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Characterisation of silicate scale formation: The effect of pH on silica/silicate scale formation

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Abstract

One of the challenging situations that engineers have been encountered in the oil and gas industry is the formation of silicate scale during alkaline surfactant polymer (ASP) flooding which could be throughout the overall production system, from upstream to downstream processes. In the ASP flooding, the ASP slug is injected into the reservoir at a high pH, which is later mixed up with the connate water causing the pH condition inside the reservoir to be reduced. This reduction of pH will aggravate the precipitation of silicate scaling as this type of scale is very much pH-dependent, with the presence of the metallic ion worsening the situation. The main objectives of this study are to characterise and compare (with the pure commercial) the precipitates produced from the Si/Mg system at various pH conditions using spectroscopic analysis, as well as to study the mechanism of silicate scale formation. The precipitates were reproduced from a-Si/Mg scaling brine at room temperature under various pH test conditions. The spectroscopic results confirmed the variation of pH as it propagates within the reservoir from the injection to the production well, producing a different type of morphology. The results obtained showed that pH affects the types and morphology of the precipitation significantly, where the formation of silica/silicate scale is most severe when the value of pH increases whilst Mg-silicate scaling favours pH 8.5.

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1.0 Introduction

Scale is defined as deposit of inorganic compounds that are caused by the presence of fluids in a system at least partially man-made, by which silicate scale formation is nothing uncommon encounter in the oil and gas industry. Silicate scaling usually occurs and found mainly in three main fields which are alkaline surfactant polymer (ASP) flooding, geothermal brine, and industrial water (Kashpura & Potapov, 2000; Rodríguez, 2006; Amjad & Zuhl, 2008; Basbar et al., 2013; Umar & Saaid, 2014; Guo et al., 2017; Lu et al., 2018).

Generally, during alkaline flooding, the water usually has a pH of 11 or higher when it sweeps the fluid within the reservoir. This high pH water usually dissolves quartz in the formation, producing dissolved monomeric silica ($Si(OH)_3O^-Na^+$) ion, along with the waterflood which remains soluble and stable in this high pH environment. However, this pH is reduced as this high pH ASP sludge propagates along the reservoir and comingles with the neutral pH connate water (Alexander et al., 1954; Gill, 1993; Gill, 1998).

As the ASP flood starts maturing, most of the ASP flooded oilfields experienced serious silicate scale in the production wells. Problems related to the silicate scale are different from the previous traditional scales (Arensdorf et al., 2010). The mechanism of silica formation scale is complex and requires high and in-depth knowledge of scaling fundamentals (Meyers, 1999; Ning, 2002; Meyers, 2004; Icopini et al., 2005; Brown, 2011; van den Heuvel, 2018) which can be explained by the following stages:

i. Silica Dissolution: As the water flooding is alkaline, thus it creates high pH conditions at the reservoir and around the wellbore. The alkaline water with high pH will dissolve quartz in the formation resulting in monomeric silica as shown in Eq. (1)

$$Si(OH)_4\,(aq.) + OH^{-}(aq.) \leftrightarrow (OH)_3SiO^{-}(aq.) + H_2O\,(aq.) \qquad (1)$$

ii. Silica Polymerisation: The polymerisation of silica is controlled by pH. When the ASP water is mixed with the connate water that has neutral pH near the wellbore, the previously dissolved silica will start to polymerise, then form colloidal silica as the pH is partially neutralised.

$$Si(OH)_3^{-}\,(aq.) + Si(OH)_4\,(aq.) \leftrightarrow (OH)_3Si\text{–}O\text{–}Si(OH)_3\,(s) + OH^{-}\,(aq.) \qquad (2)$$

iii. Metal-silicate scale formation: The formation of the metal-silicate scale is due to the existence of magnesium in the solution. In ASP injection, water is softened to provide a buffer in the reservoir between the existing waters. After the softening process, the presence of remaining magnesium will precipitate as $Mg(OH)_2$. The magnesium presented was then interacted and precipitated with the neutral pH connate water, forming a magnesium silicate scale.

iv. Co-precipitation of silicate scale by other minerals: The introduction of calcium to the high pH ASP water will cause calcium carbonate to occur. Apart from that, it provides nuclei for the development of silicate scale.

The silicate scaling can occur at many points in the formation or within the wellbore and this will give adverse effects on the oil production facilities. This scaling can be found in the perforation tunnels, within the pipe, and clogging the equipment (Gunnarsson & Arnórsson, 2003; 2005).

The purpose of this research is to investigate the relationship between the silicate scale formation and the pH, to understand the complex mechanism of the silicate scale formation. Fig. 1 depicts the factors affected the rate of scaling, which pH gives the most significant effect on the rate of scaling as well as the types and morphology of the scale produced (Ahmed & Elraies, 2015; Ahmed et al., 2015; Basbar et al., 2013; Kazempour et al., 2012; Sonne et al., 2012).

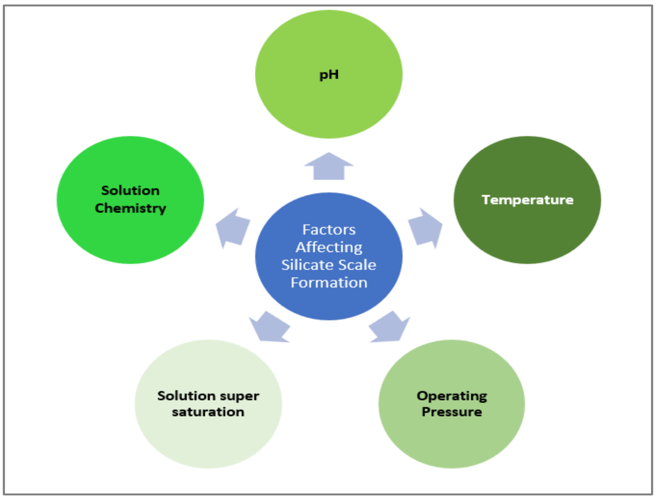

Fig. 1: Factors affecting the formation of silica scale

The function of the solution's pH is to determine the degree of alkalinity or acidity of the solution. As has been discussed before, the pH of a solution affects the rate of saturation and precipitation greatly (Utami et al., 2014; Sazali et al., 2015). Sazali et al. (2015) and Sazali (2018) reported that the scaling precipitation or behaviour is greatly affected by the pH, producing different results even though there are swift changes in the pH.

2.0 Methodology

2.1 Material

Magnesium chloride hexahydrate, $MgCl_2.6H_2O$, 99.0–101.0 % (CAS# 7791-18-6) from Merck; sodium metasilicate pentahydrate $Na_2SiO_3{\cdot}5H_2O$, ≥ 97% purity from (CAS# 10213-79-3), pure commercial magnesium silicate, $MgSiO_3$ (CAS# 1343-88-0) magnesium hydroxide, $Mg(OH)_2$, reagent grade 95% (CAS# 1309-42-8) and amorphous silicon dioxide, (SiO_2) 99.8% (CAS# 112945-52-5) were acquired from Sigma Aldrich.

2.2 Methods

Few references reported that the magnesium ions concentrations in the formation water varied from as low as 190 ppm to more than 3200 ppm (Fathi et al., 2010; Gupta el., 2011; Chandrasekhar & Mohanty, 2013) whereas Merdhah & Yassin (2009) reported the magnesium ion can be presented in the formation water in the range of 100 to 10,000 ppm. Moustafa & Shedid (2018) analysed the formation of water in the Gulf of Suez, Egypt where they measured the magnesium ions appeared to be about 1090 ppm. Akstinat (2019) reported that the amount of silica ion can be in the range of 1 to 100 ppm, which this amount can be escalated in the high pH as reported by Wei et al. (2011) where about 700 ppm was dissolved (from grundite and, alone) after only 40 days introduced in the NaOH. It is well known that the frame minerals in the sandstone reservoir are composed of kaolinite, grundite, chlorite, feldspar, and quartz.

The ASP system involved injecting the slug at an extremely high pH up to 12 that most of the silica is dissolved and stable at this high pH as a monomeric silicate ion (Sheng, 2014). But it was also reported that the solubility of monomeric silica is pH-dependent and decreases significantly below pH 10.5 (Amjad and Zuhl, 2008). The presence of the magnesium ions in the formation water (connate water) will further alter the solubility of the dissolved silica where Meyers (1999) reported that solubility decreases with pH when divalent cations were present. It is known that the magnesium will be softened to $Mg(OH)_2$ if reacted at a pH of more than 9 and allowing reacting at a pH of 8.5 will promote most of the Mg-silicate to form where Demadis (2010) reported that at pH less than 8, magnesium silicate is rarely observed in the deposit.

In this study, mixed brines of magnesium brine (MB) and silicon brine (SB) were prepared to replicate the ASP leachate where 1800 ppm magnesium brine was prepared by dissolving 75.3 gram of magnesium chloride hexahydrate $MgCl_2{\cdot}6H_2O$ salt in 5 L of distilled water, while 1880 ppm of silicon brine was prepared by dissolving 71 grams of sodium metasilicate pentahydrate $Na_2SiO_3{\cdot}5H_2O$ salt in another 5 L of distilled water.

In the first duplicate sample, 50 mL of MB was added to 50 mL of SB to produce an initial Si/Mg mixed concentration of 900 ppm of Mg^{2+} and 940 ppm Si^{4+} (940Si:900Mg). The pH of these brines was then adjusted to pH 8.5 and left reacted at room temperature for 22 hours. The second duplicate of Si/Mg brines with the same initial mixed concentration were left reacted at their natural pH, approximately ~11, and room temperature for 22 hours. While the third duplicate Si/Mg sample was left to react exactly in the same condition 2 for 22 hours before these mixed brines were adjusted to pH 8.5 and allowed to react for another 22 hours i.e., in a total reaction time of 44 hours. The test conditions are tabulated in Table 1.

Table 1 : Test conditions (all mixed brines allowed to react at room temperature)

Test condition	Adjusted pH @0 hr	Adjusted pH @22 hr	Total reaction time (hour)
1	pH 8.5		22
2	Not adjusted (~pH 11)		22
3	Not adjusted (~pH 11)	pH 8.5	44

The scale produced was then filtered and analysed by using Spectrum one Fourier transform infra-red (FTIR), and X'Pert PRO PANalytical X-ray powder diffraction, (XRD) (Keshavarz and Ahmad, 2013; Zulfiqar et al., 2015). The pure commercial compound of magnesium silicate, magnesium hydroxide, and silicon dioxide were also analysed to be used as a reference to the produced precipitates.

In the FTIR analysis, all samples, i.e., the three precipitates formed in the three test conditions and three pure commercials were crushed into a fine powder and tested on the base plate of the FTIR equipment. Acetone was used to clean up the base plate surface. This process was repeated before testing a new sample to ensure the sample did not mix up with the previous sample and affect the results.

All the samples were examined by X'Pert PRO PANalytical using Cu-κα radiation at a scan speed of 2.5/min. All samples were crushed and ground to a fine powder and sieved to ensure to be less than ~10 μm (or 200-mesh) in size. Then the sample was put into, packed, and pressed into the sample holder. They were then smeared uniformly onto a glass slide and placed into the spring-loaded clip and ready for scanning process. Upon completion, the result displayed on the display monitor was recorded.

3.0 Results and discussion

The deposition of silicate scale in downstream section and production facilities need to be superintended properly, otherwise, clogging will occur, hence damaging the equipment (Gunnarsson &Arnórsson, 2003; 2005). In turns added cost is expected for repair and cleaning of the scale formation to remove other impurities. Frequent shut down for cleaning and maintenance not only costs more money but also will cause a loss in profit as the production and process need to be stopped during maintenance (Bello, 2017). Apart from that, it also decreases the performance and the efficiency of the equipment.

3.1 Determination of the functional groups by using Fourier transform infra-red (FTIR)

Generally, an intense Si-O covalent bond can be observed at around 1000 to 1200 cm^{-1} which indicates the immense silica network. Next, the range from 780 to 810 cm^{-1} shows the Si–O–Si stretching. At 1000 to 1080 cm^{-1}, it shows the presence of the Si–O stretching while at 515 cm^{-1}, represents the Si–O–Mg bending.

Based on the spectra obtained in Fig. 2, shows that there was the presence of an inorganic compound at the main peak, 1006.78 cm^{-1} which was silicate and possibly hydrated. This phenomenon happened in regions 1100–950 cm^{-1}. The possible compounds that were present were silicates ions, SiO_4^{4-} and a water molecule, H_2O. The peak appearing in the 1636 cm^{-1}

band in Si/Mg scaling solutions belongs to the bending vibration of water molecules.

Comparing the precipitates formed in Test Condition 1 with the spectra of commercial magnesium silicate in Fig. 3; similar inorganic compound peak was present at 1014.80 cm^{-1}, either silica SiO_2 or silicate at region 1000–1130 cm^{-1}.

This region of 1000–1225 cm^{-1}, also indicated the presence of inorganic oxyanion, $(M_xO_y)z^-$, where M could be metal or non-metal. There are peaks at 789.14 and 675.00 cm^{-1} respectively, indicating the presence of a functional group of Mg–O–Si.

For the spectra shown for commercial amorphous silica (Fig. 4), the main peak at region 950–1100 cm^{-1} indicates the presence of the inorganic compound, possibly silicate and hydrated, SiO_4^-. This main peak is attributed to the Si–O bending vibration and the asymmetric vibration stretching of the bond of siloxane (Si–O–Si). The peak observed at 807.72 cm^{-1} is associated with vibration bending mode in O–Si–O.

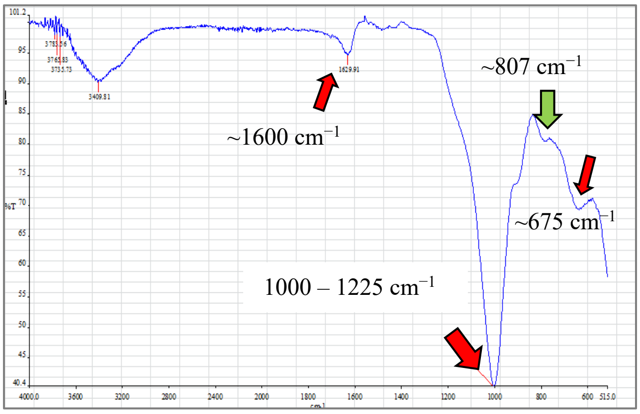

Fig. 2: FTIR spectra for precipitates formed in 940Si:940 Mg at room temperature (Test Condition 1)

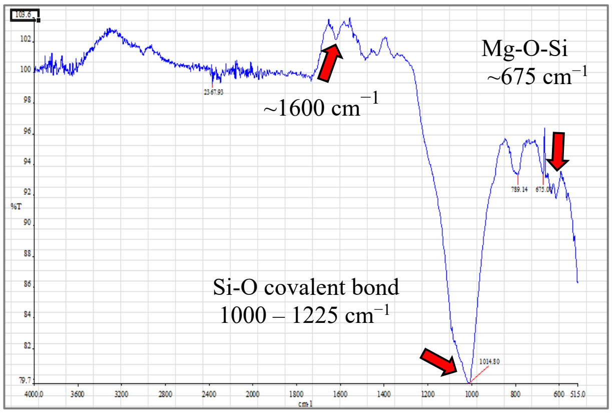

Fig. 3: FTIR spectra for commercial magnesium silicate

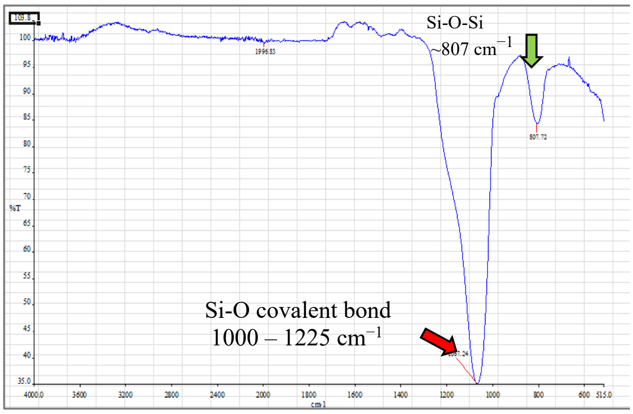

Fig. 4: FTIR spectra for commercial silica dioxide (amorphous silica) SiO_2

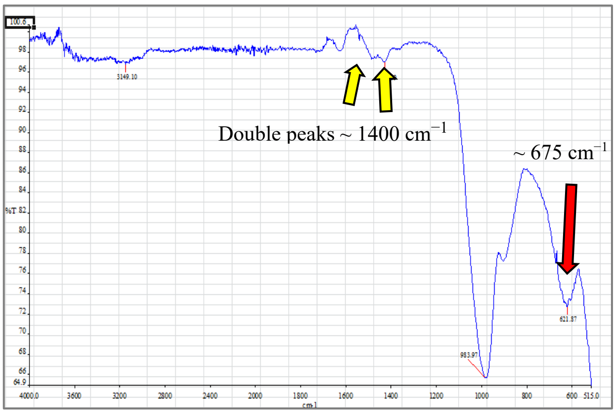

Fig. 5: FTIR spectra for precipitates formed in 940Si:940 Mg at room temperature (Test Condition 2)

Fig. 6: FTIR spectra for commercial magnesium hydroxide, $Mg(OH)$

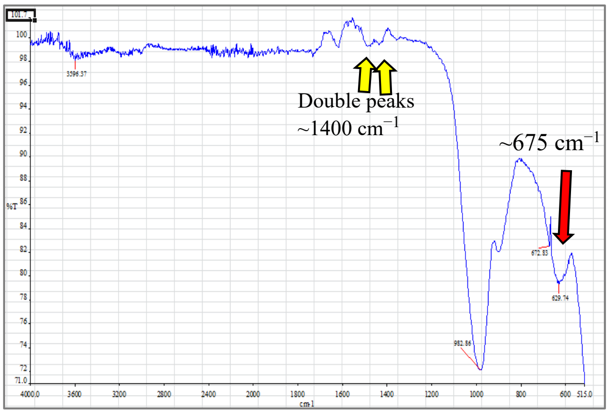

Fig. 7: FTIR spectra for precipitates formed in 940Si:940 Mg at room temperature (Test Condition 3)

Based on this evidence, we can postulate that the precipitate produced in Si/Mg at pH 8.5 (T_{room}) may resemble the Mg-silicate scale and amorphous silica. Based on the spectra obtained in Fig. 5, it shows that there were double peaks observed at 1400 cm^{-1} that matched the one observed in spectra Fig. 6 for commercial $Mg(OH)_2$. The Si–O covalent bond at around 1000–1200 cm^{-1} was seen to shift to ~980 cm^{-1}. It is proof that the precipitate produced in high pH11 (T_{room}) was a mixture of Mg-silicate scale, amorphous silica, and magnesium hydroxide. From the spectra recorded in Fig. 7, the precipitates that were produced in test condition 3, were the same as being produced in test condition 2.

3.2 Crystallographic study of the precipitates formed by using X-ray diffraction (XRD)

XRD is a powerful non-destructive technique used to find out the nature of the materials It provides information on structures, phases, preferred crystal orientations (texture), and other structural parameters, such as average grain size, crystallinity, strain, and crystal defects (Kohli & Mittal, 2019).

Based on the diffraction pattern plotted in Fig, 8 for the precipitates formed in Si/Mg brine at pH8.5, the diffraction signals at 2θ = 36.55°, 44.01°, 63.41°, and 76.53° were observed. The signals showed that the sample presented as a crystalline magnesium silicate (Joni et al, 2018) that complement the diffraction pattern observed in commercial magnesium silicate (Fig. 9). When Fig. 8 was further analysed, the broad peak at ~20 θ (Fig. 10) was not observed, which dictated that no amorphous silica was formed in this pH 8.5, or the Mg ions had bridged the silica backbone forming the Mg-silicate scale. The diffraction pattern for amorphous silica recorded in Fig. 10 illustrates that there was only one successive graph and does not have many peaks as others. This is because silica dioxide or also known as amorphous silica does not has a shape or is unstructured. In addition, the graph portrays that the peak was broad compared to other patterns.

From the diffraction pattern observed in Fig. 11, the diffraction signals appeared at 2 θ = 25.11°, 33.73°, 58.72°, and 70.48°. The signals can be said as broad and have asymmetric shapes, the elongation of the tails to higher at 2 θ. This shows that the material structure is disordered (Demir et al., 2014). When comparing these peaks with the commercial samples, it may be suggested that $Mg(OH)_2$ was also formed (Fig. 12) in addition to the amorphous silica and Mg-silicate scale.

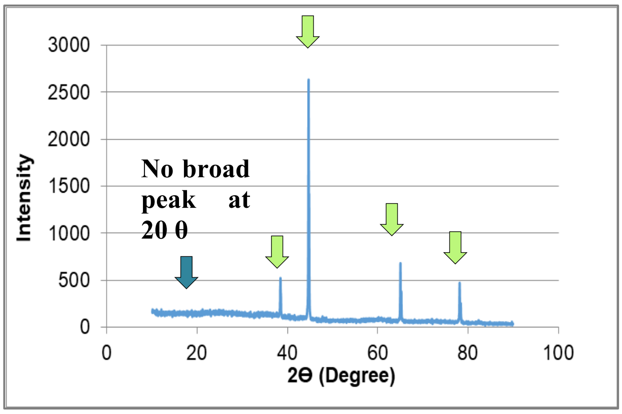

Fig. 8: Diffraction pattern for precipitate formed in 940Si:940 Mg at room temperature & pH 8.5 (Test Condition 1)

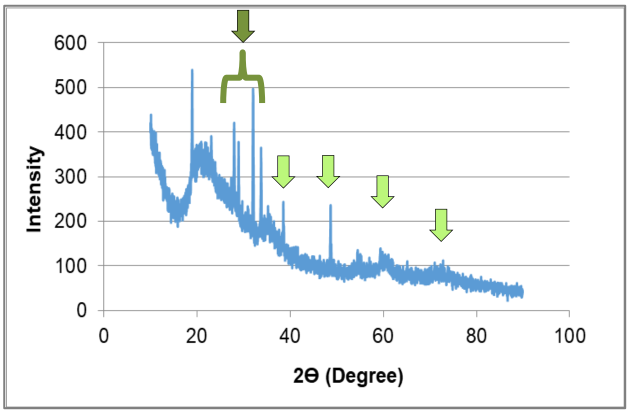

Fig. 9: Diffraction pattern for commercial magnesium silicate

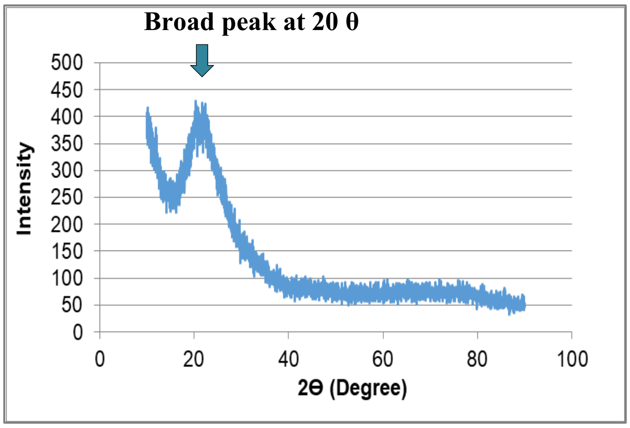

Fig. 10: Diffraction pattern for commercial amorphous silica

The precipitate formed in Test condition 3 produced similar peaks as observed in Fig.11, suggesting a similar type and morphology of the scale produced. However, it has slightly higher intensities as compared to Fig. 11; this may be due to the mixed brine being adjusted to pH 8.5 (after 22 hours) which promotes more microcrystalline Mg-silicate scale being produced as compared to precipitates formed in Test Condition 2. It is proven that time does give a great impact on crystallisation, but pH gives the most significant effect on precipitation and crystallisation. Prolonged idle time will broaden the peaks and decrease the intensities agreed to by Tavangarian & Emadi (2010).

Apart from that, the successive peaks from the sample at pH8.5 (Fig. 8) were higher compared to precipitates produced in a higher alkaline condition of pH 11 (Fig. 11 & Fig. 13). Thus, the hypothesis that can be made from this result is that Mg-silicate scaling formation does not favour alkaline conditions. More successive peaks may indicate a more crystallised structure as the structure diffract the signals. Apart from that, diffraction recorded in Fig. 8 showed that the intensities were higher, more than twice the intensities of prepared samples in the 'alkaline' (Fig. 11 & Fig. 13) condition.

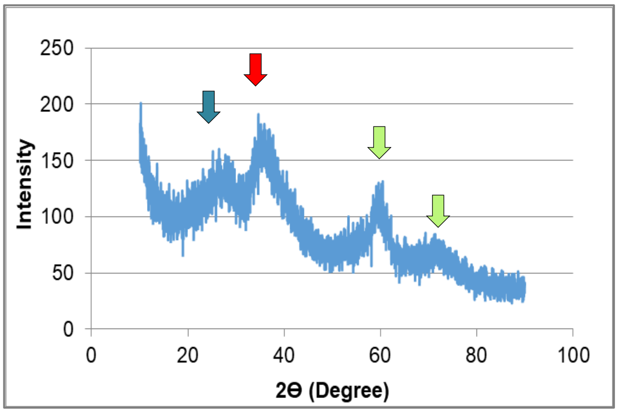

Fig. 11: Diffraction pattern for precipitate formed in 940Si:940 Mg at room temperature & pH 11 (Test Condition 2)

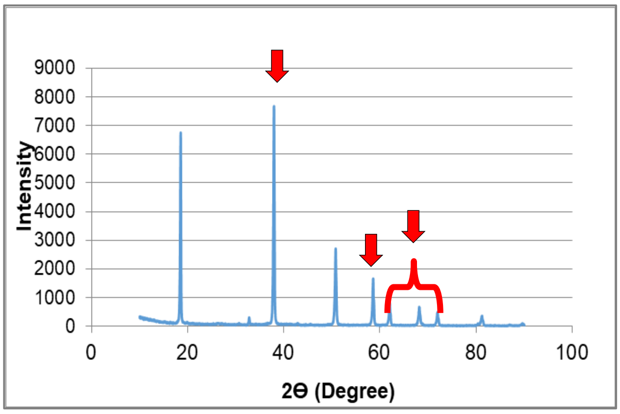

Fig. 12: Diffraction pattern for commercial magnesium hydroxide

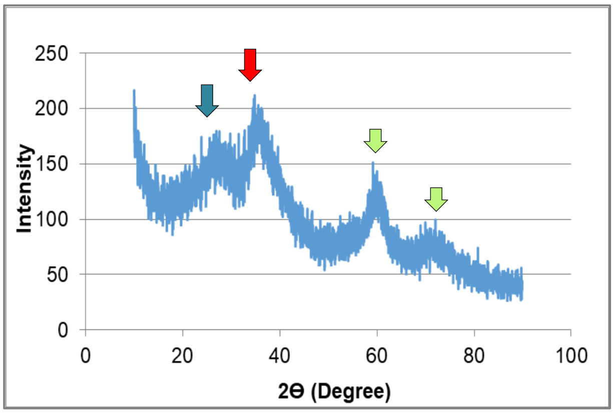

Fig. 13: Diffraction pattern for precipitate formed in 940 Si:940 Mg at room temperature (Test Condition 3)

4.0 Conclusions

The formation of silicate scale is a crucial issue that must be highlighted and worked on in the oil and gas industry. This research has achieved its main objective which is to study the effect of pH on silicate scale formation by characterising the scale produced by various spectroscopic analyses. FTIR and XRD analysis were able to analyse the chemical moieties and functional groups present in the test samples. It can be concluded that pH greatly affects the mechanism of silicate scale formation. However, a more comprehensive analysis should be done by extending the analysis using other spectroscopic techniques such as mass spectrometry, environmental scanning electron microscopy/energy dispersive X-ray (ESEM/EDAX), and X-ray fluorescence (XRF). It can be concluded that pH affects the formation of silicate scale greatly when the value of pH value increases. Based on the results obtained, it showed that the scale produced in pH 8.5 has more successive peaks, in both the FTIR and XRD analyses. This may indicate a more crystallised structure or more scale produced as the structure diffract the signals when compared to higher pH graphs. Thus, the conclusion that can be drawn from these results is that Mg-silicate scaling favours pH 8.5.

Generally, the pH condition of the reservoir is different from one point to another, and varied pH will result in a different rate of scaling, and the scale produced may differ morphologically. Apparently, the silicate deposits are difficult to be removed once they are formed, hence it is crucial to understand their formation mechanism and their morphology so that systematic management of this scale could be designed.

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