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To cite this article: Adeline Norlee *et al* 2019 *IOP Conf. Ser.: Mater. Sci. Eng.* **495** 012113

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Investigating the Effectiveness of Emulsified Acid on Sandstone Formation under High Temperature Conditions

Adeline Norlee¹, Tan Inn Shi¹, Hisham Khaled Ben Mahmud, Henry Foo Chee Yew and Mian Umer Shafiq¹

¹Department of Petroleum Engineering, Curtin University Malaysia, CDT 250, Miri 98009, Sarawak, Malaysia

tan.s@curtin.edu.my

Abstract. Acid stimulation supports the oil and gas industry as a versatile mean in enhanced oil recovery to fulfil the world energy demand. Although hydrochloric acid can significantly improve the reservoir permeability, its rapid reaction rate at high temperature has created a barrier for acid penetration. Subsequently, emulsified acid has slowly gain its popularity due to its retardation effect which allows deeper penetration of acid into the formation and achieves minimal corrosion issues. Nonetheless, emulsified acid has rarely applied on sandstone formation. Since a large portion of reservoirs are made up of sandstone, the effects of emulsified acid on sandstone under high temperature conditions should be studied to unlock the effective usage of emulsified acid in restoring the hydrocarbon recovery from the potential sandstone reservoirs. Besides, it is also crucial to explore cheaper and greener substitute to formulate innovative emulsified acid in minimizing the high acidizing budget along with environmental concerns. In this project, 10 different emulsified acid combinations are prepared using hydrofluoric acid, hydrochloric acid, phosphoric acid, formic acid, acetic acid, cationic surfactant, diesel and waste oil. The pre-flush treatment (5 % CH₃COOH: 10 % HCl) is followed by the main flush (emulsified acids) saturation for 3 days. The thermal stability of emulsified acids and their effect on the Berea sandstone properties are evaluated. Major outcome is that the emulsified acids have the capability to remain stable at 275 °F up to 6 hours with uniform and fine droplet size. It is also proven that most of the emulsified acids can improve the porosity and permeability of Berea sandstone core samples except for HF: H₃PO₄. Regardless of the types of oil used for emulsified acid formulation, HF: HCl acid combination shows the best performance enhancement for both diesel-based and waste oil-based emulsified acids. In sandstone acidizing, emulsified acid dissolves the minerals and create acid transport pathway with close connectivity between pore spaces, causing the formation of large and conductive channels within the rock. Hence, these positive results clearly reflect on the feasibility of emulsified acid application in sandstone matrix acidizing and the effectiveness of waste oil as a replacement fluid for diesel.

Keywords: Emulsified acid; sandstone matrix acidizing; thermal stability; porosity; permeability.

1. Introduction

Formation damage due to the drilling, workover and completion processes causes detrimental effects to the well productivity. The effective removal of the near wellbore impairment can increase the



permeability to many folds which ultimately improves the hydrocarbon production from the reservoir. As discussed by Al-Harbi et al. (2013), matrix acidizing renowned as one of the most commonly used technique to increase the well productivity and injectivity by removing the detrimental formation damage materials [1]. Well stimulation treatments for these highly heterogenous formations have traditionally relied upon the use of plain mineral acid (HCl). However, fast rock-acid reaction rates and corrosion issues pose a significant challenge to the longevity and practical application of such treatments [2].

Alternative approaches have been proposed to slow down this process, among the most popular being emulsified acids. Emulsified acid is typically made up of acid and diesel in which diesel acts as a diffusion barrier between acid and rock [3]. It delays the contact of acid with the formation and has a higher efficiency in preventing rapid spending and reaction of acids [4]. In other words, diesel, as the continuous phase is in direct contact with the well tabular instead of the dispersed phase, acid. In this case, the release mechanism of live acid is governed by droplet contact with the formation surface in conjunction with suitable downhole conditions such as temperature, pressure and pH. Upon release, the acid-in-diesel emulsion proceeds to rapidly react with the carbonate formation thereby unveiling a conductive wormhole network, minimizing well tabular corrosion and significantly increasing the production of reservoir fluid.

By having a high viscosity up to 50 cP, the emulsified acid can significantly improve the zonal coverage, sweep efficiency, acid penetration in a heterogeneous reservoir [5]. This property of emulsified acid allows it to reduce the leak-off rate of acid and thus further improve the deeper penetration of the acid [6]. In plain acid case, the injected acid is more favourable to the formation with high permeability and has the tendency to flow through it [7]. Therefore, the low permeability zone is untreated and lead to failure of acid treatment due to incomplete coverage of acid in the reservoir.

Emulsified acids have primarily been used on carbonate formations and it is proven to be effective in enhancing well performance and real field applications [8, 9]. By combining theoretical and experimental studies, the effects of emulsified acid on sandstone reservoir need to be investigated by utilizing the combinations of emulsified acid applied in carbonate reservoir as a reference and apply on sandstone formation by adding hydrofluoric acid. However, emulsified acid is more extensively applied on carbonate reservoirs. Since approximately 60 % of all petroleum reservoirs are made up of sandstone formation, this project aims to fill the research gap to unlock the potential of emulsified acid application on hydrocarbon recovery of siliciclastic reservoir [10].

Apart from that, acid stimulation jobs typically consume high operational budget in which the average treatment cost is approximately \$ 1,000,000 [11]. Several studies showed the success of replacing diesel with crude oil and xylene in emulsified acid system [12]. Hence, this project will explore the unwanted waste oil as the substitute for diesel to promote cost effective and resource conservation practice. The effect of acid in waste oil emulsion on the properties of sandstone formation has not been reported in the literature. To the best of my knowledge, the researchers only tested the stability of the waste oil emulsion and the positive results show its potential to be applied as an acidizing fluid in the real oil and gas field. By addressing the performance of waste oil emulsion on sandstone properties in term of porosity and permeability, it will comprehensively apprehend more value for the petroleum industry.

Therefore, the objectives of this study are to, (1) investigate the thermal stability and characterize the droplet size of emulsified acid in oil; (2) study the effect of different formulation of emulsified acids on the porosity and permeability of Berea sandstone core; (3) determine the effectiveness of acid-in-waste oil emulsion for acidizing performance enhancement on the properties of porous sandstone.

2. Materials and Method

2.1. Materials

The chemicals used in this project are listed in **Table 1**.

Table 1. List of chemicals.

Name of Chemical	Purity	Source
Hydrofluoric acid (HF)	40 %	Merck
Hydrochloric acid (HCl)	37 %	Merck
Phosphoric acid (H ₃ PO ₄)	85 %	Merck
Formic acid (HCOOH)	98 %	Merck
Acetic acid (CH ₃ COOH)	100 %	Merck
Hexadecyltrimethylammonium bromide (CTAB)	99 %	Sigma Aldrich
Diesel	N/A	Petronas
Waste oil	N/A	Miri Crude Oil Terminal

2.2. Determination of Critical Micelle Concentration (CMC) of Cationic Surfactant

8 sets of 20 mL CTAB solution with different concentrations ranging from 1 wt% to 8 wt% were prepared and the conductivity measurements were carried out using the conductivity meter (Mettler Toledo SevenMulti™) under ambient temperature. The CMC was indicated by the inflection point of the slope of the lines in the conductivity versus total surfactant concentration plot.

2.3. Preparation of Emulsified Acid Samples

The oil phase and CTAB were then mixed at 4000 rpm using homogenizer (Fisher Scientific Silverson). The respective volume of acid, oil and distilled water were then calculated accordingly. The acid solution was slowly added into the mixture with uniform drop-wise nature while blending for 90 min. 10 different emulsified acid samples were prepared using the same approach and the combination of the emulsified acids were tabulated in **Table 3**.

2.4. Thermal Stability Test

The emulsified acid samples were poured into an acid-resistant measuring cylinder and placed in an oil bath which was heated by using a hot plate at 275 °F for 6 h. The stability condition of each sample was constantly monitored by observing the phase separation at every 30 min interval. Conductivity and pH test were also conducted to further characterize the stability of emulsified acids.

2.5. Optical Microscopy Studies of Emulsion Morphologies

After the post emulsion mixing stage, a small volume of synthesized emulsified acid droplet was loaded on the glass slide and its microscopic behaviour was observed thoroughly by using polarizing microscope (Nikon LV100N) with camera (Nikon DS-Fi2) through magnification as 10 and 20 times. The liquid droplet size measurement was read at five distinct locations across the magnified image to obtain the average droplet diameter with the aid of NIS software.

2.6. Acidizing Treatment on Berea Sandstone

10 Berea sandstone core samples were cut into the length of 1.5 inch and labelled accordingly. Initially, core samples 1-10 were then immersed into beakers containing pre-flush fluid of 5 % CH₃COOH: 10 % HCl and heated at 275 °F. After 3 days, the core samples were cleaned and dried thoroughly. Followed by main flush stage, the core samples 1-10 were saturated with 10 respective emulsified acid combinations under the temperature of 275 °F for 3 days in the fume hood.

2.7. Porosity Determination

The porosity of the core samples at three distinct stages: initial condition, after pre-flush saturation and after emulsified acid saturation were determined by the saturation method with distilled water. The experiment was began by saturating the core samples in distilled water for 3 days in a vacuum oven under room temperature with a suction pressure of 0.07 MPa. After 3 days, the core samples were taken out from the distilled water for wet weight measurements. Next, the drying of core samples were carried out in the furnace for 24 h at 185 °F. The dry weight of core samples was then measured and the porosity of the core samples was calculated using the Equation (1).

$$\emptyset = \frac{(W_{\text{wet}} - W_{\text{dry}}) / \rho_f}{V_b} \quad (1)$$

where \emptyset = porosity (dimensionless); W_{wet} = wet weight of the core after saturation with distilled water (g); W_{dry} = dry weight of the core sample (g); ρ_f = fluid density of distilled water which assume as $\rho_f = 1 \text{ g/cm}^3$ and V_b = bulk volume of the sample (cm^3).

2.8. Evaluation of Permeability

Similar to porosity test, permeability of the core samples at initial condition, after pre-flush saturation and after emulsified acid saturation were measured by using Gas Permeameter (Model TKA-209). By substituting the experimental data obtained into Equation (2) and (3), the permeability of the core sample was determined.

$$K_a = \frac{2\mu Q_b P_b L}{A(P_1^2 - P_2^2)} \times 1000 \quad (2)$$

$$\mu = \frac{13.85 \times T^{1.5}}{T + 102} \times 0.0001 \quad (3)$$

where K_a = gas permeability (mD); μ = viscosity of the nitrogen (cP); Q_b = flow rate (cc/sec); P_b = atmospheric pressure (Assume as $P_b = 1 \text{ atm}$); L = length of the core sample (cm); A = area of the core sample (cm^2); P_1 = inlet pressure, the sum of the upstream pressure and the atmospheric pressure (atm); P_2 = outlet pressure, the sum of downstream pressure and atmospheric pressure (atm) and T = temperature where this experiment takes place (Assume as room temperature = $25 \text{ }^\circ\text{C} + 273 \text{ K} = 298 \text{ K}$).

3. Results and Discussion

3.1. Critical Micelle Concentration of Cationic Surfactant

When the CMC of solution is determined by conductivity measurement, the conductivity of the solution increased linearly with total surfactant concentration as shown in **Figure 1**. CMC is the lowest concentration at which micelles first appear and is found out to be 4 wt% in this case. Hence, for this experiment, the surfactant is injected at 5 wt% which is slightly more than the critical micelle concentration to ensure sufficient interfacial tension reduction for stable emulsion formulation. According to Apaydin & Kovscek (2001), the surfactant concentration used in experiment cannot be too high [13]. Otherwise, it may lead to building of pressure gradient due to end effect. This gradient is against the direction of flow and thus pose difficulties for the hydrocarbon to flow from upstream to downstream.

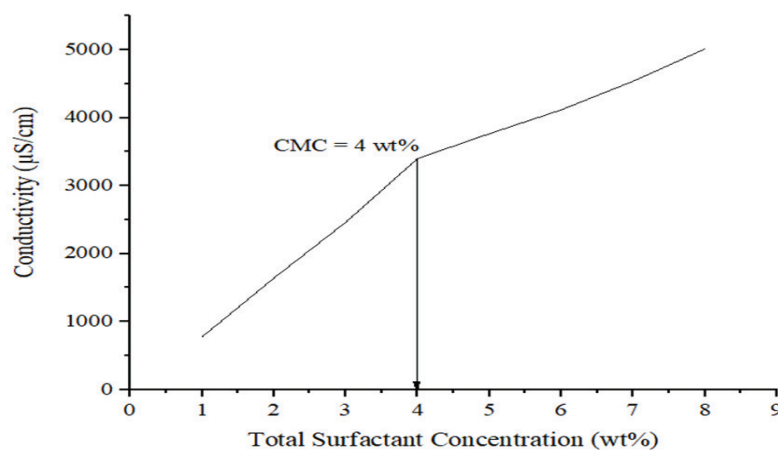


Figure 1. Graph of conductivity against total surfactant concentration.

3.2. Thermal Stability Investigation of Emulsified Acid

Stability of emulsified acid is of the primary concern because when the emulsified acid is able to stay in stable emulsion form for an appropriate period of time, it will allow a deeper penetration into the formation without separating the two immiscible fluids (acid and oil) before reaching to the targeted zone. After heating up at the elevated temperature of 275 °F for 6 h, all the emulsified acid samples prepared are able to remain in stable form as there is no oil layer formed on the top of the emulsion which indicates that diesel/ waste oil does not escape from the emulsion as displayed in **Figure 2**. Hence, the emulsified acids are considered stable as it does not experience a complete phase separation between the oil phase and the aqueous phase.

One possible explanation for the observation of two stratification layers of emulsion is that the upper layer is acid-in-oil emulsion while the lower layer is oil-in-acid emulsion. This statement is verified by the pH and conductivity results. The upper layer is less acidic than the lower layer due to its higher pH value, indicating acid is dispersed in oil. As a continuous phase, both diesel and waste oil have a relatively lower density than acid and thus explaining the presence of emulsified acid in the upper layer. In addition, Cairns et.al (2016) stated that the electrical conductivity of a good and stable emulsified acid is close to zero or zero [14]. The upper layer emulsion showed a conductivity close to zero whereas the lower layer depicted higher conductivity values ranging between 40 $\mu\text{s}/\text{cm}$ to 95 $\mu\text{s}/\text{cm}$. In short, this result has validated the successful formation of acid-in-oil emulsion.

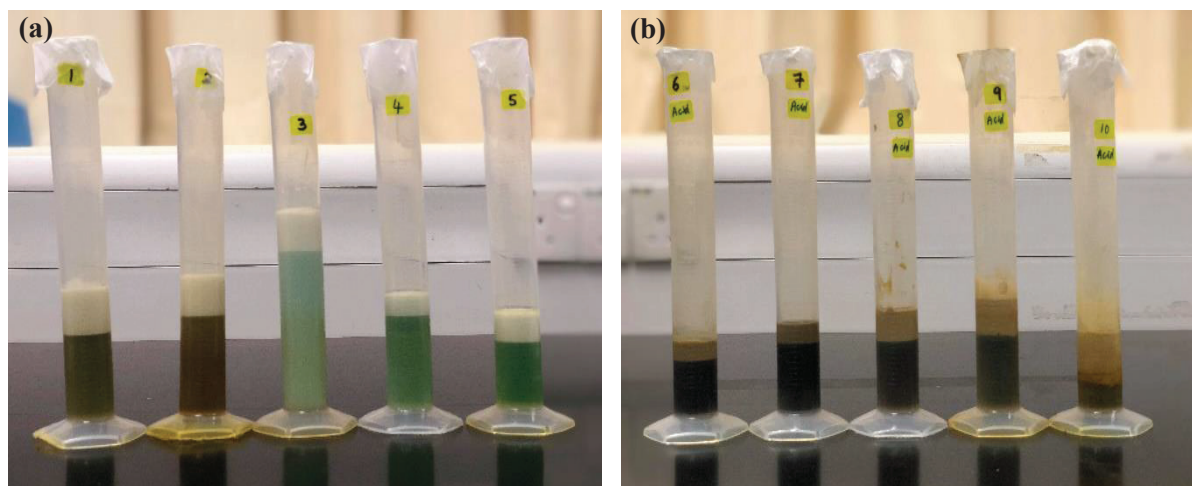
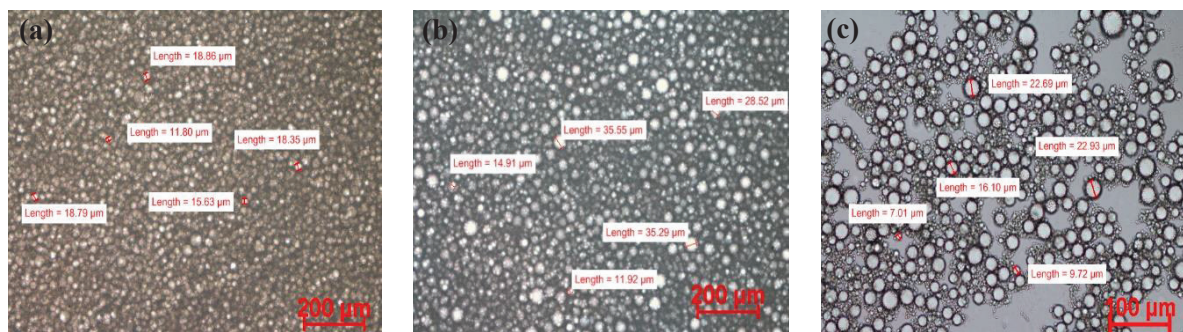


Figure 2. Emulsified acid (a) Sample 1-5 and (b) Sample 6-10 after 6 h of thermal stability test.

3.3. Droplet Size Analysis

Emulsified acid with good emulsion performance should have a few features such as uniform droplet size, tight placement of droplets and small droplet diameter [15]. According to the microscopic photographs attached in **Figure 3**, Sample 1, 2, 6 and 7 that consist of HF: HCl showed the best microscopic result as the emulsion displayed uniform, tight and narrow droplet size. The remaining samples showed less superior results in term of droplet size diameter and distribution.



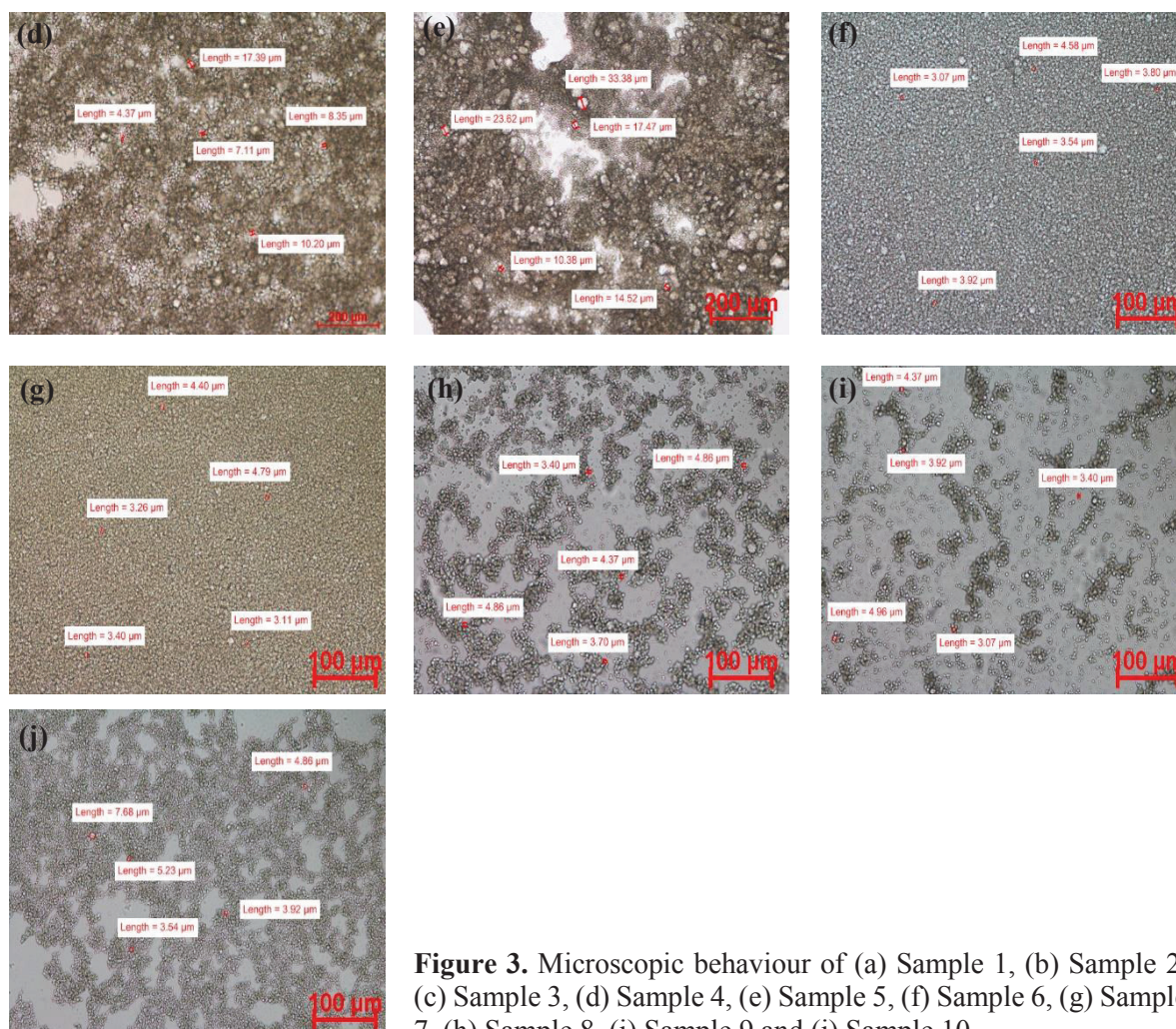


Figure 3. Microscopic behaviour of (a) Sample 1, (b) Sample 2, (c) Sample 3, (d) Sample 4, (e) Sample 5, (f) Sample 6, (g) Sample 7, (h) Sample 8, (i) Sample 9 and (j) Sample 10.

However, the droplet size of all samples was in accordance with the emulsion droplet size obtained from other researchers. The droplet size of diesel-based and waste oil-based emulsified acid is ranging from 9.48 μm – 25.24 μm and 3.78 μm - 5.05 μm respectively. **Table 2** summarizes the average droplet size of emulsified acids. It is evident that the size of the particles for all samples was compatible with those produced by the previous researchers. To add on, the droplet size of acid-in-waste oil emulsion was relatively smaller than that of emulsified acid in diesel. This has also proven the practicability of waste oil as an effective alternative for stable emulsified acid formulation.

Table 2. Average droplet size of emulsified acid systems.

Author and year	Type of emulsion	Average droplet size (μm)
Al-Anazi et al. (1998) [16]	Diesel	77
Al-Mutairi et al. (2009) [17]	Diesel	6 - 12.4
Sayed et al. (2013) [18]	Diesel	2.8 - 8.1
Sidaoui & Sultan (2016) [19]	Waste oil	1.47 - 3.09

Furthermore, emulsified acid is classified into micro-emulsion and macro-emulsion [20, 21]. Micro-emulsions are composed of droplet size of 0.01-0.1 μm whereas any size larger than that range will be categorized under macro-emulsion [22]. Hence, the emulsified acid of this project is considered as macro-emulsion and the stability of emulsified acid is proven once again by the droplet size characterization results.

3.4. Porosity Enhancement of Berea Sandstone

As observed from **Table 3**, the porosity of core samples increased after pre-flush and further enhanced after saturating core samples with 10 different emulsified acid combinations. The porosity enhancement is up to 5.19 % after pre-flush acid treatment and this result is in accordance with the experimental findings obtained by Shafiq et al. (2017) in which the same pre-flush fluid (5 % CH₃COOH: 10 % HCl) achieved a porosity increment of 6.24 % by removing the calcium, potassium and sodium ions [23].

On the other hand, HF: HCl provided the most significant porosity improvement, followed by HF: HCOOH, HF: CH₃COOH and HF: H₃PO₄. In the case of diesel-based and waste oil-based emulsified acid, conventional mud acid (HF: HCl) demonstrated best performance in improving the porosity of core samples from 15 % to 22 %, experiencing total porosity enhancement of approximately 7 % after pre-flush and main flush acid treatment. As HCl can dissolve siliceous minerals and clay which made up most of the minerals in Berea sandstone, more new pores were created within the core and thus leading to the significant increment in porosity. By comparing with the weak organic-HF acids, HCl is a stronger acid to act as the buffer acid to provide a low pH region that could keep the reaction products soluble in the spent acid [24].

Contradictory, HF: H₃PO₄ showed the least effectiveness in porosity enhancement on Berea sandstone. Despite of the fact that sample 3 and 8 experienced a total porosity increment, it is obvious that their final porosity values decreased up to 1.42 % when compared to post pre-flush porosity. This could be due to the formation of unwanted precipitates under high temperature conditions after emulsified acid treatment, causing undesired fine migration issues. As a result, precipitates re-damage the core samples by creating isolated pores due to bridging effect of mobile particles and eventually lead to effective porosity reduction.

Generally, both emulsified acids with 0.7 acid: 0.3 oil had similar yet high capability in mineral dissolution regardless of the types of oil. The right combination of acids plays a more vital role in affecting porosity as it is necessary to remove potential ions that could form insoluble precipitation that choke pore throats as well as initiate more pore spaces caused by rapid acid-rock reaction.

Table 3. Porosity results.

Sample	Acid Combinations	Initial ϕ (%)	Post Pre-Flush ϕ (%)	Post Main Flush ϕ (%)	Change in ϕ After Pre-Flush (%)	Change in ϕ After Main Flush (%)
0.7 acid: 0.3 diesel						
1	3% HF: 9% HCl	14.96	18.79	22.39	3.83	7.43
2	3% HF: 12% HCl	15.58	19.67	22.21	4.09	6.63
3	3% HF: 9% H ₃ PO ₄	15.40	19.88	19.38	4.48	3.98
4	3% HF: 12% HCOOH	15.80	19.55	21.55	3.75	5.75
5	3% HF: 12% CH ₃ COOH	15.50	19.83	19.88	4.33	4.38
0.7 acid: 0.3 waste oil						
6	3% HF: 9% HCl	15.02	19.63	22.29	4.61	7.27
7	3% HF: 12% HCl	14.97	20.16	21.98	5.19	7.01
8	3% HF: 9% H ₃ PO ₄	14.68	19.56	18.14	4.88	3.46
9	3% HF: 12% HCOOH	14.65	18.99	19.81	4.34	5.16
10	3% HF: 12% CH ₃ COOH	14.99	19.79	19.84	4.80	4.85

3.5. Permeability Alteration in Berea Sandstone

Alluding to **Table 4**, it is clearly shown that the permeability of all core samples increased by 17.51 % - 85.84 % after being treated by pre-flush fluid. Hence, 5 % HCl: 10 % CH₃COOH is proven to be an effective pre-flush fluid for Berea sandstone as it can successfully remove fine particles in the core and minimize precipitation issues.

After emulsified acid treatment, most of the core samples experienced increment of permeability ranging from 80.71 % - 467.27 % with the exception of core samples that undergone HF: H₃PO₄ saturation. When phosphoric acid is used, the permeability of sample 3 and 8 were significantly reduced

by 29.34 % and 19.59 % respectively. Shafiq & Mahmud (2016) used the same acid formulation to treat Berea sandstone under ambient temperature and its permeability value was improved by 135 % [25]. Hence, it can be concluded that HF: H₃PO₄ is not practicable for the real reservoir conditions as it will have detrimental impact on rock permeability at elevated temperature. Similar to porosity findings, HF: HCl had the most substantial permeability enhancement, followed by HF: HCOOH and HF: CH₃COOH. The conventional mud acid which is widely used in the industry increased the core samples' permeability by five folds. Application of organic acids such as formic and acetic acid also has successfully raised the permeability by about 1 to 2 times as they are effective in removing aluminosilicates from sandstone.

Permeability enhancement is critical in improving the well productivity and the working mechanism of emulsified acid will lead to significant increment in core permeability. The dispersed acid encapsulated in the oil phase will be slowly released as it goes deeper into the targeted zone, dissolving the minerals, forming long conductive wormholes that induce a pathway with high permeability with minimal corrosion issues. On the other hand, the similar amount of permeability incremental using acid-in-waste oil emulsion indicated the promising feasibility of waste oil in substituting diesel as the diffusion barrier between acid and rock.

Table 4. Permeability results.

Sample	Acid Combinations	Initial k (mD)	Post Pre-Flush k (mD)	Post Main Flush k (mD)	Change in k After Pre-Flush (mD)	Change in k After Main Flush (mD)
0.7 acid: 0.3 diesel						
1	3% HF: 9% HCl	123.42	206.29	628.89	82.87	505.47
2	3% HF: 12% HCl	113.32	193.66	642.83	80.34	529.51
3	3% HF: 9% H ₃ PO ₄	109.00	202.57	77.02	93.57	-31.98
4	3% HF: 12% HCOOH	115.88	142.86	369.27	26.98	253.39
5	3% HF: 12% CH ₃ COOH	137.88	173.19	284.73	35.31	146.85
0.7 acid: 0.3 waste oil						
6	3% HF: 9% HCl	131.29	192.00	714.83	60.70	583.54
7	3% HF: 12% HCl	143.30	222.14	731.33	78.85	588.03
8	3% HF: 9% H ₃ PO ₄	91.57	108.98	73.63	17.41	-17.94
9	3% HF: 12% HCOOH	79.63	94.73	250.10	15.10	170.47
10	3% HF: 12% CH ₃ COOH	97.07	114.07	175.42	17.00	78.34

4. Conclusion

The conventional mud acid (HF:HCl) is identified to be the most effective acid combination that provides the best improvement in sandstone properties which includes 7 % porosity increment and permeability enhancement by 5 folds. Emulsified acid that contains HF: HCOOH is ranked second best, followed by HF: CH₃COOH and HF: H₃PO₄. In addition, HF: H₃PO₄ is ineffective in improving sandstone permeability, albeit it caused slight increment in porosity. The emulsified acids are proven to remain stable up to 6 h at 275 °F, supported by the tight and small droplet size of emulsion. Another important highlight is that waste oil is proven to be an effective substitute to replace diesel in formulating greener and cheaper emulsified acid.

5. Recommendations

In order to examine the effect of emulsified acid on sandstone formation, wormhole propagation and mineral dissolution mechanism should be studied by performing Computer Tomography (CT) Scan and Inductively Coupled Plasma (ICP). Nuclear Magnetic Resonance (NMR) should be conducted to determine the porosity, permeability and topology of acid-reacted core samples as well. Apart from that, another possible improvement is to carry out core flooding to mimic the high pressure high temperature (HPHT) condition in the reservoir so that the results of emulsified acid on sandstone formation will be

more significant and realistic to the oil and gas industry. Not to mention, the acid injection rate can be manipulated and the hydrocarbon recovery factor can be identified directly.

6. Acknowledgement

The authors acknowledge Curtin University Malaysia for providing financial support as well as technical resources for this research paper.

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