SPE-204266-STU

Formation Integrity and Pore System Evaluation for Stimulated Sandstone Formation Using In-Situ Generated Acid Catalyzed by A Fused Chemical Reaction

Ibrahim Gomaa, King Fahd University of Petroleum & Minerals

This paper was prepared for presentation at the SPE Annual Technical Conference & Exhibition originally scheduled to be held in Denver, Colorado, USA, 5 – 7 October 2020. Due to COVID-19 the physical event was postponed until 26 – 29 October 2020 and was changed to a virtual event. The official proceedings were published online on 21 October 2020.

Abstract

This study aims at evaluating the mechanical integrity and the changes in the pore system of illitic sandstone formations being treated with HF acid generated in-situ and thermochemical fluids (**TCF**). Firstly, the acid-generating fluids were injected separately inside the core within a typical core flooding experiment. After that the core permeability, NMR porosity and rock static and dynamic parameters such as rock strength (**UCS**), dynamic Young's modulus, Poisson's ratio, shear modulus and bulk compressibility were evaluated before and after the treatment. Sand production prediction model was used to examine the potential of sand production problems for the cores treated with the in-situ generated acid (**ISGA**) and the **TCF**.

Treating Scioto cores with the ISGA only led to permeability enhancement with final to initial permeability ratio $\frac{K_{final}}{K_{initial}}$ of 1.2. In addition, treating the same cores with acid generating fluids and thermochemicals resulted in higher permeability enhancement with $\frac{K_{final}}{K_{initial}}$ of 485. This higher enhancement in the formation permeability was attributed to the creation of some microfractures as was proved using the NMR scans. There was a reduction of the cores' static and dynamic parameters after being treated with the ISGA. This reduction was even higher after treating the core with thermochemical fluids. However, the sand production prediction model stated that the reduction in rock mechanical parameters would not lead to sand production problems.

Introduction

Formation damage is one of the critical problems in the oil industry. The term "formation damage" refers to any improper action that can lead to decrease the formation permeability. Formation damage problem is expected to occur at any time of the reservoir life and can be classified as natural or induced damage, Figure (1), (Civan, 2005; Amaefule et al.,1988; Radwan et al., 2019).

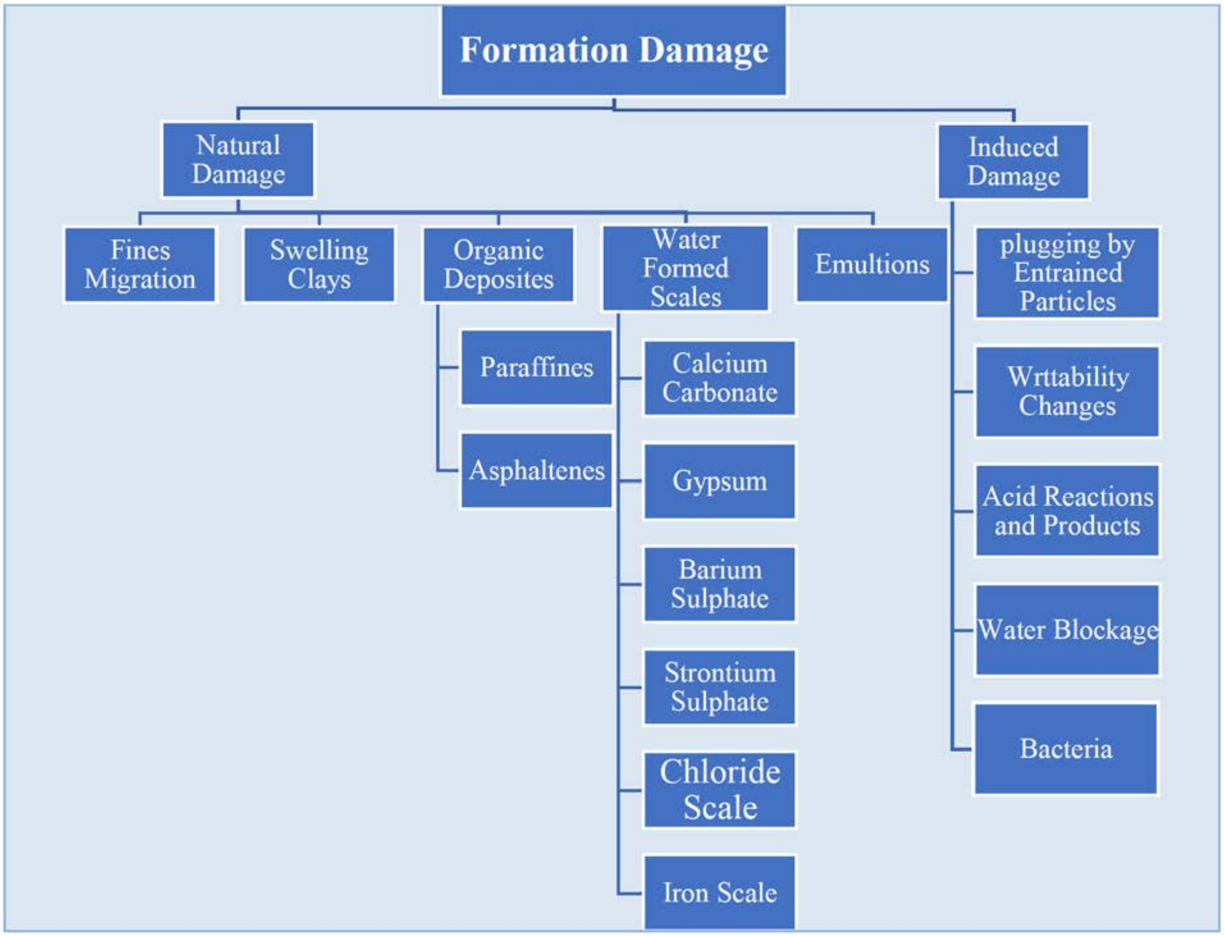

Figure 1—Formation damage classifications (Radwan et al., 2019)

Unlike carbonate formations, sandstones are distinguished by their complex mineral system with quartz as the main constituent. Different types of clays such as chlorite, illite, kaolinite and smectite can be found with different amounts and structures within sandstone rocks. Moreover, sandstone formations contain fractions of feldspars and carbonates as cementing material. Generally, the objective of the acid treatment to an oil-bearing formation is to enhance its productivity by two main mechanisms; (1) is to dissolve the rock minerals and create additional flow channels within the reservoir or (2) dissolve the formation damage and restore the formation initial conditions (Coulter, Hendrickson, & Martinez, 1987).

In 1939, the regular strength mud acid was firstly introduced to treat sandstone formation damage. Regular mud acid is simply a mixture between hydrochloric acid (HCl) and hydrofluoric acid (HF) with ratios of 12% and 3% respectively (Portier et al., 2007). Most of the mineral constituents of sandstone rocks have good solubility in the mud acid (HCl +HF) unlike the hydrochloric acid alone. However, the reaction of mud acid with sandstone formations is considered to be a very quick reaction. Once the acid mixture is injected, it reacts quickly with the near formation area and is spent rapidly. This makes the depth of acid penetration into the formation so small. As a result, fines migrate to the near wellbore area would plug the formation and restore the damage (Cheung and Van Arsdale, 1992; Chang et al. 2000).

HCl and HF acids are harmful to the environment as well as the well equipment. Their corrosion rate is very high and needs special treatment once returned to the surface. This warrants the need for a delayed acid system where live acid can be controllably released downhole and propagates deeper into the reservoir while treating formation damage (Malate et al. 1998; Leong and Ben Mahmud, 2019).

Throughout the literature, several trials adopted the idea of in-situ acid generation. In-situ acid generation allows high control of the acid placement against the target formation. The amount of acid released into the formation can be highly monitored as well. Moreover, the well tubular corrosion problems due to their long contact with live acids during conventional acidizing jobs are highly reduced. Besides, the safety hazards that are related to acid handling on the surface are eliminated. Since the amounts of acids released are stoichiometrically controlled, almost all the produced acids will be spent. This reduces the over cost of the acid neutralization process once returned to the surface before disposal (Gomaa and Mahmoud, 2020).

Injecting a mixture of water-soluble fluoride salt namely (ammonium fluoride) and organic acid ester namely (methyl formate) is an approach to generate in-situ hydrofluoric acid adopted by (Templeton et al.,1975). This technique gives enough time for the generated acid to be in contact with the target formation and hence enhances the stimulation process. It is claimed that retarded HF acids (RHF) systems can achieve deeper acid penetration due to their slower reaction rate with the sandstone formation minerals, especially the alumino-silicates (clays and feldspars) (Al-Dahlan et al., 2001). One of the retarded methods to generate HF is by hydrolyzing fluoboric acid HBF_4 in aqueous solution. This process takes place at a slow rate and has different stages. The slowness of this process allows the acid to penetrate deeper into the formation. HBF_4 has a positive impact on clay stabilization and the prevention of fines migration (McBride et al. 1979; Thomas and Crowe 1981).

Fused chemical reactions are those reactions that can be retarded by either chemical or physical means. This kind of reaction has a wide range of applications in the oil industry such as acid diversion, enhanced oil recovery processes, scale removal, and even more. One example of the fused reactions is the reaction between sodium nitrite and ammonium chloride, (Eq. 1). This reaction is a highly exothermic reaction with a change in enthalpy of $\Delta H_{Rx} = -79.95\ Kcal.\ mol^{-1}$. It is a highly controllable irreversible reaction with $K_{eq} = 3.9\times10^{71}\ Pa.mole\ m^{-3}$at 25°C, (Nguyen et al., 2003).

$$NH_4Cl + NaNO_2 \rightarrow NaCl + 2H_2O + N_2 + \Delta H \tag{1}$$

This exothermic reaction was firstly introduced to the oil industry for removing the wax, asphaltene, hydrate and paraffin deposits in low temperature (Nguyen et al., 2001; Singh et al., 2000). Further development, later on, led to the use of the same reaction for more wide applications in the oil industry. Hassan et al., (2019) used the technique of thermochemical fluids for treating tight sandstone reservoirs suffering from water blockage problems. Gomaa et al. (2020) introduced for the first time the concept of combining the thermochemical fluids (TCF) reactions, (Eq. 1), with ammonium fluoride oxidation reaction (Eq. 2) for the sake of producing in-situ hydrofluoric acid (HF). The produced HF is a strong function in the amount of TCF fluids used as well as the oxidizing agent concentration. Since the HF generation reaction only occurs downhole, the contact between the acid and the well tubulars is highly decreased and the acid corrosion problems are minimized.

$$2NH_4F + NaBrO_3 + \Delta H \rightarrow 2HF + NaBr + 3H_2O + N_2 \tag{2}$$

Once the HF acid is formed, it starts to react with the sandstone minerals and dissolve the rock matrix as well as the cementing materials while trying to remove the formatting damage. These dissolution reactions have to be evaluated and monitored in order not to cause any adverse effects. If the matrix and cementing material dissociation rate is high, then problems such as sand production, fines migration, and sand un-consolidation can be anticipated. The objective of this paper is to use the rock elastic parameters such as Young's modulus (E), Poisson's ratio(ν), and shear modulus (G) for evaluating the sandstone rock stiffness after being acidized in the presence/absence the thermochemical fluids. In addition, the use of nuclear magnetic resonance (NMR) scans for monitoring the changes in the pore system after core flooding experiments.

Methodology and Materials

Core Flooding Experiments

Linear core flooding experiments were carried out on 6" × 2.5" Scioto sandstone cores having an illite clay content of 18 wt.%. Firstly, the core liquid permeability was measured using a potassium chloride brine solution with a concentration of 3 wt.%. The core flooding set-up is shown in Figure (2) is used to carry out all the core flooding experiments throughout this study. The components of the system are 6-inch core holder and 3 high capacity stainless steel transfer cells that can operate simultaneously and are connected to a syringe pump. The core inlet and outlet pressures are monitored by pressure transducers connected to "OMEGA" software. For heating purposes, the system is fixed in a heating oven.

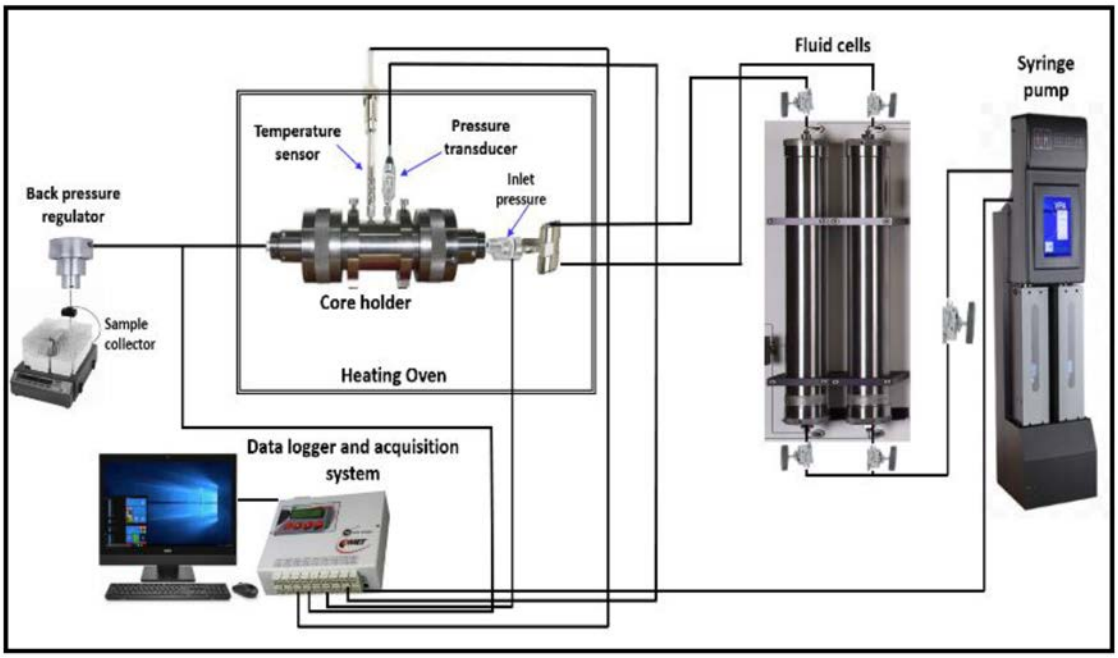

Figure 2—The core flooding set-up

Firstly, the acid-generating chemicals (NH_4F and the oxidizer) are kept in separate transfer cells and then injected simultaneously into the Scioto cores for three consecutive cycles of one pore volume (PV) each. This stage was preceded with a pre-flush stage of 3 wt.% KCl solution where the core permeability was measured and the clay minerals got stabilized by the brine solution. Finally, the core was flooded back using 3 wt.% KCl brine solution and the core final permeability was measured using Darcy's law for steady-state flow.

In order to investigate the influence of the pressure and temperature generated from the TCF, the same previous core flooding sequence was followed except for the main flooding stage. In this stage, the acid-generating fluids are kept in one cell and the TCF fluids are kept separately in individual cells. Firstly, 0.5 PV of the acid-forming chemicals was injected followed by another 0.5 PV of the TCF. Then the core was left for about half an hour before the second injection cycle. This was repeated for 5 consecutive cycles where the pressure drop across the core was measured in a timely manner.

Rock Mechanical Properties Measurements

Sonic waves have wide applications in the oil industry. Calculating the rock elastic moduli in non-destructive tests using sonic waves is getting high interest in the petrophysics related areas. The primary compressional wave (P-waves) is a mechanical wave that propagates in all the solid and fluid media. For P-waves, the oscillations of the particles are of the same direction as wave propagation direction. The shear waves (S-waves) are secondary waves that propagate only through solids. For shear waves, the oscillations of the particles are perpendicular to the direction of the wave propagation. Measuring the velocity these two waves (V_P & V_s) is very helpful for calculating the rock elastic moduli such as elastic Young's modulus (E), Poisson's ratio (v) and shear modulus (G).

Elastic Young's modulus is a measure of the rock resistance to the axial stress. It gives an indication of the rock stiffness and is defined by the ratio of the axial stress over the corresponding axial strain. Using sonic waves' velocities, elastic Young's modulus can be calculated through Eq. (3).

$$E = \rho V_p^2 \frac{3V_p^2 - 4V_s^2}{V_p^2 - V_s^2} \tag{3}$$

Poisson's ratio is the negative unitless ratio of transverse strain to axial strain. It gives an indication of the rock compressibility and can be calculated from Eq. (4).

$$v = \frac{V_p^2 - 2\ V_s^2}{2\left(V_p^2 - V_s^2\right)} \tag{4}$$

Shear modulus, Eq. (5), is the indication of the rock resistance to shear force. It is a useful input for sand production prediction models as we will explain more in this paper.

$$G = \frac{E}{2(1+v)} \tag{5}$$

All the previous elastic parameters were measured using the sonic mode attached to the scratch test device.

Pore System Evaluation

Evaluating the rock pore system by identifying the pore system distribution along the core is one of the main objectives of this study. NMR scans using the Oxford Geospec rock core analyzer were carried out before and after the acid treatment. This can give us an indication of the rock dominant pore system and show the presence of any secondary pore system that might be created after the acidizing treatment. All the measurements were made using the 2 MHz frequency under ambient conditions while the core samples were saturated with 3 wt.% KCl brine solution. The relaxation time (T2) distribution along the core was recorded for the fresh Scioto core and final acidized core with TCF as well.

Results and Discussion

Permeability Enhancement Due to The In-Situ Generated Acid (ISGA) And the Thermochemical Fluids (TCF)

$K_{initial}$ was measured at the rate of 1 ml/min using the potassium chloride brine solution. Using the information shown in Table (1) and by applying Darcy's law, the core initial permeability was found to be about 0.55 mD.

Table 1—The parameters of Scioto core used to calculate the initial permeability

	Scioto core parameters
Diameter (in)	1.494
Core Length (in)	5.970
Fluid salinity	3 wt.% KCl
Fluid density (g/cm^3)	1.0166
Flow rate (cm^3/min)	0.5
Fluid viscosity (cP)	1

Injecting the in-situ generating acid fluids inside the cores for 3 successive cycles led to dissolve the rock minerals. This, in return, resulted in an enhancement in the connectivity of the pores reflected by an increase in the sample permeability. The final permeability measurement was carried out by injecting the KCl brine solution in the reverse direction of the acid injection direction in order to simulate the actual flow back process after well stimulation. The brine injection with a rate of 0.5 ml/min continued until the steady-state conditions were achieved, see Figure (3) that shows the pressure drop (ΔP) across the core during the different flooding stages. Applying Darcy's law resulted in a core final permeability of 0.66 mD and a permeability ratio $\frac{K_{final}}{K_{initial}}$ of 1.2. The enhancement in the core permeability proves the concept of the HF generation by the oxidation of the ammonium fluoride salt. HF reacted with the rock minerals and dissolved them including quartz due to its high ability to react with silica compounds.

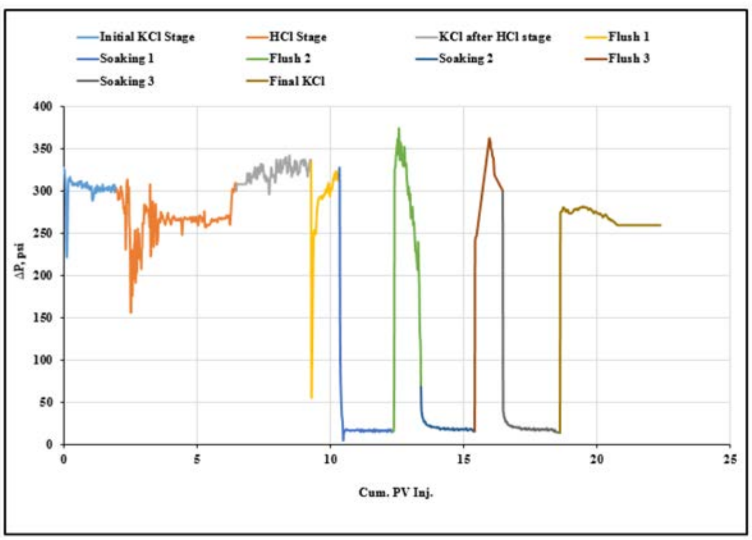

Figure 3—Pressure drop across the Scioto core during the different flooding stages with the ISGA

The same core flooding sequence was repeated using the HF acid-producing chemicals along with the TCF as a main flush stage. This injection sequence allows deeper penetration for the acid-generating fluids. The temperature generated from the TCF should catalyze the acid generation reaction, and dissolve any unwanted organic deposits on the rock surface. Moreover, the high pressure produced from the TCF will drive the generated acid further into the formation, generate some microfractures and make it easier to flow back the well.

The final permeability was measured using potassium chloride brine. The brine was injected along the core back-flow direction to simulate the actual well flow after acid treatment. Different flow rates were tested until the steady-state conditions were achieved. The final core permeability reached about 267 mD resulting in a permeability ratio $\frac{K_{final}}{K_{initial}}$ of 485, see table (2).

Table 2—Core permeability and final/initial permeability ratio after different treatments

	Permeability (mD)	$\frac{K_{final}}{K_{initial}}$
Initial permeability	0.55	NA
Permeability after treating the core with ISGA	0.66	1.2
Permeability after treating the core with ISGA +TCF	267	485

The Effect of the Stimulation Process on the Core Pore System

Enhancing the core permeability after acidizing is definitely a positive response. However, identifying the reasons of permeability enhancement has a tight relation to the changes in the pore system. Permeability can be enhanced due to the enlargement in the core initial pore system or the creation of a secondary pore system through the generation of some microfractures. NMR scans using T2 distribution can give an indication of the changes in the core pore system after the acidizing process. Figure (4) shows the NMR scan results for the acidized Scioto core with the acid-generating fluids only. It is clear from the area under the curves that the core porosity was enhanced after the acidizing process. This is attributed to the mineral dissolution occurred by the action of the HF acid generated. In the meanwhile, both curves maintained almost the same behavior having only one major peak. This indicates that the core original pore system was maintained with only some enlargement in the major pores.

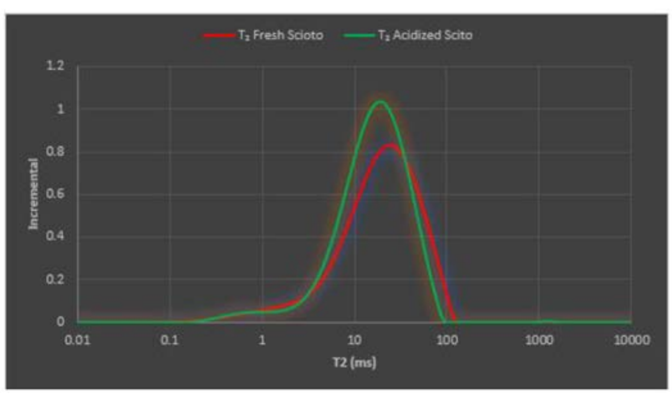

Figure 4—NMR scans for both fresh and acidized Scioto core showing the T2 distribution

As mentioned above, stimulating Scioto cores with the <SC>ISGA and the TCF resulted in higher permeability enhancement. As expected from the behavior of the TCF, microfractures might be anticipated in the tight formation such as Scioto sandstone. Figure (5), shows the NMR scans for the Scioto core before and after being treated with the <SC>ISGA along with the TCF. The same observation found in Figure (4) can be concluded here about the porosity enhancement as the area under the curve for the acidized sample is higher than the fresh Scioto sample. However, there is an additional pore system was created after using the TCF. This is clear from the additional hump observed in the T2 distribution for the acidized core (highlighted by the red circle). This hump indicates the presence of other pore system created upon treating the core with TCF. This indicated the generation of some microfractures inside the core due to the high-pressure pulse generated from the core.

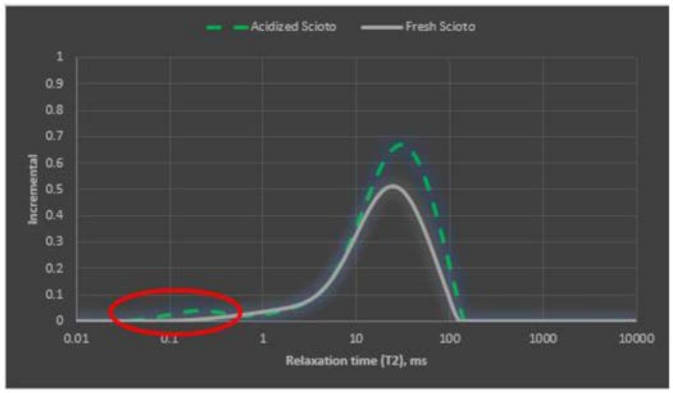

Figure 5—NMR scans for both fresh and acidized Scioto core with TCF showing the T2 distribution

The Effect of The Stimulation Process on Core Integrity

Scratch tests were conducted to measure the core uniaxial compressive strength along with the rock elastic parameters such as dynamic Young's modulus (E), dynamic Poisson's ratio (ν) and the shear modulus. These values can give us an indication of the effect of the acidizing process on rock integrity. Due to the dissolution of some rock minerals by the action of the hydrofluoric acid generated the core UCS was reduced by 12.7%. This reduction in the rock strength was reflected by the reduction in the elastic parameters e.g; elastic Young's modulus was reduced by 4.6%, the shear modulus was reduced by 3.63% and finally, the Poisson's ratio was reduced by about 5%. All these measured values can be found in Figure (6).

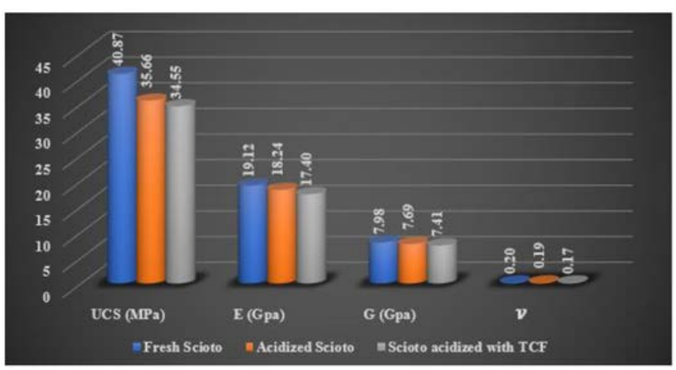

Figure 6—The core mechanical properties measured by the scratch test

On the other hand, reduction in the core mechanical and elastic properties was more in the case of using the TCF with the <SC>ISGA. This higher reduction is expected due to the effect of the pressure pulse generated on the core structure as well as due to the microfractures generated inside the core. In this case, the reductions in the core UCS, E, G and (ν) were about 15.5%, 9%, 7.14%, and 15% respectively. In order to investigate the effect of the reduction in the rock mechanical properties after acidizing on the core integrity.

A sand production prediction model after (Tixier et al., 1975) based on the ratio of the shear modulus (G) over the bulk compressibility (C_b). Rock bulk compressibility is another elastic modulus than can be calculated from Eq. (6). Based on Tixier model, if the $\frac{G}{C_b}$ ratio is greater than $38 \times 10^{18}\ Pa^2$, then the sand production problem is less reliable to take place. $\frac{G}{C_b}$ ratio was calculated for the fresh Scioto core, the one that was treated with the <SC>ISGA only and the one that was treated with the <SC>ISGA and the thermochemical fluids, see Table (3).

Table 3—The shear modulus to bulk compressibility ratio for the tested cores

	G/C_b (10^{18} Pa^2)
Fresh Scioto	84.21
Acidized Scioto	74.48
Scioto acidized with TCF	66.04
Tixier Safe limit	38.00

It was expected that the reduction in the rock Young's modulus, Poisson's ratio and bulk modulus would cause a decrease in the $\frac{G}{C_b}$ ratio. Based on the results shown in Table (3), the sand production problem is not expected to take place after the proposed treatment. This means the reduction in the rock mechanical properties after the acid treatment and even after the use of TCF could not harm the core integrity.

$$C_b = \frac{3(1-2v)}{E} \tag{6}$$

Conclusions

A novel sandstone acidizing technique was developed by generating in-situ hydrofluoric acid in the presence of thermochemical fluids. The following conclusions can be drawn from this study:

- Oxidizing ammonium halide salts such as (NH_4F) with a suitable oxidizer is capable of generating hydrofluoric acid that can be used for treating damaged sandstone reservoirs.
- The reaction of the thermochemical fluids can generate enough heat and pressure that catalyzes the acid-generating reaction and create some microfractures. This leads to higher porosity and permeability enhancement.
- NMR scans show the presence of microfractures generated after the use of TCF.
- There were a reduction in the rock static and dynamic mechanical parameters after the acid treatment. However, this reduction was still in the safe limit and will not lead to sand production problems.

Acknowledgment

All acknowledgment and appreciation go to the College of Petroleum & Geosciences, KFUPM for their support and funding this research. The author thanks his advisor, Prof. Mohamed Mahmoud, for his generous guidance and consultancy. Thanks and gratitude are extended to Dr. Amjed Hassan, Dr. Zeeshan Tariq and Mr. Mahmoud Elsayed for their tremendous help with the experimental lab work.

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