

Evaluation of Green Silica Scale Inhibitors Performance Using Dynamic Tube Blocking Test

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ARTICLE INFO

Article history:

Available online xx xx xx

Keywords:

scales

scale inhibitors

dynamic tube blocking

MIC

silicate scale

ABSTRACT

The formation of scales is considered a harmful and expensive problem in the oil and gas industry. Many different types of scales can be formed during oil and gas production, such as calcium carbonate, iron sulfide, barium sulfates, and silicate scales. Scale precipitation can cause a number of issues, including reduced oil and gas production, formation damage, plugging the oil-producing formation matrix, blockages in perforations or gravel packs, restricted/blocked flow lines, safety valves, pump wear, and corrosion underneath deposits. Scale inhibitors are the most effective approach for reducing and preventing scale deposition. In this work, dynamic tube blocking is used to determine the efficiency of chemical inhibitors against silicate scales formed through the minimum inhibitor concentration (MIC). The brine water sample was prepared to simulate the scale formation, and the scale inhibitors were then used to evaluate the minimum inhibitor concentration for scale build-up. From the study, the MIC values for poly(amidoamine)-1 and poly(amidoamine)-2 were determined to be 10 mg/L and 500 mg/L for PGLU, respectively. Meanwhile, the mixture of poly(amidoamine)-1/pteroyl-L-glutamic acid and poly(amidoamine)-2/pteroyl-L-glutamic acid scale inhibitors also has shown good performance by increasing the time for scale build-up. Interestingly, it was found that the mixture of silica scale inhibitors performed better than the scale inhibitors without mixing. Based on the results obtained, the mixture of poly(amidoamine)-2/pteroyl-L-glutamic acid should be recommended as a silica scale inhibitor that can efficiently and effectively inhibit silica formation.

1. Introduction

Silicate scaling is a common problem in sandstone reservoirs when alkali is used in water flooding (Gbadamosi *et al.*, 2019). As the high pH alkaline flood moves through the sandstone formation, quartz silica is dissolved as monomeric silica (Roostaei *et al.*, 2020). This dissolved monomeric silica is stabilised by the high pH alkaline water (Park *et al.*, 2020). However, when the alkaline water reaches the production well, it encounters neutral pH connate water from other production regions. Colloidal silica is formed as a result of a fall in pH that significantly lowers the solubility of monomeric silica in the mixed fluids (Lunevich, 2019). In addition, this colloidal silica prefers to form a metallic silica scale when exposed to metal ions.

In connate water, magnesium has been shown to bridge colloidal silica particles and form a magnesium silicate scale. In addition, the production of calcium carbonate may also have an impact on the deposition of silica. Although the calcium carbonate scale does not promote deposition by supplying nucleation sites, it does provide a matrix for silica to be trapped (Ueckert *et al.*, 2017). According to this theory, silica and silicate may also be formed and deposited in this way.

Silicate scale is very difficult to remove from oil wells, and mechanical removal was frequently the only available remedial method in the past. Many wells had rods that were tightly scaled into the tubing, and some had to be abandoned if silicate scaling became severe. As a result, numerous polymers and other chemicals such as phosphates, esters, organic phosphonates, polyacrylates, carboxylates, and sulfonates have been developed to inhibit silicate scales (Mady & Kelland, 2020). Nevertheless, most conventional scale inhibitors are toxic and harmful to human health, water sources, marine and freshwater organisms (Lusinier *et al.*, 2019). The environmental effects of scale inhibitors are exacerbated when they are mixed with other chemicals during exploration, drilling, well completion, and start-up operations. Due to stricter regulations, the industry has shifted from the use of traditional scale inhibitors.

Since the late 1990s, various attempts have been made to develop more environmentally friendly scale inhibitors, and a growing number of such scale inhibitors are becoming commercially available (Marzorati *et al.*, 2019). Over the past 15 years, recent environmental awareness has contributed to creating and implementing more environmentally friendly scale inhibitors, called 'Green Scale Inhibitors' (GSI) (Jafar Mazumder, 2020). These GSIs are designed to have high biodegradability characteristics to reduce water pollution around oil production systems. Besides, the use of cationic polymers as a green scale inhibitor has also recently been the subject of numerous inhibition studies (Tan *et al.*, 2020). The efficacy of cationic poly(amidoamine) dendrimers as scale inhibitors was found to rely strongly on the branching in the dendrimer's backbone (Martins *et al.*, 2020). Experimental findings reveal that the efficacy of inhibition largely depends on dendrimer structural characteristics, such as the nature of end groups and the number of generations, as well as the dosage levels (Dariusz *et al.*, 2017). The inhibitory effect of mixture anionic polymers and cationic polymers has also been studied by Hernandez, Hassani and Nassef, 2017. The results showed that the scale inhibitor with two or more functional groups effectively handles scale problems. The carboxylic derivatives are usually synthesised as anionic derivatives and are considered the most effective because of their high solubility (Solmi *et al.*, 2019). It was found that the interactions between dissociated and undissociated groups of the cationic and the anionic tend to prevent the crystal growth sites (Vologzhanina, 2019).

2. Literature Review

Alkaline flooding is an enhanced oil recovery technique in which an alkaline chemical is injected during waterflooding or polymer flooding operations (Lwisa, 2021). Surfactants are formed inside the reservoir when an alkaline chemical combines with certain types of oils. Surfactants eventually diminish the oil-water interfacial tension, resulting in an increase in oil production (Belhaj *et al.*, 2020). Nevertheless, Sun *et al.*, (2020) found that the employment of alkaline floods in sandstone reservoirs increased the production of silicate scales. It was found that high-pH solutions dissolved silica in the rock formation into monomeric silica, as revealed by Lunevich, (2019) in his study. According to Lucy, (2019), the hydrolysis of silica-oxygen-silica bonds results in the release of silicic acid, $[\text{Si}(\text{OH})_4]$, and silicates into the aqueous phase. In an oil production system, silicate scale can offer a significant challenge since it can form in perforation tunnels, casing surfaces, and tubular. For example, in China's Daqing field, ESP failures owing to blockage by silicate scale have caused a number of operational issues (Guo *et al.*, 2018).

In most situations, the use of scale inhibitors is the optimal strategy for preserving the productivity of the well. Scale inhibition is a chemical treatment used to monitor production well and delay or reduce the scale from forming (Mpelwa & Tang, 2019). Scale inhibitors mainly prevent precipitate from adhering to solid surfaces like pipes and vessels by absorbing onto the crystal surface. In order to manage and prevent scale formation and subsequent deposition, scale inhibitors (SI) have previously been the recommended downhole treatment. (Al-Rawahi *et al.*, 2017). Besides, Mazumder, (2020) studied that the use of scale inhibitors also could lower the rate of scale formation to nearly zero.

Previously, researchers from academia and the oil industry have made significant progress in recent years in attempting to take up the task of using “green products” that are biodegradable, low toxicity, and hydrothermally stable (Mostafa *et al.*, 2018). New groups of compounds that are not based on phosphorous chemistry and are less toxic have been introduced into the marketplace. These polymers are based on acrylates, such as polyacrylamide and polyacrylic acid (Hahladakis *et al.*, 2018). Polyaminoacids and poly aspartate also have recently been suggested, studied, and developed as silica scale inhibitors in various industries, as stated by Kelland, 2018. In addition, some new chemicals, including carboxylated plants, polysaccharides, and other natural compounds, have also been proposed by Barclay *et al.*, (2019). Although the use of green silica scale inhibitors is a relatively unexplored field in oil and gas wells, several studies have been done on this “promising alternative” (Al-Rawahi *et al.*, 2017).

Moreover, the use of cationic polymer as a silica polymerisation inhibitor has also recently been the subject of numerous studies (Li *et al.*, 2020). Asl, Baba and Demir, (2017) reported in their study that cationic-based copolymers were effective as silica polymerisation inhibitors. Similar conclusions were also reported by Kazi, (2020) in his investigation on the use of cationic polymers and surfactants under geothermal conditions in inhibiting silica polymerisation. Aside from that, extensive silica inhibition has also been done using two dendrimer inhibitors, poly(amidoamine)-1 and poly(amidoamine)-2 (Pawlaczyk & Schroeder, 2020). These inhibitors show excellent performance in the water treatment industry. The backbone of poly(amidoamine) dendrimers (PAMAM) is made of amide bonds, making them biodegradable. As a result, they are obviously innocuous compounds. The generation number of a dendrimer shows its pace of growth and branching. More precisely, dendrimers of PAMAM generations 0.5, 1.5, and 2.5 have –COOH termini, whereas dendrimers of generations 1 and 2 have –NH₂ termini. The previous studies also reported that the –COOH terminated dendrimers showed virtually no activity as silica inhibitors (Gosika & Maiti, 2018). In contrast, the –NH₂ terminated analogues (generations 1 and 2) were potent SiO₂ scale inhibitors (Mady *et al.*, 2021). However, no comprehensive studies have been conducted in testing the effectiveness of cationic poly(amidoamine) in reducing silica scale formation under dynamic conditions for use in oilfields. Moreover, the effect of other operational parameters such as pH, temperature, pressure and metal ions on the formation of silica scale is also not widely studied in dynamic conditions. Hence, this study is important in finding a starting point in developing silica scale inhibitors that can be used in dynamic conditions without affecting oil production.

3. Methodology

The chemical reagents used for synthetic brines preparation were NaCl (99.0%), KCl (99.0%), NaHCO₃ (99.7%), Na₂SO₄ (99.0%), CaCl₂·2H₂O (99.0%), MgCl₂·6H₂O (99.0%) and BaCl₂·2H₂O (99.0%). Silica supersaturated stock solutions were prepared from Na₂SiO₃·5H₂O (95.0%) and stored in plastic bottles. These chemicals are analytical reagent grade and used without further purification. The dynamic tube blocking process was carried out in this study using Process Measurement and Control (PMAC) Systems Ltd equipment. The PMAC method is used to assess the efficacy of scale inhibitors in actual simulated conditions (de Souza *et al.*, 2019). This system's main premise is to combine cationic and anionic solutions in a scaling coil and measure the pressure differential across the scaling coil (Al Helal *et al.*, 2019). The increased differential pressure indicates the presence of scale in the test coil (Martins *et al.*, 2020). The system's operating principle is visualised in Figure 1.

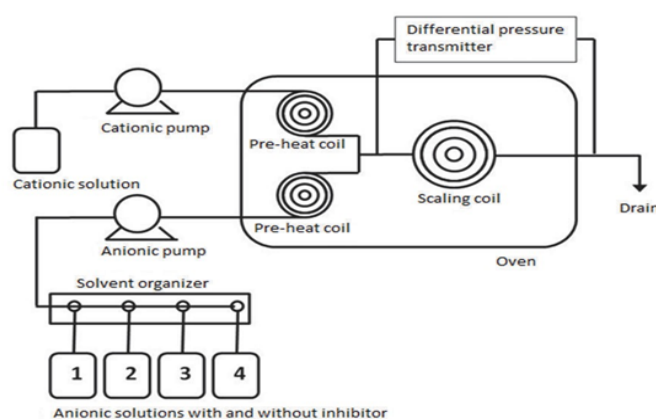


Figure 1: Operational principle of the PMAC instrument.

In PMAC, the anionic and cationic pumps are HPLC piston pumps. The solvent organiser prior to the anionic pump enables dosage adjustment of the scale inhibitor. Additionally, the solvent organiser contains an anionic solution free of inhibitors, an anionic solution containing the necessary concentration of inhibitors, deionised water, and a cleaning solution. In this study, the brine waters were pumped into the heating coil separately. This ensures that brines achieve the test temperature before they mix. After passing the heating coil, the anion and cation brine were mixed at the T-junction of the scaling coil's inlet. Then, the difference in pressure between the intake and output was measured. Increases in differential pressure have been linked to scale accumulation in the scaling column. The standard test conditions are set as below:

Scaling coil length	: 1 m
Scaling coil internal diameter	: ~1 mm
Coil	: Alloy 600
Brine flow rate	: 10 ml/min
Back pressure	: 200 psi
pH	: 7
Pass criteria	: < 1psi rise in DP over set time
Temperature	: 100 °C

In this experiment, the scale rig was designed to test the inhibitors automatically. It was programmed to complete three stages in each experiment. Blank runs without the scale inhibitor were performed first to determine the brine scaling time. If the scaling rate was too slow, the silicate concentration would be adjusted. The test duration for subsequent inhibitor runs was about three times the blank scaling time. The brines' pH was adjusted to pH 7, and then the two brines were filtered and preheated through coils and pumped at a combined flow rate of 10 ml/min (5 ml/min for each). Scale inhibitor concentration was increased stepwise, starting from blank until injection pressure (DP) remained constant, and no blocking of the tube was observed for at least 15 minutes of flow. The minimum inhibitor concentration (MIC) was defined as being between the highest inhibitor concentration, which failed to inhibit scale formation and the lowest inhibitor concentration, which prevented scale formation (Martins *et al.*, 2020). The MIC was recorded for all inhibitors used in each brine system. The scale inhibitors' concentrations used were 10, 50, 100, 500, 1000, and 5000 ppm. The system was cleaned thoroughly with acetic acid and deionised water after each run with the given inhibitor concentrations. These steps were then repeated by using mixture scale inhibitors.

4. Finding and Analysis

Precipitation of various inorganic salts is an issue that occurs during oilfield operations in reservoirs, downhole equipment, and wellheads (Khurshid *et al.*, 2020). This could result in pump failures, a decrease in output rate, and formation damage. The precipitation of silica scale in the reservoirs and producing wells frequently happens for a variety of reasons. The silica scale was formed primarily as a result of a pH change that increased the concentration of ions in the solution above their equilibrium concentration. In this study, a series of dynamic tube blocking tests were performed to assess the performance of PAMAM-1, PAMAM-2, PGLU, and mixture PAMAM-1/PGLU and PAMAM-2/PGLU scale inhibitors in preventing silica scale precipitation. The dynamic tube blocking tests have been carried out to determine the minimum inhibitory concentration (MIC) of the scale inhibitor package. The MIC is defined as the minimum inhibitor concentration required to prevent a predefined increase in ΔP over a particular time. During the solution's injection into the tube, differential pressure was increased by silica formation in the coil with time. In this method, the test duration for each inhibitor concentration was fixed at 120 minutes. Results obtained from this dynamic measurement often give rise to a different ranking than tests performed in static conditions. The differences are explained in terms of residence times which are much longer in the static method (a few hours) than in the dynamic method (a few minutes).

In this study, the changes in ΔP for the blank sample without the scale inhibitor were noticed after 7 minutes. Therefore, the scale inhibitor must resist scale deposition (determined to be a rise in DP of 1 psi) for 21 minutes (3 times the blank scale-up time) according to evaluation criteria. In the absence of scale inhibitors, the formation of silicate scale occurred rapidly.

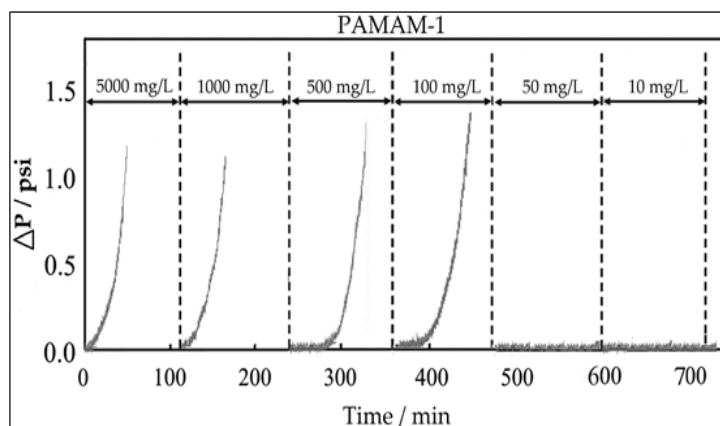


Figure 2: MIC for PAMAM-1.

However, with scale inhibitors in the solution, it was observed that the scaling time had been extended. Wang *et al.*, (2018) discussed that the extended scaling time was caused by either the postponed silica precipitation or the changes of precipitate's shape and surface property, which delayed the silica deposition or slowed the growth rates. In this research study, the tests were started with a concentration of 5000 ppm and gradually decreased to 1000, 500, 100, 50, and 10 ppm. The MIC of PAMAM-1 and PAMAM-2 are plotted in Figures 2 – 3.

From the results obtained, it can be seen that PAMAM-1 inhibitor failed at 5000 mg/L after 45 minutes, 1000 mg/L after 60 minutes, 500 mg/L after 72 minutes, 100 mg/L after 86 minutes, and 10 mg/L after 81 minutes. However, PAMAM-1 passed at 10 and 50 mg/L, as shown in Figure 2. There was no deposition in the scaling coil at 10 and 50 mg/L, as indicated by the ΔP profile. Meanwhile, the MIC value for PAMAM-2 inhibitors was also found at 10 and 50 mg/L as well, as shown in Figure 3. This was demonstrated by no increase in the ΔP profile for both concentrations. However, it was observed that the increases in ΔP exceeded 1 psi after 60 minutes at 5000 mg/L, 82 minutes at 1000 mg/L, 103 minutes at 500 mg/L, and 115 minutes at 100 mg/L. At 50 mg/L, ΔP was 0.2 psi after 120 minutes. This experimental work found that the scaling was not prevented even at the highest inhibitor concentration. Previously, it was found that the excessive use of PAMAM dendrimers will cause agglomeration with silica and thus deactivate the scale inhibitor. This deactivation causes an increase in the silicate scale formation in the solution. Although, Lunevich, (2019) discovered that cations were extremely effective at inhibiting silicate scale formation due to their ability to attract silicate anions. However, in this study, the excess of cationic scale inhibitors in the mixing waters has created an oversaturated cations solution for salt content. As a result, the solution can no longer hold this amount of salt in the water and form an insoluble complex that precipitated out of the solution as a solid. Mahmud & Ermila, (2020) also demonstrated that the insoluble salts could precipitate in the mixing zone due to chemical species in the injection water reacting with excess scale inhibitors as a result of temperature change.

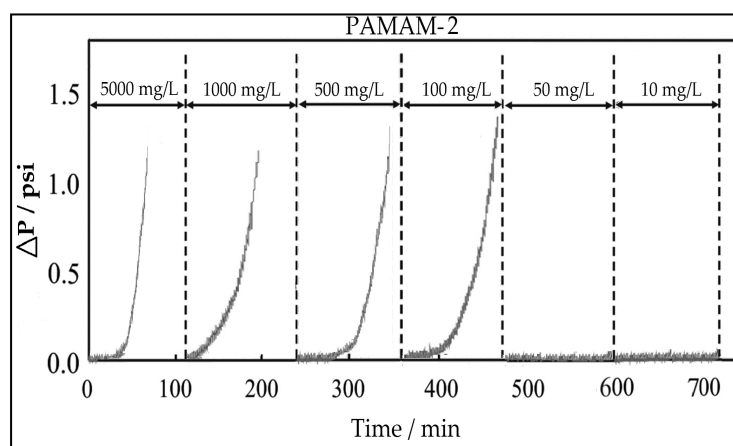


Figure 3: MIC for PAMAM-2.

Therefore, it can be concluded that a sufficient amount of scale inhibitor was important to prevent the formation of scales effectively. Scale inhibitors in excess of what is required to prevent scale formation successfully will create flocculation between the scale inhibitor and the scale formation. In this study, the excess of cationic PAMAM-1 and PAMAM-2 scale inhibitors in the solution had caused the inhibitor's flocculation with the colloidal silica matrix. Previously, Farrell *et al.*, (2018) proposed a mechanism that showed an increase in turbidity was due to flocculation between treated silicate scales with untreated silicate scales, where the untreated silicate scales were blocked by steric crowding, as illustrated in Figure 4. It can be seen that from Figure 4, the excessive cationic scale inhibitors in the solution have attracted more anionic silicate, resulting in flocculation. This indirectly deactivated the cationic scale inhibitor to interact with other untreated silicate anions. Lazaro *et al.*, (2017) also discovered that despite the excellent performance of PAMAM-1 and PAMAM-2 as colloidal silica growth inhibitors, these dendrimers had disadvantages where the uninhibited silicate led to the formation of large colloidal silica particles that entrapped the dendrimers. As a result, the active inhibitor depleted from the solution, leading to a decrease in bulk inhibitory action. Visual examination of the test containers reveals that these particles appear as white flocculant precipitates at the bottom.

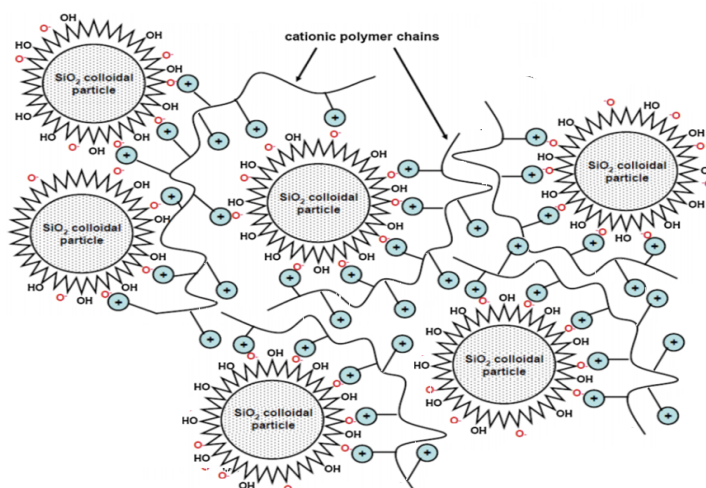


Figure 4: Schematic diagram of the formation of cationic inhibitor-colloidal silica flocculation.

Meanwhile, for PGLU, it was found that scaling times increase gradually with increasing inhibitor concentrations, as shown in Figure 5. At 10 mg/L, ΔP started to increase after 80 minutes, at 50 mg/L, ΔP started to increase after 99 minutes, while at 100 mg/L, ΔP started to increase after 80 minutes and reached 0.2 psi at the end of the test run. However, it can be seen that no ΔP increases were observed at 500, 1000, and 5000 mg/L. This was in contrast to PAMAM dendrimers. PGLU is a zwitterion compound composed of cationic and anionic groups compared to PAMAM-1 and PAMAM-2, composed of the cationic group only. It was believed that cationic and anionic groups' presence assists the scale inhibitory action by alleviating flocculation and formation of insoluble precipitates. The cationic group aid in preventing the formation of colloidal silica in the solution, whilst the anionic group assist in dispersing silicate anions. Therefore, PGLU exhibits the highest and more effective silica scale inhibitor than PAMAM-1 and PAMAM-2. In this study, the MIC values for PAMAM-1 and PAMAM-2 were 10 mg/L, while the MIC value for PGLU was 500 mg/L, respectively. Nevertheless, a high amount of PGLU was required to inhibit silica polymerisation effectively. Besides, Zhou *et al.* (2019) discussed that the high concentration was unsuitable for reservoirs because it might create other problems such as aggregation with minerals in the reservoir.

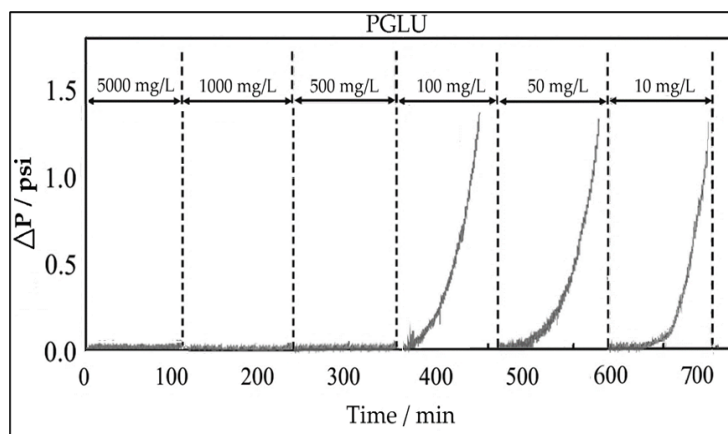


Figure 5: MIC for PGLU.

Furthermore, in this research study, PAMAM-1 and PAMAM-2 also have been mixed with PGLU to enhance the inhibition of the silicate scale. Zhang, (2020) previously reported that effective silica scale inhibitors could be achieved by combining cationic scale inhibitors with an anionic polymer. This is based on a delicate balance structure of cationic–anionic charges. Therefore, in this study, 40 v/v % of 5000 ppm of PGLU was utilised with 60 v/v% of 50 ppm of PAMAM-1 and 10 ppm of PAMAM-2 to enhance the inhibition performance. Figure 6 shows the effect of formulated PAMAM-1/PGLU and PAMAM-2/PGLU on differential pressure obtained against the silicate brine system. From the results obtained, it can be seen that both the mixture of PAMAM-1/PGLU and the mixture of PAMAM-2/PGLU were passed after 240 minutes. It was found that the differential pressure increments for 10 mg/L of mixture PAMAM-1/PGLU started at 290 minutes, while the differential pressure increments for 10 mg/L of mixture PAMAM-2/PGLU started at 320 minutes which were 41 and 45 times higher than the blank scale-up time. The mixtures PAMAM-1/PGLU and PAMAM-2/PGLU showed superior effectiveness as a silicate scale inhibitor by prolonging the time required for scale formation compared to PAMAM-1 PAMAM-2, PGLU, and blank. It was believed that PGLU assists the inhibitory effect of PAMAM-1 and PAMAM-2 by alleviating the flocculation of SiO₂-PAMAM precipitates. This is most likely due to colloidal silica dissolution by the mixture of PAMAMs/PGLU, as discussed by Spinthaki, Kamaratou, Skordalou, *et al.*, (2021).

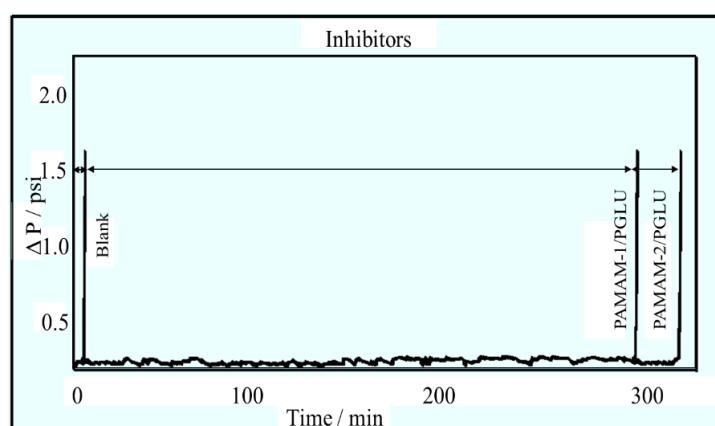


Figure 6: The effect of 60:40 v/v% of PAMAM-1/PGLU and 60:40 v/v% of PAMAM-2/PGLU on differential pressure obtained against silicate brine system.

Figure 7 shows the possible mechanism of dissolution of colloidal SiO₂ as studied by Chen *et al.*, (2018). The initial silica-inhibitor interaction is an electrostatic attraction between the negatively charged silica particle and the cationic ammonium of the PAMAM dendrimer. The positioning of cationic ammonium in such a way deprotonated the carboxylate group in PGLU. The negatively charged carboxylate group then swings and binds to the Si centre's surface and enhances the Si-O bond's cleavage. This function is "mimicking" the action of OH⁻ anions in the Si-O-Si network's hydrolysis, as studied by Issa and Luyt (2019). Sarver *et al.*, (2019) also confirmed

in their study that the first two generations of $-NH_2$ terminated analogues were potent SiO_2 scale inhibitors. PAMAM-2 has more $-NH_2$ terminated groups (cationic) than that of PAMAM-1. Thus, PAMAM-2 has more active sites to interact with anionic silicate and reduces scale formation.

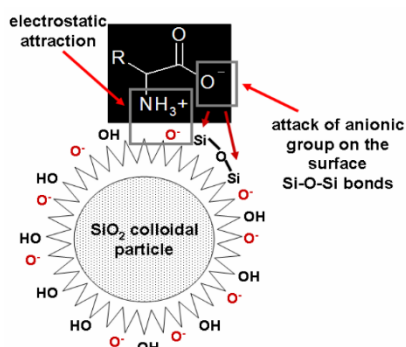


Figure 7: The possible dissolution mechanism of colloidal SiO_2 in the presence of mixture PAMAM/PGLU inhibitors (Chen *et al.*, 2018).

In addition, the study found that the mixtures of scale inhibitors were extremely stable and effective at preventing silica scale formation at high temperatures (100 °C). According to Tomasz and Waks, (2018), temperature affects the formation of silica in two ways; the solubility of monomeric silica increases with increasing temperature, or the rate of silica polymerisation increases with increasing temperature. From the results obtained, it can be observed that temperature also affects the dispersion of colloidal silica. However, no significant studies from the literature can support this hypothesis. Besides, most of the present studies involved surface modification of silica to facilitate agglomeration. Therefore, this study could be a benchmark for future research on the dispersion of silica in the solution at static conditions as well as at dynamic conditions. Furthermore, compared to other methods, the dynamic tube blocking test successfully simulates oilfield production in this study.

5. Conclusion

NH_2 -terminated PAMAMs showed superior inhibitory activity on the silicate scale in this study, as compared to the control. PAMAM-2 has more $-NH_2$ terminated groups than PAMAM-1, based on dendrimer structure. Because of this, the results suggest that PAMAM-2 is the most effective in silica polymerisation. It is proven that the cationic charge on the dendrimer backbone is required for effective silica scale inhibition. Additionally, it is demonstrated that inhibitor dosage has an effect on inhibition performance. Excess dendrimers may result in the production of large colloidal particles, depleting the active inhibitor from the aqueous medium. In this study, PAMAM-1 and PAMAM-2 has a MIC value of 10 mg/L while PGLU has a MIC value of 500 mg/L. In addition, it was discovered that the cationic–anionic interactions also helped the inhibitors to work better. PAMAM-1 and PAMAM-2 in combination with PGLU have been demonstrated to have a strong inhibitory effect on the formation of SiO_2 . By decreasing flocculation and the development of insoluble SiO_2 -PAMAM precipitates, it was hypothesised that PGLU enhanced PAMAM-1 and PAMAM-2 inhibitory effect. This was confirmed by the positive results of the dynamic tube blocking tests. Mixtures of PAMAM-1/PGLU and PAMAM-2/PGLU have been found to be superior silicate scale inhibitors in this study by significantly increasing the time required for scale build-up, compared to PAMAM-1, PAMAM-2, PGLU and blank.

Acknowledgment

The authors would like to acknowledge the Politeknik Tun Syed Nasir Syed Ismail and Universiti Teknologi PETRONAS Petroleum Engineering Department and FRGS Grant for providing the fund and facilities to accomplish the experimental work for this research.

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