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ADSORPTION AND SQUEEZE PERFORMANCE OF PAMAM-PGLU INHIBITORS FOR SILICATE SCALE MITIGATION DUE TO ASP FLOODING

Bee Chea Tan[1*], Ismail Mohd Saaid[1], Siti Qurratu' Aini Mahat[2], Suzalina Zainal[3], Astriyana Anuar[4], Petrus Tri Bhaskoro[3]

[1]Department of Petroleum Engineering Department, Universiti Teknologi PETRONAS, Malaysia
[2]Faculty of Chemical and Process Engineering Technology, Universiti Malaysia Pahang, Malaysia
[3]PETRONAS Research Sdn Bhd, Bangi, Malaysia
[4]Project Delivery and Technology Division, PETRONAS, Malaysia

*E-mail: tan_22000121@utp.edu.my

ABSTRACT

Silica and silicate scale deposition in the production systems resulted in reduced production rates and equipment damages. Chemical scale inhibition using squeeze method is advantageous for petroleum reservoir conditions. This paper aims to assess the adsorption and squeeze performance of developed scale inhibitors that consist of polyamidoamine (PAMAM) dendrimers and pteroyl-L-glutamic acid (PGLU). Commercial inhibitor, diethylenetriamine penta(methylene phosphonic acid), also known as DETPMP, was also used to benchmark the laboratory findings. Static adsorption test and core flooding were performed to investigate the adsorption characteristics and retention of the inhibitors in Berea sandstone cores. Simulation was also conducted to estimate the squeeze performance of the inhibitors. Results revealed that PAMAM-2-PGLU scale inhibitor exhibited the highest adsorption and retention in the sandstone core. The treatment of PAMAM-2-PGLU and PAMAM-1-PGLU inhibitors increased the core permeabilities. SQUEEZE IV model predicted that both PAMAM-PGLU inhibitors yielded longer squeeze lifetime than DETPMP inhibitor. Both experimental and simulation results showed good fit in terms of adsorption and squeeze lifetime. In this paper, the tested PAMAM-PGLU scale inhibitors have demonstrated better adsorption, retention, and squeeze lifetime in sandstone formation. They could offer effective and low-toxicity alternatives to commercial inhibitors due to raising environmental concerns.

Keywords: *Amine, green scale inhibitor, polyamidoamine dendrimer, formation damage, sandstone, cEOR*

INTRODUCTION

Chemical-enhanced oil recovery (cEOR) is known for recovering residual oil remains in the reservoir. Surfactants, polymers, and alkalis are commonly used in the cEOR processes. Combining these chemicals is also employed to suit the reservoir characteristics and overcome shortcomings during the process [1]-[2]. Alkaline surfactant polymer (ASP) flooding is a promising oil recovery technique for mature oilfields. The main purpose of the alkali in the ASP slug is to react with crude oil to increase its pH and generate soap for interfacial tension reduction. Sarbast et al. [3] summarise that sodium hydroxide alkali, sodium dodecyl sulfate surfactant, and hydrolysed polyacrylamide polymer were used in a typical composition of ASP slug. However, alkaline solutions in sandstone formations have raised concerns about silicate scaling. This situation has been reported in several oilfields where silicate scale was deposited inside the tubing, such as in Daqing and Alberta [4]-[5].

Fink [6] mentions that the alkaline slug in ASP is commonly at pH 11 and above. When it contacts the rock surfaces for a sufficient time, it consequently dissolves the quartz mineral on the rock surfaces [7]. The dissolved quartz mineral is present in the form of monomeric silica in a solution ($Si(OH)_3O^-Na^+$)

that is usually stable in a high pH environment [8]. Unfortunately, the solubility of monomeric silica decreases as it flows towards the production region. The alkaline solution, which consists of monomeric silica, is partially neutralised by the pH-neutral formation water. In this case, the soluble monomeric silica polymerises at a reduced pH and forms colloidal silica. This colloidal silica then aggregates further and creates amorphous silicate scales. This scenario is illustrated in Figure 1. Sazali et al. [9] added that colloidal silica tends to form metallic silicates in the presence of excessive divalent cations in water formation, such as magnesium and calcium ions. Hence, it is essential to manage silicate scaling when deploying an alkali-based chemical flooding.

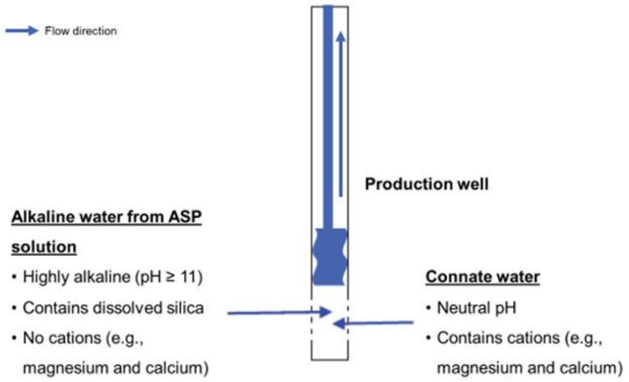

Figure 1 Mixing of two different waters in a production well (modified) [10]

Chemical treatment is one of the scale control techniques in petroleum production operations. Different scale inhibitors are available, including chelating agents, dispersants, and anti-scalants. To name a few, Liu et al. [11] mention the usage of diethylenetriamine penta(methylene phosphonic acid) (DETPMP) and polyphosphino-carboxylic acid (PPCA) are common in oilfield operations. Although they had demonstrated promising scale inhibition over a wide range of pH and higher temperatures, they were not intended to mitigate the formation of silicate scales in the first place. Instead, DETPMP and PPCA are primarily used to inhibit carbonate, sulfate, and phosphate scales [12]. On top of that, it is also reported that the inherent biodegradability of DETPMP was less than 40% in 28 days, while PPCA was reported as non-biodegradable [13].

Owing to rising environmental concerns and discharge restrictions, environment-friendly or green scale inhibitors were developed for silicate scale control. Spinthaki et al. [14] listed some examples, such as phosphonated chitosan, cationic polyethyleneimine, anionic carboxymethyl inulin, neutral polyethene glycol, and methacrylate-based polyampholyte. This paper is interested in investigating another green scale inhibitor developed from polyamidoamine (PAMAM) dendrimers and pteroyl-L-glutamic acid (PGLU). The developed scale inhibitors by Mahat and Saaid [15] were found effective in silicate scale inhibition with the bottle test method. This paper aims to investigate further the adsorption and squeeze performance of these PAMAM-PGLU scale inhibitors.

ADSORPTION AND RETENTION OF SCALE INHIBITOR

Scale inhibitor squeeze treatment is an effective technique to mitigate the deposition of scales inside the wellbore and production tubing. Several factors need to be considered when designing the treatment program, such as scale types, formation characteristics at near wellbore regions, and chemical properties of scale inhibitors. Squeeze treatment is performed in four stages: pre-flush, treatment slug, over-flush, and shut-in [16]. A specific scale inhibitor concentration, usually above its minimum inhibitory concentration (MIC), is injected into the formation to prevent scale deposition adequately. A successful scale inhibitor squeeze treatment can prevent expensive scale removal operations and maintain good productivity.

Adsorption and desorption equilibrium are among the many criteria for scale inhibitor application [6]. Adsorption occurs due to interactive forces between the adsorbate and absorbent, including Van der Waals forces, electrostatic attractions, ionic bonding, covalent bonding, and hydrogen bonding [17]. During squeeze treatments, the adsorption of scale inhibitor is prevalent for its retention in the formations, which is directly related to its squeeze lifetime [18]. During the shut-in stage, the scale inhibitor is anticipated to adsorb onto the grain surface and be evenly distributed throughout the formation to form a protective layer. Besides that, it shall also remain in the formation when the well starts producing again. Scale inhibitors with strong retention would yield a longer squeeze lifetime, thus indicating an effective scale inhibition.

Adsorption of scale inhibitors in sandstone formation is a complex process that depends on various factors such as the concentration of scale inhibitors, the rock's chemical composition, the fluid's pH, and the temperature [19]. Although the previously developed PAMAM-PGLU scale inhibitors had shown effective silicate scale inhibition, their applicability for squeeze treatment is still yet to be evaluated. Therefore, this work investigates the adsorption and retention of the developed scale inhibitors on sandstone formation on the laboratory scale and later predicts their squeeze lifetime based on the laboratory results obtained.

METHODOLOGY

Materials

Berea sandstone cores were used in this paper, and one was crushed for a static adsorption test. Synthetic formation water was prepared with salt composition provided in Table 1. Different concentrations of scale inhibitors were prepared in synthetic formation water. The formulation of green silicate scale inhibitors was adapted from Mahat and Saaid [15], namely PAMAM-1-PGLU and PAMAM-2-PGLU. Meanwhile, DETPMP was used for the experimental benchmark. Then, the scale inhibitor solutions were adjusted to nearly pH 7.0 before any experiment to mimic near-wellbore conditions.

Static Adsorption Test

25 grams of the crushed Berea core was soaked in 5000-ppm scale inhibitor solutions and stirred for 15 minutes before placing them in the oven. The volume of the scale inhibitors solution was 75 ml. The treatment process lasted for 24 hours at 100°C. The adsorption capacity of scale inhibitor on rock surface can be calculated using the following equation:

$$q = \frac{(C_0 - C_e) \times V}{m}$$

where q is the adsorption capacity in milligrams per gram (mg/g), C_0 is the initial concentration of adsorbate in milligrams per litre (mg/L), C_e is the equilibrium concentration of adsorbate in milligrams per litre (mg/L), V is the volume of the adsorbate in a litre (L), and m is the mass of adsorbent in gram (g).

Inductively coupled plasma-optical emission spectroscopy (ICP-OES) was used to determine the concentration of silica from Berea core filtrate before and after the treatment of PAMAM-1-PGLU and PAMAM-2-PGLU scale inhibitors. Recalculation was done to determine the adsorption capacity of these scale inhibitors. Meanwhile, the concentration of DETPMP inhibitor solution before and after the treatment process was characterised using ultraviolet-visible spectroscopy (UV-Vis).

Core Flooding Experiment

Core flooding was performed to determine the adsorption and retention of the scale inhibitors in sandstone formation. This experiment used the Formation Evaluation System (FES350, Vinci Technologies) at 100°C under 1000-psi net confining stress and 200-psi backpressure. This experiment condition was consistent with this paper's static adsorption test and the previous work's dynamic experiment [20]. The schematic diagram of the setup is provided in Figure 2.

Three Berea cores were cleaned and dried overnight. Then, their initial porosity and absolute permeability were measured and provided in Table 2. After that, the cores were saturated with formation water for seven

Table 1 Composition of synthetic formation water and scale inhibitors adapted from Mahat and Saaid [15]

Synthetic Formation Water	Concentration (ppm)	Scale Inhibitor (ppm)	Concentration
Sodium chloride	2950	Polyamidoamine, first generation (PAMAM-1)	50
Sodium bicarbonate	1800		
Magnesium chloride	1500	Polyamidoamine, second generation (PAMAM-2)	10
Calcium chloride	300		
Sodium sulfate	300	Pteroyl-L-glutamic acid (PGLU)	5000
Potassium chloride	80		
Barium chloride	30		
pH	8.59	Volume ratio (v/v%)	60:40

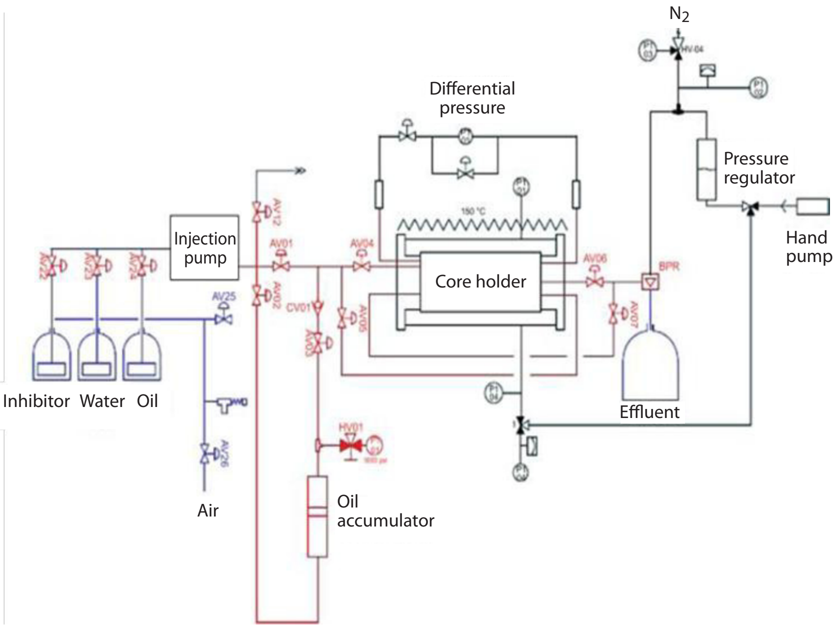

Figure 2 Schematic diagram of Formation Evaluation System (FES350)

days using a saturator. Lithium chloride was added into the formation water and scale inhibitor solutions at 10 ppm and 50 ppm, respectively, for tracing before the core flooding experiment. The concentration of scale inhibitors was prepared at 5000 ppm in formation water.

To begin with, Berea core F1 is installed in the core flooding system. The sequence of fluid injection is divided into four stages: preparation, pre-treatment, main treatment, and post-treatment, as illustrated in Figure 3. The fluids were injected into the core at 5 mL/hr until steady-state pressure was obtained. Then, the core was shut in for 24 hours after the main treatment stage. The effluents were evaluated before and after scale inhibitor treatment with the aid of ICP-OES spectrometry. The same procedure was repeated for Berea cores F2 and F3.

Table 2 Porosity and permeability of Berea cores and scale inhibitors tested

Berea Core	Porosity (%)	Absolute Permeability (mD)	Scale Inhibitor
F1	23.90	1019	PAMAM-1-PGLU
F2	23.01	1068	PAMAM-2-PGLU
F3	25.50	1076	DETPMP

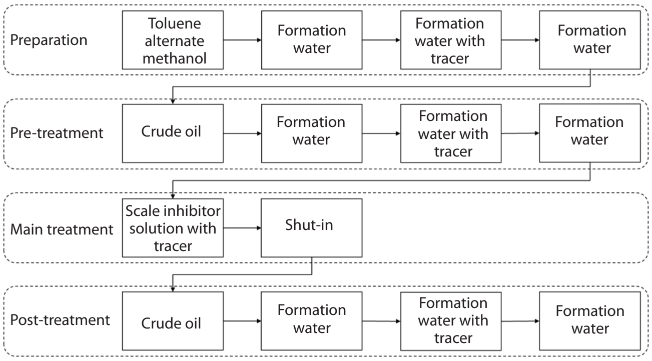

Figure 3 Sequence of fluid injection in core flooding

Prediction of Squeeze Performance

The squeeze performance of scale inhibitors was predicted using SQUEEZE IV software. This software assumes a linear isotherm in the adsorption and desorption process. In order to develop and validate an isotherm, the required input parameters were obtained from core flooding data that includes the total injection volume of scale inhibitors and concentration of scale inhibitors in the effluent. The minimum inhibitory concentration (MIC) for scale inhibitors is set at 10 ppm, adapted from Mahat et al. [20]. The input parameters for squeeze prediction are provided in Table 3.

Table 3 Inputs for SQUEEZE IV modelling obtained from core flooding experiment

Parameter	PAMAM-1-PGLU	PAMAM-2-PGLU	DETPMP
Core dimensions, L × D (cm)	7.52 × 3.82	7.47 × 3.82	7.56 × 3.82
Effective water porosity of core (fraction)	0.24	0.23	0.25
Density of rock grain (g/cm^3)	2.65	2.65	2.65
Volume of inhibitor injected (ml)	21.5	20.5	21.0
Active concentration of scale inhibitor injected (ppm)	5000	5000	5000
The concentration of the first sample after shut-in (ppm)	4600	4550	4700
MIC (ppm)	10	10	10
Throughput to reach MIC (PV)	1200	1420	800
Flow rate (mL/hr)	5	5	5
Duration (hr)	2	2	2
Temperature (°C)	100	100	100
Shut-in (hr)	24	24	24

RESULTS AND DISCUSSIONS

Adsorption Capacity of Scale Inhibitor

Table 4 summarises the adsorption capacity of scale inhibitors on crushed Berea and their respective zeta potential values. PAMAM-2-PGLU scale inhibitor adsorbed the most to grain surface with an adsorption capacity of 0.94 mg/g. Then, PAMAM-1-PGLU scale inhibitor ranked the second highest adsorption with 0.85 mg/g. Meanwhile, the DETPMP scale inhibitor absorbed the least on the sandstone core with an adsorption capacity of 0.63 mg/g.

Table 4 Adsorption capacity of scale inhibitors on crushed Berea

Scale Inhibitor	Zeta Potential (mV)	Adsorption Capacity (mg/g)
PAMAM-1-PGLU	– 0.3	0.85
PAMAM-2-PGLU	– 0.7	0.94
DETPMP	– 2.3	0.63

Scale inhibitors tend to adsorb onto the grain surface through physical adsorption, which is Van der Waals and electrostatic forces [21]-[22]. The maximum adsorption occurs when one is a positive charge, and the other is a negative one. However, the scale inhibitor solutions were found to have a negative charge, and water-wet sandstone also has a negative charge in nature. Thus, the repulsion force seems dominant in this experiment. Hence, the scale inhibitors and sandstone are expected to repel each other, resulting in a small adsorption capacity of less than 1.0 mg/g. In addition, PAMAM-2-PGLU scale inhibitor had the highest adsorption capacity even though its zeta potential magnitude is higher than the PAMAM-1-PGLU scale inhibitor. It might happen because the second-generation polyamidoamine (PAMAM-2) contains more surface primary amino groups than the first-generation polyamidoamine (PAMAM-1), so it has a more extensive functional surface area to adsorb onto the gain surface, thus increasing its adsorption capacity in the crushed Berea core.

Adsorption and Retention Potential of Scale Inhibitor in Berea Core

Figure 4 plots the normalised concentration profiles of scale inhibitors and lithium-ion tracers in effluents (C/C_o) for core floods F1, F2, and F3 throughout the main treatment and post-treatment stages. In general, all scale inhibitors fully saturated the core at a smaller inhibitor volume, denoted by a steep increment of concentration profiles from zero to 5 PV inhibitor injections, and in the middle, inhibitor concentration profiles remained plateau after 5 PV, which indicates that scale inhibitors had achieved their maximum adsorptions on the grain's surface. After the 24-hour shut-in period, the reduction in concentration profiles represents the scale inhibitors' desorption from the grain's surface. It can be observed from the exact figure that scale inhibitors detached rapidly from the grain's surface in the early phase of the post-treatment process.

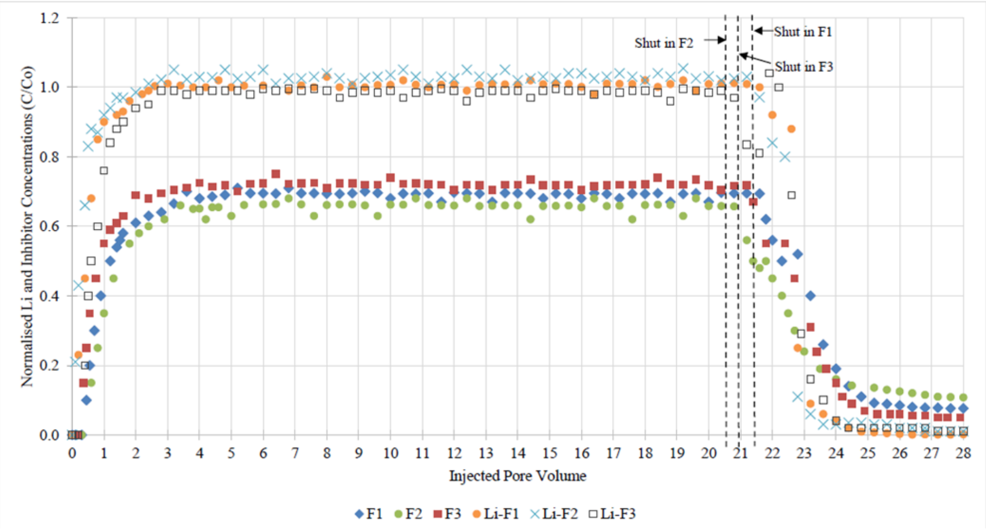

Figure 4 Normalised concentrations of PAMAM-1-PGLU (Li-F1), PAMAM-2-PGLU (Li-F2), and DETPMP (Li-F3) scale inhibitors with lithium-ion tracer (C/C_o) against their injected pore volumes (PV) for core floods F1, F2, and F3

Based on the normalised concentration profile above, the PAMAM-2-PGLU scale inhibitor (denotes Li-F2) demonstrated the highest adsorption in the Berea core. Its large retardation was inferred with lithium-ion tracer effluent before shut-in. Then, PAMAM-1-PGLU scale inhibitor (denotes to Li-F1) had the second highest adsorption, and finally, DETPMP scale inhibitor (denotes to Li-F3) possessed the least adsorption in Berea core based on their return concentration of lithium-ion tracer. This dynamic core flooding result supports the earlier static adsorption test that the PAMAM-2-PGLU scale inhibitor had the highest adsorption, followed by the PAMAM-1-PGLU scale inhibitor, and lastly, the DETPMP scale inhibitor that had the least adsorption in Berea sandstone core.

Adsorption of scale inhibitors onto the grain's surface is often the predominant mechanism in scale inhibitor squeeze treatment. Moreover, scale inhibitors with stronger adsorption are also expected to possess lower desorption and could retain longer in rock formation when a well starts to operate again. This scenario usually happens in the later phase of the post-treatment stage, and a high volume of post-flush brine is typically needed to detach the strongly adsorbed scale inhibitor from the grain's surface.

Due to this, the potential of scale inhibitor retentions in the later phase of the post-treatment stage can be interpreted from Figure 5. It presents the concentration profiles of scale inhibitors that remained in the core during the last phase of the post-treatment stage until they reached the 10-ppm minimum inhibitor concentration (MIC). In this figure, the PAMAM-2-PGLU scale inhibitor (denotes Coreflood F2) showed the slowest desorption in the core. It required about 1420 PV of post-flush brine to drop until 10 ppm MIC. On the other hand, the PAMAM-1-PGLU scale inhibitor (denotes Coreflood F1) had the second slowest desorption, which needed about 1200 PV of post-flush brine to drop to 10 ppm. Lastly, the DETPMP scale inhibitor (denoted Coreflood F3) had the highest desorption in the core, which required about 800 PV of post-flush to drop until its MIC. Scale inhibitors with higher retention potential can be deduced from slower desorption in the core during the post-treatment process.

The core flooding result reveals that scale inhibitors with higher adsorption capacity would exhibit slower desorption and higher retention in the Berea core. PAMAM-2-PGLU scale inhibitor is anticipated to possess the most extended squeeze lifetime among the tested inhibitors. Although DETPMP is used for corrosion and scale inhibition, its performance may be hampered by sandstone instead of carbonate, which has been discussed in previous works [23]-[24].

Potential of Formation Damage Due to Treatment

Formation damage or permeability impairment is also a concern to ensure good productivity after inhibitor squeeze treatment. The brine permeability of the Berea cores was measured before and after the main

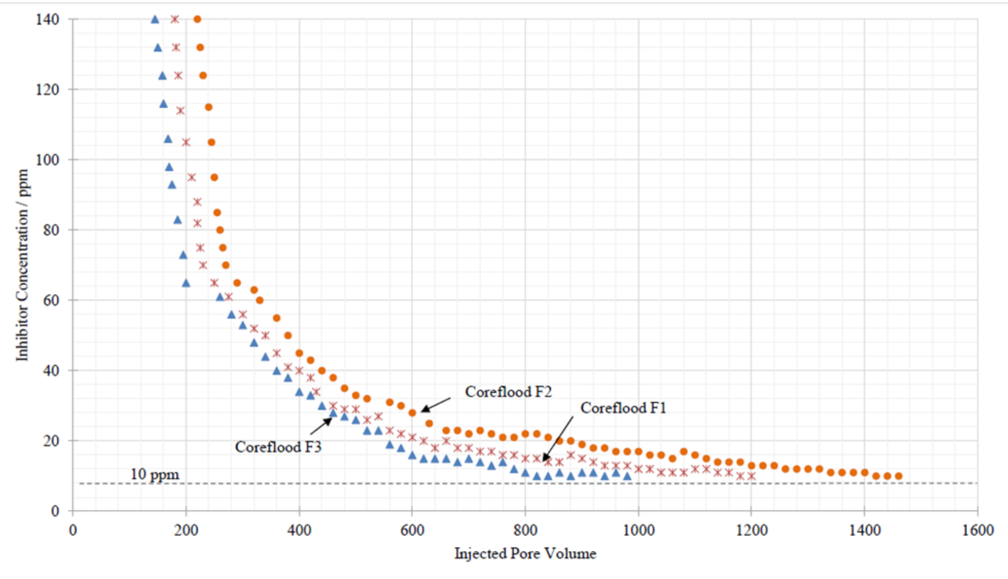

Figure 5 Concentration of PAMAM-1-PGLU, PAMAM-2-PGLU, and DETPMP scale inhibitors against injected pore volume in the later phase of the post-treatment stage for core floods F1, F2, and F3, respectively

treatment stage. All three cores showed permeability increments after the main treatment. The changes in the core's permeability are recorded in Table 5. Core flood F2 (PAMAM-2-PGLU) had the highest permeability increment, followed by core flood F1 (PAMAM-1-PGLU), and lastly, core flood F3 (DETPMP) had the least increment in permeability. The causes of permeability increment due to inhibitor injection remain unclear and require further assessment.

Table 5 Changes in permeability of Berea core before and after the main treatment stage

Berea core	Permeability before inhibitor injection (mD)	Permeability after inhibitor injection (mD)	Changes in permeability (%)
F1	634	954	50.47
F2	645	998	54.73
F3	576	762	32.29

Prediction of Scale Inhibitor Squeeze Performance

The corresponding prediction curves from the core flooding experiment are shown in Figure 6. The excellent correlation between the experimental and calculated data suggests that the derived adsorption isotherm for scale inhibitor selection was valid.

To elaborate further, core floods F1 (PAMAM-1-PGLU) and F2 (PAMAM-2-PGLU) demonstrated a closer alignment between their modelling and experimental results compared to core flood F3 (DETPMP). This is likely due to the potent influence of divalent ions in the interaction between the scale inhibitor's functional groups and the rock, which are magnesium and calcium ions (Mg^{2+} and Ca^{2+}), in this paper. This is apparent in the case of injecting PAMAM-PGLU scale inhibitors in a core flooding experiment. Therefore, a single adsorption isotherm may not entirely portray the process in this scenario.

On top of that, the retention of scale inhibitors can also be predicted from the simulation, as shown in Figure 6. The retention of scale inhibitors can be interpreted by determining the brine required to reduce the scale inhibitor concentration to 10 ppm in the post-flush. A higher volume of post-flush is expected for scale inhibitors with stronger retention in the formation. Based on the predictions, the PAMAM-2-PGLU scale inhibitor had the strongest retention, requiring 1350 PV of post-flush to reach the minimum 10-ppm concentration. PAMAM-1-PGLU scale inhibitor required 800 PV of post flush, while DETPMP scale inhibitor required 520 PV of post flush to reach the same minimum concentration. Hence, using SQUEEZE IV software, the PAMAM-2-PGLU scale inhibitor is expected to exhibit the longest squeeze lifetime in

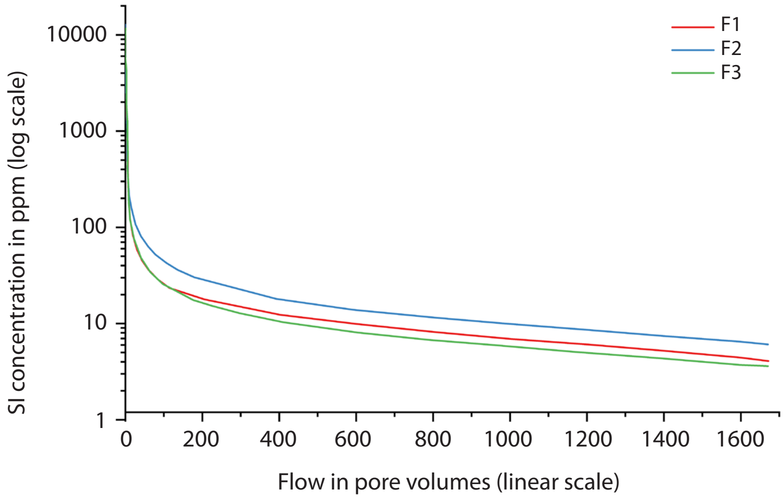

Figure 6 Predicted post-flush brine injected in pore volumes for PAMAM-1-PGLU (F1), PAMAM-2-PGLU (F2), and DETPMP (F3) scale inhibitors to reach 10-ppm inhibitor concentration (MIC)

sandstone formation. This prediction is also in line with the core flooding experiment.

CONCLUSION

The adsorption and squeeze performance of scale inhibitors were investigated in this work. It was found that the previously developed PAMAM-2-PGLU scale inhibitor yielded the highest adsorption and retention in sandstone formation. Based on modelling, it is also expected to have the longest squeeze lifetime among the tested inhibitors. Meanwhile, the phosphonate scale inhibitor, DETPMP, showed slightly poor adsorption and squeeze performance compared to the formulated PAMAM-PGLU scale inhibitors. The formulated scale inhibitors also improved the permeability of the Berea core. Based on the laboratory results, PAMAM-PGLU scale inhibitors could be offered as environment-friendly alternatives due to phosphonate disposal regulations. Nonetheless, more studies can be carried out to understand the characteristics of the formulated PAMAM-PGLU for future application of scale inhibition.

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