

Research Article

Application of Ultraviolet Radiation to Control the Calcium Carbonate Scale Formation and Deposition on the Membranes

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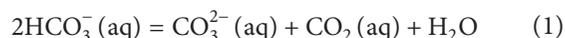
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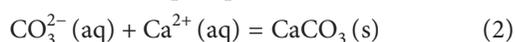
Scale formation on surfaces in contact with water supersaturated with calcium carbonate creates technical problems, including heat transfer hindrance, energy consumption, and equipment shutdown. Thus, nowadays, there is an increasing need for new approaches that are environmentally friendly and economically feasible. In this work, for the first time, calcium carbonate growth was investigated using UV light exposure, and the growth rate was compared with control and commercial antiscalant. Saturated calcium carbonate samples were exposed to UV radiation; the growth rate of calcium carbonate crystals was monitored at different time intervals. Results clearly show that about 85% decrease in crystal growth rate was observed when compared to 43% after the addition of 3 mg/L of amino tris(methylene phosphonic acid) antiscalant. Calcium carbonate scale deposition on hydrophobic and hydrophilic membranes was investigated. The amount of scale deposited in the case of a UV-treated sample is insignificant when compared to control samples. Thus, the exposure of UV might help to improve the membranes' lifetime. X-ray diffraction and scanning electron microscopy analyses revealed that UV light treatment produced mostly calcite crystals. The produced calcites are less dense and less adherent, and it can be easily removable when compared to other types of calcium carbonate phases. Thus, UV radiation is an efficient green approach for calcium carbonate scale mitigation on membrane surfaces.

1. Introduction

Calcium carbonate scale formation is a major challenge in the transportation and operational oil pipelines in oil industries. The risk of scale deposition occurs during the course of injection operations mostly because of change in temperature that aid solution rapid supersaturation, the process that encourages the decomposition of HCO_3^- according to the following reaction:



The mineral scale can also be formed from the reaction of two chemicals that lead to precipitation:



The calcite crystals are large, but, in the presence of impurities, they take the form of uniform, finely divided crystals. The CaCO_3 precipitate started while agglomerating Ca^{2+} and CO_3^{2-} cluster, and colloidal nuclei grow and become a stable crystal.

Inorganic mineral deposits, especially, calcium carbonate, were found in an aragonite form in nature. At ambient conditions, it forms stable calcite form while aragonite is the high-temperature polymorph. Vaterite is another thermodynamically unstable form [1]. Their transformation into stable calcite is usually problematic in oil and gas processes, paper production, thermal power plants, nuclear power stations, and desalination facilities [2].

The cost for maintenance and cleaning of scale pipelines and heat exchangers in industries is very high each year [3].

Calcium carbonate scaling is a dominant fouling mechanism bedeviling fluid-based sectors [4]. The deposit of carbonate minerals is a subject of vital interest due to its side effects in industrial processing, which has been continuing to challenge in ensuring flow assurance and continuous operation [5]. Much research has been done to evaluate a suitable scale inhibition technique that may mitigate the damage caused by scales in the oil and gas industry [6–8], among which are chemical and nonchemical based methods. It is worth mentioning that little work is reported on the role of light places in the fouling mechanism.

Calcium scale prevention involving chemicals is a common practice industry used for scale prevention in industrial fluid flow treatment. Chemical inhibitors are abundantly used in the industry. Functional groups like polyphosphate, phosphate esters, organic phosphonates, polyacrylates, and other various copolymers of phosphonates, carboxylate, and sulfonate help the inhibition of calcium carbonate [9, 10]. The following vital parameters were considered when selecting a suitable chemical inhibitor: solution composition, compatibility, stability, solubility, toxicity, hydrolytic stability, degradation level, and minimum inhibitory concentration (MIC) [11–13]. Temperature is another important factor considered in the choice of efficient chemical inhibitor. The MIC represents the minimum effective concentration of chemical scale inhibitors to prevent scaling in industrial processes [13, 14]. Recent findings have shown that the chemicals, as mentioned above, pose a great concern to the environment and public health and are also very expensive [15–17].

There are also some nonchemical based scale inhibitors that serve as an alternative to magnetic, electronic, and electrolytic processes [18–20]. Magnesium was another method employed in an attempt to mitigate the problems caused by scale formation in water treatment without the use of chemicals [21]. Even though earlier works done in this area were not entirely sure about the mechanism of the magnetic field in addressing this issue, [22] provided some insight into the research field. However, [21, 23] in their work suggested that the magnetic field modifies the nucleation stage and crystal growth, which goes a long way to affect the rate of formation and crystal deposition. Various experimental designs were proposed for electromagnetic antiscaling studies [2, 24, 25]. Among these studies, the heat exchanger with a magnetic field of an orthogonal flow fluid circulating the system maximizes the scale formation at a specific temperature and pH [26, 27].

It was observed that calcium carbonate was reduced by 50% after samples exposure to the magnetic field on the equilibrium of carbonates with the change in PH. The application of a magnetic field directly influences sample pH and the formation of crystal growth in the equilibrium condition. A reduction in the size of crystals was observed [22, 26, 27].

Applying the electric field is another nonchemical method reported in the literature to regulate the crystal growth rate of calcium carbonate [28]. Various strategies are reported on the electrical effectiveness potential on the antiscaling treatment of water, where [28, 29] focused on the

way of preventing the scaling in water treatment. They used a pulsed spark discharge approach to enhance the precipitation of dissolved calcium ions in cooling towers. This process led to heat reduction in water hardness close to 20–26% and led to the conclusion that electrolysis was the reason for this observation. Applying high electric potential also helps to enhance bicarbonate dissociation [30]. Dhanasekaran and Ramasamy [31], in their work, asserted that the tendency for nucleation to occur is reduced with an increase in the strength of the electric field at certain angles.

According to [3, 24, 32], the effect of the electric field on calcium carbonate scale formation has been studied by applying an electric field on two graphite electrodes immersed in the working solution at different levels of voltage, frequency, and degree of supersaturation.

Little is known about the efficacy of ultraviolet light on calcium scale, and this particular work is to shed light on this area. Among the earlier mentioned methods, ultrasonic is tested to be more effective in the crystallization process [33–40]. The effect of applied ultrasonic on a liquid medium is that it exerts alternating and rarefaction within the liquid, generating bubbles at rarefaction stage. The bubbles outlive repeated cycles of compression and rarefaction until a critical size is reached, and breakdown occurs, starting what is known as cavitation. That is, the rapid formation, growth, and violent disruption of bubbles and chemical and physical changes are promoted due to powerful ultrasound. The mechanism of single or multibubble cavitation indicates that the collapsing bubble behaves as a particular microreactor where ions and radicals in the excited state are involved in the reaction outcome. Near the liquid-solid interface, cavity collapse leads to boundary layer destruction and mass/heat transfer improvements. The ultrasonic technique is seen as an effective way of achieving faster, proper, and uniform nucleation, comparatively easy nucleation of particles at lower supersaturation levels, and reduced agglomeration [41, 42].

However, deliberate efforts are geared toward developing more fouling retarding techniques that are low cost and remarkably benign to the environment. The finding shows that Dallas [31] has studied the effect of UV light on calcium carbonate scale formation, reporting inhibition of calcium carbonate scale compared to the untreated water.

2. Materials and Methods

2.1. Materials. Sigma-Aldrich, Germany, supplied calcium chloride ($\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$), sodium chloride (NaCl), sodium bicarbonate (NaHCO_3), and ethylenediaminetetraacetic acid (EDTA) of analytical grade. Hydrex 4102 RO antiscalant and polypropylene and polysulfone membranes are supplied by VEOLIA WATER STI, Khobar, Saudi Arabia. Hydrex 4102 chemical constituents are amino tris(methylene phosphonic acid) and phosphonic acid with a pH of 11.00–12.00 and a specific gravity of 1.35–1.45.

2.2. Methods. Synthetic seawater solution was prepared by mixing 86.0 mM sodium chloride, 4.50 mM calcium chloride, and 9.50 mM sodium bicarbonate solutions prepared

from standardized stock solutions prepared using deionized water using a Millipore Q-Plus 185 system. The pH of the solutions was measured by a glass/saturated calomel electrode (Metrohm), calibrated before and after each experiment with 4, 7, and 10 standard buffer solutions. The pH of the working solution was adjusted by the addition of hydrochloric acid and ammonia buffer. Calcium carbonate precipitation started when calcium chloride and sodium bicarbonate were added.

UV light was generated by a UV lamp source having a 15 mm diameter, $106 \mu\text{W}/\text{cm}^2$ intensity, 54 V operating voltage, and 12 W output power, emitting broadband of UV light, with eight filters for specific wavelength selection, producing an average intensity of $2 \text{ mW}/\text{cm}^2$ at a distance of 2 cm from the working solution. Figure 1 shows the experimental setup of the UV treatment experiment. For the determination of the calcium ion concentration, 10 mL of the working solution was taken every 15 min, filtered by $0.025 \mu\text{m}$ membrane filter, and titrated with EDTA standardized solution.

At the end of the experiments, the solutions were filtered, and the precipitates were collected and examined by XRD for the determination of crystal types of the deposited CaCO_3 scales. Sample sizes of $10 \times 10 \text{ mm}$ were prepared by manual cutting using a saw blade for each fouled tube and were analyzed at diffraction angles of 10 to 110° .

For the calcium carbonate scale deposition on membranes study, hydrophobic porous membrane (polypropylene) and hydrophilic membrane (polysulfone) were placed in the sample solution. The number of crystal deposits on the membrane was monitored at different intervals of time.

3. Results and Discussions

Calcium carbonate scale mitigation in water using UV light was only reported by [31]. Calcium carbonate is a sparingly soluble salt, growing by a parabolic rate law. Hence, the rate-limiting step of its crystal growth involves the dehydration of the growth units and the surface diffusion of these dehydrated growth units into the lattice from the adsorption site [31]. Calcium carbonate scale inhibition (CCI %) can be calculated by

$$\text{CCI \%} = \frac{[\text{Ca}^{2+}]_{\text{sample}} - [\text{Ca}^{2+}]_{\text{blank}}}{[\text{Ca}^{2+}]_{\text{initial}} - [\text{Ca}^{2+}]_{\text{blank}}} \times 100\%, \quad (3)$$

where $[\text{Ca}^{2+}]_{\text{blank}}$ is the calcium ion concentration of the blank solution, $[\text{Ca}^{2+}]_{\text{initial}}$ is the calcium ion concentration at $t=0$, and $[\text{Ca}^{2+}]_{\text{sample}}$ is the calcium ion concentration at time (t), respectively. The effect of antiscalants on calcium carbonate scale formation has been investigated by using ATMP (amino tris(methylene phosphonic acid)) as a scale inhibitor. ATMP, commercially named as Hydrex 4102, is one of the commonly used antiscalants used for calcium carbonate scale inhibition in water since it has an excellent chelating ability with calcium ions, low threshold inhibitory dosage, and powerful lattice distortion process. In chemical treatment experiments, different dosages of ATMP have

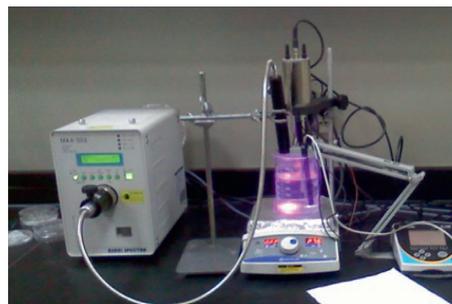


FIGURE 1: Experimental setup of UV treatment experiment.

been used, ranging from 1 to 10 mg/L, to test for the threshold concentration suitable for calcium carbonate scale inhibition.

Starting with 1 mg/L, CCI has increased by about 1% compared to the untreated system, as shown in Figure 1. When the dosage is 2 and 3 mg/L, the scale inhibition has increased by about 4%. Meanwhile, adding 5 mg/L has increased CCI to about 80% compared to 65% obtained for the control experiment. The 10 mg/L dosage achieved the best performance regarding scale inhibition, increasing CCI% to about 82% after 60 minutes. This finding indicates that 10 mg/L is the threshold inhibitory dosage of ATMP as calcium, a scale inhibitor for calcium carbonate (Figure 2).

The effect of the addition of ATMP during UV (265 nm) irradiation of the working solution has been investigated by adding antiscalant at different dosages from 1 to 10 mg/L. The addition of 1 and 2 mg/L increased the precipitation of calcium carbonate (Figure 3). Applying UV radiation dissociates the antiscalant into fragments that were unable to inhibit the scale formation. When increasing the dosage to 3 mg/L, the scale inhibition has been enhanced because the antiscalant molecules have blocked some of the active sites that are available for crystal growth. Meanwhile, increasing the antiscalant concentration to 5 mg/L and 10 mg/L has raised the scale inhibition to 96% compared to 36% for the untreated case.

3.1. Factors Affecting Scale Inhibition

3.1.1. Effect of pH. Figure 4 depicts the effect of pH on calcium carbonate scale inhibition using UV light of 265 nm radiation for 60 minutes. At a pH of 7.0, the scale inhibition was almost steady, reaching 97.1% after 60 minutes, while decreasing to 91.8% at a pH of 8. On the other hand, increasing pH to 9 had a drastic effect on the scale inhibition behavior, falling slowly in the first 15 minutes and then sharply decreasing in the next 15 minutes. After 60 minutes of exposure, the scale growth rate drops to 71% when compared to control samples.

This dramatic effect of pH can be interpreted based on the pH dependence of carbonate concentration in solution. Bicarbonate ions were dissociated into carbonate according to the following equation:

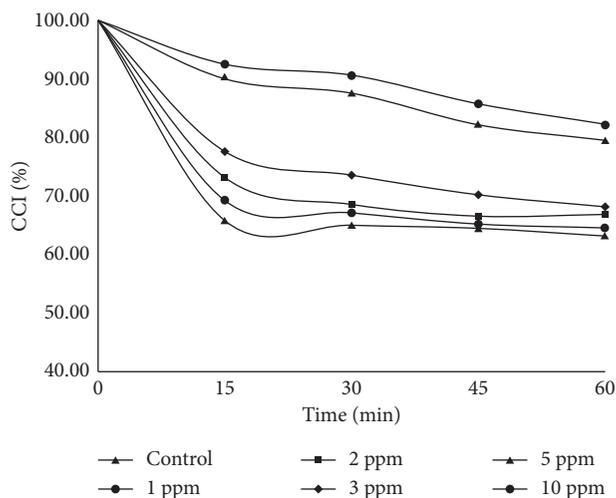


FIGURE 2: Effect of ATMP concentration on calcium carbonate scale formation changes the legend for 1 mg/L and 10 mg/L, as a function of time.

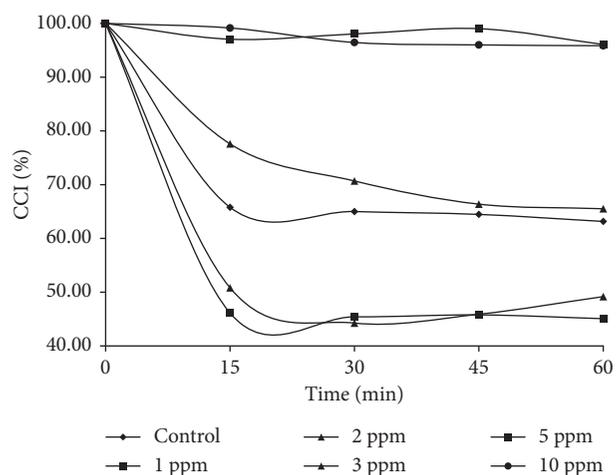


FIGURE 3: Influence of UV radiation on the ATMP concentrations on calcium carbonate scale inhibition, as a function of time.



According to the Henderson-Hasselbalch equation, as pH increases, the carbonate concentration increases as indicated in Table 1:

$$\text{pH} = \text{p}K_a + \text{Log} \frac{[\text{CO}_3^{2-}]}{[\text{HCO}_3^-]} \quad (5)$$

By increasing sample pH, an increase in the carbonate concentration by two orders of magnitude was noticed. An increase in sample pH also changes carbonate ions recombination, hence increasing the rate of calcium carbonate precipitation.

3.1.2. Effect of Radiation Energy. The impact of radiation energy was studied in UV and visible regions. For the visible region, the solution was irradiated with 385 nm radiation,

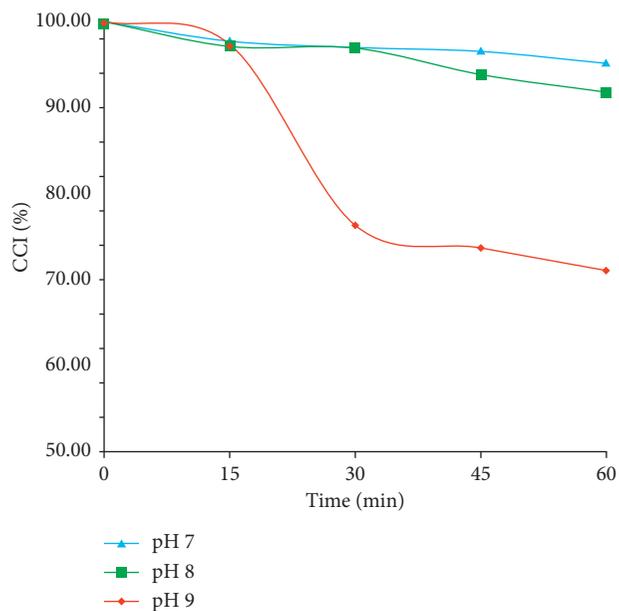


FIGURE 4: Effect of pH on calcium carbonate scale inhibition, as a function of time.

TABLE 1: Effect of pH on carbonate concentration in 9.50 mM bicarbonate solution.

pH	$[\text{CO}_3^{2-}]$ (mM)
7	4.44×10^{-9}
8	4.44×10^{-8}
9	4.44×10^{-7}

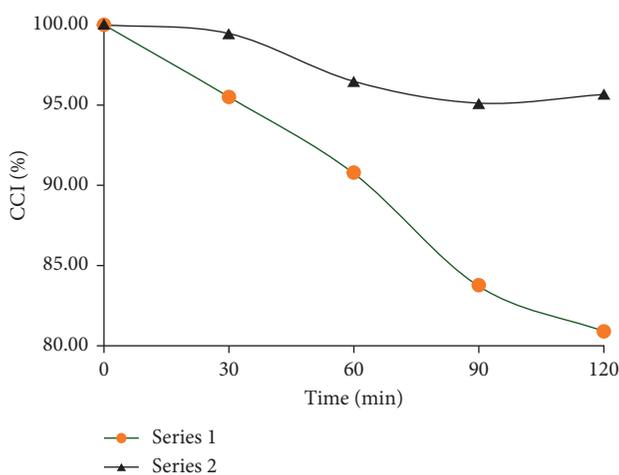


FIGURE 5: Effect of radiation energy on calcium carbonate scale inhibition, as a function of time.

providing scale inhibition of 80.9% after 120 minutes, compared to 95.6% when irradiated with 265 nm UV radiation, as shown in Figure 5.

The effect of UV radiation can be explained through the calcium ion acid-base character. The calcium ion is a weak acid, and when excited, it becomes much more vulnerable.

TABLE 2: Rate of calcium carbonate crystal growth for UV-treated and UV-untreated water.

Water sample	$f(s)$	$R (\times 10^{-8} \text{ mol}^{-2} \text{ s}^{-1})$
Untreated	1.05	7.36
Hydrex 4102	0.92	4.22
385 nm	0.92	1.87
265 nm	0.68	1.12

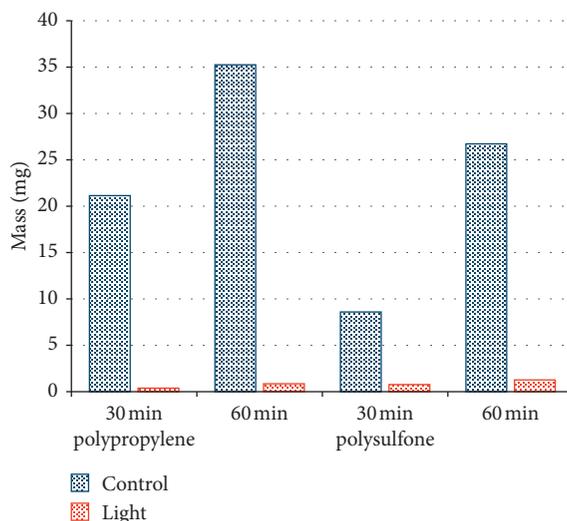


FIGURE 6: Weight measurements of calcium carbonate deposits with and without UV light treatment.

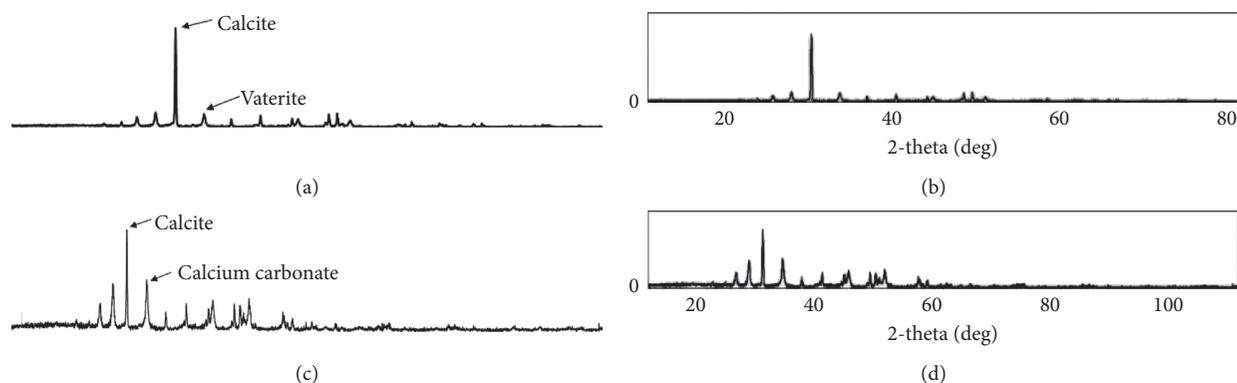


FIGURE 7: XRD spectra for (a) untreated case and (b) standard peaks of calcite and Vaterite and for (c) UV-treated case and (d) standard peaks of calcite and calcium carbonate.

Applying 385 nm visible light excites some of the energetic calcium ions becoming weakly acidic, and these acidic ions prevent recombination. When shifting from visible to UV region, the radiation energy increases, exciting more calcium ions, resulting in much more efficient scale inhibition.

3.2. Calcium Carbonate Crystal Growth Rate. Calcium carbonate scale formation involves supersaturation, nucleation, crystal growth, and precipitation. Thus, affecting any of these steps can retard the crystal growth rate. Table 2 shows the percentage of crystal growth of calcium carbonate for the untreated, chemically treated, and irradiated water samples. The rate of crystal growth decreased by about 43% when

adding 3 mg/L of Hydrex 4102 antiscalant. The chemical antiscalant might be blocking some of the active sites of the calcium carbonate nuclei, therefore, decreasing the number of calcium carbonate crystals precipitated. The effect of UV radiation can be explained through the calcium ion acid-base character. When the calcium ions are subjected to UV radiation, some of the excited, energetic calcium ions become weakly acidic, and it reduces the recombination of calcium carbonate crystal growth. The crystal growth rate decreased by about 75% when water was irradiated with 385 nm. When shifting from visible to UV region, 265 nm, the radiation energy increases, exciting more calcium ions, degrading the crystal growth by about 85%. Thus, 265 nm UV radiation was used for further studies. Hence, UV light

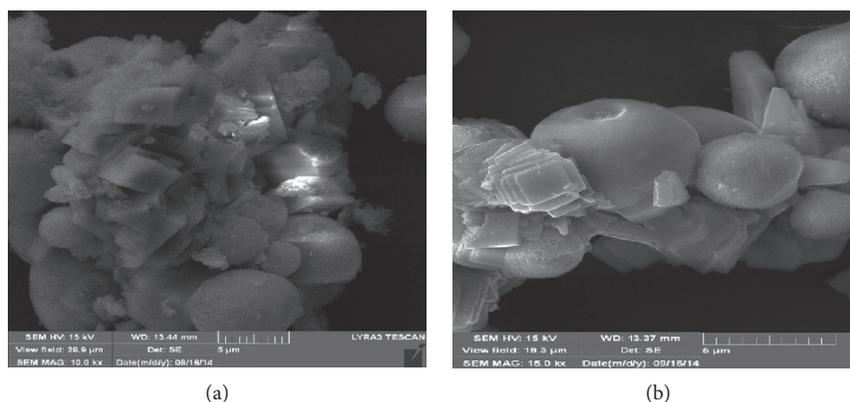


FIGURE 8: SEM images for deposits of (a) untreated water and (b) UV-treated water.

treatment is much more efficient in calcium carbonate scale inhibition compared to commercial antiscalants. The application of UV light has more advantages than chemical treatment. The UV radiation is commonly used in water treatment to get rid of pathogens without having any harmful effect when compared to chemical antiscalants.

The rate of crystal growth depends on the relative supersaturation and the number of the available surface active sites according to the following equation:

$$R = k_g \cdot f(s) \cdot \sigma^n, \quad (6)$$

where k_g is the crystal growth rate constant, $f(s)$ is a function of the number of available active sites for crystal growth, σ is the relative supersaturation, and n is the order of crystal growth, respectively.

3.3. Characterization of Scale Deposits

3.3.1. Membrane Scale Deposit Measurements. The amount of calcium carbonate deposited on polypropylene and polysulfone membranes was investigated by immersing the membrane separately inside the working solutions from 30 to 60 minutes. The amount of scale deposited for UV-treated samples is very low compared to the untreated one (Figure 6). This observation is precious for predicting the lifetime of the desalination membranes that are affected by the amount of scale deposited. Thus, using a UV light for scale inhibition increases the lifetime of the membrane due to the reduced membrane fouling.

3.3.2. XRD Analysis. Figure 7 shows XRD images of the deposits obtained from untreated and UV light treated water. The untreated control sample produces calcite and vaterite form of crystals. Interestingly, only calcite was formed in the case of the UV light treated samples. These crystals are less dense, less adherent, and easily removable when deposited compared to aragonite [11].

3.3.3. SEM Analysis. SEM images of the deposits obtained from untreated and UV light treated water are shown in Figure 8. The SEM images reveal that calcite crystals with

small particle sizes for the UV radiation samples and vaterite form control samples.

4. Conclusion

In the reported work, we investigated the scale inhibition property of ultraviolet light on calcium carbonate. An outcome is an excellent approach to the calcite scale problem that is bedeviling the oil and liquid-based industries over the years. The following are the conclusions drawn from this study.

- (1) Applying UV light, the calcium carbonate crystal growth rate decreased by about 85% when compared to 43% for 3 mg/L spiked amino tris(methylene phosphonic acid) antiscalant.
- (2) Experimental results on polypropylene and polysulfone membranes deposition study indicate that the amount of scale deposited in the case of a UV-treated sample is insignificant when compared to the untreated one. Hence, it is expected that UV treatment might increase the lifetime of the membrane.
- (3) XRD and SEM analyses revealed that UV light treatment produced mostly calcite crystals, which are less dense, less adherent, and easily removable than other types of calcium carbonate phases, hence increasing the lifetime of the membrane.
- (4) We believe that UV radiation is an efficient green approach for calcium carbonate scale mitigation on membrane surfaces.

Data Availability

The data used to support the findings of this study are included in the article.

Disclosure

The manuscript is part of the M.S. thesis "Calcium Carbonate Scale Inhibition by Non-Chemical Methods."

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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