A LABORATORY STUDY TO IMPROVE ACID STIMULATION IN SANDSTONES

by

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I. INTRODUCTION

The main purpose of acidizing is to improve well productivity. Acids are useful for this reason because of their ability to dissolve undesired formation minerals and materials which may either be intrinsic in nature or be introduced into the formation during the processes of drilling, completion, and production. The effectiveness of acids in improving productivity in a particular well essentially depends on an accurate analysis of the problem and the selection of acid.

Prudent judgment in acid to be used should be confirmed by laboratory tests. Apart from the analysis on the nature of the formation damage itself, acid selection should be based on study of reservoir rocks mineralogy and characteristics in general and accordingly the relevant material/minerals to be dissolved or removed. Improper diagnostics may result in inefficient, and even damaging, acidizing. Various studies have been conducted in this highlight (e.g. Crowe, 1984; Gidley, 1971; Crowe in Economides and Nolte, 1989; Daccord in Economides and Nolte, 1989; Ali, 1981; and Piot and Perthuis in Economides and Nolte, 1984).

Those studies conducted in the past reveal that in comparison the success ratio of acidizing for limestone reservoir is almost 90%, whereas for sandstone reservoir the success ratio is only 30%. Undoubtedly, this disparity in success ratios is caused by the fact that appropriate acids dissolve limestones more properly due to limestones generally simpler mineral composition and by the fact that sandstones usually have more complex mineralogy hence providing less simple materials to dissolve. From this point of view it is considered that more acid studies in sandstones, especially sandstones that exist in Indonesian reservoirs, are required in order to improve our understanding. The works, which results are presented in this paper are a part of the efforts to reach that objective.

Table 1
Selected samples for acid response test

Well	Depth, feet	Sample no.	Porosity, %	Permeability,mD
X-2	4601.30	1	25.71	176
X-1	4605.01	2	27.00	275
X-3	5079.00	3	23.67	253

II. SAMPLE PREPARATION AND METHODS

The acid test study has been designed, prepared, and conducted in LEMIGAS laboratory core testing facilities. The study is basically to check whether an acid design for a field application is appropriate for the reservoir rocks of interest.

For the purpose of the acid response test, three (3) samples were taken from three wells penetrating a sandstone reservoir of Lower Sihapas Formation in a field in Malaka Strait. The rocks are of hard consolidated sandstone, which were prepared to have dimension of 1.5 inches in diameter and 1.3 to 1.7 inches in length. After all samples have been thoroughly extracted, leached of all salts, and dried, the porosity and air permeability were then measured. All of the results of porosity and permeability measurement can be seen in Table 1.

The design of the laboratory test is a simulation of real acidizing to be implemented in the field. Before tests, samples were loaded in core flow apparatus and cleaned with toluene and ethanol mixture in order to expose clay surfaces to acid. Then samples were flushed with ammonium chloride (NH_4Cl) brine to remove ethanol mixture and saturate the samples, and were then measured their liquid permeability (before acid injection). In the acid test itself, before injection of the acid (commercial HCl - HF 'mud acid' commonly used for sandstone acidizing), the samples were pre-flushed with hydrochloric acid (HCl) in order to dissolve carbonate materials. The mud acid was then injected at a carefully designed flow rate based on real desired rate of pump, depth of acid penetration, porosity and permeability of rock. After mud acid injection, samples were finally flushed with NH_4Cl to remove the acid. Liquid permeability after acid injection was then measured.

For monitoring results of the acid tests, an integrated petrographic analysis including thin section, scanning electron microscope (SEM) with energy dispersive x-ray (EDX) and x-ray diffractometer (XRD) was used. The petrography tests were carried out prior and after the acid test. The main objective of the test and analysis at initial condition prior to the acid test is to determine reservoir characteristics including mineralogical compositions, clay mineralogy, texture and framework grains and diagenesis before the introduction of acid into core samples. The tests after acid injection are to identify the likely problems or the reworks of the reservoir characteristics that may occur in the reservoir due to the presence of sensitive diagenetic minerals under acid response test. The combined petrographic and permeability tests before and after acid tests will serve as an indicator for the nature of the acid – rock interactions during acid injection.

In detail, the three samples are described, before acid injection, as follows:

A. *Sample #1 from X-2 well (depth 4601.3 ft)*

The main framework grain composition is mainly composed of monocrystalline quartz (54.25%), rock fragments (4.5%), feldspar (1.75%) with additional glauconite (1%), carbonaceous materials (2.75%), micas (0.75%) and heavy minerals (0.5%). Pore-filling materials such as matrix and pseudo-matrix are observed in little amount (1.5%). Clay minerals are composed of kaolinite (3%) and illite (1%). This sample is classified as *sublitharenite* (Folk, 1974). Mean grain size is 0.13 mm (fine-grained sand) and the grains are well-sorted. Roundness of the sample varied from subangular to subrounded, whereas the grain boundary is dominated by planar with additional concave-convex and semi suture.

The sample shows several diagenetic events including compaction (moderate degree) indicated by the presence of pseudo-matrix, planar, concave-convex and semi suture grain boundaries; cementation such as quartz overgrowths (1.75%), clays i.e. kaolinite (1.25%), siderite (7.5%); dissolution and alteration of unstable grains include feldspar, rock fragments, matrix and mica creating secondary porosity (0.75%) and diagenetic minerals such as clay i.e. kaolinite (0.75%), and siderite (0.5%). Authigenic illite was detected in minor quantity.

In term of reservoir quality the sample is apparently moderate to well. Visible porosity is 21.25%, consisted of intergranular (20.5%) and partial dissolution of unstable grains such as feldspar, rock fragments and mica (0.75%). Interconnectivity between pores is poor to moderate due to cementation of quartz overgrowths, siderite and kaolinite, and occasionally by the presence of matrix and compaction. Quartz overgrowths have made some pores to be isolated.

B. *Sample #2 from X-1 well (depth 4605.5 ft)*

Monocrystalline quartz (55.0%) is the dominant component followed by rock fragments (4.25%), feldspar (1.75%) with additional glauconite (1.25%), carbonaceous materials (1.75%), micas (0.75%) and heavy minerals (0.5%). Pore-filling materials such as matrix and pseudo-matrix were observed in little amount (1.75%).

Clay minerals are composed of kaolinite (5%) and illite (1%). This sample is classified as *sublitharenite*. Texture is well sorted, fine-grained sand and the roundness is subangular to subrounded with planar and concave-convex grain boundaries.

The main diagenetic processes are moderate degree compaction indicated by the presence of planar and concave-convex grain contacts, and the presence of pseudo-matrix; cementation i.e. quartz overgrowths (2.50%), clays i.e. kaolinite (4.25%); dissolution and alteration of unstable grains i.e. feldspar, rock fragments, matrix and mica creating secondary porosity (1.25%) and diagenetic minerals such as clays i.e. kaolinite (1.5%), siderite (0.5%) and minor amount of authigenic illite.

The quality of the sample as reservoir quality is moderate to relatively good. Visible porosity is 23.25% consisted of intergranular (19.5%) and partial dissolution of unstable grains i.e. feldspar, rock fragments and mica (1.25%) and microporosity (2.5%). Interconnectivity between pores is poor to moderate caused by cementation of quartz overgrowths and kaolinite, and may also by matrix and compaction. Intensive quartz cementation has caused some pores to be isolated.

C. *Sample #3 from X-3 well (depth 5082.1 ft)*

The main framework grain composition is composed of monocrystalline quartz (54.25%), rock fragments (4.5%), feldspars (1.75%) with additional glauconite (1%), carbonaceous materials (2.75%), micas (0.75%), heavy minerals (0.5%); and planktonic foraminifers (0.25%). Pore-filling materials such as matrix and pseudo-matrix are observed in little amount (1.5%).

Clay minerals include kaolinite (3%), chlorite (2%) and illite (1%). The proportion of clay matrix is low (2.25%), and mainly consists of dispersed (1.75%) and pseudo types (0.5%).

Sample #3 is classified as *sublitharenite* (Folk, 1974). Fine sand characterized the mean grain size of 0.127 mm with well-sortation. The grain boundary is dominated by planar and subordinate concavo-convex.

The main diagenetic processes are slight to moderate compaction, indicated by the presence of pseudo-matrix, and planar and concave-convex grain boundaries; cementation i.e. quartz overgrowths (3%), siderite (1.75%) and kaolinite (1.5%); dissolution and alteration of unstable grains i.e. feldspars, mica, rock fragments and matrix creating secondary porosity (0.5%) and diagenetic minerals i.e. clays (1%) and siderite (0.5%).

Quality of the sample as reservoir rock is moderate to relatively good. Visible porosity is 20%, and it composed of intergranular (19.5%) and dissolution (0.5%). Interconnectivity between pores is poor to moderate due to compaction and cementation. The reduction of reservoir quality is mainly controlled by the combination of compaction and cementation and slightly by matrix content.

III. RESULTS

A. *Sandstone composition and texture after acid test*

Sample #1

Monocrystalline quartz (56.25%) is dominant followed by subordinate rock fragments (6%), feldspars (0.75%) and additional glauconite (1.5%), carbonaceous materials (1.75%), micas (1.25%) and heavy minerals (0.25%). Pore-filling materials such as matrix and pseudo-matrix were observed in small amount (1.25%).

Clay mineral is only composed of trace amount of illite. This sample is classified as *sublitharenite* (Folk, 1974). Mean grain size is 0.13 mm (fine-grained sand) and the grains are well-sorted with subangular to subrounded roundness, whereas the grain boundary is dominated by planar with additional concave-convex and semi suture types.

The main diagenetic events including moderate degree compaction indicated by the presence of pseudo-matrix, planar, concave-convex and semi suture grain boundaries; cementation includes quartz overgrowths (1.75%), clays i.e. kaolinite (0.5%), siderite (0.5%); dissolution and alteration of unstable grains i.e. feldspar, rock fragments, matrix and mica creating secondary porosity (0.75%) and diagenetic minerals such as clays i.e. kaolinite (0.50%). Authigenic illite was detected in minor quantity; other component found after acid test is amorphous material (4.75%). Visible porosity after test is 22.25% consisted of intergranular (20.5%) and mould (0.75%).

Sample #2

The sample is mainly composed of monocrystalline quartz (56.75%), rock fragments (5.75%), feldspars (0.75%) with additional glauconite (0.75%), carbonaceous materials (2.25%), micas (1.5%) and heavy minerals (0.5%). Matrix occurred in small amount (1.25%).

Clay mineral is only composed of minor amount of kaolinite (1%). This sample is classified as *sublitharenite* (Folk, 1974). Mean grain size is 0.14 mm (fine-grained sand) and the grains are well-sorted. Roundness of the sample varies from subangular to subrounded, whereas the grain boundary is dominated by planar with additional concave-convex types.

The main diagenetic events are moderate degree compaction indicated by the presence of planar and concave-convex grain boundaries; cementation i.e. quartz overgrowths (2.5%), clays i.e. kaolinite (1%); dissolution and alteration of unstable grains i.e. feldspar, rock fragments, matrix and mica creating secondary porosity (1%) and diagenetic minerals such as clays i.e. kaolinite (0.50%) and illite (0.25%); other component found after acid response test was small amount of amorphous material (0.5%). Visible porosity is 25.75%, consisted of intergranular (24.75%) and secondary (1.0%).

Sample #3

The sample is mainly composed of monocrystalline quartz (55.0%), rock fragments (5.5%), feldspars (0.5%) with additional glauconite (1.25%), carbonaceous materials (4.5%), micas (1%) and heavy minerals (0.25%). Matrix was observed in minor to moderate amount (4.5%).

Clay mineralogy includes only trace amount of illite. This sample is classified as *sublitharenite* (Folk, 1974). Texture is fine-grained sand and shows well-sorting. Roundness of the sample varies from subangular to subrounded, whereas the grain boundary is dominated by planar with additional concave-convex types.

Table. 2
Water permeability before and after acid injection at various rate, Sample #1

Rate, ml/min	Kw, mD (before acid injection)	Kw, mD (after acid injection)
0.25	10.84	15.76
1.0	17.57	17.17
4	25.16	27.18

Table 3
Measured water permeability at various flow rates for Sample#2 before and after acid injection

Rate, ml/min	Kw, mD (before acid injection)	Kw, mD (after acid injection)
0.25	4.68	31
1.0	12.9	42.84
4	16.94	48.17

The main diagenetic events are moderate degree compaction indicated by the presence planar and concave-convex grain boundaries; cementation i.e. quartz overgrowths (2.75%), kaolinite (0.5%); dissolution and alteration of unstable grains i.e. feldspar, rock fragments, matrix and mica creating secondary porosity (0.75%) and diagenetic minerals such as clays i.e. kaolinite (0.25%) and illite (0.25%). Authigenic illite was detected only in trace amount; other component found after acid test was amorphous material (1.75%). Visible porosity is 22.0%, consisted of intergranular (21.25%) and secondary (0.75%).

B. *Results of brine throughput test (permeability before and after acid tests)*

Sample #1

Initially, in the process, after the sample was being cleaned with toluene and ethanol mixture, the sample was flushed with 3% NH_4Cl brine until fully saturated. Cell was heated at 80R deg C, at which it was flushed using the same brine. At this condition, water permeability was measured as a function of volume throughput of injected brine. Throughput test results show very low liquid to air permeability ratio remaining at terminal condition indicating that the tested sample is very sensitive to brine (liquid permeability=16.99 mD, air permeability=176 mD). As the flow was reversed at terminal condition, water permeability at various rates was also measured in this reversed direction. Results are presented in Table 2.

The acid injection process was performed by injecting 10 PV HCl at 5.76 ml/min, 10 PV acid at 5.76 ml/min, and 10 PV 3% NH_4HCl at 5.76 ml/min sequentially. The water permeability after acid injection (after acid injection, sample has changed slightly in color and texture, at various rates was also measured (see Table 2). As in the case of brine volume throughput before the acid test, for brine throughput test after acid test flow was also reversed at terminal condition and the water permeability was measured as a function of brine volume injected. In this reversed flow condition water permeability increased sharply to 89.47 mD, but however, as brine was continuously injected water permeability was again reduced to 20.24 mD.

Sample #2

Following the same procedure as in the case of Sample #1, brine throughput test was carried out before and after acid test. Similar to results for Sample #1, very small liquid to air permeability ratio was observed after brine throughput before acid test (liquid permeability = 15.31 mD, air permeability = 275.3 mD. Then flow was reversed and water permeability at various rates was measured (see Table 3). Water permeability increases as the rate rises. The acid injection process was performed by injecting 9 PV HCl at 5.9 ml/min, 9 PV Acid at 5.9 ml/min, and 9 PV 3% NH_4HCl at 5.9 ml/min sequentially. The water permeability after acid injection (after acid injection, the sample has changed slightly in color and texture, at various rates was also measured (see Table 3). After that flow was reversed back to production point, in this condition, water permeability was measured as a function of brine volume injected. Water permeability increased to 33.69 mD, and the net reduction in permeability is just by 4.4% at end of test.

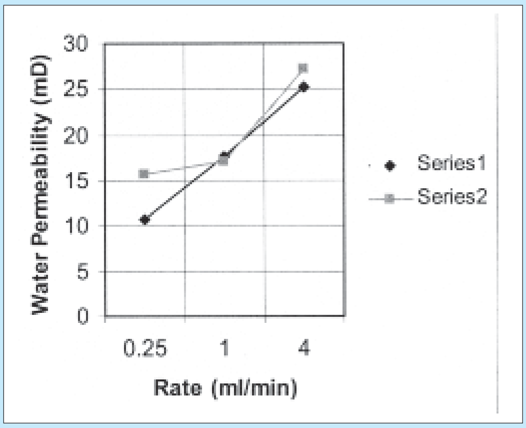

Figure 1
Plot of water permeability vs. flow rate for Sample #1 before and after acid injection

Sample #3

Results show very low liquid to air permeability ratio at the end of brine throughput indicating the sample's strong sensitivity to brine (liquid permeability=13.05 mD, air permeability=253 mD). After flow had been reversed, various flow rates were used and water permeability was measured. Water permeability increases as the rate rises. Overall results are presented in Table 4.

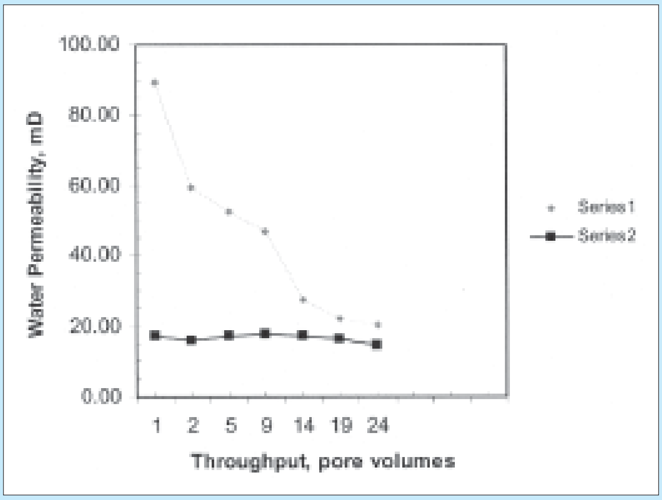

Figure 2
Water permeability as a function of brine volume injected for Sample #1 before and after acid injection

The acid injection process was performed by injecting 13 PV HCl at 5.9 ml/min, 13 PV acid at 5.9 ml/min, and 13 PV 3% NH4HCl at 5.9 ml/min sequentially. After acid test, brine throughput test was again carried out and water permeability was measured at various flow rates (see Table 4). At the end of the test flow was reversed back to production point and water permeability was measured as a function of brine volume injected. It was observed that water permeability increased to 130 mD, but further brine throughput reduced the water permeability down to 98 mD.

Table 4
Water permeability for Sample #3 at various flow rates before and after acid injection

Rate, ml/ min	Kw, mD (before acid injection)	Kw, mD (after acid injection)
0.25	13.13	63.94
1.0	18.51	80.06
4	25.06	120

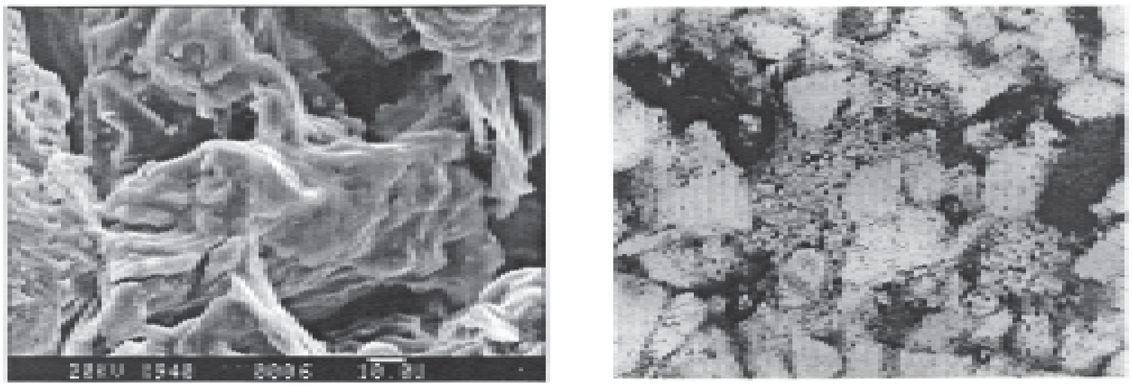

Figure 3
SEM (left) and thin section (right) show silica gel precipitated in pore-throats due to the dissolution of siderite causing the pH of the environment become higher. This causes permeailiaty reduction

IV. FURTHER DISCUSSIONS

As shown in Figure 1 and Table 2, the results for Sample #1 show that there is no significant change in water permeability value at various rates after acid injection. Nevertheless, when flow was reversed into opposite direction the figure shows differently. As presented in Figure 2, water permeability value increases after acid injection from 16.99 mD to 89.47 mD initially but then drops sharply to value of 20.24 mD after further brine injection.

Permeability reduction during fluid flow, as acknowledged, could be caused by various causes but in this case it is fairly acceptable that the plugging occurrence was likely caused by precipitation of silica. The occurrence of the precipitation of silica gel is indicated by the integrated petrographic analysis as described in these following paragraphs.

After acid injection test the amount of subangular grains decreased as quartz overgrowths have partly been dissolved. Grain boundary is mainly planar with minor point types. Concave-convex and suture contacts are still observed, but have decreased significantly due to acid test. Some feldspar grains and matrix have also been dissolved. Only trace amount of illite is detected by XRD. Kaolinite and illite have totally been removed by acid test. Quartz cement partial dissolution caused the enhancement of porosity. Siderite was also dissolved (as confirmed by XRD). However, as shown by SEM-EDX and thin section analyses some amorphous material (silica gel) (4.75%) has occurred. It was found out the gel was formed due to the increase of pH of the reservoir causes by the dissolution of siderite as iron bearing minerals (for more detail explanation see to Piot and Perthuis in Economides and Nolte, 1989). The increase of pH has led into the possibility of the precipitation of dissolved silica in the form of silica gel. The precipitation of the silica gel seemingly reduced the permeability of the tested sample (Figure 3).

As mentioned earlier, the reverse is true for porosity. Porosity increased with the dissolution of cement and other diagenetic minerals i.e. quartz overgrowths, kaolinite and illite. This dissolved material eventually precipitates in the form of silica gel, which mainly took place in pore-throats (Figure 3).

The results of test on Sample #2 indicate a successful acid injection test. As shown in Table 3 and Figure 4, water permeability as a function of various flow rates increased sharply after acid injection. Permeability value gained at least twice after acid injection. Moreover, water permeability as function of injected volume figure also increase after acid injection and only drop 4.4% as brine injected further (see Figure 5). The test shows no presence of pore-throat plugging material such as precipitation of gelatinous substance such as one formed in the acid injection on

Sample #1. This conclusion is supported by petrographic analysis result.

After acid response test the amount of subangular grains have been reduced due to partial dissolution of quartz cement. Grain boundary is mainly planar type. Concave-convex grain contact is still observed, but has moderately decreased. Some feldspar grains and matrix have also been dissolved. Minor and trace amount of kaolinite (1%) and illite are detected. Kaolinite and illite have mostly to totally been dissolved by acid test. The dissolution of these cements caused increase in porosity. Minor amount of siderite were also dissolved, however minor amount of amorphous material (silica gel) (0.75%) occurred. Similar to in the case of Sample #1, the dissolution of siderite during acid test has caused the pH of reservoir become higher. This causes the precipitation of silica gel. Porosity increases with the dissolution of cement and other diagenetic minerals i.e. quartz overgrowths, kaolinite and illite. Permeability of the sample may not be affected since only minor amount of the gel developed (Figure 6).

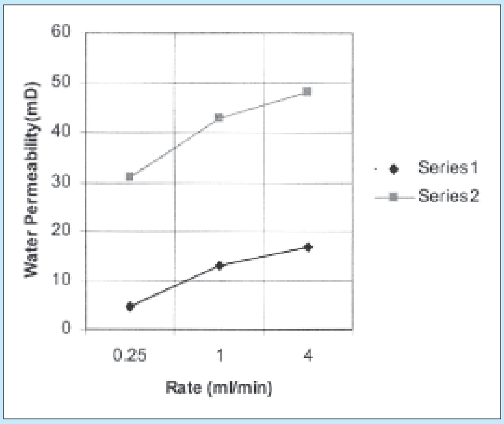

Figure 4
Water permeability for Sample #2
as function of flow rate before
and after acid injection

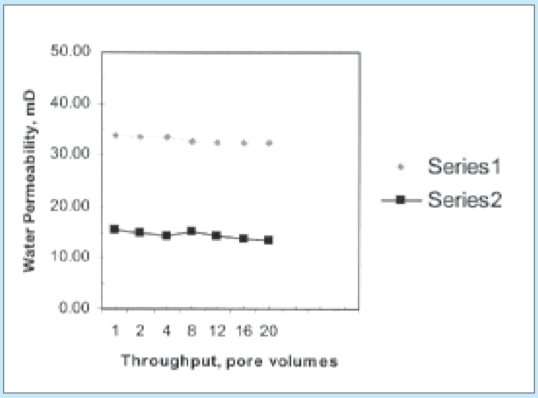

Figure 5
Water permeability for Sample #2
as a function of injected brine volume before
and after acid injection

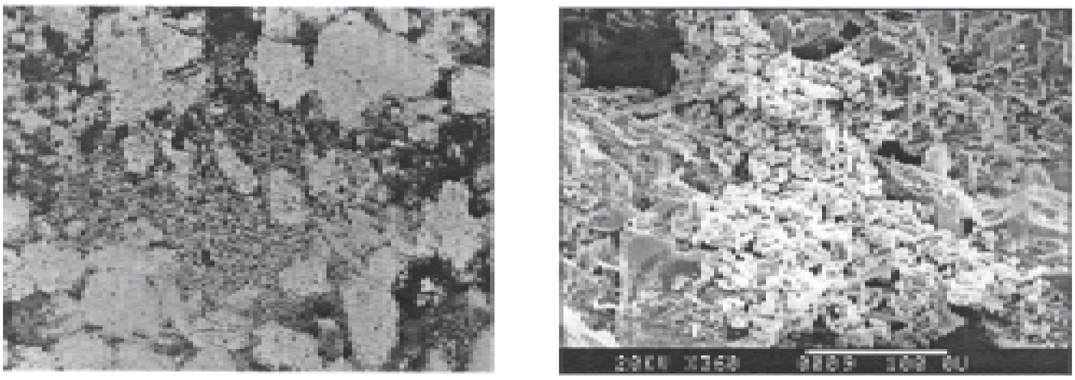

Figure 6
Only minor amount of silica gel detected under thin section (black area; left).
Minor amount of kaolinite still remains after acid test (right)

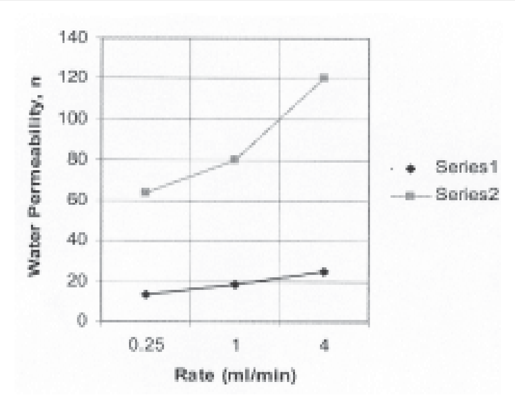

Figure 7
Water permeability for Sample #3 as a function of flow rates before and after acid injection

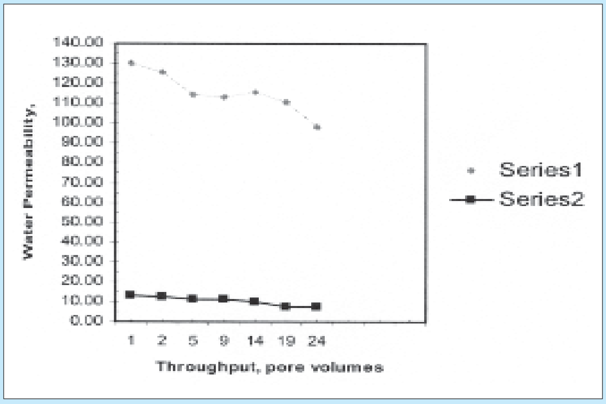

Figure 8
Water permeability for Sample #3 as a function of injected brine volume before and after acid injection.

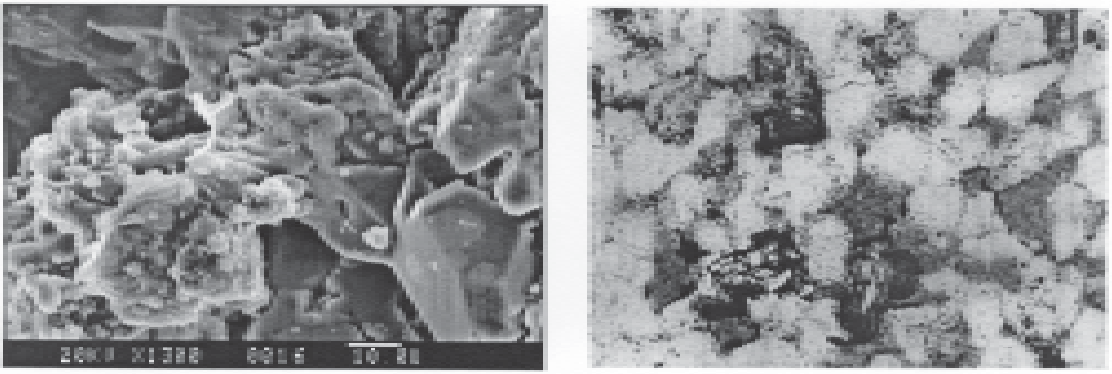

Figure 9
Precipitation of silica gel caused by the dissolution of siderite during acid test. The development of the gel caused permeability reduction

Acid response test on Sample #3 is characterized by gain in permeability after acid injection. As seen in Figure 7 and Table 4, water permeability as function of flow rates after acid injection increases sharply compared to water permeability before acid injection. Water permeability as function of injected volume figure also increases after acid injection. However, as brine volume injected further, water permeability tends to decrease faster than its corresponding figure before acid injection (Figure 8). As in the case of Samples #1 and #2, the main causes of the permeability reduction is precipitation of silica gel produced after acid injection. The existence of the precipitation of silica gel is supported by petrographic analysis results.

After acid response test the quantity of subangular grains has been reduced due to dissolution of quartz overgrowths. Grain boundary is mainly planar with minor point types. Concave-convex type was still observed, but has moderately decreased. Some feldspar grains and matrix have also been dissolved.

Only trace amount of illite was detected by XRD, whereas kaolinite, chlorite and illite have entirely been removed by acid test. Quartz cement has also partly been dissolved causing increase in porosity. Siderite is also dissolved. However, according to SEM-EDX and thin section analyses some amorphous material (silica gel; 1.75%) took place. As iron (Fe) rich minerals, siderite ($FeCO_3$) and chlorite $(Mg, Al, Fe)_{12}$; $Si\ Al)_8\ O_{20}; OH)_{16}$ have been dissolved during acid test causing the increase of pH, which has caused the precipitation of silica gel (Figure 9). As in the case of Samples #1 and #2, the precipitation of this gel reduced the permeability of the tested sample.

V. CONCLUSIONS AND RECOMMENDATION

A. Conclusions

Several conclusions can be drawn from the test,

1. Dissolution of siderite within rock samples increases pH that causes the precipitation of silica gel Precipitation of the gel is the major factor in reducing permeability of the tested samples.
2. Result of acid response test on Sample #1 has not shown good acid performance. The dissolution of siderite as iron bearing mineral during acid test has caused the pH of the environment become higher, causing the precipitation of dissolved silica.
3. Acid response test on Sample #2 has shown good results in the form of only minor gel precipitation occurred after acid injection.
4. Result of acid response test on Sample #3 has shown increase in permeability despite the petrographic evidence showing precipitation of the silica gel.
5. Variation of response shown by the three samples tested in this study has served as evidence that a careful examination over acid – rock interaction is essential before any attempt to implement acid stimulation treatment in the field.

B. Recomendations

1. Sample #1 contains a high percentage of siderite. A great caution should be taken before deciding for an acidizing using commercial mud acid on this kind of sandstones.
2. Problem of silica gel production and precipitation may be reduced by increasing the volume HCl during pre-flush. The volume of pre-flush HCl should be greater than the mud acid proportionally.
3. Lowering HF/ HCl ratio is one way to retard precipitation. A low HF content reduces the precipitation of silica Si(OH) (as suggested by Walsh et al., 1982).
4. Keep the pH low by adding chelating agent (organic acid; as hinted by Ali, 1981 and Piot and Perthuis in Economides and Nolte, 1984).

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