Journal of Energy Research and Reviews

9(3): 36-45, 2021; Article no.JENRR.80666
ISSN: 2581-8368

Evaluating the Effectiveness of Red Onion Skin Extract Derivatives as Oilfield Scale Inhibitors

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

Article Information

DOI: 10.9734/JENRR/2021/v9i330234
Editor(s):
(1) Sreekanth. K. J., Kuwait Institute for Scientific Research, Kuwait.
Reviewers:
(1) Mian Muhammad, University of Malakand, Pakistan.
(2) Mihai Todica, Babes-Bolyai University, Romania.
Complete Peer review History, details of the editor(s), Reviewers and additional Reviewers are available here: https://www.sdiarticle5.com/review-history/80666

Original Research Article

Received 08 October 2021
Accepted 16 December 2021
Published 17 December 2021

ABSTRACT

With growing awareness of the environmental impact of some conventional production chemicals and concerns about the depletion of non-renewable natural resources, increased efforts are being made to use renewable and non-toxic materials in the oilfield. In this study, a potential green scale inhibitor was developed from the skin of red onions and evaluated for calcium sulphate, calcium carbonate and barium scale inhibition. Based on the different extraction processes utilized, two products were obtained and characterized using FTIR and SEM and evaluated using a static jar test procedure. The FTIR results confirmed the bands that make up the major constituents (quercetin) and other important compounds, which supports the present study. Laboratory evaluation show that ROSE can efficiently inhibit calcium sulphate scale and barium sulphate scales with a good inhibition rate of greater than 75% at an optimum dosage. Effect of temperature and dosage on inhibition performance revealed that ROSE is stable at higher temperatures and can effectively inhibit calcium and barium sulphate scales at nearly the same rate without degradation but requires additional dosage to produce same result for calcium carbonate scale. Also, the effect of time reveals that scale inhibitor performs a continuous $CaSO_4$ and $CaCO_3$ inhibition. Not only

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does ROSE perform excellently in the laboratory condition as a green scale inhibitor, but it also show a relatively close performance rate when compared to an existing commercial inhibitor which indicate that ROSE has a high potential for use in the oil industry.

Keywords: *Calcium sulphate scale; calcium carbonate scale; barium sulphate scale; red onion skin; green scale inhibitor; scale inhibition.*

GLOSSARY

ROSE : *Red Onion Skin Extracts;*
FTIR : *Fourier transform infrared spectroscopy;*
NaOH : *Sodium hydroxide;*
SEM : *Scanning Electron Microscope;*
$CaSO_4$: *Calcium sulphate;*
$CaCO_3$: *Calcium carbonate;*
$BaSO_4$: *Barium sulphate;*
$SrSO_4$: *strontium sulfate;*
NaCl : *Sodium chloride;*
$CaCl_2 \cdot 2H_2O$: *Calcium Chloride, Dihydrate;*
$MgCl_2 \cdot 6H_2O$: *Magnesium chloride hexahydrate;*
Na_2SO_4 : *Sodium sulfate;*
$BaCl_2 \cdot 2H_2O$: *Barium chloride, dihydrate.*

1. INTRODUCTION

Scale inhibitor is a term generally used to describe a class of chemicals used to mitigate the formation or deposition of scale. Scale is an inorganic precipitation formed on metal or rock surfaces, wellbore tubulars and other wellbore equipment [1,2]. Scale inhibition can occur when one or more aspects of the crystallization process is disrupted [2,3]. Sulfate and carbonate scales have been identified as the most common inorganic scales found in oilfields worldwide [4-6] While Sulfate scales, which include calcium sulfate ($CaSO_4$), barium sulfate ($BaSO_4$) and strontium sulfate ($SrSO_4$) are formed due to the mixing of incompatible brines, specifically formation brine and injected seawater, carbonate scales are deposited due to change in operating conditions, such as drop in pressure in production wells [4,5,7,8]. Mineral scale formation can be described with the following reactions [3].

$$Ca^{2+}_{(aq)} + 2HCO^{3-}_{(aq)} \rightarrow CaCO_{3\,(s)} + CO_2 + H_2O$$
$$Ca^{2+}_{(aq)} + SO_4{}^{2-}_{(aq)} \rightarrow CaSO_{4\,(s)}$$
$$Ba^{2+}_{(aq)} + SO_4{}^{2-}_{(aq)} \rightarrow BaSO_{4\,(s)}$$

To mitigate the problem of mineral scaling, chemical inhibitors are widely used. However, some of these chemical inhibitors are toxic and environmentally unsafe for application. With the increasing awareness of environmental impact caused by hazardous production chemicals, efforts have been made to utilize green scale inhibitors. Recently, green inhibitors, have raised great interest because they are biodegradable, non-toxic and do not affect environment non-bioaccumulation [9-11].

Quercetin, as shown in Fig. 1a has been discovered to have good inhibiting effect due to its high antioxidative and excellent metal chelation properties like other flavonoids [12-18]. The metal chelating property is attributed to the presence of five (5) hydroxyl groups at positions 3,3',4'5,7, and a carbonyl group at position 4 as shown in Fig. 1b [3]. Red onion skin (ROS) contains the highest concentration of "quercetin" [19,20] and other polyphenolic compounds [21,22] which can easily be extracted and used in the oil industry. However, "onion skins" are usually discarded as wastes which becomes a nuisance to the environment especially when they are disposed improperly the discovery of this active ingredient in red onion skin prompted this investigation to harness the properties of quercetin as a potential green scale inhibitor.

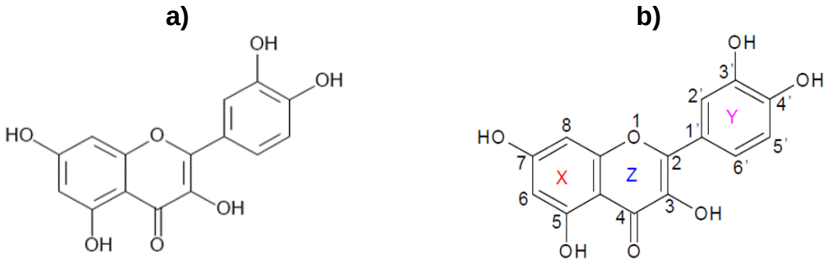

Fig. 1. (a) Structure of quercetin, (b) Functional group positions of quercetin [3]

2. MATERIALS AND METHODS

2.1 Collection and Pretreatment of ROS

Discarded onion skins from processed onions were collected from markets in Port Harcourt, Rivers State, Nigeria. They were carefully selected and thoroughly washed with distilled water to remove impurities. Thereafter, air dried at ambient temperature and pulverized into powder form using a fabricated grinder and then stored in airtight plastic containers for further use.

2.2 Extraction Process

This study adopted two extraction processes: a solvent extraction mechanism [3,23] and cold extraction process [24].

2.2.1 Solvent extraction method

Solvent extraction mechanism involves the use of a low boiling point liquid to get out extract from materials. The choice of this technique is its ability to extract up to 99.5 % of the extract from its bearing material, leaving 0.5 % as a residual [25]. In this study, Soxhlet extractor apparatus was used while acetone served as the extracting solvent.

2.2.2 Cold Extraction method

The second extraction method involved soaking clean red onion skin in 1 percent NaOH for 24 hours. After that, the soluble extracts were filtered. To separate red onion skin extract from water, a rotary evaporator was used. Thereafter, the extract was air dried for approximately 30 days to allow any remaining moisture to evaporate.

2.3 Characterization of Scale Inhibitor and Deposited Scale Crystal

Fourier transform infrared spectroscopy, FTIR (Agilent Cary 630, US in transmission mode from 4000 to 600 cm^{-1}) was used to determine the presence of various functional groups in the proposed scale inhibitors. Further investigation to assess the morphology of the scale deposition with and without inhibitor was carried out using Scanning electron microscope, SEM (HITACHI S-4800, Japan). Calcium sulphate, calcium carbonate, barium sulfate, depositions were carefully sampled at low/no inhibitor dosage.

2.4 Evaluation of the Inhibition Efficiency

To evaluate the inhibition efficiency, a static jar test was done using National Association of Corrosion Engineers (NACE) Standard TM0374-2007 procedures [26,27]. Table 1 shows the solutions prepared for evaluating the scale inhibition performance. After weighing a specific amount of scale inhibitor (20 ppm, 40 ppm, 60 ppm, 80 ppm and 100 ppm) into the Erlenmeyer flask, 50 mL of cation solution (as specified in NACE standard) was added and agitated for proper mixing, followed by another 50 mL of anion solution. The test bottle was sealed with a plastic stopper and wrapped in polyethylene film. The wrapped Erlenmeyer flasks were placed in the oven for a predetermined time at a constant temperature. The inhibitor efficiency was calculated based on the remaining Ca^{2+} and Ba^{2+} ions in solution using the following equation.

$$\textbf{\textit{Percentage inhibition}}\ (\%) = \frac{m_2 - m_0}{m_1 - m_0}\ X\ 100\%$$

where m_2 is the mass concentration of Ca^{2+} and Ba^{2+} ions after inhibitor functions, m_1 is the mass concentration of Ca^{2+} and Ba^{2+} ions in the solution of 50 mL deionized water without the addition of anions solution, m_0 is the mass concentration of Ca^{2+} and Ba^{2+} ions of the solution with no inhibitor. The test of the mass concentration of Ca^{2+} and Ba^{2+} ions also followed the method specified by the standard, TM0374-2007.

Table 1. Static jar test conditions

Scale type	Brine	Concentration (g/L)	Condition
$CaSO_4$	cation	NaCl = **7**.50; $CaCl_2 \cdot 2H_2O$ = 11.10	70 ^oC & 90 ^oC
	anion	NaCl = **7**.50; Na_2SO_4 = 10.66	4 Hrs & 22 Hrs
$CaCO_3$	cation	$CaCl_2 \cdot 2H_2O$ = 12.15; $MgCl_2 \cdot 6H_2O$ = 3.68; NaCl = 33.0	70 ^oC & 90 ^oC 4 Hrs & 22 Hrs
	anion	$NaHCO_3$= 7.36; NaCl = 33.0	
$BaSO_4$	cation	NaCl = 7.50, $BaCl_2 \cdot 2H_2O$ = 0.66	70 ^oC & 90 ^oC
	anion	NaCl = 7.50, Na_2SO_4 = 0.80	4 Hrs & 22 Hrs

3. RESULTS AND DISCUSSION

3.1 Properties of ROSE and its Derivatives

Solubility of ROSE in aqueous medium and other parameters investigated are presented in Table 2. The pH measurement of the scale inhibitors, labelled ROSAC, ROS, were 8.5 and 8.0. This implies that the two products pass the criteria of a pH greater than five (>5). ROS was more soluble in water than ROSAC. This informed the decision to use ROS for further evaluation.

3.2 Characterization of Scale Inhibitor

The FTIR spectra shown in Fig. 2b was used to confirm the structure of the tested inhibitor (ROS). The peak value around 3257 cm^{-1} is attributed to the broad O-H stretching vibration for phenolic compounds. The aromatic C-H stretching vibrations are confirmed by peaks at 2918 cm^{-1} and 2851 cm^{-1}. The band at 1756 cm^{-1} is caused by the C=O stretching vibration, and the band at 1562 cm-1 is caused by the C=C and C=O stretching vibrations of the gamma-pyrone in ring C of the quercetin structure. The peak at 1439 cm^{-1} results from C-H asymmetric deformation vibration, while the peak at 1410 cm^{-1} results from the O-H deformation vibrations and the C-O stretching vibration. Peaks 870 cm^{-1}, 803 cm^{-1} and 784 cm^{-1} are due to the C-H out of plane deformation vibrations of the isolated aromatic C-H hydrogen atoms in the quercetin structure.The wavelength around 1071 cm^{-1} confirms the C-O-C stretching vibration. These observations agree with those of other works [28,3] and are consistent with the bonds that make up the major constituents (quercetin). The presence of quercetin and other important compounds supports the present study.

3.3 Evaluation of Scale Inhibition Performance

Fig. 3 depicts the inhibitor's inhibition performance of calcium sulfate (Fig. 3a), calcium carbonate (Fig. 3b), and barium sulfate (Fig. 4) with different inhibitor dosages at 71 ^{o}C. It was generally observed that increasing the inhibitor dosage increases the inhibitor's inhibition efficiency. When the dosage reaches a critical level, the inhibition rate remains constant or slowly decreases.

In the case of calcium sulfate (Fig. 3a), the inhibitor demonstrated a good inhibition efficiency at a low dosage of 20 ppm, and the best inhibition efficiency of 70 % was obtained at a dosage of 80 ppm. The inhibition rate was low across all dosages in the calcium carbonate inhibition performance test (Fig. 3b). Based on the scope of this work, an inhibition efficiency of only 50% was obtained at the optimum dosage of 100 ppm. However, barium sulphate inhibition evaluation provided the highest inhibition efficiency of 83 % at a dosage of 80 ppm before reaching a plateau state (Fig. 4). As a result, ROS has a relatively good inhibition performance on both the calcium and barium sulfate scales. Figs. 3,4,5 (b) shows a close relationship in the performance of ROS and a commercial scale inhibitor (CSI) evaluated at the same laboratory condition.

3.4 Effects of Temperature and Time on the Inhibition Performance of the Antiscalant

3.4.1 Temperature

For inhibition property on calcium sulfate (Fig. 5a), when a dosage of 60 ppm was added, an identical inhibition rate of 65 % was obtained at 71 ^{o}C and 90 ^{o}C. From Fig. 5b, the scale inhibition efficiency on calcium carbonate is greater than 40% with a dosage of 20 ppm at 71 ^{o}C and nearly 60 ppm at 90 ^{o}C. This could be attributed to the fact that high temperatures facilitate the formation of calcium carbonate; as a result, an additional inhibitor dosage is required for improved efficiency. When the dosage is increased to a certain level (60 ppm), the inhibition rate at 90 ^{o}C is greater than at 71 ^{o}C, indicating that more inhibitor adsorption occurs on the calcium carbonate crystal at higher temperatures [29].

Table 2. Properties of inhibitors

ROSE Derivatives	ID	Solubility in water	Colour	pH
ROSE Extracted with Acetone	ROSAC	Insoluble	Light Brown	8.5
ROSE Extracted with 1% NaOH in water	ROS	Soluble	Reddish Brown	8.0

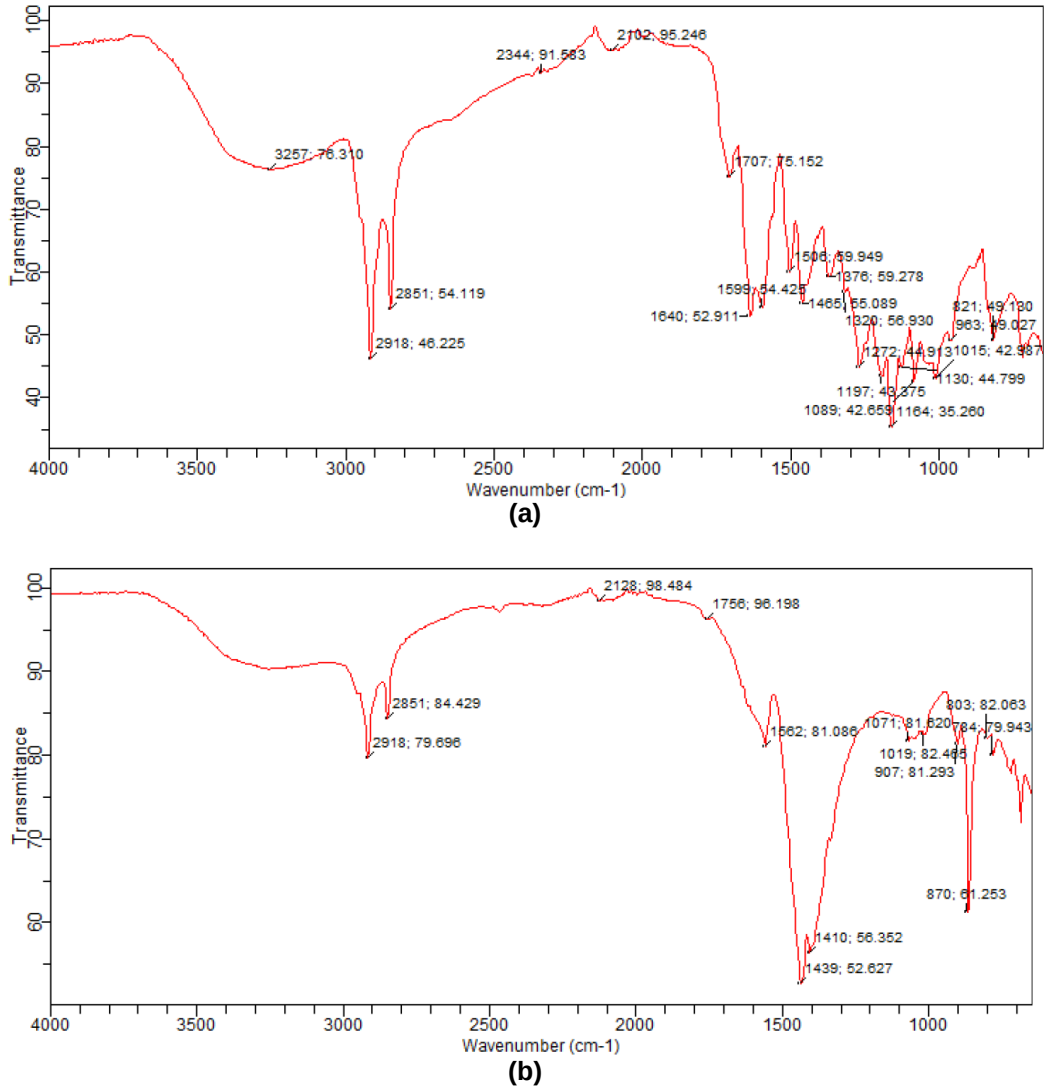

Fig. 2. FTIR spectrum of (a) ROSAC and (b) ROS

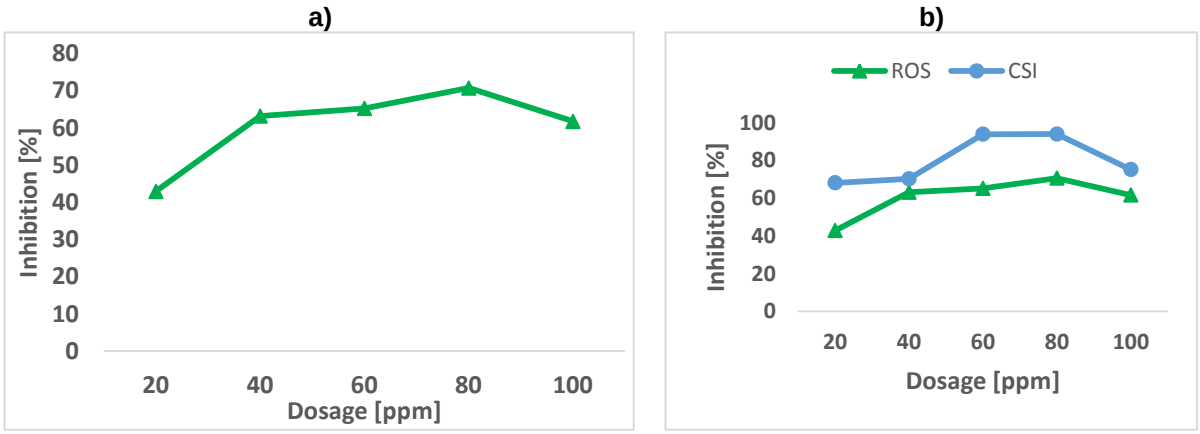

Fig. 3. a) Relation curve of dosage to inhibition efficiency of ROS on calcium sulfate scale, (b) comparison of ROS and CSI performance on calcium sulphate scale

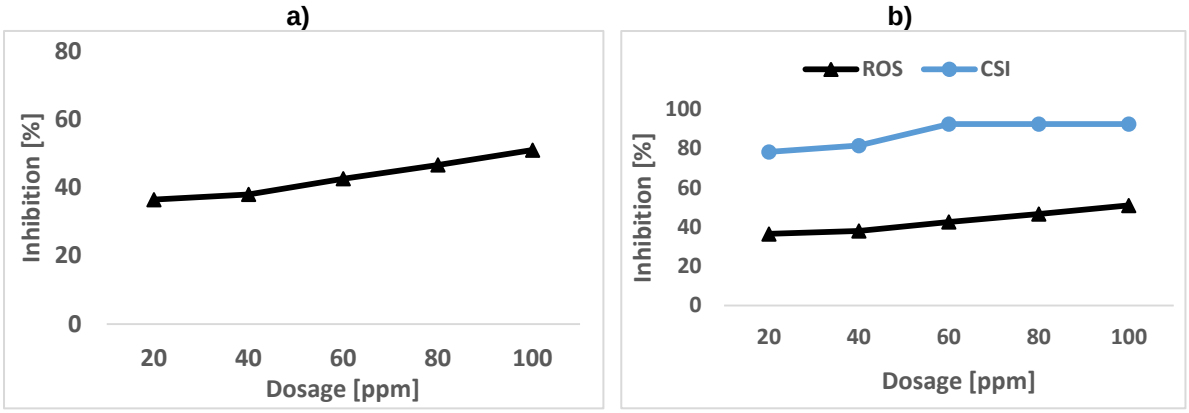

Fig. 4. a) Relation curve of dosage to inhibition efficiency of ROS on calcium carbonate scale, (b) comparison of ROS and CSI performance on calcium carbonate scale

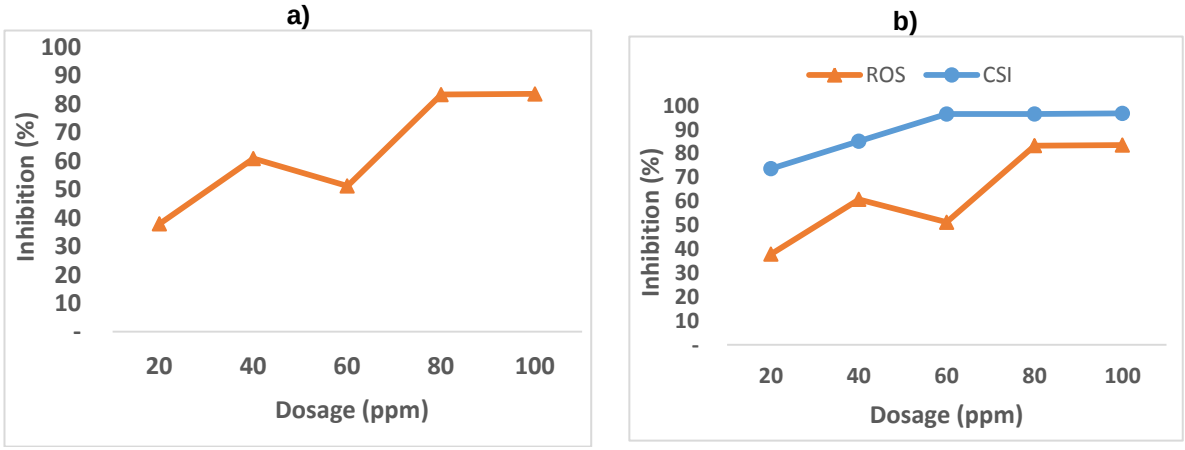

Fig. 5. a) Relation curve of dosage to inhibition efficiency of ROS on barium sulfate scale, (b) comparison of ROS and CSI performance on barium sulphate scale

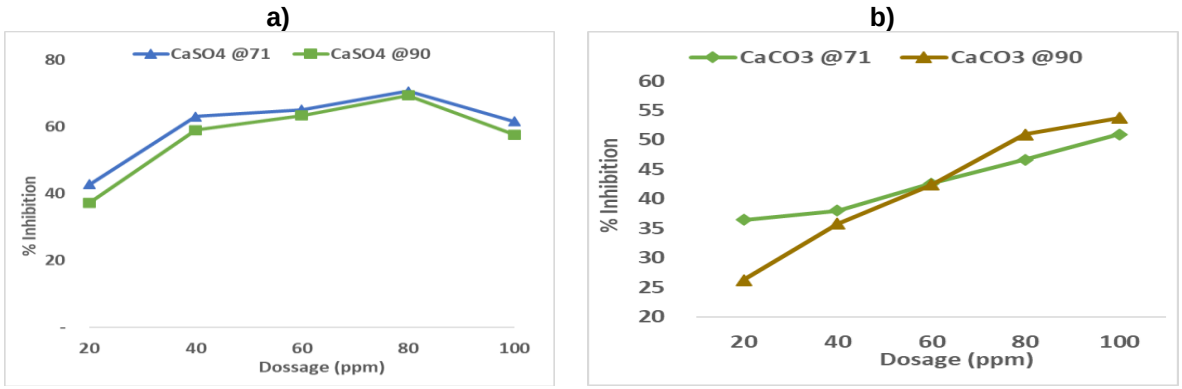

Fig. 6. Effects of temperature on the inhibition performance of ROS on (a) Calcium sulphate (b) calcium carbonate

For barium sulfate (Fig. 6), when the optimal dosage at 60 ppm was added, almost identical inhibition rate of about 60 % was obtained at both 71 ^{o}C and 90 ^{o}C. Therefore, it can be inferred that at higher temperatures, additional inhibitor dosage is required to maintain a high inhibition rate for $CaCO_3$ inhibition, whereas $BaSO_4$ control requires no additional dosage in the scope of this work.

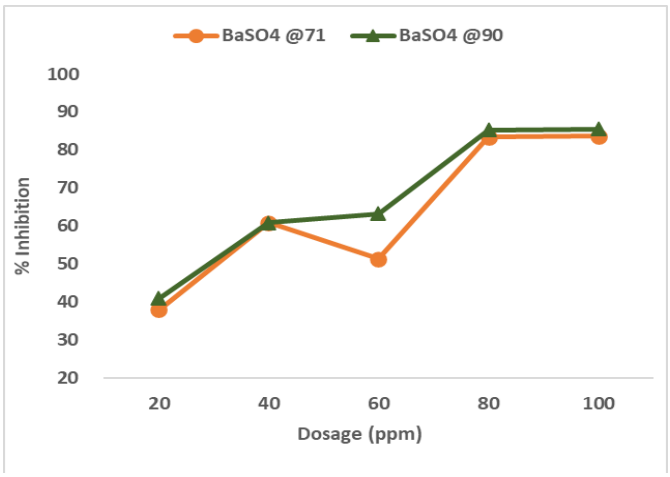

Fig. 7. Effects of temperature on the inhibition performance of ROS on barium sulphate

3.4.2 Time

Effect of inhibition time on the performance of scale inhibitor is shown in Fig. 7. For calcium sulphate deposition inhibition experiments (Fig. 7a), the scale inhibition rate increases at a higher time duration. The inhibition efficiency on calcium carbonate is also increasing at a higher time duration (Fig. 7b). At an evaluation time of 22 hrs, $CaCO_3$ has an inhibition rate of 50 % which is higher when compared to 39 % obtained for 4hrs inhibition time. This indicates that the inhibitor plays a continuous $CaSO_4$ and $CaCO_3$ inhibition performance.

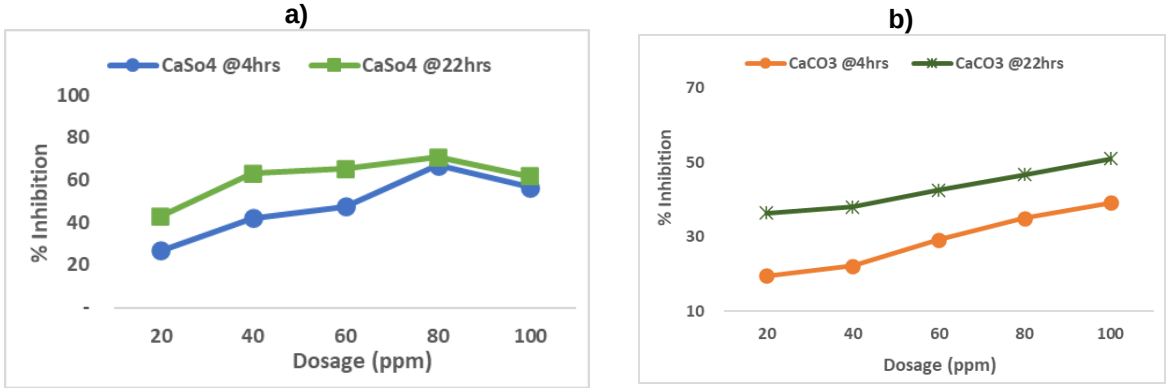

Fig. 8. Effects of time on the inhibition performance of ROS on (a) Calcium sulphate (b) calcium carbonate

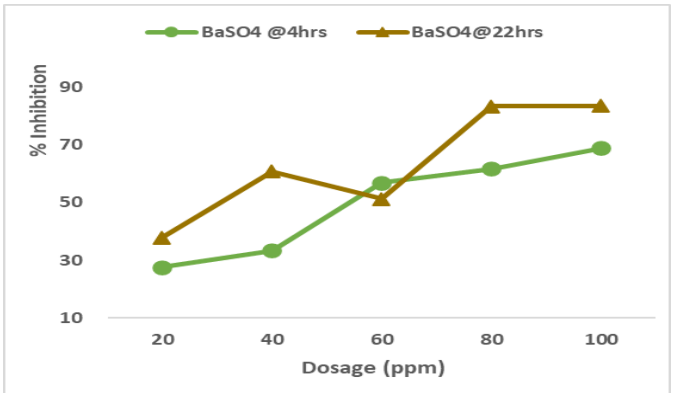

Fig. 9. Effects of time on the inhibition performance of ROS on barium sulphate

For barium sulphate deposition (Fig. 8), there was also an increase in inhibition efficiency for 22 hrs up until 60 ppm, before a sharp decrease in the inhibition rate and thereafter, a rise with increased dosage. This phenomenon suggests that a sufficient dosage for controlling barium sulfate scale deposition may be required in a long time.

3.5 SEM Analysis

SEM images of calcium sulphate, calcium carbonate, and barium sulfate, obtained from the absence and low dosage of inhibitor is shown in Figs. 9, 10, 11 respectively. When an inhibitor was added to calcium carbonate crystals, they became looser (Fig. 10c), an indication of crystal

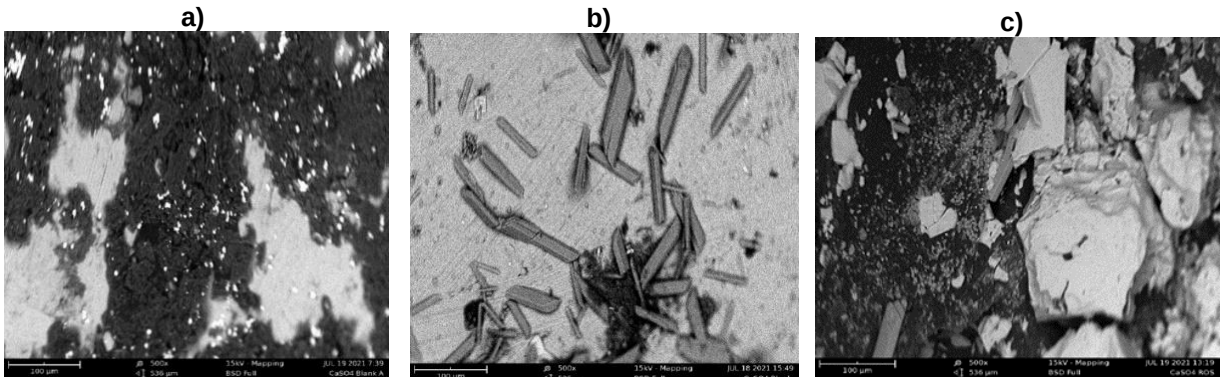

Fig. 10. SEM images of $CaSO_4$ without inhibitor before and after conditioning (a, b) and with inhibitor (c)

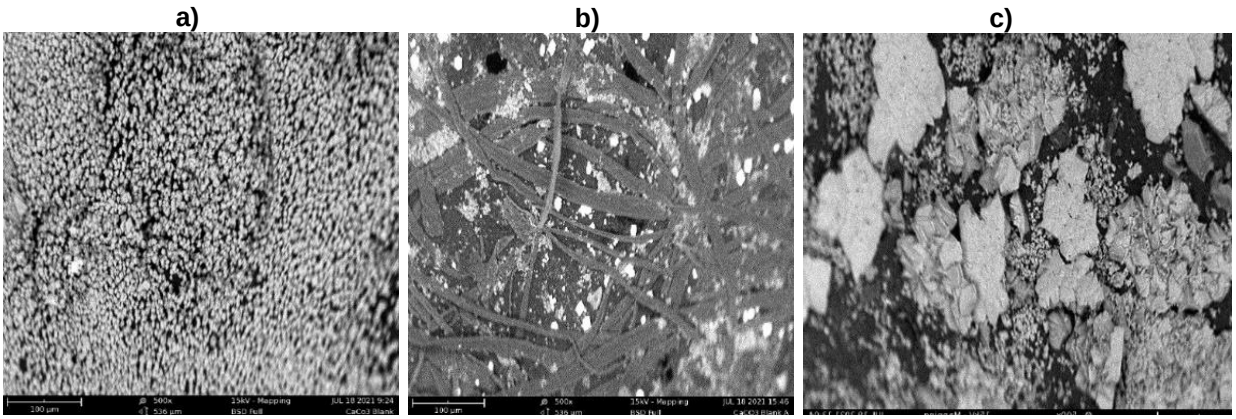

Fig. 11. SEM images of $CaCO_3$ without inhibitor before and after conditioning (a, b) and with inhibitor (c)

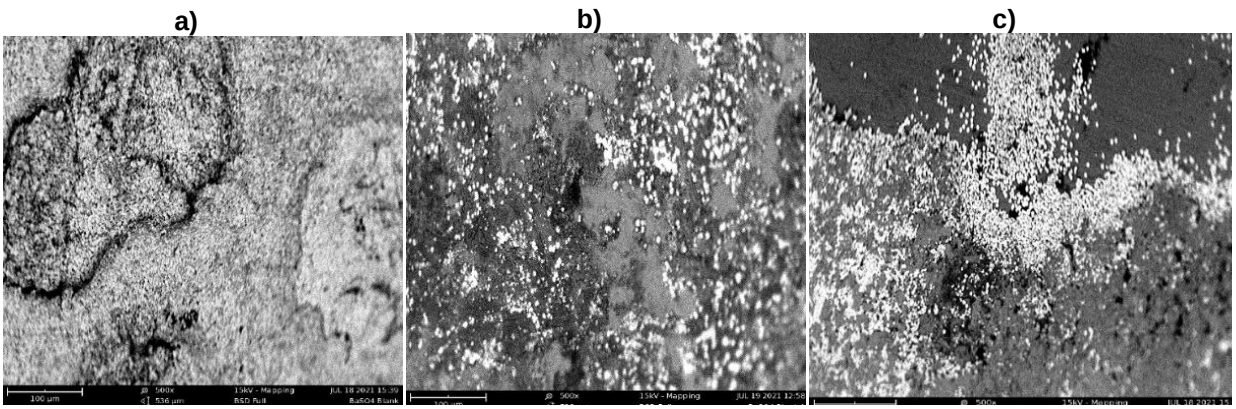

Fig. 12. SEM images of $BaSO_4$ without inhibitor before and after conditioning (a, b) and with inhibitor (c)

growth suppression. A similar phenomenon was observed for calcium sulfate (Fig. 9c), where the dense structure changed to lose lump. Fluid turbulence could easily disrupt the loose material, causing it to become suspended in the solution [29,30]. Also, in the presence of the inhibitor, the diameter of the calcium carbonate and calcium sulfate crystals increases, indicating that the inhibitor may have some influence on the surface phase of these scales [31]. Fig. 11 depicts a densely packed crystal of barium sulphate before conditioning and without inhibitor (Fig. 11a). Fig. 11c shows that the addition of the inhibitor resulted in lose particles. This demonstrates that the preferential growth of barium sulphate crystals was strongly inhibited.

4. CONCLUSION

1. A potential green scale inhibitor for oilfield is developed and evaluated on calcium sulfate, calcium carbonate, and barium sulfate scales. For calcium sulphate, the inhibition efficiency could be as high as 69%, and for barium sulphate, it could be as high as 85%. Calcium carbonate had a lower inhibition rate than the other scales evaluated. The highest inhibition rate of 53% was obtained at a dosage of 100 ppm.
2. The developed inhibitor has good chemical stability, high-temperature resistance, and good scale inhibition effects at low dosage.
3. Extra inhibitor is required for good inhibition of calcium carbonate deposition at higher temperatures, but it is not required for calcium sulphate and barium sulfate scale inhibition.
4. Because the diameter of calcium sulfate and calcium carbonate formed when inhibitor is present is larger than that formed at blank conditions, proper dosage is critical for calcium sulfate and calcium carbonate prevention.
5. Comparison with an existing commercial inhibitor (CSI) show that ROS has a good potential as green scale inhibitor in the oil industry.

ACKNOWLEDGEMENTS

The authors are grateful to Tetfund for the Institute Based Research (IBR) grant received which enabled the completion of this work.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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