

ACID TREATMENT ON CARBONATE ROCK: AN EFFECT OF HCL CONCENTRATION ON ROCK PROPERTIES AND FLUID FLOWRATES

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Abstract

A series of analyses techniques were performed to study the influence of different acid concentrations on the rock properties during the acidizing process. Based on the Thin section and Routine Core Analysis (RCA), the calcite content have effect on the reservoir quality from the aspect of rock-fluid properties. In this paper, the physical and mineralogical responses to rock acidizing of carbonate rock are evaluated. This study found that calcite content decreases approximately 25% from the total calcite content of rock samples after the addition of HCl. Scanning Electron Microscope (SEM) Analysis show that samples treated using HCl 15% provide a wider pore size distribution, resulting in the increasing permeability fluid flow rate.

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1. Introduction

Oil and gas production still facing numerous challenges due to the different type of reservoir found around the globe. Common challenge that still attracted is the carbonate reservoirs, which have low capacity flow rate for hydrocarbon to pass through rock reservoir. They are commonly termed as low-permeability reservoirs with <10 millidarcy in permeability [1,2]. These made optimal oil production is hard to achieved, because of the limited flow hydrocarbon fluid that can be passed through [3]. Therefore, stimulation method become one of the best solutions to tackle the problem.

Stimulation method is one of the ways to increase the productivity of a reservoir by tuning the rock properties, such as porosity and permeability. Porosity is an important rocks properties which can be used to estimate the amount of hydrocarbons that can be stored inside the reservoir [4,5]. Another important rock properties is permeability, which is the ability of the rock to pass fluid through its pores [6]. Porosity and permeability can be used to determine the amount of fluid that can be produced to the surface of a reservoir, or to estimate Original-Gas-In-Place (OGIP) [7,8]. One of the reliable stimulation methods to stimulate the carbonate reservoir is acid treatment method [5].

Acid treatment is one of the most widely used acidification techniques to stimulate carbonate reservoir. The conventional acid treatment model is widely used to develop fracture in low – permeability - gas carbonate reservoir [9]. The acid stimulation methods are advantageous in carbonate formation, because of the presence of acid soluble minerals in form of calcite [3]. In this method, acid fluid were contacted into the rock formation at a certain temperature and pressure conditions [10]. However, acid treatment still has many challenges to be optimally done. Acid treatment method has been extensively researched in the laboratory. Because acid treatment experimental studies are commonly using outcrop samples deposited on the surface; therefore, the results obtained are not fully representative of the actual situation beneath the surface [11]. In those cases, it required further study to observe influencing factors of acid stimulation method.

Although an acid treatment model can provide some initial stimulation, it is still relatively difficult to achieve stable production rate result [12]. Variable condition such as acid volume and concentration, during the acid treatment method in carbonate reservoir, usually more difficult to control than in other reservoir type of

reservoir, like dolomite [11]. Ameri et al. reported that the higher HCl concentration which contact to rock will produce more significant effect on fracture damage [2]. However, Li et al shows the greater HCl concentration will provide more challenge in terms to control in micro scale damage, leading to weaken the rock structure and made it brittle [3]. They also suggested the acid system that should be chosen is the one that provide a wider range of fractures or pores distribution, not because based on its magnitude of fracture damage. Based on those facts, we used variations in HCl concentrations of 10%, and 15 % to observe the resulting impact on rock properties (e.g., porosity and permeability). In this study, we investigate the relationship of rock properties to the fluid flowrate through several analysis such as RCA (Routine Core Analysis), Thin Section 2D Analysis, and SEM Images. The permeability was calculated based on theoretical of Kozeny – Carman [13,14]. While Darcy equation was used to calculate the fluid flowrate of gas that passed through the rock [15,16]. Moreover, fracture is related to the rock-fluid properties that could influence on reservoir productivity because it could increase permeability of a reservoir [17]. Therefore, more research is needed to investigate the effect of acid concentration on rock properties and the resulting fluid flowrate, especially on changes in rock properties.

Samples were obtained from outcrop samples at Kutai Basin, East Kalimantan. The Kutai Basin carbonate platform (Oligocene Berai Limestone) covers a subsurface area measuring approximately 11 by 16 km in the westernmost Kutai Basin, Central Kalimantan. The Kutai Basin platform is approximately 1,000 m thick and comprises of three aggrading seismic sequences identified by the downlap of basal strata at the platform margin and downlap of transgressive strata within the platform. The platform rim is characterized by interbedded bioclastic wackestones, packstones, grainstones and boundstones, with grainstones increasing toward the platform margin [18].

In this work, we correlated the effect of changes in crystallinity (e.g., calcite content) and diameter of the pore size distribution due to differences in HCl concentration. With the rock-fluid properties produced, the determination of a more suitable acid system can be considered to be applied to the reservoir by considering the crystallinity of calcite content so that the results obtained can be used to improve reservoir quality from the aspect of rock-fluid properties.

2. Experimental Method

A. Samples Preparation

We collected several carbonate samples (KR - 1 to - 14), which are crop out at the Kutai Basin, East Kalimantan. We prepared the samples for several analysis, 1) petrographic (thin section analysis), 2) Routine Core Analysis (RCA), 3) SEM Images Analysis, and 4) Acid Treatment. Prior to the analysis, acid treated samples undergo washing procedure using DI water to neutralize the sample.

First, samples were prepared for petrographic analysis and were sectioned into blue dyed thin section of rock at Obsidian Geo - Laboratory. The dimension of the thin sections is approximately 30 micrometers (μm) in thickness for 2D porosity analysis using a free software of ImageJ [19].

Second, sample was prepared as cylindrical cores for porosity and permeability measurement using RCA method at Geoservice, Ltd. The dimension of core samples is 2 x 2 cm in length and diameter. Then, sample KR-1 to 14 was crushed to smaller size and weighed about 2 grams before acid treatment. The same dimension was also used in SEM analysis.

In this study, thin sections were scanned using EPSON LV - 600 Film Scanner with polarized film. Scanned thin sections were used to observe the component of carbonate rocks (e.g., micrite, fossil) and were analyzed using an ImageJ software under 8-bit thin section images. We use JPOR feature on imageJ software to calculate the actual porosity of fresh and treated samples [20].

B. Routine Core Analysis

Before treatment, porosity of the sample was measured by Porosimeter - Permeameter instrument (AP – 608 Automated, Poretest System Inc.) using helium inert gas as fluid. Then, permeability before treatment was also measured using the same instrument but with nitrogen inert gas fluid. This analysis method is generally referred to RCA with the same principles as previous report [21].

C. SEM Images Analysis

We conducted SEM Images analysis to observe fracture in micrometer scale using Phenom ProX Dekstop SEM Image. The operation condition was 15-kV at the magnification at 500x. This method was similar to what has previously reported in the microstructure of shale [17].

D. Acid Treatment

Samples is heated in an oven with temperature of 120 °C for 1 hour, to model the actual reservoir temperature [18]. The sample was immediately reacted with HCl solution at various concentration (10% and 15%) for the acid treatment process. In typical preparation 81.08 and 54.05 ml HCl (37%, SmartLab) was dissolved in 200 mL distillate water to make 15% and 10% HCl solution respectively. The rock sample then react with HCl solution at volume 25 ml for 30 seconds at ambient pressure of 1 atm.

E. Theoretical Approach in calculating Permeability and Fluid Flowrate

After acid treatment, we use theoretical approach using Darcy and Kozeny-Carman Equation to estimate permeability and fluid flowrate changes. Due to the limitation of the RCA and the availability of samples, permeability (K) of each samples was calculated using equation (1) and (2) [13,14].

$$K = c\phi^3/S^2 \tag{1}$$

$$c = \left(4 \cos\left(\frac{1}{3} \arccos\left(\phi \frac{8^2}{\pi^3} - 1\right) + \frac{4}{3}\pi\right) + 4\right)^{-1} \tag{2}$$

Kozeny-Carman factor (c) was obtained from equation (2), while the surface area (S) was obtained from previous research about carbonate reservoir [14].

$$Q = \frac{KS}{\mu} \frac{(p_1 - p_2)}{L} \tag{3}$$

After permeability data is obtained, the fluid flowrate was calculated using the Darcy's Law as shown in equation (3) [15,16]. Where μ is the fluid viscosity and pressure gradient, $(p_1 - p_2)/L$, was obtained from the previous research at the Kutai Basin [22]. These reservoir scenarios and variable approach is used to calculate the permeability and rate of fluid produced after the acid treatment by using a reference paper that is close to the actual field conditions.

3. Results and Discussion

A. Sample Rock Properties Profile

Fig. 1. shows that sample has various range of permeability before acid treatment. We could observe that samples are divided into 3 regions of range, depict by region (a), region (b) and region (c). Region (a) indicates that there are some samples that has permeability far below than 1 millidarcy and the porosity below than 5 %, these samples are KR – 9, KR – 11 and KR – 14, so the region could classified as low – permeability reservoirs [2]. Region (b) shows where most of the samples are located, these region ranging 0.5 – 2 millidarcy in permeability and porosity 10% - 15%, this indicates that some of the samples has natural fracture that allows pore opening to be formed because geological circumstances [23]. Region (c) shows there is a sample (KR – 5) that overlapped from the rest region with permeability 4.7 % and porosity 23.5 %, that indicates in same type reservoir we could have very broad range of permeability and porosity, although the rock components is relatively the same, the amount of the contents are very depends on the location of sample obtained. But in common, we could classified that these samples can be categorized as low – permeability reservoir because the overall permeability of each sample doesn't exceed 10 millidarcy [22].

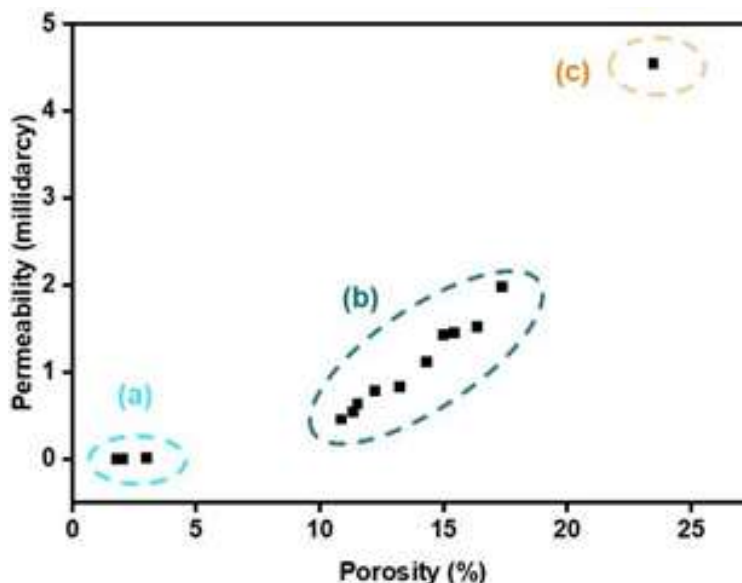


Figure 1. Porosity and permeability profile of the samples before acidizing treatment.

B. Thin section analysis

Based on Dunham's classification, three textural features fundamental in classifying carbonate rocks hold their depositional texture. (1) Presence of carbonate mud, which distinguishes muddy carbonate from grainstone; (2) Abundance of grains to subdivide muddy carbonate into *mudstone*, *wackestone*, and *packstone*; and (3) presence of signs of binding during deposition, which characterized boundstone. The distinction between packstone and wackestone can be seen in grain support and mud support. Packstone contains plenty of grain support, while wackestone contains mainly mud support. The packstone's depositional texture is related to crystalline carbonate content [24].

Fig. 2. shows the thin section of the KR – 1 and KR – 9 before treatment. The white area of KR-1 shows that the sample is dominated by fossil (1) and coral (5), which have turned into calcite after undergoing the rock recrystallization process. In contrast, the brown one (3) is a matrix structure commonly called micrite [24,25]. Thus, KR-1 is classified as packstone because it is dominated by calcite. It also can be observed from Fig. 2b that KR – 9 is dominated by brownish color, as indicated by number (2), which is called the micrite matrix. As suggested by number (4), KR - 9 still contains a small amount of calcite, then KR – 9 is categorized as wackestone by its dominated micrite matrix [26].

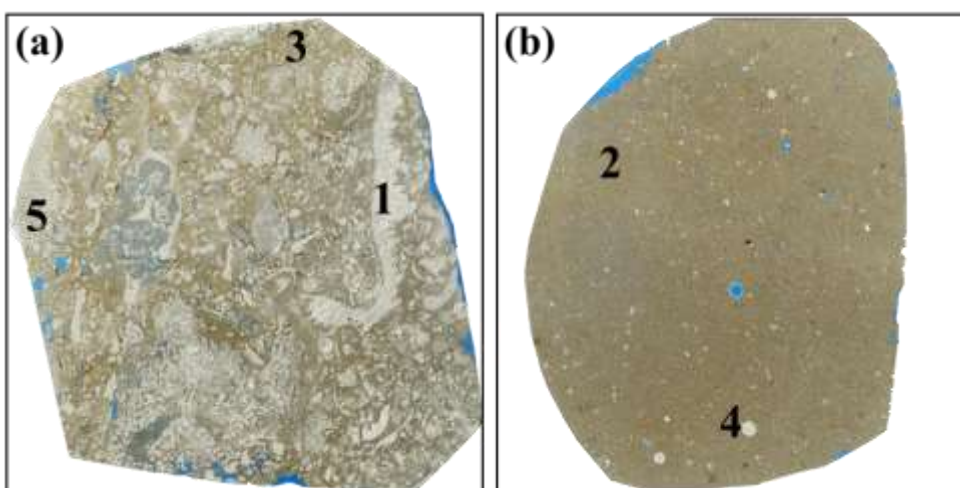


Figure 2. Thin section image of (a) KR - 1 before treatment; (b) Thin section image of KR – 9 before treatment.

Fig. 3. shows that the thin section of KR-1 component components is calculated using ImageJ software, by utilizing the difference in color thresholds displayed. The difference in color thresholds was clearly seen where the red one is part of KR - 1 which is dominated by the micrite matrix. While those that are not red are fossils

and corals that have become calcite [26]. Thus, based on the software, micrite matrix content in KR – 1 is 22.82%. The same procedure was applied to sample KR-2 to KR-14 and then micrite matrix content along with calcite content were summarized in Table 1. Based on Dunham classification KR - 1 and 2 are packstone with high calcite content (76.83 and 75.39 %, respectively). Meanwhile, KR-3 to 14 classified to wackestone with lower calcite content.

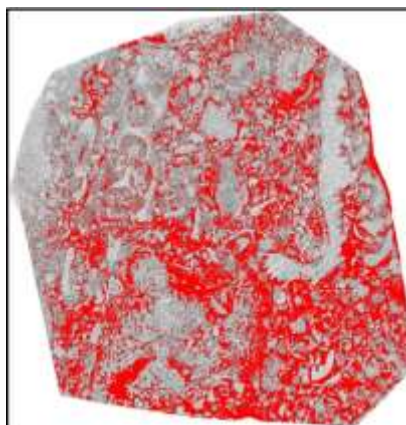


Figure 3. KR - 1 components analyzed by using ImageJ software.

Table 1. Summary of proportion of rock components before acidizing treatment

Sample Name	Component		Qualitative Description
	Matrix	Calcite	
KRC-1	22.82%	76.83%	Packstone
KRC-2	24.22%	75.39%	Packstone
KRC-3	37.68%	61.73%	Wackestone
KRC-4	34.47%	64.99%	Wackestone
KRC-5	33.31%	66.17%	Wackestone
KRC-6	32.27%	67.22%	Wackestone
KRC-7	31.35%	68.16%	Wackestone
KRC-8	38.29%	61.11%	Wackestone
KRC-9	37.84%	61.56%	Wackestone
KRC-10	34.33%	65.13%	Wackestone
KRC-11	38.08%	61.32%	Wackestone
KRC-12	32.82%	66.66%	Wackestone
KRC-13	39.68%	59.70%	Wackestone
KRC-14	37.94%	61.46%	Wackestone

Fig. 4. shows thin section of sample KR – 9 after 15% HCl treatment which has been given blue dye. As can be seen in Fig. 4a. the blue color fills the voids in the rock which show the porosity of the KR – 9. Meanwhile Fig. 4b. is a thin section that has been processed with ImageJ by using the difference in color threshold, the amount of porosity will be represented by the red area. By using this method, the calculated porosity in KR – 9 is 10%. Further analysis using same technique can be done to another sample and the result is presented in Table 2. All sample show increasing in porosity by HCl 10% treatment due to pore opening. This might be due to the decreasing calcite content as the result of carbonate reaction with HCl. Further increase in HCl concentration to 15% lead to the increase in porosity as corelated with calcite decreasing.

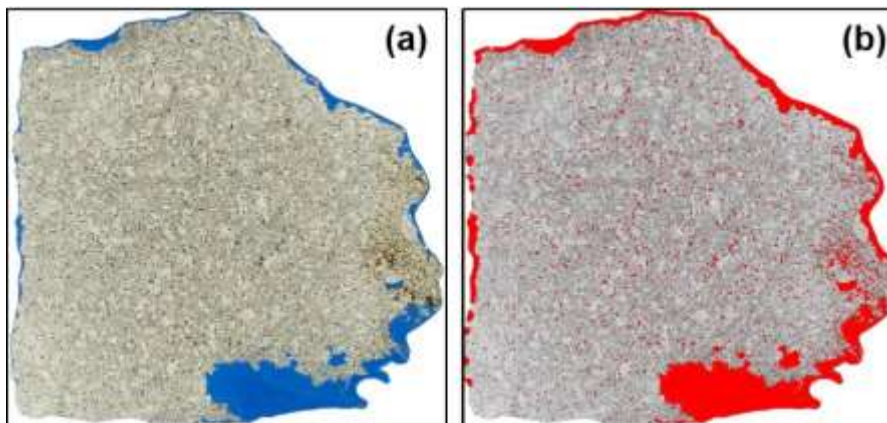


Figure 4. shows Porosity of KR - 9 after HCl 15 % treatment using ImageJ. (a) Thin section of KR – 9 after HCl 15 % treatment before color thresholding. (b) Thin section of KR – 9 after HCl 15 % treatment after color thresholding using ImageJ.

Table 2. Porosity and calcite contents after acidizing treatment

Sample Name	Porosity			Calcite		
	HCl 0 %	HCl 10 %	HCl 15 %	HCl 0 %	HCl 10 %	HCl 15 %
KRC-1	15.46%	24.52%	25.77 %	76.83%	51.07%	52.31%
KRC-2	10.86%	18.10%	37.34%	75.39%	57.29%	38.05%
KRC-3	15.02%	25.03%	43.83%	61.73%	36.69%	17.90%
KRC-4	17.36%	28.94%	44.08%	64.99%	36.05%	20.90%
KRC-5	23.49%	39.15%	40.56%	66.17%	27.01%	25.60%
KRC-6	14.33%	23.89%	38.67%	67.22%	43.33%	28.55%
KRC-7	13.24%	22.06%	27.54%	68.16%	46.09%	40.62%
KRC-8	12.23%	20.38%	30.63%	61.11%	40.73%	30.48%
KRC-9	3.00%	5.00%	10.00%	61.56%	56.56%	51.56%
KRC-10	11.54%	19.23%	33.25%	65.13%	45.90%	31.88%
KRC-11	1.79%	2.98%	4.92%	61.32%	58.34%	56.40%
KRC-12	10.41%	11.34%	18.90%	66.66%	56.25%	47.76%
KRC-13	16.37%	27.28%	51.60%	59.70%	32.42%	8.10%
KRC-14	2.07%	2.10%	2.54%	61.46%	58.49%	58.91%

C. Surface SEM – Analysis

Fig. 5a. shows KR-14 before HCl treatment which natural fractures has been observed (1). However open pores are still very small and far apart. After acidizing process Fig. 5b., we can see that the surface texture is damaged after adding HCl due to the dissolution of calcite. Moreover, after addition 10% HCl, the new cracks or pores began to form due to the dissolution of the calcite content by HCl. However, the distance between the pores was still relatively far apart as indicated by blue circle (2). At higher acid concentration Fig. 5c. and Fig. 5d., as indicated by number (3), the change in surface texture looks very significant compared to the sample that treated by 10% HCl. Moreover, the new pores are larger compared to 10% HCl treatment and have varying sizes with fairly close distances. From Fig. 5d. depicted by number (3) we could observe that elongated fracture was formed after acidizing treatment. This can happen because the dominance of soluble calcite is located between the elongated fossil grains, so that when the calcite is dissolved, elongated fractures are formed.

Also, we could observe that SEM observation indicate that 15% HCl gave a significant change to sample texture compared to 10% HCl. HCl 15% tends to form more pores, relatively larger in size and the distance between pores is closer than the samples treated with HCl 10% which form pores that are relatively smaller and spaced apart. This can be explained because the sample by treating with 15% HCl have higher dissolution of calcite compared to sample treated by 10% HCl. From the reaction stoichiometry, 15% HCl will have a higher dissolving power than 10% HCl in higher temperature than 100 °C and pressure 1 atm where more calcite with

CO₂ gas is formed [5]. The presence of calcite will cause the cementation effect which closing the pores that lead to poor productivity of the reservoir.

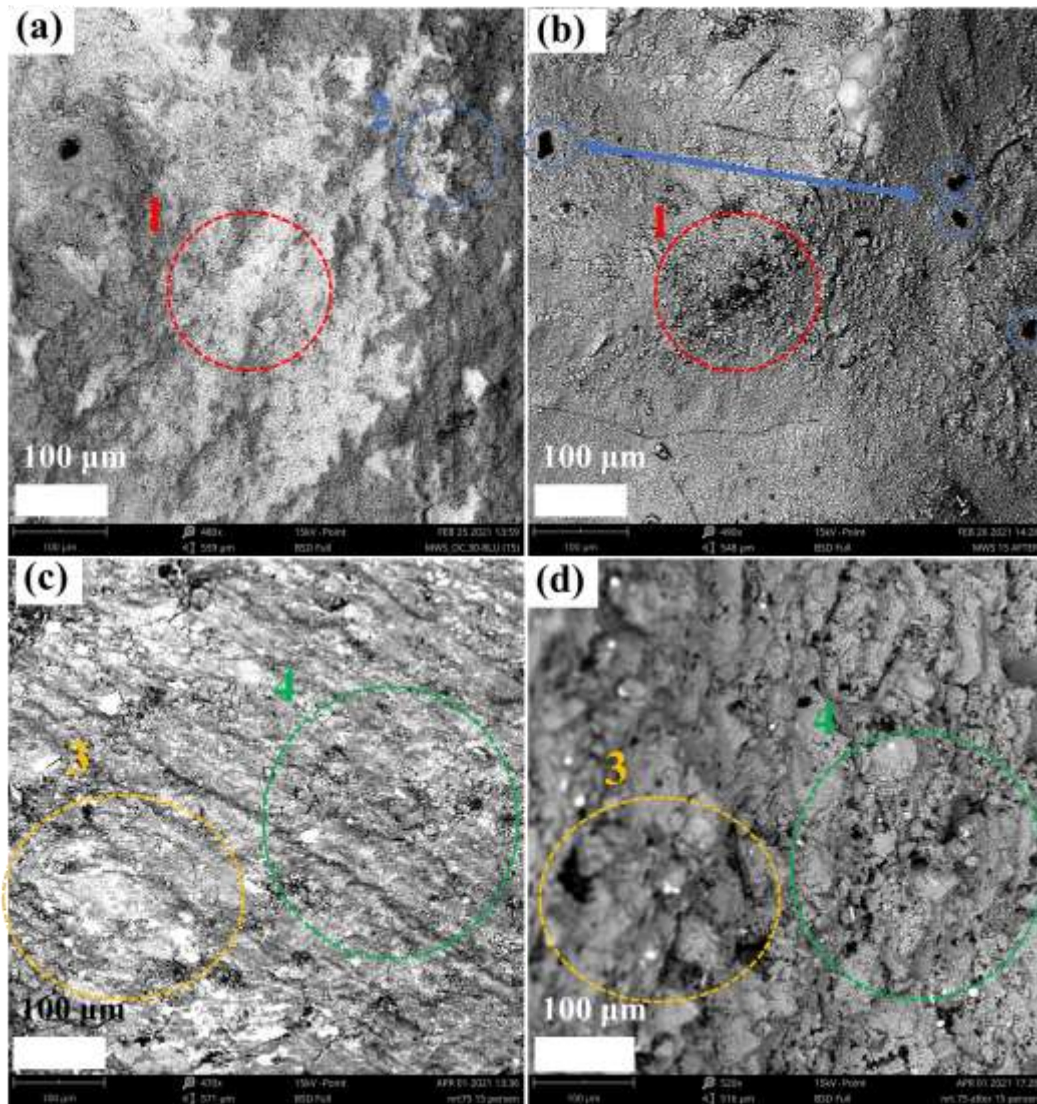


Figure 5. SEM Images of (a) KR-14 before acidizing, (b) KR – 14 after acidizing by HCl 10 %, (c) KR – 14 before acidizing, (d) KR – 14 after acidizing by HCl 15 %.

D. HCl Concentration effect on permeability

Fig. 6. shows the effect of HCl concentration on rock permeability. The rock sample before HCl treatment has high calcite content, which is above 50%. While the permeability is relatively low, which is around 0.1 - 5 millidarcy. The high calcite content in the rock will have a cementation effect, where the result of this cementation is the closing of the pores in the rock thereby reducing its permeability. The data in red color shows the rock samples that have been treated with 10% HCl. The calcite content begins to decrease because it is dissolved in HCl, thereby increasing the permeability due to the presence of new open pores as confirmed by the SEM analysis result. Meanwhile, the data in green color represent samples that have been treated with 15% HCl. The permeability has increased higher than the samples treated with 10% HCl. This is because the amount of calcite that decreases in the rock is getting bigger. The higher the concentration of HCl for acidizing, the higher the permeability of the rock, so that 15% HCl is recommended to increase the permeability of the rock. The SEM result also suggest that there is certain increase in the permeability of the rock because the porosity cavities formed are interconnected. These indicate that an increase in permeability as a result of the acid treatment process [3].

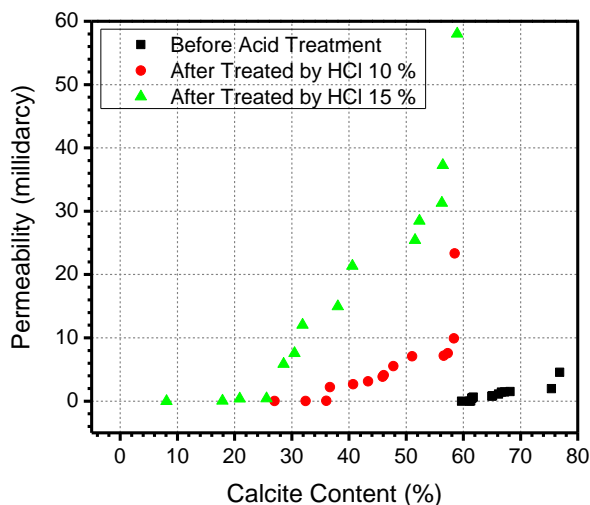


Figure 6. Effect treatment using HCl on various concentration on permeability.

E. HCl concentration on Fluid Flowrate Reservoir

The fluid flowrate is calculated using a theoretical approach such as the Darcy equation [16] as shown in equation (1) which was mentioned in the experimental section. The calculated fluid flowrate data is presented in Fig. 7. This study uses several variables from previous studies that are relevant to the Kutai Basin and other carbonate reservoirs to calculate the fluid flowrate. The surface area data was obtained using the data presented by Fabricius on the typical carbonate reservoir [14]. The fluid flowrate calculation was done using the assumptions that fluid pass to the rock is inert nitrogen gas, which was also used in RCA to measure permeability [21]. The platform temperature at Kutai Basin is approximately 120 °C with pressure of 1 atm [18]. The viscosity of nitrogen gas is 0.000021681 Pa.s calculated from equation (4) which was used by Johansson using similar operating conditions [15].

$$\mu = \mu_{ref} \left(\frac{T}{T_{ref}} \right)^\omega \tag{4}$$

Where μ is viscosity in this cases is nitrogen, T is temperature that used in this study (120 °C), Tref is Temperature reference at 273.15 K and ω is viscosity index as well as explained by Johansson [15], where they studied the microporous flow on low - gas reservoir.

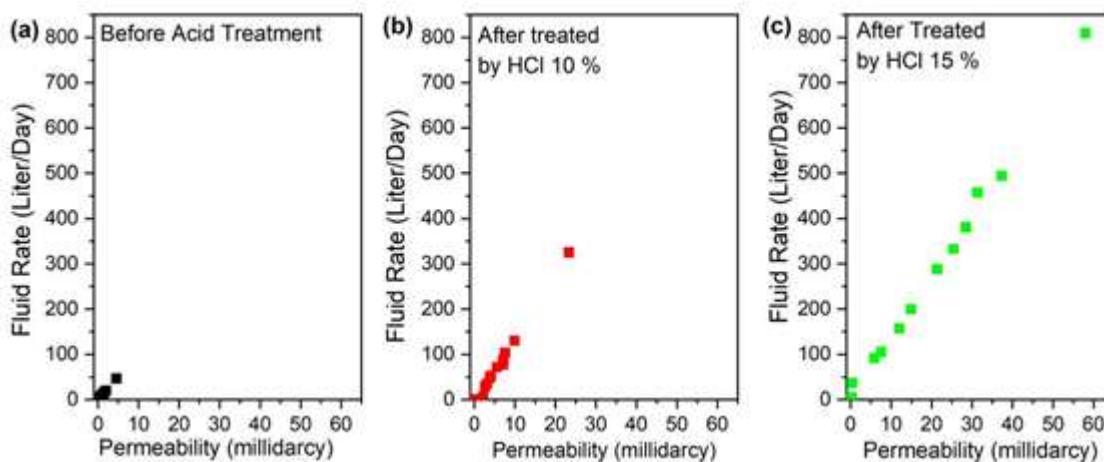


Figure 7. HCl Concentration effect on fluid flowrate of the rock sample. The data in black color shows the fluid flowrate of the sample before being

The data in black color shows the fluid flowrate of the sample before being treated with HCl. From this data, the rate of fluid that can be passed by rocks is still relatively small below 100 Liter/Day, this is due to the small permeability of the sample because the pores of the rock are still closed by calcite. After acidizing treatment with 10% HCl (red color), the fluid flowrate begins to increase with increasing permeability due to dissolution of calcite. The addition of 10% HCl formed a population of fluid flowrates that gathered in the range of 0.07 – 325 Liters/ Day as shown by red data with an average increase in permeability below 10%. The data also shows that the fluid flowrate increases significantly as the permeability increases sharply. After treatment in 15% HCl (green color), the variation of the fluid flowrate and permeability range in each sample, namely 0.09 - 809 Liters/Day as shown by green data. The difference in the range of fluid flowrate produced by 10% and 15% HCl treatment, may occur due to the difference in pore size diameter resulting from the two acid concentrations as well. After treatment in 15% HCl (green color data), the variation of the fluid flowrate and permeability range is longer in each sample than after treatment HCl 10 % This is because sample treated by 15% HCl gives wider distribution range of pore size diameter compared to rocks treated with 10% HCl as show on Fig. 7.

Fig. 8. shows pore size diameter distribution of 100 point taken from samples treated with HCl 10 % and 15 %. These porosity data was measured using ImageJ to calculated pore size diameter from SEM Images as similarly suggested by Rishi [27]. The samples treated with HCl 10 % has pore size diameter with the range of 3.93 – 45.97 μm , where most of the pore size diameter are in 3 – 10 μm . While samples treated with HCl 15 % has pore size diameter within range at 6.5 – 144.1 μm , where the most of pore size diameter are in 20 – 40 μm .

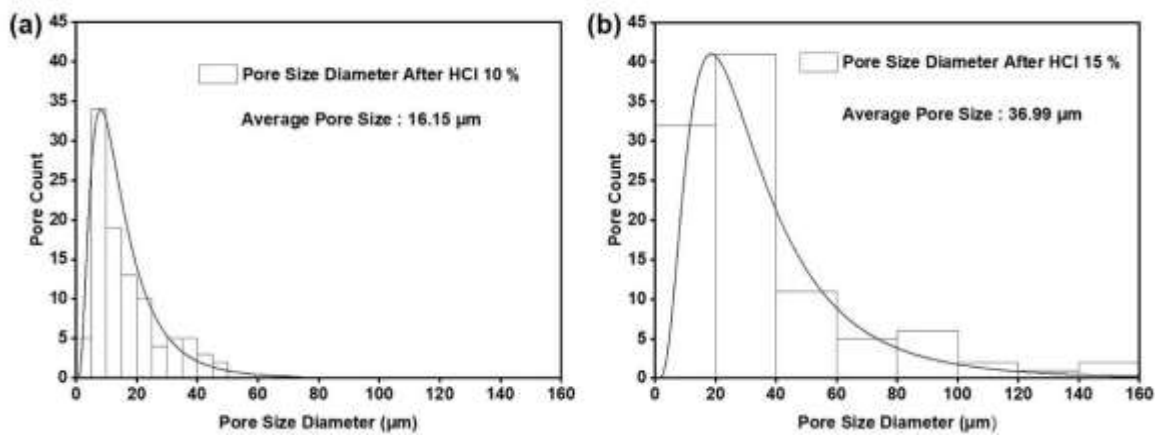


Figure 8. Graphs shows (a) pore size diameter distribution of HCl 10 % treated samples, and (b) pore size diameter distribution of HCl 15 % treated samples.

Further data analysis revealed that the samples treated with HCl 15 % has average pore size 36.99 μm which was higher than samples treated with HCl 10 % (16.15 μm). These tell us that there is difference on the pore size distribution after different acid treatment. Pore size diameter distribution data shows that sample treated with HCl 15 % gives wider fluid flowrate distribution range compared to sample treated with HCl 10%. The comparison between green and red color data show that even with low - permeability, fluid rate can be significantly increase with the wider pore size distribution. This might be due to the changes of surface texture or pore size of the samples treated with higher HCl concentrations. The wider range of pore diameter distribution will allow a larger amount of fluid to pass through it without having to experience a significant increase in permeability [10]. The effect of the pore size on fluid flowrate is also explained by the Poiseuille equation that relates permeability to pore size in general:

$$Q = \frac{\pi P r^4}{8 \mu L} \tag{5}$$

Where Q is fluid flowrate, P/L is pressure gradient along the area, r is pore radius and μ represents the fluid viscosity. The equation (5) tells us that amount of fluid passed through a material will affect the size of the open pores in the material [28]. This is in agreement with SEM showing 15% HCl will provide larger pores which then have higher fluid flowrate compared to the 10% HCl treated sample.

4. Conclusions

Based on this study, we conclude that HCl treatment give significant impact on carbonate reservoir microscale damage and rock properties. We suggested that high HCl concentration will provide better increase in permeability. In this case, HCl 15 % give permeability increase better than HCl 10 %. From rock-fluid aspects, HCl 15 % shows better results than HCl 10 % in increasing the fluid flowrate in reservoir samples. These phenomena are caused by the dissolution of the calcite content of the reservoir which can reduce the reservoir quality. Moreover, the study results shows that the pore size diameter also has significant effect on increasing fluid flowrate. Where HCl 15 % provides a wider pore size distribution in rocks compared to HCl 10 %. These lead to the higher gas fluid flow rate that can be passed on the sample after 15% HCl treatment than 10% HCl. The findings of this study indicate that 15% HCl will provide good stimulation results in terms of changes in rock-fluid properties. However, further studies are still required related to other variables that can be optimized such as volume of HCl and acidizing treatment reaction time.

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