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Development and Assessment of the Synergistic Inhibition Efficiency of IA/AMPS Copolymers for Inhibiting $CaCO_3$ and $CaSO_4$ Co-production in Oilfield Scale

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Abstract. Polycarboxylate-scale inhibitors (PSI) are widely utilised in managing oilfield mineral scales, not only due to their high complexation, increased dispersion, and high thermal stability, but also due to their environmental compatibility, as they are phosphorus-free. However, they are effective in inhibiting only one specific scale. Thus, new non-phosphorus multifunctional SI is in demand immediately, where the scale can be made up of a combination of two or more minerals. Itaconic acid/ 2-acrylamido-2-methylpropane sulfuric acid (IA/AMPS) novel copolymer is synthesised via a free radical polymerisation to inhibit the $CaCO_3$ and $CaSO_4$ scales. The functional groups believed to be effective in inhibiting the scales were characterised using FTIR. The IA/AMPS performance was investigated to determine the optimal synthesis parameters/ conditions for the dispersion property. Results show that the best conditions for IA/AMPS copolymer preparation included a 1:2 molar ratio of IA to AMPS, 7% of initiator dosage and monomer total mass, a 4:1 molar ratio of ammonium persulfate (APS) to sodium bisulfite, 90 °C of reaction temperature, and 7 hours of the reaction period. Based on the qualitative observations, the IA/AMPS scale inhibitor effectively reduces the formation and surface adherence of both $CaCO_3$ and $CaSO_4$ scales. It significantly lowers the scale density and promotes the formation of less adherent, finer particles, especially under elevated temperatures. The combined effect of IA/AMPS and heat demonstrates a synergistic inhibition, particularly evident in the complete prevention of $CaSO_4$ scale at 80 °C.

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INTRODUCTION

One of the most important flow assurance issues affecting the oil industry is the oilfield scale. It is defined as the organic and inorganic compounds that form during deposition and are found in the produced brine. In oil and gas production systems, scale deposition is the accumulation of various materials in undesirable locations. These build-ups will prevent fluid from flowing through the reservoir's pore throats and block them. They not only have the potential to damage the formation, but also can and do coat perforations, casing, production tubulars, valves, pumps, and down-hole completion equipment, such as safety equipment and gas lift mandrels. It is crucial to predict component failure before it occurs, as it may lead to significant permeability losses, pressure drops, and severe formation damage issues (A. Rawahi *et al*., 2017; M. Merdhah, 2008; Hu *et al*., 2022). Scale deposition is a significant issue in the oil and gas industry because it can lead to problems that compromise the integrity of multiple pieces of production equipment and negatively impact production.

The sulphate and carbonate scales, formed by two distinct mechanisms, are the most common inorganic scales found in oil fields worldwide (A. Rawahi *et al*., 2017; M. Merdhah, 2008; Hu *et al*., 2022). As a result of the mixing of incompatible brines, specifically injected seawater and formation brine, sulphate scales such as barium sulphate ($BaSO_4$), strontium sulphate ($SrSO_4$), and calcium sulphate ($CaSO_4$), precipitate. Pressure drops in the production wells caused carbonate scales to form, releasing the dissolved CO_2 and causing the scales to precipitate. Calcium carbonate ($CaCO_3$), calcium sulphate ($CaSO_4$), and calcium phosphate ($Ca_3(PO_4)_2$ are the three most frequent species of scale that are found in the industrial water system (Cui and Zhang, 2019; Hosny *et al*., 2019; Mohammadinezhad *et al*., 2018; Olajire, 2015; Vazquez *et al*., 2016).

The use of scale inhibitors (SI), acidification, electrochemical methods, electromagnetic methods, and other strategies is employed to prevent scale formation. As electrochemical and electromagnetic methods are not relevant to large and complex systems like the oil and gas industries, adding custom-made scale inhibitors is currently an effective and reliable method of scale inhibition.

Polycarboxylate SI is widely used due to its phosphorus-free composition and has been found to combat the scale above effectively. However, depending on the circumstances, the scale may be composed of two or more different mineral scales. As a direct consequence, there is an immediate need for novel non-phosphorus multifunctional scale inhibitors. Itaconic acid (IA), a green monomer produced through biological fermentation, is recognised for its biodegradability and features two carboxylic groups, which indicate a good Ca^{2+} chelating capacity and effective prevention of $CaSO_4$/$CaCO_3$ deposition (Willke and Vorlop, 2001). However, the IA homopolymer's dispersion characteristic is poor, restricting its application. Whilst 2-acrylamido-2-methylpropane sulfonic acid (AMPS) is an anionic monomer having a sulfonic acid and an amide group, that is widely utilised. Copolymerization of IA with AMPS can

substantially improve its dispersion behaviour. By utilising molecular design to combine diverse functional groups, such as carboxyl, amide, and sulfonic acid, we hypothesise that the IA-AMPS copolymer not only exhibits excellent scale inhibition efficiency but also possesses a strong dispersing ability as an additive.

This hypothesis is grounded in Liu *et al*. (2019), which tested the IA/AMPS to inhibit $CaCO_3$ and $Ca_3(PO_4)_2$, finding that both scales were fully inhibited at dosages of 10 mg/L and 30 mg/L, respectively. Whilst Cui and Zhang (2019) incorporated allyl polyoxyethylene ether (APEG) into IA-AMPS copolymer, they found that the formulation of IA-AMPS-APEG was able to inhibit $CaCO_3$ and $CaSO_4$ scales at 94.35% and 85.69%, respectively, at 24 mg/L. Allyl polyoxyethylene ether (APEG), which is non-toxic and non-irritating, has reactive groups and is believed to react with active hydrogen or double bonds. For instance, studies have shown that the IA/AMPS is efficient in inhibiting $CaCO_3$/$Ca_3(PO_4)_2$ at high concentrations (Liu *et al*., 2019) and, by further modifying with APEG, and supplied at high dosages, is able to achieve more than 80% $CaCO_3$/$CaSO_4$ inhibition. Both studies suggest that a high dosage of either IA/AMPS or IA-AMPS-APEG may have the ability to stop the scaling reaction.

However, not all reported work specified the severity of the scaling conditions. In this research, we synthesised the IA/AMPS scale inhibitor under various synthesis conditions, specifically at 90°C for 7 hours. This scale inhibitor is further studied and evaluated for its effectiveness in inhibiting $CaCO_3$ and $CaSO_4$ scales in the oilfield environment. Following this, we tested the scale inhibitor under high scaling conditions, specifically with ratios of 3630 ppm Ca^{2+}:18035 ppm HCO_3^- and 1841.25 ppm Ca^{2+}:3603.75 ppm SO_4^{2-}, respectively.

To test this hypothesis, IA/AMPS copolymers were synthesised in this study to inhibit the formation of these inorganic scales. Based on what is known about these polymers, it is suggested that a combination of IA and AMPS groups can also effectively dissolve and prevent scale, as well as prevent the weak hydrophilic groups from forming insoluble calcium gel (Wang *et al*., 2018). IA is an unsaturated dicarboxylic acid. In the IA molecule, in addition to the functional group of the carbon double bond needed for free radical polymerisation, a carboxylic acid group is attached on both sides of the double bond, giving the copolymer excellent negative dispersion performance and the capacity for complexation with other ions. The presence of only carboxylic groups is insufficient to inhibit the growth of scale effectively, so carboxylic acid-type polymer scale inhibitors are modified with sulfonic acid groups. This combination of acidic groups effectively inhibits the formation of calcium gel (Wang *et al*., 2018). The introduction of AMPS, which contains sulfonic acid groups and utilises IA as the primary raw material, is proposed in this study, assuming it will not compromise the biodegradability of IA.

Itaconic acid contains two carboxylic acid groups (-COOH) that can effectively chelate with metal ions such as calcium, magnesium, and barium. IA can easily copolymerize with other monomers, i.e., such as AMPS, allowing the creation of specialised polymers with enhanced scale-inhibiting properties. IA is relatively inexpensive to produce, especially when derived from renewable sources, such as glucose, sucrose, molasses, corn starch, or other carbohydrate-rich agricultural by-products, through fermentation.

AMPS contains a sulfonic acid group (-SO_3H), which provides strong ionic interactions. This group is highly effective at binding to metal ions, such as calcium, magnesium, and barium, which are common components of scale. By binding these ions, AMPS prevents them from forming insoluble salts that would otherwise precipitate as scale. IA and AMPS are known for their stability under harsh conditions, including high temperatures, high salinity, and a wide pH range. This makes it particularly suitable for applications such as oilfield operations, where these conditions are frequently encountered. The sulfonic acid group also imparts hydrophilicity, and IA-derivatives, i.e., IA/AMPS, are highly soluble in water, allowing them to remain soluble in aqueous environments. This solubility is crucial for its effectiveness as a scale inhibitor, as it needs to interact with dissolved ions in water to prevent scale formation. Compared to some other scale inhibitors, AMPS-based inhibitors are considered to have a lower environmental impact, which is increasingly important in regulatory environments.

IA/AMPS copolymer, a novel scale inhibitor, was synthesised in this study by using itaconic acid (IA) and 2-acrylamido-2-methylpropane sulfonic acid (AMPS), whilst the ammonia persulfate and sodium bisulphite were used as the initiators, with a specific concentration of sodium hydroxide solution added for the neutralisation treatment of this process. Following that, the recently formulated scale inhibitor (SI) was characterised, and its performance in inhibiting the growth of calcium carbonate and calcium sulphate was evaluated. In the scale-inhibition performance evaluation, Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) was used to analyse the remaining calcium ion, Ca^{2+}, concentration in the IA/AMPS-inhibited scaling solution.

The primary objective of this study is to synthesise and characterise the IA/AMPS copolymer as a scale inhibitor. The effects of reaction time/temperature, monomer ratio, dosage, and initiator ratio on the dispersion properties and scale inhibition performance of the copolymer were systematically investigated to obtain the optimised synthesis conditions. The evaluation of IA/AMPS copolymer's inhibition performance against calcium carbonate and calcium sulphate is the ultimate goal of this study. FTIR spectroscopy was used to characterise the functional groups present in the synthesized IA/AMPS copolymer whilst the ICP-OES was used to determine the concentration of the calcium metallic ions in the scaling solution in either with IA/AMPS-inhibited scaling solution (known as inhibited brine) or without the presence of the newly developed scale inhibitors (blank noninhibited scaling solution known as blank solution). A static inhibition efficiency (IE) test method was conducted to evaluate the performance of IA/AMPS copolymer as a scale inhibitor in inhibiting the formation of calcium carbonate and calcium sulphate scale. A comparison was made between two distinct experiments: one conducted in an IA/AMPS-inhibited scaling solution and another in a blank, non-inhibited scaling solution.

METHODOLOGY

Material

In the experiments, pure chemicals which were the analytical grade of Itaconic acid (IA) from Sigma, analytical grade of 2-acrylamido-2-methylpropane sulfonic acid (AMPS) from Sigma, analytical grade of

sodium hydroxide, NaOH from R&M, calcium chloride, $CaCl_2$ (93%) from R&M, ammonium persulfate, $(NH_4)_2S_2O_8$ (98%) from System, sodium chloride, NaCl (99%) from ChemCenter, soluble starch, sodium sulphate, Na_2SO_4 (98%) from HmBG, calcium carbonate, $CaCO_3$ (95%) from Systerm, sodium bisulfate, Na_2SO_4 (95%) from Thermo Scientific, hydrochloric acid, HCl (99%) from Fisher, potassium hydroxide, KOH (85%) from Thermo Scientific, sodium bicarbonate and $NaHCO_3$ (99-100.5%) purity from Arm & Hammer were acquired.

Experimental Methodology

Synthesis and Formulation of IA/AMPS Copolymer

In a three-necked round flask containing 30 mL of ultrapure water and a water bath equipped with a magnetic stirrer and a reflux condenser, 10 g of AMPS and 3.1387 g of IA were added, resulting in a 1:2 molar ratio. The electric heating jacket was activated concurrently. The reactants were then heated for 30 minutes, bringing the temperature up to between 75 and 80 °C. For neutralisation treatment, a sodium hydroxide solution (NaOH) was added using a constant-pressure dropping funnel. The pH of the copolymer determined the amount of NaOH to be added. The copolymer's pH value was evaluated using a pH probe before NaOH was added. Then, 0.9197 g of initiators, consisting of sodium bisulphite and ammonium persulfate (APS), representing 7% of the total mass of the monomers, were added. A constant-pressure dropping funnel was used to slowly add 0.8256 g of ammonium persulfate and 0.0941 g of sodium bisulphite at a 4:1 molar ratio. An aqueous copolymer solution with a solid content of approximately 30% was then produced by heating the mixture to 90°C and maintaining this temperature for 7 hours. Figure 1 illustrates the reaction equation for the synthesis of the copolymer, while the apparatus is set up as shown in Figure 2.

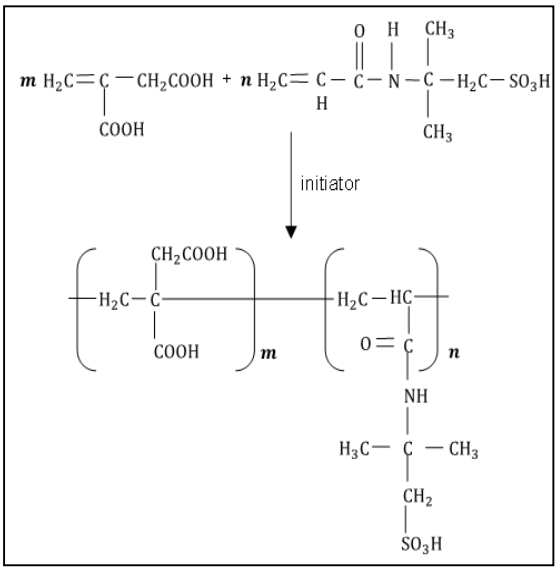


Fig. 1: The reaction equation for the synthesis of the IA/AMPS copolymer

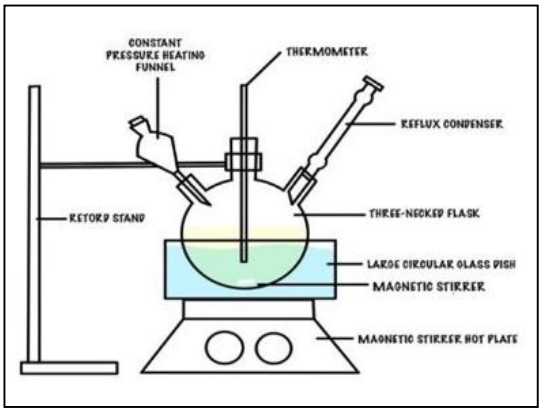


Fig. 2: Laboratory experimental set-up for synthesis of IA-AMPS copolymer

FTIR Analysis of the Newly Developed IA/AMPS

In the Instrumentation Laboratory of the Faculty of Chemical Engineering, UiTM Shah Alam, an FTIR spectrometer (PerkinElmer Spectrum One) was used to analyze the infrared spectra of the IA/AMPS copolymer. The newly formulated scale inhibitor,

IA/AMPS copolymer, has its chemical structure verified using FTIR by analysing the functional groups present, which can be confirmed by the characteristic peaks (Yang *et al.*, 2017).

The diamond crystal surface and the mounting plate are ensured to be clean by wiping them with an alcohol-moistened tissue. To scan the liquid sample using the attenuated total reflectance (ATR) method, first press the 'liquid scan' icon. Then, a few drops of liquid are placed over the ATR crystal, and the pressure arm is rotated so that it just touches the sample. No additional pressure is needed with this liquid sample, as there is good contact between the crystal and the liquid. However, the pressure arm is still placed over the top of the sample to prevent or slow down the evaporation of more volatile liquids from the crystal surface. This instrument covered the wavelength range from 2.5 µm to 20 µm (wavenumber range 4,000 cm−1 to 500 cm−1).

IA/AMPS Copolymer Performance Evaluation Against Calcium Carbonate and Calcium Sulphate Scales in Static Scale Inhibition Efficiency Test (SIE)

A static scale inhibition efficiency test method was conducted to evaluate the performance of IA/AMPS copolymer as a scale inhibitor in inhibiting the formation of calcium carbonate and calcium sulphate scales.

In this SIE test, the concentrations of the scaling ions, Ca^{2+}, in the blank solution (without the presence of IA/AMPS copolymer) and the inhibited brine (with the presence of IA/AMPS copolymer) were analysed. The static inhibition efficiency percentage (SIE%) was then calculated as part of the evaluation using Eq. (1) (Liu *et al.*, 2019; Merdhah and Yassin, 2008; Yang *et al.*, 2017).

$$SIE\ \% = \frac{(Ca^{2+}{}_1 - Ca^{2+}{}_2)}{(Ca^{2+}{}_0 - Ca^{2+}{}_2)} \times 100\% \quad (1)$$

Where,

$Ca^{2+}{}_0$ = the theoretical calcium ion concentration in the blank noninhibited scaling solution before the experiment (at time = 0 hours).

$Ca^{2+}{}_1$ = the water's calcium ion concentration in the IA/AMPS-inhibited scaling solution at sampling time (t=2 hours/22 hours).

$Ca^{2+}{}_2$ = the water's calcium ion concentration in the blank noninhibited scaling solution at sampling time (t=2 hours/22 hours).

Static Scale Inhibition Efficiency Test (SIE) of IA/AMPS Copolymer in Laboratory-reproduced Calcium Carbonate Scale

The individual brines were prepared separately, which contained 14520 ppm of Ca^{2+}(CB1) by dissolving 10.02 grams of $CaCl_2$ into 250 ml of DW, and 36070 ppm of HCO_3^- (HB) by dissolving 12.5 grams of $NaHCO_3$ into 250 ml of DW, respectively. A 40:60 ratio of the $CaCO_3$ scaling brine (i.e., blank solution) was prepared by mixing 10 ml of CB1 with 20 ml of HB to produce a final mix of 4840 ppm Ca^{2+}: 24050 ppm HCO_3^- (Table 1).

A 10 ml of the newly synthesised IA/AMPS copolymer scale inhibitor was then added to the mixed scaling brine (the final concentration of IA/AMPS is 109.490 g/L). Following vigorous shaking to ensure a homogeneous solution was achieved, they were placed in an 80°C water bath for 10 hours (Abbasi *et al.*, 2020; Kapse *et al.*, 2024; Khodabakhshi and Bijani, 2024). A blank noninhibited scaling solution, called a blank sample (without IA/AMPS copolymer scale inhibitor) is run concurrently. The methodology is summarised in Figure 3, and four (4) various conditions were prepared as

tabulated in Table 2.

The study by Ghaffari et al. (2023) investigates the efficiency of Microbial Enhanced Oil Recovery (MEOR) using the bacterium Rhodococcus erythropolis under reservoir conditions, with a particular focus on the impact of temperature (80°C). The study by Kapse et al. (2024) focuses on characterising reservoir properties and their impact on Microbial Enhanced Oil Recovery (MEOR) through laboratory simulations. A key aspect of the research is the investigation of microbial activity and efficiency under reservoir temperatures ranging from 80°C to 101°C, which are typical of high-temperature oil reservoirs. The study by Khodabakhshi and Bijani (2024) focuses on predicting scale deposition in oil reservoirs using machine learning (ML) optimisation algorithms, with a specific emphasis on reservoir conditions, including a tested formation temperature of 80°C. Most producing reservoirs in Malaysia operate at temperatures between 80°C and 120°C (Bishop, 2002).

Table 1. The concentration of Ca^{2+} ion, HCO_3^- ion, and IA/AMPS in solution

Individual brine concentration	
CB1 (Ca^{2+})	14520 ppm
HB (HCO_3^-)	36070 ppm
IA/AMPS Stock Solution	
IA/AMPS	437.96 g/L
Mixed brine concentration (in blank noninhibited scaling brine)	
Ca^{2+}	4840 ppm
HCO_3^-	24050 ppm
Mixed brine concentration (IA/AMPS-inhibited scaling brine)	
Ca^{2+}	3630 ppm
HCO_3^-	18035 ppm
IA/AMPS	109.49 g/L

Table 2. List of samples (at varied conditions) prepared from laboratory-reproduced calcium carbonate scale

Samples	**Calcium carbonates, $CaCO_3$**
Samples 1	Blank + no heat (T_{room})
Samples 2	Blank + 80 °C
Samples 3	IA/AMPS + no heat (T_{room})
Samples 4	IA/AMPS + 80 °C

Static Scale Inhibition Efficiency Test (SIE) of IA/AMPS Copolymer in Laboratory-reproduced Calcium Sulphate

The individual brines were prepared separately, containing 4910 ppm of Ca^{2+} (CB2) by dissolving 3.4 grams of $CaCl_2$ into 250 ml of DW, and 9610 ppm of SO_4^{2-} (SB) by dissolving 3.55 grams of $NaSO_4$ into 250 ml of DW, respectively. The final concentration of each ion in the mixed solution is tabulated in Table 3.

Table 3. The concentration of Ca^{2+} ion, SO_4^{2-} ion, and IA/AMPS in solution

Individual brine concentration	
CB2 (Ca^{2+})	4910 ppm
SB (SO_4^{2-})	9610 ppm
IA/AMPS Stock Solution	
IA/AMPS	437.96 g/L
Mixed brine concentration (in blank noninhibited scaling brine)	
Ca^{2+}	2455 ppm
SO_4^{2-}	4805 ppm
Mixed brine concentration (in IA/AMPS-inhibited scaling brine)	
Ca^{2+}	1841.25 ppm
SO_4^{2-}	3603.75 ppm
IA/AMPS	109.49 g/L

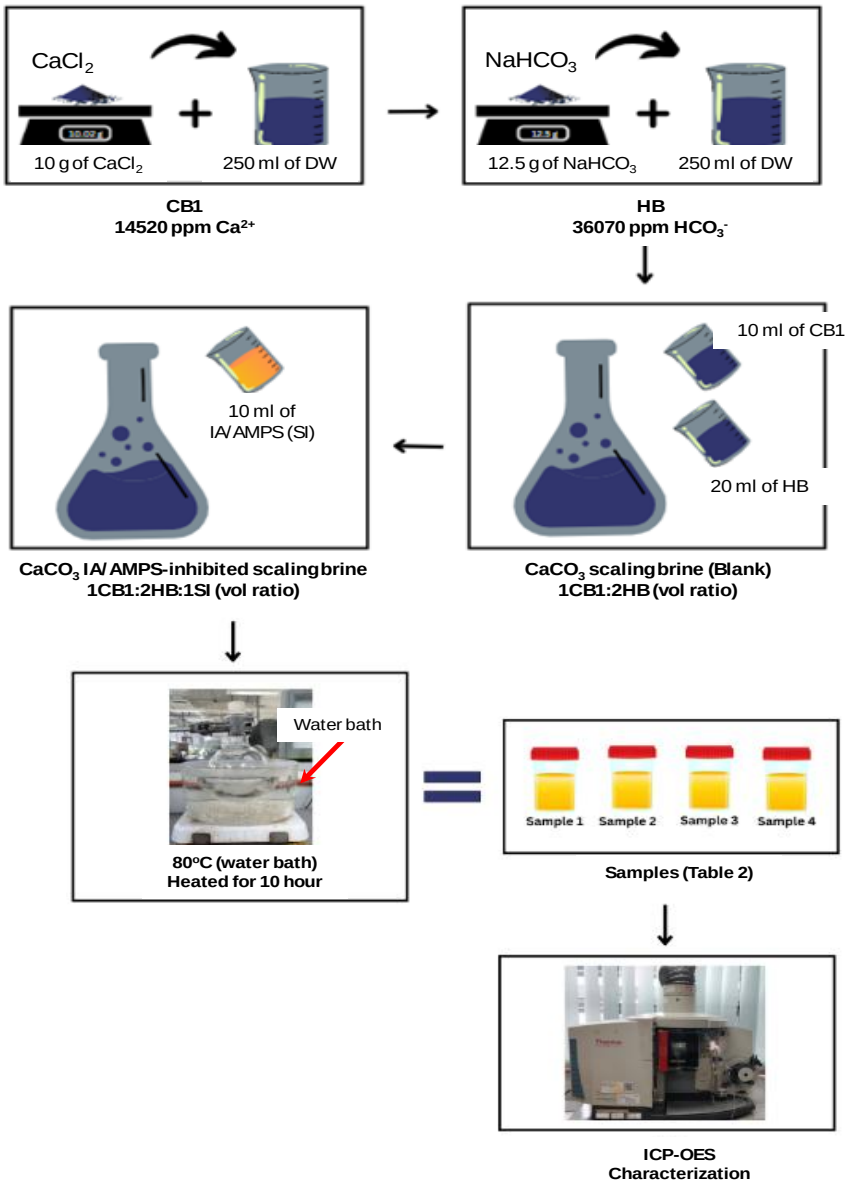


Fig. 3: The procedure of performance study of IA/AMPS copolymer in combating calcium carbonate scale: Noninhibited scaling brine (blank) and IA/AMPS-inhibited scaling brine

In a volumetric flask, 15 mL of CB2 and 15 mL of SB were mixed, and the pH was adjusted to neutral with a hydrochloric acid solution (pH=7). The cooling water was heated to 80°C for 10 hours, while the dosage of IA/AMPS was maintained at 10 mL (resulting in a final concentration of IA/AMPS of 109.5 g/L) for all testing conditions. This procedure is illustrated in Figure 4 with four (4) various conditions as shown in Table 4.

Table 4. List of samples (at varied conditions) prepared from laboratory-reproduced calcium sulphate scale

Samples	Calcium sulphates, $CaSO_4$
Samples 5	Blank + no heat (T_{room})
Samples 6	Blank + 80 °C
Samples 7	IA/AMPS + no heat (T_{room})
Samples 8	IA/AMPS + 80 °C

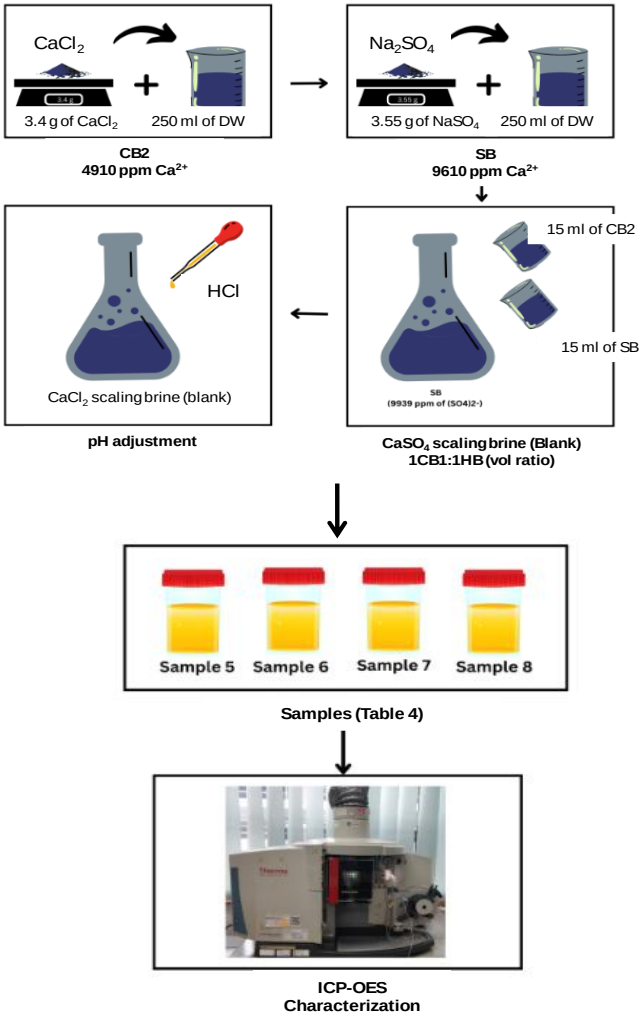


Fig. 4: The procedure of performance study of IA/AMPS copolymer in combating calcium sulphate scale: Noninhibited scaling solution (blank solution) and IA/AMPS-inhibited solution

Performance Evaluation of IA/AMPS against $CaCO_3$ and $CaSO_4$

The inhibition mechanism of scale inhibitors in oilfield scale formation is complex. It involves several processes that work together to prevent the precipitation and deposition of scale-forming minerals, such as calcium carbonate ($CaCO_3$) and calcium sulphate ($CaSO_4$). Scale inhibitors may prevent oilfield scales through various mechanisms, including nucleus inhibition, crystal growth retardation, dispersion of precipitated particles, threshold inhibition, modification of crystal morphology, and chelation of scaling ions. In oilfield operations, these mechanisms were crucial for preventing scale formation in pipelines, wellbores, and production equipment, where scale can lead to blockages, reduced flow rates, and increased maintenance costs. To address specific scaling challenges, scale inhibitors are typically injected into production fluids, with their selection tailored to the water chemistry and operational conditions.

This study aims to evaluate the effectiveness of IA/AMPS in inhibiting the formation of $CaCO_3$ and $CaSO_4$ under simulated field conditions.

The Qualitative Performance Study of IA/AMPS via Physical Observation

The performance of this copolymer was evaluated through physical observations made over 22 hours, including changes in colour, turbidity, and the formation of any visible precipitate at specific time intervals (i.e., 0 hours and 22 hours). These observations are crucial in providing initial insights into the inhibitor's performance.

The Quantitative Performance Study of IA/AMPS via EDTA Titration

To determine the Ca^{2+} concentration in the supernatant, an EDTA titration is performed after the solution has been cooled to room temperature. These concentrations are then used to determine the SIE% as shown in Eq. (1). In the scale-inhibition performance evaluation, EDTA titration was used to calculate the concentration of the scaling ion, i.e., calcium ion (Ca^{2+}) (Sazali, 2015; Sazali *et al.*, 2015). Metal cations can be directly determined using the EDTA titration method (Sazali *et al.*, 2021). For this method, 5 mL of supernatant was measured with a pipette into an Erlenmeyer flask. Hydrochloric acid was added, with a volume ratio of concentrated hydrochloric acid to water of 1:1. The mixture was then heated to micro-boiling for 30 seconds. After cooling to room temperature, 5 mL of a 20% potassium hydroxide (KOH) solution was added. Upon adding the calcein indicator, the test solution turned fluorescent green. A standard EDTA solution (0.0124 $mol \cdot L^{-1}$) was then used to titrate the solution. The endpoint was reached when the green fluorescence gradually faded, and a purple colour appeared. The calcium ion content was determined based on the mass of calcium carbonate, as calculated using Eq. (2) (Sazali, 2015).

$$X(mg \cdot L^{-1}) = \frac{V_1 \times c(EDTA)}{V} \times M \times 1000 \quad (2)$$

Where,

c (EDTA) = EDTA solution concentration, $mg \bullet L^{-1}$

V_1 = the volume of EDTA solution consumed in titration, ml

V = the volume of supernatant taken

M = the molar mass of calcium carbonate, $g \bullet mol^{-1}$

The Quantitative Performance Study of IA/AMPS via Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES)

Alternatively, the Ca^{2+} concentration can be determined using ICP-OES instead of the EDTA titration. These concentrations are then used to determine the SIE, as shown in Eq. (1). The calcium standard was monitored at 317.933 nm (Cui and Zhang, 2019) and 393.366 nm, where the metal cation concentrations can be directly determined using ICP-OES. The ICP-OES in the Science Material Laboratory (ICAP 6300 DUO) was used to analyse the concentration of Ca^{2+} in the blank non-inhibited scaling solution and the IA/AMPS-inhibited scaling solution.

RESULTS AND DISCUSSION

Characterisation Analysis of IA/AMPS Copolymer via FTIR Spectra

After reacting for 7 hours, the formulated copolymer appears as a liquid with a light-yellow colour, as shown in Figure 5. This copolymer was allowed to cool to room temperature before being analysed and characterised using FTIR. The IR spectra of the IA/AMPS copolymer can be seen in Figure 6. The peak between 2500-3000 cm^{-1} represents the stretching vibration of the carboxylic acid (COOH) group in the IA monomer. The symmetric stretching vibration of the carboxylate group (COO^-) appears at

1400-1500 cm^{-1}. The stretching vibration of the amide group (CONH) in the AMPS monomer occurs at a wavenumber between 2500 and 1600 cm^{-1}. The stretching vibration of the sulfonic acid (SO_3H) group in the AMPS monomer is represented by a wavenumber in the 600–700 cm^{-1} region of the IR spectrum.

According to Figure 6, a broad absorption band is observed at 3350.50 cm^{-1}, indicating the presence of the hydroxyl group (-OH) in the copolymer (Kealey and Haines, 2002). Both the carboxylic acid (IA) and sulfonic acid (AMPS) units include hydroxyl groups. The C=C stretching vibration in the IA/AMPS copolymer has an absorption band at 1634.81 cm^{-1}, indicating the presence of unsaturated carbon-carbon (C=C) bonds. This range exhibits a significant absorption band due to the presence of a double bond in the IA monomer. (Refer to Table 5).

Fig. 5: The newly synthesised IA/AMPS scale inhibitor

The performance of the IA/AMPS copolymer

IA/AMPS Copolymer Inhibition Efficiency against $CaCO_3$ and $CaSO_4$ by Physical Observation (Qualitative Analysis)

The samples of the non-inhibited scaling brine (blank) and the IA/AMPS-inhibited scaling brine in both $CaCO_3$ and $CaSO_4$ environments were carefully observed and photographed. Table 6 shows the results obtained based on physical changes of $CaCO_3$ and $CaSO_4$ that include four categories; blank samples at room temperature (Samples 1 and 5), blank samples at the elevated temperature of 80 °C (Sample 2 and 6); and IA/AMPS-inhibited samples at room temperature (Samples 3 and 7) and 80 °C (Samples 4 and 8), respectively.

Observing the $CaCO_3$ and $CaSO_4$ scale under different conditions reveals distinct physical characteristics, which are explained in the following details:

(a) The $CaCO_3$ scale formed in an unheated, blank sample shows a layer of scale that appears as a white, powdery material. The $CaCO_3$ particles are only weakly adhered to the surface and can be easily removed. The structure and light-scattering characteristics of the scales created prevent them from displaying any transparency, making them appear opaque.

(b) When 80 °C is applied to the blank samples, the $CaCO_3$ scales become more compact, closely adherent, and develop a hard texture at the bottom of the surface. The disruption of weak bonds due to thermal energy at higher temperatures facilitates the formation of stronger bonds between carbonate ions, resulting in a more compact and closely adherent structure (Vallapragada *et al*., 2011).

(c) In the presence of the IA/AMPS scale inhibitor (without the application of heat), there is evidence showing that a smaller amount of $CaCO_3$ scales is produced compared to the conditions in (a) and (b). It was observed that the scale becomes thinner and less widespread. This indicates that IA/AMPS can prevent calcium carbonate particles from adhering to each other, resulting in scale formations that are less dense and smaller.

(d) The sample becomes cloudy with the formation of tiny, suspended particles when the IA/AMPS-inhibited scaling solution is heated to a temperature of 80 °C. Compared

to the blank samples' scale, a less adherent layer to the surface is observed. The IA/AMPS scale inhibitor, when used at the specified temperature, helps prevent strong bonding and adherence of the scale to the surface. The precipitation of insoluble $CaCO_3$ is responsible for the cloudiness detected in the solution (Nandiyanto *et al*., 2019). The higher temperature may force $CaCO_3$ to dissolve more than it normally would, resulting in the formation of much smaller solid particles that contribute to the cloudiness.

(e) The $CaSO_4$ scale was visible as an off-white crystalline deposit with a granular texture in the unheated, blank sample. Because of its loose attachment to the surface, it was easy to remove.

(f) The amount of scale produced was decreased when 80 °C was applied to blank $CaSO_4$. This suggests that the elevated temperature has a positive effect on reducing the development or size of the $CaSO_4$ scale. By promoting $CaSO_4$ solubility and inhibiting its deposition, the temperature increase reduces the amount of scale that forms. Furthermore, the increase in temperature reduces the amount of scale that forms. Additionally, the higher temperature may encourage the breakdown or dispersion of existing scale deposits, thereby reducing their apparent presence.

(g) The $CaSO_4$ scale formed with the addition of the IA/AMPS copolymer at room temperature appeared to be less widespread compared to the blank solution. The results indicate that even in the absence of heat, the IA/AMPS scale inhibitor is effective in inhibiting the development of the $CaSO_4$ scale.

(h) The $CaSO_4$ scale was completely inhibited (no $CaSO_4$ scale was observed) when the IA/AMPS scale inhibitor was added at a temperature of 80 °C. In contrast to either factor acting alone, the synergistic combination of IA/AMPS and elevated temperature efficiently inhibited scale production and facilitated scale management.

The observations imply that a temperature of 80 °C has a beneficial effect on reducing the production of $CaCO_3$ and $CaSO_4$ scales.

IA/AMPS Copolymer Inhibition Efficiency against $CaCO_3$ and $CaSO_4$ using EDTA Titration (Quantitative Analysis)

The volume of EDTA consumed in all eight samples is tabulated in Table 7. The presence of the IA/AMPS scale inhibitor in the absence of heat (Sample 3) resulted in much lower EDTA consumption (8.33 ml). The amount of EDTA solution consumed in a calcium titration using EDTA is proportional to the quantity of Ca^{2+} present. A lower EDTA consumption implies a lower Ca^{2+} concentration in the $CaCO_3$ solution. Sample 3's lower EDTA consumption indicates that fewer Ca^{2+} were available for complexation. In contrast, the observed EDTA consumption increased to 12.33 ml when the sample was heated to 80°C in addition to the IA/AMPS in Sample 4.

We observe that the Ca^{2+} concentrations in the IA/AMPS-treated samples (3, 4, 7, 8), as measured by the EDTA titration technique, are unexpectedly lower than those in the untreated blank samples (1, 2, 5, 6). The addition of IA/AMPS as a scale inhibitor should prevent Ca^{2+} from precipitating into solid scale. As a result, a higher concentration of Ca^{2+} is expected to remain in the solution, meaning treated samples should show increased Ca^{2+} levels in the supernatant. The reversed trend suggests something went wrong in the procedure.

The analysis of $CaSO_4$ revealed different behaviour compared to $CaCO_3$, according to the results. The temperature had a significant

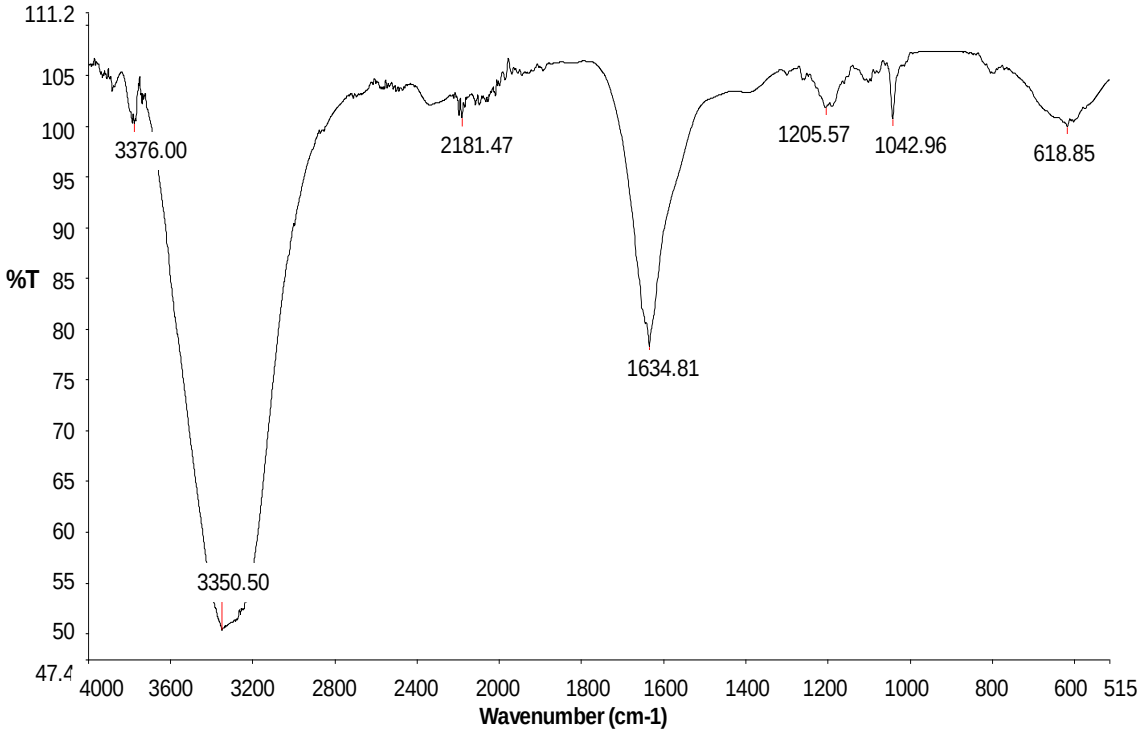


Fig. 6: IR spectra of the IA/AMPS copolymer

Table 5. FTIR spectra (IA, AMPS, and IA/AMPS**)**

Peaks (cm^{-1})	**IA** (Mohammed Ali Alsalami et al., 2017)	**AMPS** (*Greesh et al., 2008) ; (#Cui and Zhang, 2019b)	**IA/AMPS**
COOH stretching vibration	3450, 2900		3350.50
C-H stretching (alkene and alkene side)	2950-3050	*2950, *2848	3376
O=C-O (carboxylic acid)	1705		1634.81
C=C stretching	1565		Very weak peak ~1500
C-O stretch (alkene)	1166		Very weak peak ~1100
stretching vibration of the amide group C-N (CONH)	1398	*3235, *1542, #1564.56	3350.50, Very weak peak ~1500 (#3434.97)
stretching vibration absorption peak of C=O in the amide group		#1656.79	1634.81
S=O		*1372, 1656.79	1205.57, very weak peak at ~1350 (#1192.58)
Stretching vibration of S=O		#1048.34	1042.96
S-C stretching vibration peak		#629.41	618.85

Table 6. IA/AMPS copolymer inhibition efficiency against calcium carbonate, $CaCO_3$ and calcium sulphate, $CaSO_4$ scales (Physical Observation

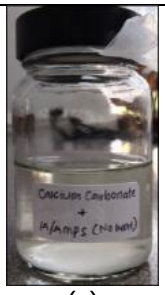
Brine	Blank + no heat	Blank + 80 °C	IA/AMPS + no heat	IA/AMPS + 80 °C
CaSO3	 (a)	 (b)	 (c)	 (d)
$CaSO_4$	 (e)	 (f)	 (g)	 (h)

Table 7. The volume of EDTA solution consumed, V_1, for each sample

	Samples	Volume of supernatant, V (ml)	Volume of EDTA solution consumed, V1 (ml)			
			Reading 1	Reading 2	Reading 3	Average reading
$CaCO_3$	Sample 1	15	15	16	15	15.33
	Sample 2	15	10	9	11	10.00
	Sample 3	15	8	9	8	8.33
	Sample 4	15	12	12	13	12.33
$CaSO_4$	Sample 5	15	16	14	15	15.00
	Sample 6	15	17	16	18	17.00
	Sample 7	15	14	15	14	14.33
	Sample 8	15	15	15	16	15.33

impact, as the blank sample subjected to 80°C (Sample 6) consumed more EDTA (17.00 ml), suggesting that heat-induced changes may have increased interference. The IA/AMPS scale inhibitor reduced the EDTA usage (14.33 ml) when used without added heat. In the $CaSO_4$ samples, there was only a slight difference in EDTA usage between the IA/AMPS at 80°C (15.33 mL) and the IA/AMPS without heat.

As shown in Table 8, the Ca^{2+} concentration measured via this EDTA titration varied according to various conditions tested. Eq. (2) was used to calculate the Ca^{2+} content, providing a thorough understanding of the potential effects on scale inhibition effectiveness.

A significant trend is evident when analysing the Ca^{2+} content of the $CaCO_3$ samples. The calculated concentration is 1268.70 ppm in the blank sample without heat (Sample 1), which serves as a standard for the general level. The concentration drops to 827.41 ppm in Sample 2, indicating that heat may cause changes that reduce the availability of Ca^{2+} ions. In Sample 3, the concentration is further reduced to 689.51 ppm with the addition of the IA/AMPS scale

inhibitor. Unexpected outcomes were observed in this experiment, wherein the concentration of Ca^{2+} in sample 3 was lower than in sample 1 despite the addition of the IA/AMPS scale inhibitor. The fundamental idea was that the IA/AMPS scale inhibitor would prevent the formation of $CaCO_3$ scale by inhibiting the reaction between Ca^{2+} ions and HCO_3^- ions, which is known to result in the precipitation of $CaCO_3$. Therefore, the concentration of Ca^{2+} ions in the solution was expected to be higher than in sample 1. However, the actual outcomes did not match this original expectation.

Table 8. IA/AMPS copolymer inhibition efficiency against calcium carbonate, $CaCO_3$ and calcium sulphate, $CaSO_4$ (via Ca^{2+} ion measured)

	Samples	Calcium ion content, Ca^{2+} (ppm)
$CaCO_3$	Sample 1	1268.70
	Sample 2	827.41
	Sample 3	689.51
	Sample 4	1020.47
$CaSO_4$	Sample 5	1688.14
	Sample 6	1913.22
	Sample 7	1613.11
	Sample 8	1725.65

For the $CaSO_4$ samples, a particular pattern is observed. A baseline is established by Sample 5, which measures a concentration of 1688.14 ppm. Sample 6 shows a higher concentration of 1913.22 ppm, indicating that heat increases the presence of calcium ions, possibly due to increased solubility. The concentration then falls to 1613.11 ppm in Sample 7. Similar to sample 3, this comparison with sample 5 yields unexpected results. However, the concentration in Sample 8 remains quite high at 1725.65 ppm.

The performance of this IA/AMPS is further calculated using Eq. 1 and presented in Table 9. However, as expected, all results were negative (i.e., efficiency is 0%), indicating that the absence of quenching solution in all the samples caused the brine to continue scaling, resulting in these reversed results.

The primary issue observed in this EDTA Titration method is that the samples were not quenched before titration, allowing precipitation of $CaCO_3$ or $CaSO_4$ to continue in the IA/AMPS-treated solutions during or after heating. Although IA/AMPS slows down precipitation, it does not completely prevent it without strict control. Therefore, an extended reaction time without quenching resulted in more Ca^{2+} being removed from the solution, i.e., reacted and formed the scale.

Table 9. IA/AMPS copolymer inhibition efficiency against calcium carbonate, $CaCO_3$, and calcium sulphate, $CaSO_4$ (EDTA Titration Method)

Oilfield Scale	Room Temperature	80°C
$CaCO_3$	-29.2%	4.81%
$CaSO_4$	-9.8%	-34.6%

IA/AMPS Copolymer Inhibition Efficiency against $CaCO_3$ and $CaSO_4$ using ICP-OES (Quantitative Analysis)

The concentration of calcium ions was quantified using ICP-OES. To establish a calibration curve, standard solutions with concentrations of 5 ppm, 10 ppm, 15 ppm, and 20 ppm were employed. During the characterisation analysis, two wavelengths, 315.887 nm and 393.366 nm, were initially used for measurement.

The signal at 393.366 nm was affected by detector saturation, resulting in unreliable results (Figure 8). Because this line also lies in a crowded spectral region with overlaps from Al, Fe, and V, as well as a broad molecular wing

that requires complex corrections, it was discarded. All quantitative work, therefore, utilised the 315.887 nm line (Figure 7), where interferences are minor and easily corrected. The calibration at 315.887 nm remained strictly linear, yielding an R^2 of 0.9982, whereas the 393.366 nm curve flattened under the same conditions.

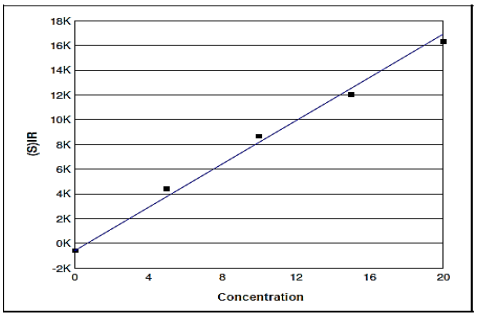


Fig. 7: The calibration curve of 315.887 nm, standard solutions with concentrations of 5 ppm, 10 ppm, 15 ppm, and 20 ppm

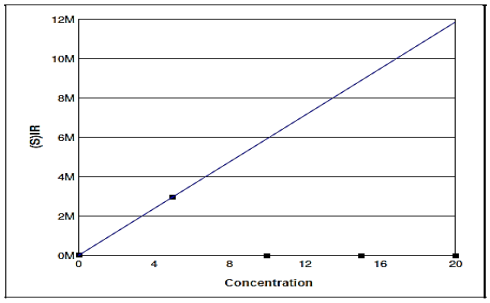


Fig. 8: The calibration curve of 393.366 nm, standard solutions with concentrations of 5 ppm, 10 ppm, 15 ppm, and 20 ppm

In summary, ICP-EOS was used to determine the concentration of calcium ions by analysing standard solutions with varying calcium concentrations. The 315.887 nm wavelength was selected for analysis due to its strong correlation coefficient, whereas the 393.366 nm wavelength was discarded because of saturation-induced unreliability in the data.

The data presented in Table 10 show the outcomes of $CaCO_3$ and $CaSO_4$ brine characterisation using ICP-OES analysis under different conditions and with an IA/AMPS inhibitor present. The Ca^{2+} concentrations obtained from each sample provide information about the efficiency of the inhibitor and the impact of temperature on Ca^{2+} levels.

The concentration from the ICP-OES analysis has been adjusted to account for the dilution applied to each sample, enabling the precise determination of the true concentration of Ca^{2+} ions. Each sample was diluted to a predetermined concentration (20 ppm) before ICP-OES analysis. It was necessary to account for sample dilution prior to ICP OES analysis in order to determine the precise Ca^{2+} ion concentration in each sample. Dilution was intentionally performed to ensure that all samples fell within the instrument's analytical range and to minimise potential matrix effects. All samples were diluted uniformly before analysis to enhance measurement precision and reliability. After obtaining the analysis results from ICP-OES, the dilution factor used for each sample is multiplied to determine the reported concentration of Ca^{2+} ions, as shown in Table 10. The dilution factor corrects the concentration to accurately represent the actual Ca^{2+} content, ensuring that the reported values reflect the deliberate dilution employed in the experimental technique.

The concentrations of Ca^{2+} in Samples 3, 4, 7, and 8 were expected to be higher than those in Samples 1, 2, 5, and 6, respectively. Based on the data in Table 10 and the proposed hypothesis, it was anticipated that the IA/AMPS scale inhibitor would prevent the formation of $CaCO_3$ and $CaSO_4$ (Niu *et al.*, 2022), leading to increased Ca^{2+} concentrations in the treated samples (Karunadasa *et al.*, 2019).

Table 10. The concentration of Ca^{2+} in $CaCO_3$ and $CaSO_4$

Mixed brine	Samples	Ca^{2+} Concentration by ICP-OES (ppm)	Dilution	Actual Ca^{2+} Concentration after 70 days (ppm)
$CaCO_3$	Sample 1	1.8653	240.8	449.16
	Sample 2	0.332472	240.8	80.06
	Sample 3	0.439156	180.6	79.31
	Sample 4	0.663508	180.6	119.83
$CaSO_4$	Sample 5	1.857429	122.8	228.09
	Sample 6	2.06403	122.8	253.46
	Sample 7	1.24636	98.24	122.44
	Sample 8	1.80627	98.24	177.45

Unexpectedly, Sample 3 (79.31 ppm) had a lower Ca^{2+} content than Sample 1 (449.16 ppm). This result differs from expectations because the IA/AMPS scale inhibitor was predicted to reduce $CaCO_3$ formation and raise the Ca^{2+} concentration. In the case of $CaSO_4$ samples, the Ca^{2+} concentration in Sample 7 (122.44 ppm) was lower than in Sample 5 (228.09 ppm), contrary to the hypothesis that the IA/AMPS scale inhibitor would prevent $CaSO_4$ formation and lead to a higher Ca^{2+} concentration. Likewise, Sample 8 exhibited a Ca^{2+} concentration (177.45 ppm) lower than in Sample 6 (253.46 ppm), which contradicts the expected inhibitory effect of the IA/AMPS inhibitor on $CaSO_4$ formation.

The absence of quenching solution at predetermined intervals following the initial mixing of the brine solutions was a critical procedural oversight that led to unexpected results in the Ca^{2+} ion concentrations found in samples treated with the IA/AMPS scale inhibitor. The samples were not exposed to the quenching solution, which is typically used to stop ongoing reactions in progress, at the specified times (2 hours and 22 hours). This oversight unintentionally allowed the reactions to continue longer than intended, affecting the Ca^{2+} ion concentrations and resulting in unexpected outcomes. Due to the absence of a quenching solution, which halts the reaction between Ca^{2+} ions and HCO^{3-} ions (Merdhah and Yassin, 2008; Raharjo *et al*., 2019), the addition of IA/AMPS to a chemical reaction yielded unforeseen results. This allowed the reaction to proceed beyond the intended point, altering the overall Ca^{2+} ion concentration and resulting in outcomes that deviated from earlier predictions. The absence of the quenching solution highlighted the need for procedural consistency in accurately determining the effects of scale inhibitors and the importance of paying close attention to every little detail of the experiment to ensure reliable and significant results.

As a result, the values shown in Table 9 were measured over an extended 70-day period, during which further reactions and equilibration processes occurred. Consequently, these concentrations do not accurately represent the system's status at the relevant time points. Due to the prolonged timescale, the equilibrium may have shifted, and subsequent reactions could have influenced the recorded concentrations.

In essence, the unexpected outcomes result from the extended reaction times caused by the absence of a quenching step. Applying the quenching solution at the designated intervals is essential for providing a more precise representation of the IA/AMPS scale inhibitor's immediate effects. This adjustment would enhance alignment with the proposed hypothesis and allow for a more accurate evaluation of the inhibitor's

influence on the initial phases of the reactions. To test this hypothesis, duplicate samples of mixed brine (blank solution) containing 2455 ppm Ca^{2+} and 4805 ppm SO_4^{2-} were allowed to react at 80°C and ambient pressure. ICP samples were taken at 2 hours and 22 hours, with proper quenching in 1% EDTA/NaOH. The same test was repeated, and ICP-OES results were found to be repeatable and reproducible.

The data in Table 11 shows the calculated reaction percentages for various samples under different conditions, including those with the addition of the IA/AMPS scale inhibitor.

Table 11. The initial Ca^{2+} (0 hours) and actual concentration of Ca^{2+} (after 70 days), and the percentage of reaction for each sample

Samples	Initial Ca^{2+} concentration (ppm)	Actual Ca^{2+} Concentration after 70 days (ppm)	% reaction
Sample 1	4816.7	449.16	90.7
Sample 2	4816.7	80.06	98.3
Sample 3	3612.5	79.31	97.8
Sample 4	3612.5	119.83	96.7
Sample 5	2456	228.09	90.7
Sample 6	2456	253.46	89.7
Sample 7	1964.8	122.44	93.8
Sample 8	1964.8	177.45	91.0

At low temperatures, Samples 1 and 5 exhibit reaction percentages of 90.7%, resulting in the formation of $CaCO_3$ and $CaSO_4$ scales. At 80 °C, the percentages in Samples 2 and 6 are higher, indicating increased scale development due to faster reaction kinetics. Samples 3 and 4 exhibit comparable or slightly decreased reaction percentages compared to the blank samples, indicating that there is no appreciable inhibition of the Ca^{2+} and HCO^{3-} ion processes. The slightly higher reaction percentages in Samples 7 and 8 suggest that the IA/AMPS inhibitor may have affected the kinetics of $CaSO_4$ production, resulting in a modest increase in scale formation.

It is postulated that the IA/AMPS scale inhibitor would function as a scale inhibitor, reducing the degree of reaction and resulting in lower reaction percentages. However, the results for Samples 3, 4, 7, and 8 do not align with the predicted reduced reaction rates. Instead, they show either higher percentages or marginal decreases, indicating that the inhibitor did not achieve the desired inhibitory effect on scale formation under the tested conditions. The ICP-OES data are unreliable due to the absence of a quenching solution, which is required to prevent further formation of $CaCO_3$ and $CaSO_4$. As a result, the scaling reaction continued for more than 70 days. It is essential to note that the samples were only analysed after a 70-day delay due to a lengthy queue for ICP-OES analysis.

CONCLUSIONS

The physical observation of the samples demonstrated that IA/AMPS is effective at combating both scales. This physical observation is reliable since the photos were taken within 22 hours of the event. The copolymer reduced the amount of $CaCO_3$ scale formed, resulting in thinner and less dense scales, even in the absence of heat. At elevated temperatures (80 °C), the copolymer further prevented strong bonding and adherence of $CaCO_3$ scales to the surface. This indicates that the IA/AMPS copolymer also effectively inhibited $CaSO_4$ scale formation. Even at room temperature, the copolymer reduced the widespread development of $CaSO_4$ scales. At 80 °C, the

copolymer completely inhibited the formation of the $CaSO_4$ scale, displaying a synergistic effect with elevated temperature.

However, considering all unintentional errors, such as the absence of a quenching solution and the lengthy reaction time, the performance evaluation of this newly developed IA/AMPS scale inhibitor via quantitative methods, namely EDTA titration and ICP-OES, was inconclusive. The addition of the IA/AMPS scale inhibitor did not consistently maintain the freely moving Ca^{2+} ions in the samples where inhibition was anticipated using EDTA Titration and ICP-OES analyses. The Ca^{2+} ion concentrations deviated from expected values as a result of prolonged reactions in the absence of quenching prior to ICP analysis. It is highly recommended to repeat this experiment to verify the observations made. Conductivity analysis may also be conducted to study the induction period, nucleation period, and growth period of $CaCO_3$/$CaSO_4$ crystals, which would help assess the efficiency of the IA/AMPS inhibitor in combating these scales. The physical and chemical properties of the $CaCO_3$ and $CaSO_4$ scales formed under the various aforementioned conditions can also be analysed and compared using EDAX-EDS and FTIR analysis to investigate further the possible inhibition mechanisms involved. Additionally, the physical and rheological properties of the IA/AMPS copolymer may be studied for extensive reporting.

While the IA/AMPS copolymer demonstrated promising scale inhibition performance, it is important to acknowledge that this study did not include specific biodegradation or toxicity evaluations to support its environmental friendliness. Although existing literature suggests that such copolymers may have a low environmental impact, further studies are needed to confirm this. Therefore, we recommend that future work include standardised environmental assessments to validate the ecological safety of the IA/AMPS formulation under reservoir conditions.

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