's solubility in the treatment solvent. The cleanup of drilling fluids with different weighting materials has been studied and discussed in the literature. However, these researches have been limited to the most common weighting materials such as barite and calcium carbonate; in particular, hematite filter cake removal has not been investigated thoroughly. The use of hematite as a weighting material alternative to the barite is growing especially with the growing demands and the limited supplies of weighting material. As evident, some of the biggest oil companies approved the use of hematite as a drilling fluid weighting agent.

Table 1: Drilling fluid formula.

Material	Quantity	Unit
Water	245	cm^3
Defoamer	0.08	cm^3
Soda ash	0.5	gm
Xanthan gum	0.5	gm
Starch	6	gm
Bentonite	4	gm
KOH	0.5	gm
PAC-R	1.5	gm
KCl	20	gm
$CaCO_3$ (50 microns)	5	gm
Hematite	350	gm

Unlike common weighting materials such as barite and calcium carbonate, the removal of hematite containing filter cake has not been studied comprehensively. The objective of this paper is to establish a framework on hematite filter cake removal. Figure 1 presents a general sketch of the problem under study. In this work, numerous solubility tests were utilized to study the different factors that affect the treatment such as acid concentration, temperature, solid/HCl ratio, particle size distribution, and treatment duration. According to these tests, the treatment was designed and the removal efficiency was tested in the designated conditions. The next section describes the methodology that has been adopted in this research and the type of conducted tests. The third section highlights the main experimental results showing the significance of different factors which influence the treatment performance. Lastly, the forth section summarizes outcomes and future recommendations.

2. Methods and Materials

In this work, solubility tests have been applied to study the different factors that could affect treatment efficiency. Then and according to the solubility test results, the acid concentration and treatment duration have been chosen and tested in the high-pressure high-temperature (HPHT) filtration cell. In this section, the research methodology and the material utilized are presented.

2.1. Drilling Fluid. The drilling fluid formula that has been used in this study is provided from the field operations. The equivalent lab-scale formula is presented in Table 1. The mud is water-based with xanthan gum and bentonite to enhance the rheological properties. The starch and the polyanionic cellulose polymer (PAC) have been added as filtration control agents, while the calcium carbonate ($CaCO_3$) is considered a bridging agent. The alkalinity of the mud has been optimized by adding potassium hydroxide (KOH), and the potassium chloride is added as a shale inhibitor. The hematite has been utilized as the weighting material; the particle size distribution (PSD) of the used hematite is shown in Figure 2, with D50 of 17 microns and D90 of 51 microns. The drilling fluid has been tested for density, pH, and rheological properties as presented in Table 2. The mud weight is above 16 ppg which is designed for high-pressure applications.

2.2. Solubility Tests. Solubility tests were conducted to measure the dissolution rate of hematite in different conditions. A variety of factors were tested, such as the acid concentration (5 to 15 vol.%), temperature (ranging from 50°C to 125°C), and treatment duration (up to 24 hours). The solubility test setup is shown in Figure 3. In each test, 4 grams of hematite is soaked in 100 ml of the acid under the designated conditions for a specific duration. The remaining hematite after the test is filtrated in a filter paper using a vacuum pump then dried and by remeasuring its weight the solubility has been calculated with

$$\text{Solubility (wt.\%)} = \frac{\text{original hematite weight} - \text{remaining hematite weight}}{\text{original hematite weight}} \times 100\%. \quad (1)$$

2.3. Filtration and Removal Efficiency Tests. The filter cake was built over a 20-micron ceramic disc using HPHT filter press at 100°C and a differential pressure of 300 psi for thirty minutes. Then, the disc with the formed filter cake was soaked in the treatment solution for the designated period to test the removal efficiency. The weight-based removal efficiency is calculated using

$$\text{Removal efficiency (wt.\%)} = \frac{w_2 - w_3}{w_2 - w_1} \times 100\%, \quad (2)$$

where w_1 is the weight of the saturated disc (g), w_2 is the weight of the filter cake-containing disc (g), and w_3 is the disc weight after the removal process (g).

3. Results and Discussions

3.1. Acid Concentration. Using the solubility setup shown in Figure 3, the solubility of hematite in various acids has been tested. In these tests, around 4 grams of hematite was soaked in 100 ml of the acid for 24 hours at 100°C. Hematite was found to be insoluble in the tested organic acids such as citric, lactic, and glycolic acids. Then the hydrochloric acid (HCl) was tested at different concentrations ranging between 5 vol.% and 15 vol.%. The dissolution of hematite increased significantly in the range between 7.5 vol.% and 12.5 vol.%, while at 15 vol.% HCl concentration the hematite was completely dissolved in the solution, as shown in Figure 4. However, the improvement between 12.5 vol.% and 15 vol.% was modest; therefore, the rest of the tests are conducted with the 12.5 vol.% HCl concentration.

3.2. Temperature. The effect of temperature on the hematite solubility in HCl has been tested in the range between 50°C to 125°C with 12.5 vol.% HCl for 24 hours. As shown in Figure 5, the solubility values were 32 wt.% and 46 wt.% at 50°C and 75°C, respectively, and increased noticeably when the temperature increased to reach 95 wt.% at 100°C with no significant change above that.

3.3. Particle Size. As shown in the PSD in Figure 2, the sizes of the hematite particles were mostly ranging between 5 microns and 90 microns. To study the effect of the particle size on the solubility, the hematite sample has been separated by size using four sieves' sizes (25, 50, 75, and 100 microns). Then, the solubility of the four samples has been tested using 10 vol.% HCl at 100°C for 24 hours. The tests were conducted with the 10 vol.% HCl instead of the 12.5 vol.% because of the high dissolution rates with the latter which may not clearly reflect the effect of the particles' size. It is noticeable that the solubility is considerably affected by the particles' size as seen in Figure 6. The solubility decreased from around 86% for the sample with the particles finer than 25 microns to 47% for the sample with particles bigger than 75 microns.

3.4. Hematite/HCl Ratio. The effect of the solids to liquid ratio has been tested for the range from 2 to 6 grams of hematite on 100 ml of 12.5 vol.% HCl at 100°C for 24 hours. As shown in Figure 7, the hematite dissolved completely when its amount was 3 grams or less which indicated that the dissolution rate

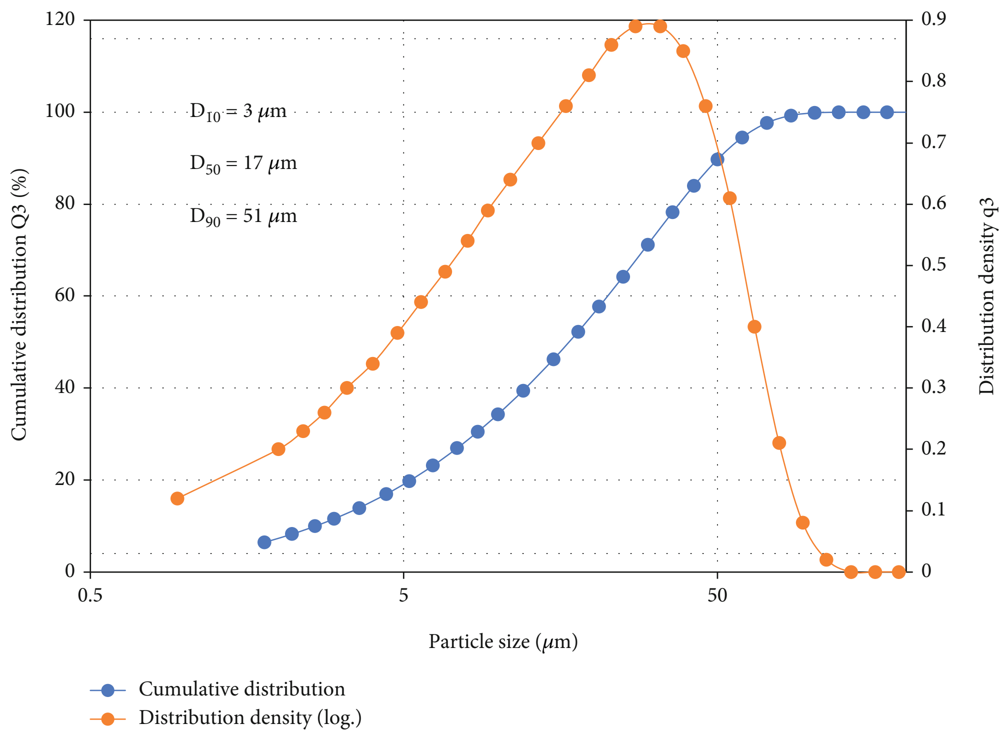

Figure 2: Particle size distribution of hematite.

Table 2: Drilling fluid properties at 120°F and atmospheric pressure.

Properties	Values
Φ_{600}	230.30
Φ_{300}	152.10
Φ_{200}	120.50
Φ_{100}	80.70
Φ_{6}	21.90
Φ_{3}	19.40
Gel Str. 10 sec, lb/100 ft^2	19.00
Gel Str. 10 min, lb/100 ft^2	54.00
Gel str. 30 min, lb/100 ft^2	72.00
Apparent viscosity	115.15
Plastic viscosity	78.20
Yp, lb/100 ft^2	73.90
pH	10.53
Density, ppg	16.30

Φ_x is the shear stress at x rpm.

is greater than 3 gm/100 ml for 24 hours. When the weight of the hematite increased to 4 grams, the solubility decreased to 95 wt.% which means that 3.8 grams were dissolved. Similarly, 78 wt.% of 5 grams (3.9 grams) and 66 wt.% of 6 grams (4 grams) were dissolved. These results indicated that the dissolution rate of hematite in 100 ml of 12.5 vol.% HCl is ranging between 3.8 grams and 4 grams per 24 hours.

3.5. Treatment Duration. Using 12.5 vol.% HCl at 100°C, the solubility has been tested for different durations up to 24 hours. As presented in Figure 8, most of the dissolution happened in the first three hours with a rate of around 0.8 g per hour; then, the reaction slowed down and almost stopped after 16 hours. According to this result, the treatment does not need to last for 24 hours; therefore, the filter cake removal test has been performed for 16 hours.

Table 3 summarizes the conditions and the outcomes of the solubility tests discussed in sections 4.1 to 4.5. Based on these results, the removal test has been conducted with 12.5 vol.% HCl and for 16 hours as in the test number 10 written in italics in Table 3.

3.6. Corrosion Test. The corrosion rate was tested using metal coupons soaked into the solution for six hours at 100°C. With 12.5 vol.% HCl, the corrosion rate was as high as 0.013 lb/ft^2. Hence, 2 vol.% corrosion inhibitor has been added, and the corrosion rate reduced significantly as shown in Figure 9. The addition of the corrosion inhibitor did not have a significant effect on the solubility (i.e., the solubility was 93% compared to 95% without the inhibitor).

3.7. Filter Cake Removal. Using the drilling fluid described in Table 1, the filtration test has been conducted on the HPHT filter press cell at 100°C. The filtration lasted for 30 minutes, and the filtration volume against time is reported in Figure 10. The total filtration volume was around 7 cm^3, which indicates that the filtration control agents performed well. The filtration stopped approximately after 25 minutes,

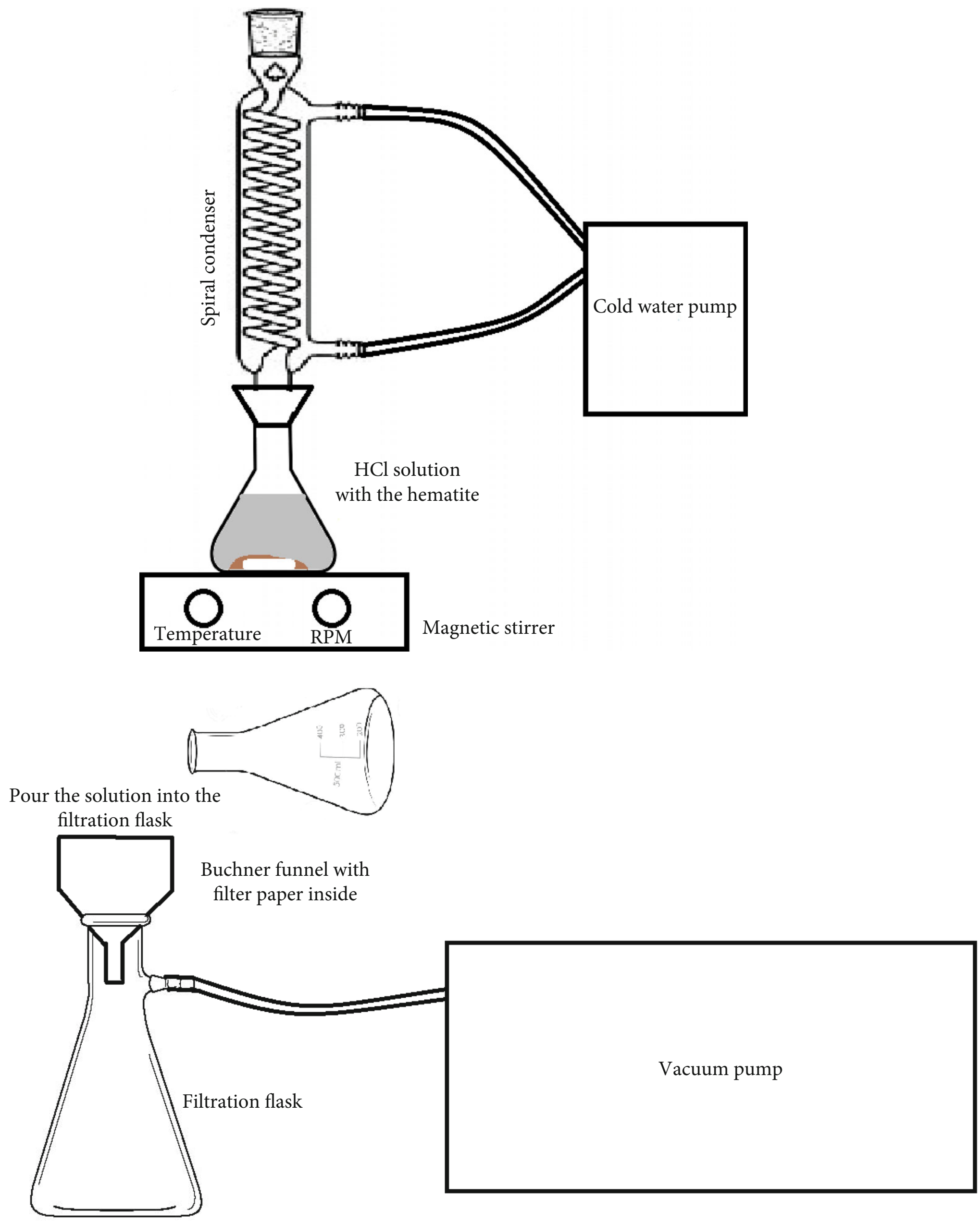

Figure 3: Solubility test setup.

which reflects the formation of impermeable filter cake on the face of the ceramic disc.

The dimensions and weight of the ceramic disc were recorded before and after the filtration test. The filter cake was thin with around 2 mm thickness, and the weight of the ceramic disc increased by 14 grams due to that, as illustrated in Table 4. After these measures have been recorded, the ceramic disc that contains the filter cake was placed back into the HPHT cell and the cell was filled with 250 ml of the 12.5 vol.% HCl with 2 vol.% corrosion inhibitor. The filter cake was soaked in the treatment solution for 16 hours at 100°C. After the treatment, the ceramic disc was extracted and remeasured, and the removal efficiency was calculated using equation (2). The obtained removal efficiency was 91%, and it could be noticed in Figure 11 that most of the filter cake has been cleaned from the face of the disc.

3.8. Additional Considerations. The following are the additional considerations:

(i) After the treatment period, the solution had a high concentration of ferrous ions which could result in formation damage unless the treatment duration is tested and designed properly

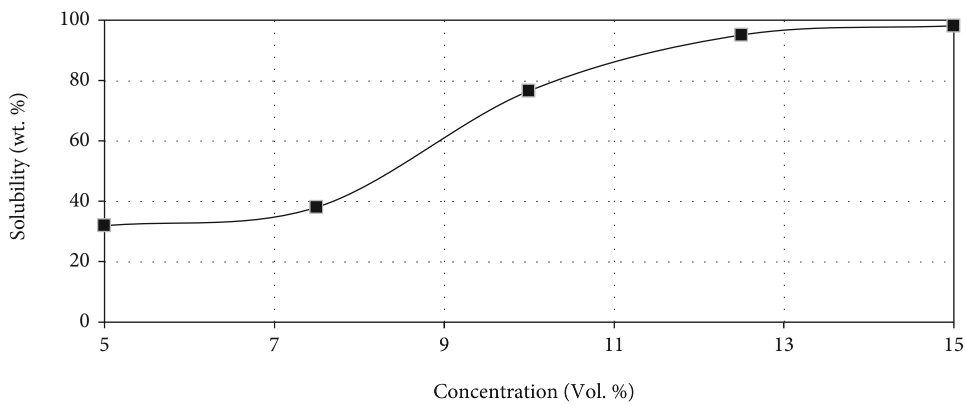

Figure 4: Hematite solubility at different HCl concentrations.

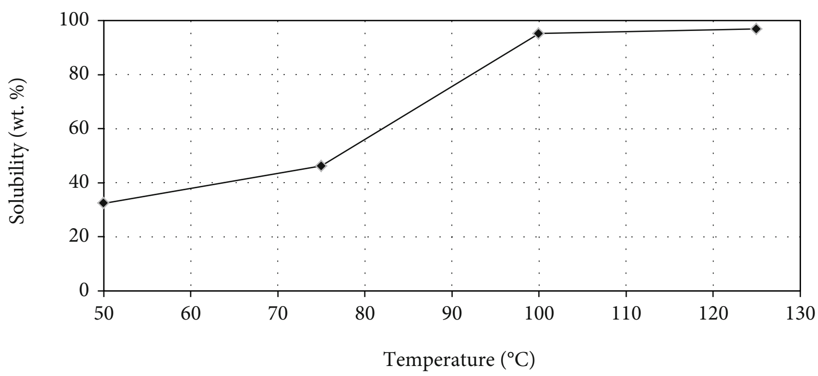

Figure 5: Hematite solubility in 12.5 vol.% HCl at different temperatures.

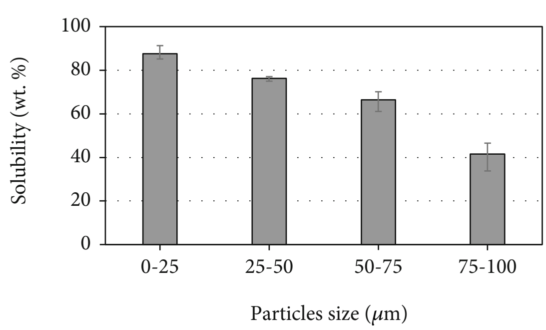

Figure 6: The effect of particle size on hematite solubility in 10 vol.% HCl.

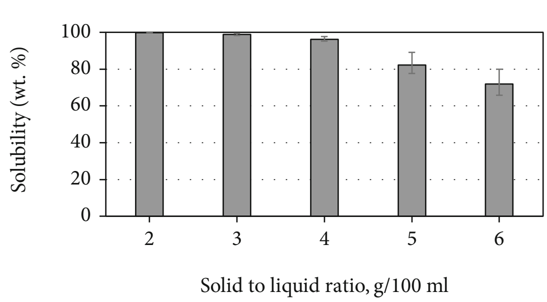

Figure 7: The effect of solid to liquid ratio.

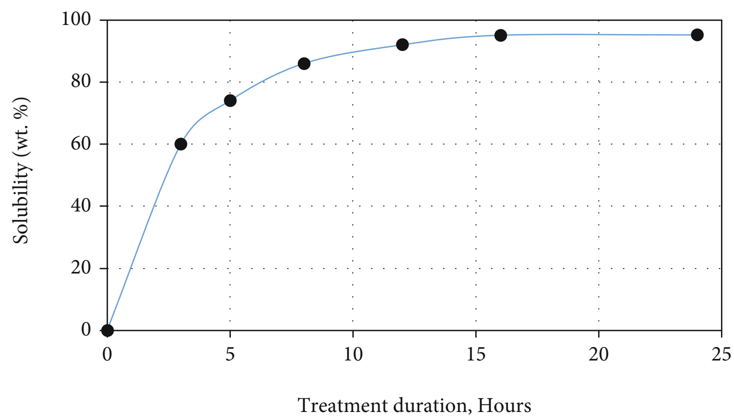

Figure 8: Solubility of 4 grams of hematite over 24 hours.

Table 3: Summary of the solubility tests.

Test index	Concentration (vol.%)	Temperature (°C)	Duration (hours)	PSD (microns)	Hematite weight (grams)	Solubility (wt.%)
1	5.0	100	24	As in Figure 2	4	32
2	7.5	100	24	As in Figure 2	4	38
3	10.0	100	24	As in Figure 2	4	77
4	12.5	100	24	As in Figure 2	4	95
5	15.0	100	24	As in Figure 2	4	98
6	12.0	100	3	As in Figure 2	4	60
7	12.5	100	5	As in Figure 2	4	74
8	12.5	100	8	As in Figure 2	4	86
9	12.5	100	12	As in Figure 2	4	92
10	*12.5*	*100*	*16*	*As in Figure2*	*4*	*95*
11	10.0	100	24	0-25	4	86
12	10.0	100	24	25-50	4	77
13	10.0	100	24	50-75	4	68
14	10.0	100	24	75-100	4	47
15	12.5	50	24	As in Figure 2	4	32
16	12.5	75	24	As in Figure 2	4	46
17	12.5	125	24	As in Figure 2	4	97
18	12.5	100	24	As in Figure 2	2	100
19	12.5	100	24	As in Figure 2	3	99
20	12.5	100	24	As in Figure 2	4	95
21	12.5	100	24	As in Figure 2	5	78
22	12.5	100	24	As in Figure 2	6	66

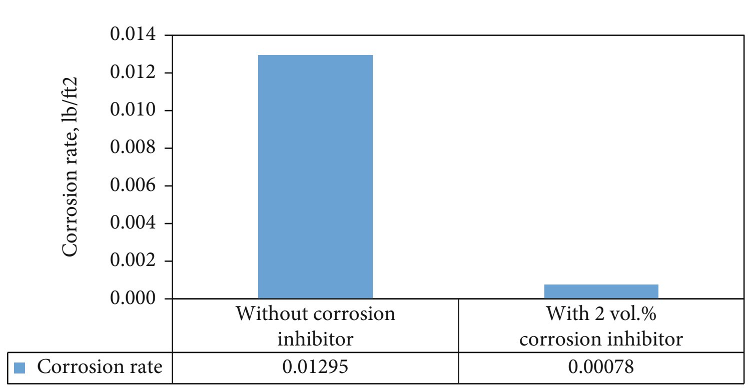

Figure 9: Corrosion rate at 100°C with and without the corrosion inhibitor.

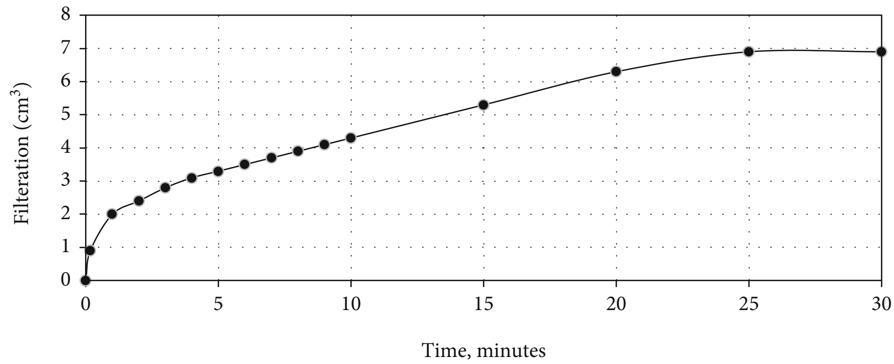

Figure 10: Filtration over 30 minutes.

Table 4: Summary of the solubility tests.

Ceramic disc properties before the filtration test	
Thickness	6.40
Dry wt. (gm)	40.79
Wet wt.[saturated with brine] (gm)	48.27
After HPHT filtration	
Thickness [disc + filter cake] (mm)	8.23
Wet wt.[disc + filter cake] (gm)	62.74
Filter cake wet wt. (gm)	14.47
Filter cake thickness (mm)	1.83
After treatment	
Wet wt. after removal [disc + filter cake]	49.56
Dry wt. after removal [disc + filter cake]	42.73
Filter cake wt. after removal (gm)	1.29
Removal efficiency	91%

Figure 11: The filter cake: (a) after 30 minutes of drilling fluid filtration and (b) after soaking in 12.5% HCl.

(ii) The study assumed uniform filter cake which may not be always the actual case as there could be thickness variation

(iii) The effect of the corrosion inhibitor on the solubility should be tested to ensure decent removal efficiency

4. Summary and Conclusions

Filter cake removal retrieves the formation productivity and facilitates the cementing integrity. In this paper, the removal of filter cake formed by a hematite-based drilling fluid was investigated. Different factors that affect the efficiency of the treatment were tested, namely, the acid concentration, temperature, particle size, and soaking duration. Based on the results presented in this paper, the followed points could be highlighted to summarize the research findings:

(i) Acid concentration is a major factor that affects the filter cake treatment efficiency; however, increasing the HCl concentration above 12.5 vol.% did not significantly improve the hematite solubility in contrast to changing the concentration from 5 vol.% to 10 vol.%

(ii) Using 12.5 vol.% HCl, increasing the temperature up to 100°C increased the solubility rates while no considerable change was noticed afterward up to 150°C

(iii) At high particle size, the hematite dissolution was considerably less compared to finer particles. The solubility in 10 vol.% decreased from around 86 wt.% for the sample with the particles finer than 25 microns to less than 50 wt.% for the sample with particles bigger than 75 microns

(iv) More than 60% of the solubility happened in the first three hours, and the dissolution rate decreased afterward so that in the next nine hours the solubility increased by 33%. After 16 hours, the maximum solubility was achieved, and no further improvement was noticed afterward

(v) Based on numerous solubility test results, 12.5 vol.% HCl was used to treat the hematite filter cake for 16 hours at 100°C. The achieved filter cake removal efficiency was 91%

Data Availability

The data are included in the manuscript.

Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

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