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Bio-Oil Dispersants Effectiveness on AsphalteneSludge During Carbonate Acidizing Treatment

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Abstract

Matrix acidizing is a remedial well stimulation that done to overcome formation damage near wellbore or improve the permeability. Although acidizing treatments are proven and abundant there is still inherit from formation damage when pumped. Acid-induced asphaltene sludging is becoming an increasing cause of oil well stimulation Failure.

The objective of the paper is to evaluate the performance of coconut oil as a bio-oil dispersant against commercial dispersants in preventing asphaltene sludge while acidizing carbonate cores with 15 wt.% HCl and a chelating agent. A Kuwaiti crude oil was used in this study has an API of 38° and 2% asphaltene content. The crude oil was characterized by a variety of analytical techniques including total acid and base numbers (TAN, TBN), saturates, aromatics, resins and asphaltene analysis (SARA), density, viscosity and elemental analysis. Indiana limestone cores were used with average porosity of 16% and permeability ranges (9-13) md. X-ray diffraction (XRD) was used to analyze the mineral and clay content in the cores. Sludge tests were used to examine the acid and oil compatibility using anaging cell under 500 psi and 160°F with oil to an acid ratio of 1:1. Coreflooding experiments under reservoir condition were done with the selected two acid systems, 15 wt. % HCl and achelating agent. Indiana limestone cores with a permeability of 7-12 md were initially saturated with the crude oil then acid was injected until breakthrough. The injected acid volume was recorded and CT-scan imaging of the cores after the acid treatment was used to evaluate the structure and the propagation of the wormhole. The effluent fluids were analyzed by inductively coupled plasma (ICP) and pH measurements.

The results for a Kuwaiti crude oil showed the formation of 13 wt% sludge with 15 wt% HCl and it increased to 19 and 30 wt% with increasing acid concentrations to 20 and 28 wt%, respectively. The presence of iron(III) in the system increased the sludge precipitation to 17.8 wt% at 15 wt% HCl and 3,000 ppm iron concentration. The sludging decreased to 7.5 wt% by adding 300 ppm coconut oil to the system. The formation of asphaltene sludge in the carbonate acidizing retards the wormhole propagation. Hence, the injected acid volume to the breakthrough decreased from 1 to 0.4 by adding 300 ppm coconut oil to the acid system. A conical wormhole was formed with the injection of 15 wt% HCl, comparing to a uniform wormhole in the presence of coconut on the acid system. In the case of stimulating the cores with achelating

agent (20 wt% GLDA), the coconut oil exceeds the expectations with the minimum pore volume needed to breakthrough compared to the GLDA alone or with the chemical dispersant B.

This study concluded that the use of dispersant can help reduce the asphaltene sludge and create better acid propagation through the core. The results can be employed to design the optimum acid formulation and create the desired wormhole in carbonate formations.

Introduction

Acidizing treatment is a common workover that done overcome near wellbore damage resulted from drilling and production. In oil wells, stimulation treatment can result in a reduction in permeability reduction or total loss of the well due to the formation of induced asphaltene sludge.

The formation of asphaltic sludge precipitates during acidizing operations, especially in the presence of Fe II and Fe III ions, has become more widely recognized over the last several years. Asphalteneis present in crude oil in the form of a colloidal dispersion which consists of an aggregate of polyaromatic molecules surrounded by lower molecular weight neutral resins and paraffinic hydrocarbons. Strong acids such as hydrochloric acid seem to destabilize the colloidal dispersion and thus cause asphaltene precipitates and rigid film emulsions. It has been found that hydrochloric/hydrofluoric acid blends seem to cause more severe sludging than hydrochloric acid of the same total concentration. Thus, greater amounts of anti-sludging additives are required with HCl/HF mixtures to get the same level of control as with straight HCl. Weaker acids such as acetic acid do not seem to cause sludging. Sludging increases dramatically with the concentration of acid, such that 28% HCl should not be used if asphaltene precipitates are possible. Even going from 15% to 20% increases the need for more additives to control the sludging process (Jacobs 1989; and Abdollahi et al. 2014)

Moore et al. (1965) investigated the asphaltene sludge after the problem was noticed in several California wells. The authors suggested using alkyl-phenol or fatty acids as an anti-sludge agent to target the asphaltene colloidal stability. Good results were obtained after field implementation. Further studies were done to understand the cause of the asphaltene sludge after the problem continued even with the use of an anti-sludge agent. Jacobs and Throne (1986) concluded that the presence of iron (Fe) contamination is one of the main causes of the sludge. They suggested using an iron control agent with the anti-sludge in the acid package. They also recommended a pre-flush with aromatic solvent.

The acid concentration and type of acid were studied to monitor their effect on the sludge. The studies showed that acid concentration threshold for hydrochloric acid is 15% and mud acid showed not exceed 12%:3% HCl: HF (Hochin et al. 1990). As the awareness of the sludging problem grown in the industry, more suggestion to design the acid treatment and test the acid additives for compatibility and their tendency for sludge. Several acid additives were proved to increase the sludge or emulsion such as some corrosion inhibitors. (Jacobs 1989; Suzuki 1993; Hochin et al. 1990; and Wong et al. 1996)

Several studies were later conducted to tackle the asphaltene sludge by reducing the iron presence. The chelating agent, such as NTA, EDTA were used as iron control agent (Fredd and Fogler 1996). Nonionic reducing agent (NIRA) also tested as acid additives against dodecyl-benzene-sulphonic acid (DBSA) (Vinson 1996).

Calcium carbonate rock as limestone has been widely stimulated with HCl. In some high-temperature HCl does not produce acceptable stimulations results because of the lack of penetration or surface reactions (Kelland 2014). Also, the high corrosion rate resulting from using strong acid as HCl at high temperature. Carbonate acidizing treatments with chelating agents such as EDTA have been reported to work better at high temperature, offering low corrosion rates and good dissolving power (Fredd and Fogler 1996; Kelland 2014).

GLDA was investigated as stimulation fluid in carbonate rock as a biodegradable chelating agent as an alternative to EDTA. GLDA found to be very effective in creating wormholes at low injection rates and low to moderate pH values. The optimum concentration for GLDA to create wormholes at minimum injected porevolume was 20 wt% (Mahmoud and Nasr-El-Din 2010). The presence of residual oil in the cores also reported decreasing the injected porevolumes needed to breakthrough while acidizing with GLDA (Mahmoud and Nasr-El-Din 2011).

The use of bio-oils as asphaltene dispersants was investigated by (Alrashidi and Nasr-El-Din 2017). The authors concluded that using coconut oil at 300 ppm as a dispersant can prevent any asphaltene aggregations. In this study, the coconut oil will be used as ananti-sludge agent against chemical dispersant in acidizing limestone cores using GLDA and HCl.

Experimental Studies

Materials

The crude oil tested in this study was provided by a Kuwaiti Oil Company (Table 1). Two chemical dispersants were provided by a local service company and were used as received. Fractionated coconut oil and andiroba oil were used and are commonly used in a household. Table 2 shows the elemental composition and molecular weights of asphaltene sample, the asphaltene sample was extracted using heptane.

Table 1—Properties of the crude oil.

Characteristic	Value
SARA Analysis	
Saturates (%)	48
Aromatics (%)	45
Resins (%)	5
Asphaltene (%)	2
Density (g/cm^3) at 25°C	0.83
Viscosity (cp) at 25°C	4.3
TAN (mg KOH/g oil)	0.1
TBN (mg HCl/g oil)	0.01
Water Content (wt%)	0.3

Table 2—Elemental analysis and molecular weight of asphaltene sample.

	Heptane-asphaltene
Composition (wt%)	
carbon	84.61
hydrogen	5.79
nitrogen	1.08
oxygen	3.17
sulfur	4.15
Molecular weight (Da)	815.37

Core Preparations. Indiana limestones cores were used in all the experiments. The permeability of the oil-saturated cores varied from 9 to 38 md. All the cores are 6 in. length and 1.5 in. diameter. The cores were dried in the oven for overnight to remove any moisture. The cores were then saturated with deionized water for 3 hours using a vacuum pump. Then at least 4 pore volumes of deionized water were injected to measure the initial permeability of the cores and fully stature them with water.

The cores were then saturated with the crude oil by pumping at least 5 pore volumes of oil through the cores at different flow rates from 0.1 cm^3/min to 1 cm^3/min over a period of 32 hours. This ensured complete saturation of the core. The weight of the cores was measured after each saturation step to estimate the porosity and effective permeability.

Fluid Preparation. The acid solution was prepared using deionized water. The concentration of the hydrochloric acid used in this experiment was 36.46 wt%. This acid was diluted to 15 wt% using deionized water. GLDA were obtained from achemical company with pH value around 3.6. Corrosion inhibitor used is added to the acid solution to prevent corrosion of the accumulators and lines. The acid system was prepared by mixing deionized water, corrosion inhibitor, and hydrochloric acid or GLDA for 30 minutes with a magnetic stirrer.

Sludge Test. The crude oil and acid system was tested for sludge tendency. The testing involves mixing 50:50 ratios of the acid system: crude oil. The mixture agitated for 15 minutes then placed in anaging cell under 500 psi pressure and placed in the oven at 160°F for an hour. The mixture then observed, if there is two separate phase indicates that there is no sludge, and if not, the sample is then filtered and the slug is weighed.

Coreflood Setup. The coreflood setup used to stimulate the matrix acidizing process has been described in Fig.1.

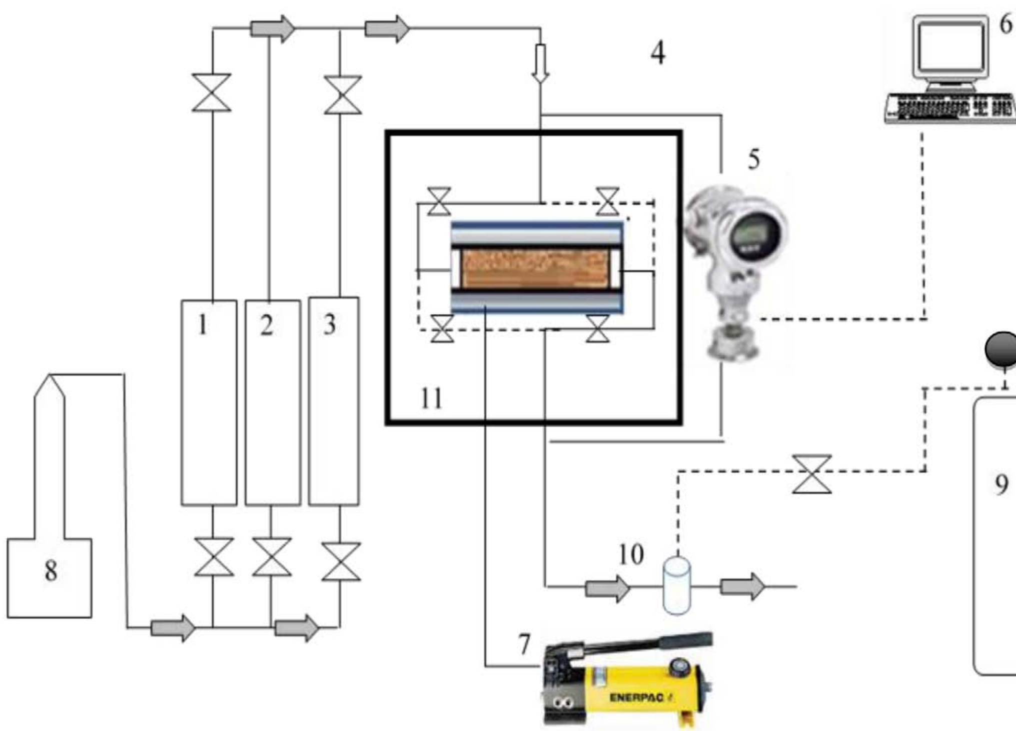

Figure 1—Coreflood experiment setup, where 1-3 Accumulators, 4-core holder, 5-pressure transducer, 6-computer LabView, 7-hand pump, 8-syringe pump, 9-nitrogen cylinder, 10-back pressure regulator, and 11-oven.

The cores were subjected to CT scan after each step in order to determine the CT number of the core. CT number served the primary source of identifying the extent of dolomitization, the presence of anhydrites, and can be used to detect wormholes propagation. The cores were scanned and imaging software named Image J was used to analyze and stack the images. The software designed to compile the images of cores taken over different cuts along the length of the core and to visualize the wormholes after the treatment.

Results and Discussions

X-ray diffraction was conducted for mineralogy examination. Indiana limestone cores identified as more than 99 wt % calcite, less than 1 wt% quartz, and clean from clays.

The sludge test was conducted to test the tendency of the oil to form sludge under different concentration of HCl and iron. The first set of the sludge test was conducted to select the optimum acid concentration to use in the experiments. The common three acid concentration used in the field were tested 15, 20, and 28 wt % HCl. Table 3 presents the Sludge test results for different HCl concentrations. As the acid concentration increases, the sludge percepitiaion increased which is agreement with the literature (Jacobs 1989; and Abdollahi et al. 2014). The decision was made to choose the 15 wt% HCl concentration for further analysis.

Table 3—Sludge test for different concentration of HCl

HCl concentration, wt %	Sludge, wt%
15	13
20	19
28	29.75

The second set of sludge test was conducted to measure the effect of the presence of iron on the formation of the sludge. The results listed in Table 4 showed that as the iron concentration increased the sludge increased. That's due to the interaction between the ferric cation with the un-stabilized negative charged asphaltene particles.

Table 4—Sludge test for 15 wt% HCl and corrosion inhibitor with different concentration of Iron

Iron Concentration, ppm + 15 wt% HCl + CI	Sludge, wt%
1000	7.625
2000	11.5
3000	16.75

The effect of each dispersant was tested at its optimum concentration (Alrashidi and Nasr-el-din 2017) in the third set of sludge tests. Acid and the crude oil completely mixed as shown in Fig. 2. The results in Table 5 showed that dispersant B prevents the sludge completely. The sample showed complete phase separation between the acid system and the oil while in dispersant A, 3 wt% sludge was recovered after filtering the sample. In the case of the vegetable oils, coconut oil reduces the sludge caused by 15 wt% HCl to almost 45%. From these results, the decision was made to select the best performance dispersant formed each category, dispersant B as a chemical dispersant and coconut oil as a bio-oil dispersant.

Figure 2—Mixture of the crude oil/HCL with 300 ppm of different dispersant at the end of the slug tests.

Table 5—Sludge test for 15 wt% HCl and 1 vol% corrosion inhibitor with different dispersants

15wt% HCl+ Dispersant+ 1 vol% CI	Sludge, wt%
Disp. A (300 ppm)	3
Disp. B (300 ppm)	Tracer
Coconut oil (300 ppm)	7.875

The last set of sludge test was conducted to test the second stimulation system that contains 20 wt% GLDA and 1 vol% corrosion inhibitor, with the selected dispersants. The results represented in Table 6 showed that there is no sludge at all form in the three cases and clear phase separation as appearing in Fig. 3. That agrees with the literature as the chelating agent should be a choice in acidizing oil wells with acid-induced asphaltene sludge problems.

Table 6—Sludge test for 20 wt% GLDA and corrosion inhibitor with different dispersants

20 wt% GLDA + Dispersant + CI	Sludge wt. %
20% GLDA	0
20% GLDA+ Dispersant B (300 ppm)	0
20% GLDA+Coconut oil (300 ppm)	0

Figure 3—Mixture of the crude oil/GLDA with 300 ppm of different dispersant at the end of the slug tests.

Coreflood Results

In all experiments, an over burden pressure at least 300 psi higher than the inlet pressure was kept to prevent the fluid from bypassing the core. The temperature of all the experiments kept at 160°F to represent the reservoir temperature. The flow rate was chosen to be as low as 0.5 cm^3/min to ensure maximum interaction between oil and stimulation fluid (Kumar et.al 2014). From the results of the sludge test, the experiments scheme was designed to test the two different sets of experiments using 6 different stimulation systems in a total of 6 coreflood experiments. Table 7 contains details about the core properties and stimulation fluid corresponded to it.

Table 7—Core properties and stimulation fluid corresponded in the different coreflood experiments.

Experiment #	Lithology Type	Porosity, vol %	Initial Permeability, md	Stimulation fluid	flow rate, cm^3/min
1	Indiana limestone	16.12%	11.30	15 wt% HCl	0.5
2	Indiana limestone	16.30%	11.26	15 wt% HCl + disp. B	0.5
3	Indiana limestone	16.25%	13.26	15wt% HCL +coconut	0.5
4	Indiana limestone	14.75%	13.00	20 wt% GLDA	0.5
5	Indiana limestone	16.00%	11.30	20 wt% GLDA + disp.B	0.5
6	Indiana limestone	15.90%	9.40	20 wt % GLDA + coco	0.5

Case I: Indiana Limestone Acidizing with 15 wt% HCl

Three different coreflood experiments were conducted to evaluate the effectiveness of the sludge disperesnt in the case of using 15 wt% HCl acid system. A base experiment was conducted with 15 wt% HCl and 1 vol % corrosion inhibitor without adding any dispersants. The second experiment was using the same system and adding dispersant B at 300 ppm, while the third experiment used coconut oil at 300 ppm as an anti-sludge agent. In all the three experiments, oil was injected first until stabilization pressure was reached, followed by acid until acid breakthrough. Deionized water was used to flush the acid after the breakthrough was reached. The pH values of the effluents samples collected during the coreflood experiments were measured using a pH meter. The analysis of the pH will help to recognize the time of breakthrough when the pH reaches near zero, while higher pH numbers represent that the acid is spent.

Fig. 4 represents the pressure drop across the core versus the cumulative injected pore volume (PV). The base case was noticed to have distinguished behavior than the two cases with the dispersants. In the base case, an increase in pressure noticed after the acid injected and that indicates that the acid reacted with the oil to form insoluble sludge. The sludge made acid penetration harder reflected in almost double acid quantity of pore volume needed in the base case to breakthrough compared to the cases with coconut oil and dispersant B. The performance of the acid systems containing dispersants B and coconut oil was almost identical. Both systems reached a breakthrough after injecting 0.37 PV while 0.9 PV is needed in the base case. The pressure drop didn't show a steep increase in the cases with the dispersants that indicates that both the coconut oil and dispersant B stabilize the asphaltene which makes the propagation of the acid smoother within the core. Fig. 5 represents the pH of the collected effluent samples versus the cumulative injected pore volume. The results in agreement with pressure drop analysis showed the breakthrough in the experiments with added dispersants occurred at the same time while in the base case double the pore volume was required.

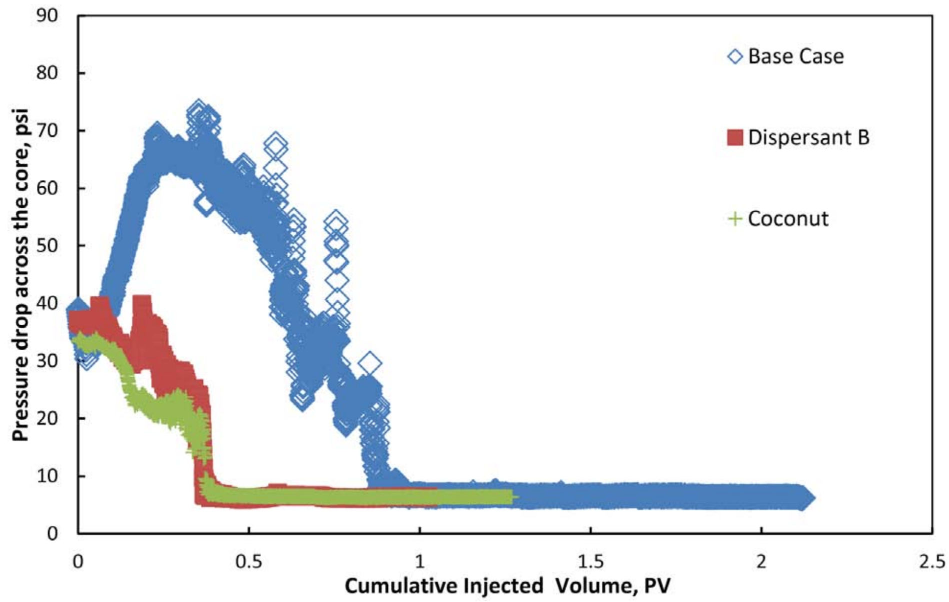

Figure 4—Pressure drop across the core vs. cumulative injected PV for limestone acidizing with 15 wt% HCl.

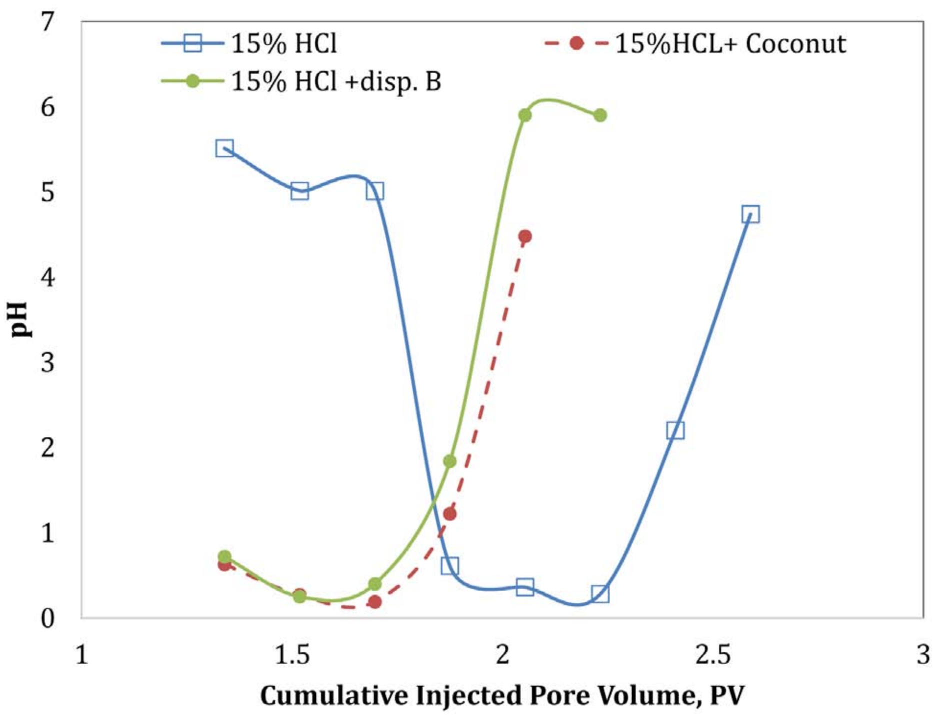

Figure 5—pH vs. cumulative injected PV for limestone acidizing with 15 wt% HCl

Fig. 6a reflects the results of the pressure drop analysis for the base experiment. The high increase in pressure drop caused by more reaction in the inlet section of the core resulted in conical shape wormhole with a wide top and a narrow end. Some face dissolution was noticed on the inlet.

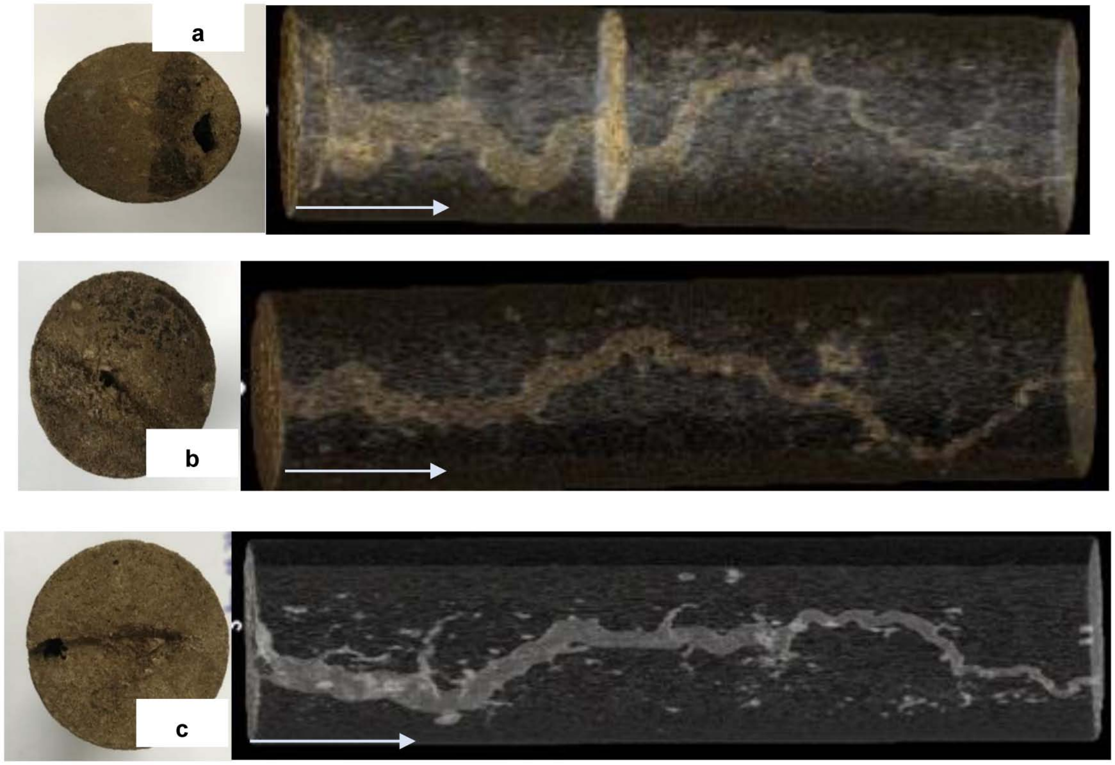

Figure 6—Wormhole profiles for the first set of coreflood experiments; a) 15 wt% HCl, b) 15 wt% HCl + dispersant B, 15 wt% HCl + coconut oil.

In Fig. 6b and c, the propagation of the acid was smooth resulted in a long uniform wormhole. The addition of dispersant B and coconut oil gave desirable results in acidizing the Indiana limestone cores.

Case II: Indiana Limestone Acidizing with 20 wt% GLDA

Three different coreflood experiments were conducted to evaluate the effectiveness of the sludge disperesnt in the case of using 20 wt% GLDA acid system. A base experiment was conducted with 20 wt% GLDA and 1 vol% corrosion inhibitor without adding any dispersants. The second was using the same system and add dispersant B at 300 ppm, while the third experiment used coconut oil at 300 ppm as ananti-sludge agent. In all the three experiments, oil was injected first until the pressure stabilized followed by the stimulation fluid until the breakthrough or maximum of 4.5 pore volume. Deionized water was used to flush the acid after the breakthrough was reached or the full four pore volume injected.

The pressure drop across the core versus the cumulative acid injection volume in Fig. 8 showed a similar trend as case I. The base case has a higher increase in pressure drop that reflects more resistance for the stimulation fluid to penetrate the rock. Even though all the results from the sludge test with GLDA showed that no sludge formation. For the base case, no breakthrough was noticed after injecting a total of 4 PV.

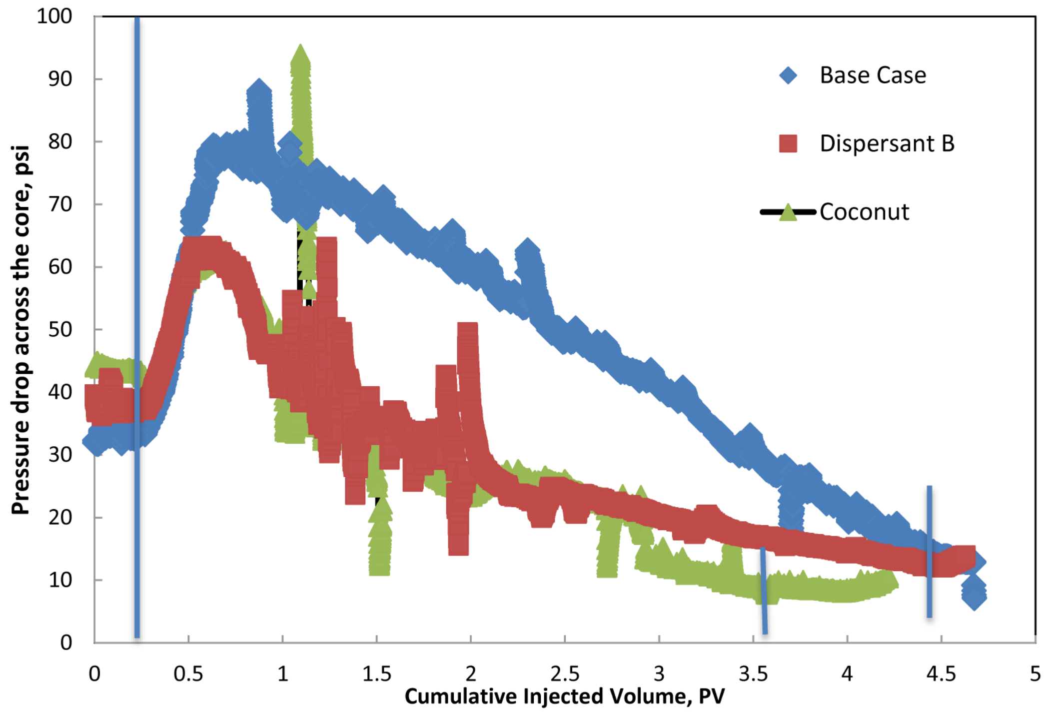

Figure 7—Pressure drop across the core vs. cumulative injected PV for limestone acidizing with 20 wt% GLDA

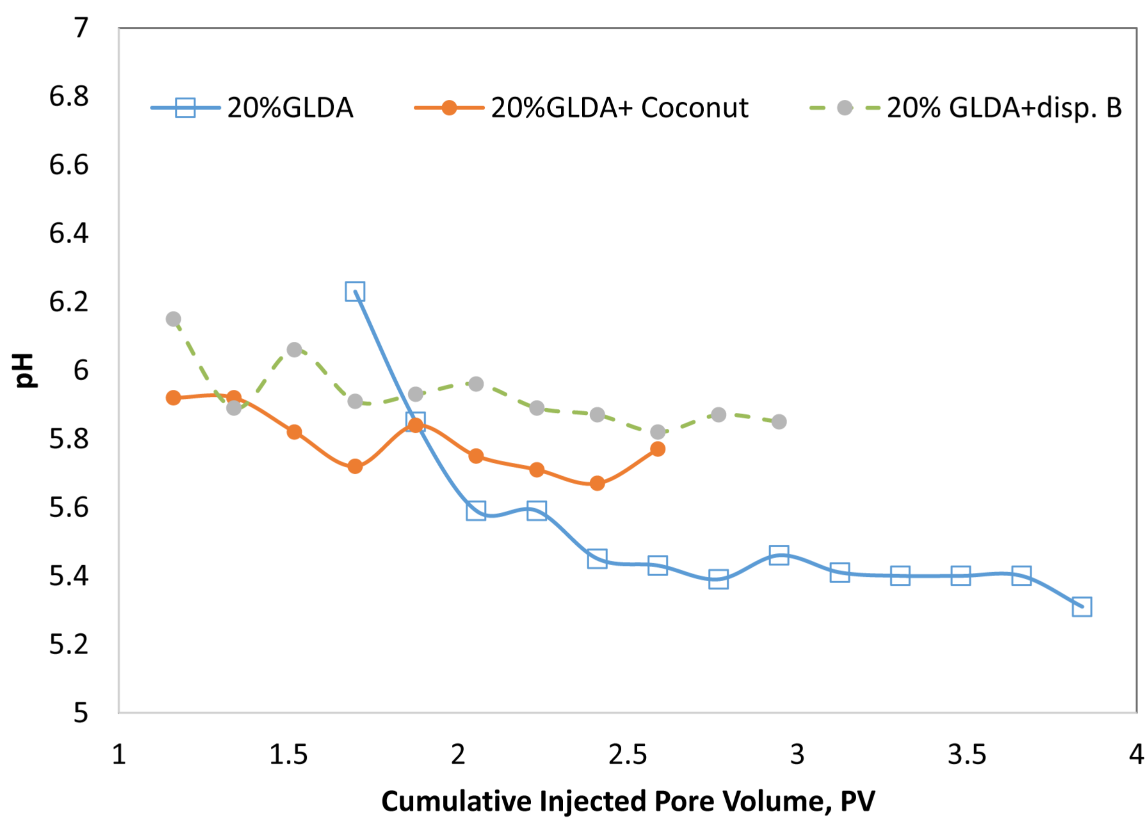

Figure 8—pH vs. cumulative injected PV for limestone acidizing with 20 wt% GLDA

For the experiments with dispersant B and the coconut oil, although both have similar pressure drop behavior, an earlier breakthrough occurred at 3.6 PV in the case with coconut as an anti-sludge agent. In Dispersant B experiment, the breakthrough occurred after 4.4 PV was injected.

The pH measurement of the effluent samples in Fig. 9 is in agreement with the pressure drop analysis. The performance of dispersant B and coconut are similar to the first case, although coconut oil outperformed the chemical dispersant B with less PV needed to breakthrough. It should be noted that the best performance system that contains GLDA and coconut is considered 100% biodegradable and eco-friendly system.

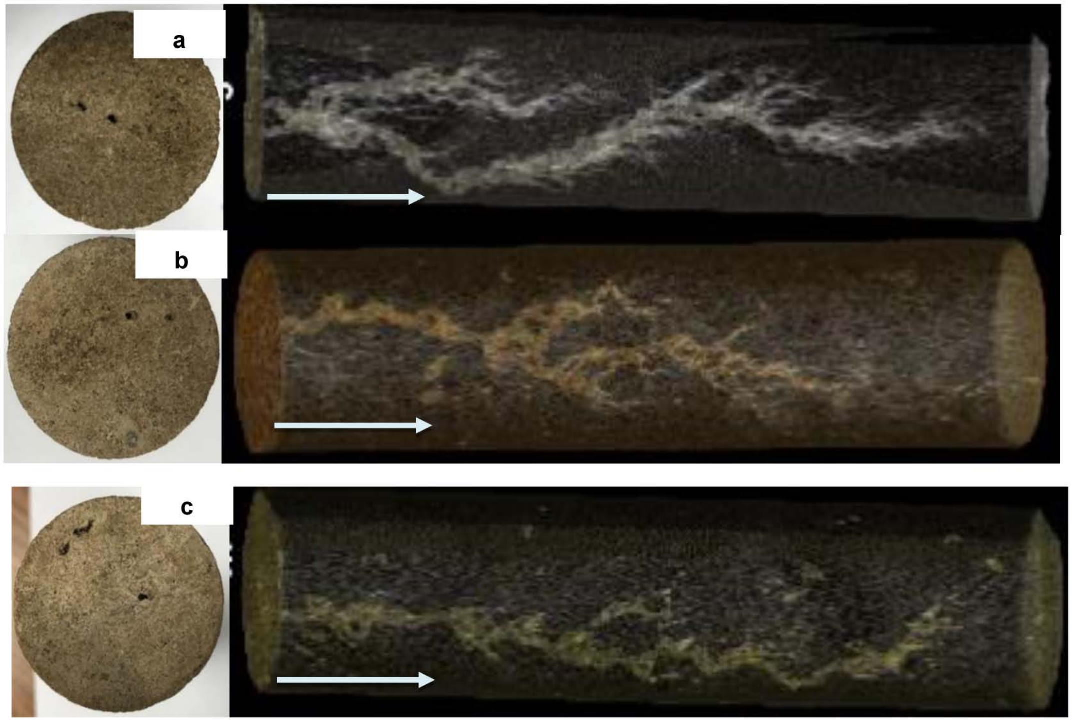

Figure 9—Wormhole profiles for the first set of coreflood experiments; a) 20wt% GLDA, b) 20wt% GLDA + dispersant B, c) 20wt% GLDA + coconut oil

The narrow wormholes in all cases indicate the reaction power of the weak GLDA since the GLDA have PH around 3.6 its needed more PV to penetrate the core. In **Fig. 10a**, the excessive branching in the wormhole reflects the high-pressure drop noticed in the base case with 20 wt% GLDA. Dispersant B wormhole profile in **Fig. 10b** show less branching in the wormhole with the very narrow end. The best performance in case II was the coconut, **Fig. 10c** showed a wormhole with a uniform width across the core, with very little branching and that's very favorable performance in matrix acidizing.

Conclusions

This study investigates the effectiveness of using the coconut oil as an anti-sludge agent against chemical dispersant in acidizing limestone cores using GLDA and HCl. The main conclusions are summarized as follows:

1. The crude oil has a tendency to sludge with 15 wt% and above concentrations of HCl and the iron increases the asphaltene precipitation. While GLDA didn't create any sludge when tested with the oil.
2. The chemical Dispersant B completely prevents the sludging of the oil, while the coconut oil reduces it by 50% when added to 15 wt% HCl.
3. For carbonate rocks, using the coconut oil and Dispersant B as an anti-sludge agent improves the wormhole propagation when added to 15wt% HCl and decrease the injected acid to breakthrough to 0.4 PV comparing to 0.9 PV with 15 wt% HCl only.
4. Adding coconut oil to GLDA reduces the injected pore volume to breakthrough to 3.6 PV comparing to 4.4 PV in the case of Dispersant B.

The coconut oil can be used as an environmentally friendly bio-dispersant to reduce the asphaltene sludge and create better acid propagation through the core. The results can be employed to design the optimum acid formulation and create the desired wormhole in carbonate formations.

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