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Evaluation of PASP–ZnO Nanocomposites as Eco-Friendly Scale Inhibitors for Calcium Carbonate Deposition

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ABSTRACT

Carbonate scaling is a significant operational issue in oil and gas production and water treatment systems, leading to blockages and reduced efficiency. Conventional scale inhibitors often face environmental restrictions, prompting a shift towards greener alternatives like polyaspartic acid (PASP). However, the performance of traditional PASP is limited. This study evaluates an eco-friendly PASP–ZnO composite as an enhanced scale inhibitor for calcium carbonate. The nanocomposite was synthesized and characterized using Energy-Dispersive X-ray (EDX) and Fourier-Transform Infrared Spectroscopy (FTIR), which confirmed the successful incorporation of zinc. The scale inhibition performance was assessed through static inhibition tests and Scanning Electron Microscopy (SEM) analysis. Results showed that the PASP-Zn composite exhibited more stable and effective inhibition than pure PASP, reaching an efficiency of approximately 44.85% at a 50 ppm concentration. SEM images revealed that the composite significantly distorted the calcium carbonate crystal morphology, indicating its effectiveness in disrupting scale formation. These results indicate that the PASP–ZnO nanocomposite is an environmentally friendly and effective inhibitor for controlling calcium carbonate scaling and may also serve as a promising modifier for polymeric membranes to enhance their antifouling properties.

Keywords: Polyaspartic Acid (PASP), $CaCO_3$ Scaling, Green Inhibitor

1.0 INTRODUCTION

Calcium carbonate ($CaCO_3$) is one of the inorganic scale that is frequently encountered in both oil and gas production facilities and membrane-based water treatment systems [8]. The deposition of this scale happened when the solubility of calcium and carbonate ion exceeded its limits as shown in Figure 1. This situation is commonly influenced by the changes in temperature and pressure during oil extraction and production processes [17, 20]. In membrane-based application, the presence of high concentration of calcium ion and bicarbonate ion in the feed water can promote the deposition of carbonate scale on the membrane surface [3]. These precipitation results in operational issues such as partial or

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complete blockages that diminish flow rates, increase maintenance costs, and potentially lead to the plugging of critical production equipment [13]. While in membrane application, these scaling reduces the efficiency of water treatment systems by lowering the flux rates and thus demands higher energy inputs to maintain desired operational pressures [26].

Based on this problem, the use of scale inhibitors in oil and gas industry as well as in water treatment processes plays a vital role in which this inhibitor can hinder the crystal nucleation and growth. In addition, this inhibitor also can alter the morphology of scale deposits and ensure that these minerals remain in the fluid phase rather than precipitate onto the surfaces. There are several types of scale inhibitors such as phosphate-based and polymeric-based such as poly acrylic acid (PAA), Amino-tris(methylene phosphonic acid) (ATMP), and 1-Hydroxyethylidene-1,1-diphosphonic acid (HEDP). However, these chemicals are harmful to the environment in which these chemicals persist in the environment for a long period, resist microbial degradation and poses risk to both marine and human health if release into open water system. Besides that, phosphate containing inhibitor also may contribute to eutrophication which may stimulate excessive algal growth that may deplete the dissolve oxygen in water. Therefore, due to the environmental concern, the application of phosphate-based and polymeric-based scale inhibitor are limited due to restricted environmental legislation [25]. Current trend in the development of scale inhibitors is the use of copolymers that are phosphorus-free and environmentally friendly. These copolymers are created by polymerizing monomers that have multiple functional groups [23]. Polyaspartic acid, or PASP, is currently being produced and used as a green scale inhibitor [14]. Even so, the conventional PASP has a relatively low molecular weight and a single functional group in its structure, leading to inadequate scale inhibition performance [24]. Besides that, the practical uses and implementation of traditional PASP is limited due to its relatively low inhibition performance and dispersion capacity which may be impacted by its linear molecular structure [6]. Thus, the combination of PASP with some extra group on the PASP's side chain or combination with other material may increase its inhibition performance [5].

In water-treatment applications, integrating polyaspartic acid (PASP) with functional nanomaterials such as zinc oxide (ZnO) nanoparticles has shown a great potential in enhancing scale-inhibition performance, particularly within membrane-based desalination systems. PASP–ZnO nanocomposites demonstrate strong potential for application either as feed-water pretreatment additives or as surface-modifying layers for anti-scaling membrane coatings, contributing to reduced mineral deposition, sustained permeate flux, and lower operational energy demand. The incorporation of nanomaterials in scale-control strategies has gained increasing attention as a sustainable alternative to conventional inhibitors, driven by their high specific surface area and reactivity toward scale-forming ions. In particular, ZnO nanoparticles exhibit superior capability in suppressing $CaCO_3$ precipitation relative to traditional chemical inhibitors. Their nano-scale characteristics facilitate effective dispersion and interaction with dissolved species, thereby disrupting ion aggregation, interfering with nucleation pathways, and hindering the subsequent growth of $CaCO_3$ crystals in aqueous systems. Consequently, PASP–ZnO nanocomposites represent a viable route toward next-generation, eco-friendly antiscalants suitable for advanced membrane processes.

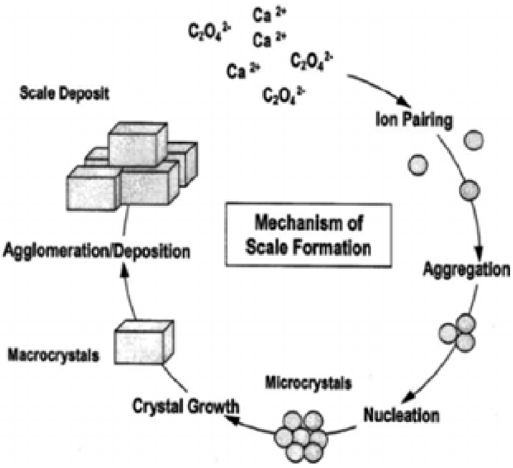

Figure 1 Steps in the process from a soluble ion to a large calcium carbonate scale layer [19]

2.0 METHODS

This study aims to evaluate the effectiveness of combining polyaspartic acid (PASP) with zinc oxide nanoparticles (ZnO-NPs) in inhibiting calcium carbonate ($CaCO_3$) scale. The experimental procedure is shown in the flowchart in Figure 2.

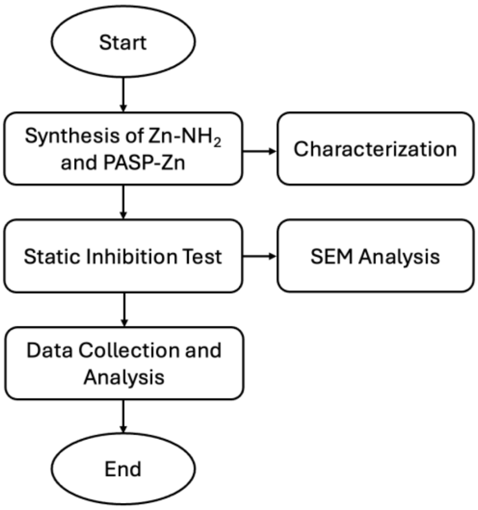

Figure 2 Flowchart for the experimental procedure

Functionalization of ZnO Nanoparticles

Zinc oxide nanoparticles were functionalized with amino groups to form Zn–NH_2. Specifically, 1 g of ZnO nanoparticles was dispersed in deionized water, followed by the addition of concentrated HCl and ethanol.

Preparation of PASP–Zn Composite

To prepare the PASP–Zn composite, 1 g of PASP was dispersed in ethanol and stirred for 1 hour at room temperature. Separately, 0.5 g of Zn–NH_2 nanoparticles was dispersed in ethanol to prevent agglomeration. Both solutions were then combined and subjected to ultrasonication for 3 hours to ensure homogeneous dispersion. The mixture was subsequently heated at 80 °C for 4 hours to promote bonding between PASP and Zn–NH_2, followed by stirring at room temperature for 12 hours for stabilization. The final PASP–Zn composite was obtained through filtration, oven-dried at 60 °C for 12 hours, and stored for further testing.

Static Inhibition Test

The static inhibition test was performed following NACE Standard TM0374-2001 to evaluate the scale inhibition efficiency of the synthesized PASP–Zn composite [16]. Synthetic brine was prepared by mixing two separate solutions: an anion solution containing Na_2CO_3 and a cation solution containing $CaCl_2 \cdot 2H_2O$. The solutions were combined to initiate $CaCO_3$ precipitation. Different concentrations of PASP–Zn inhibitor were added to assess their performance. The residual Ca^{2+} concentration in the supernatant was measured using EDTA titration, and the inhibition efficiency (η, %) was calculated as:

$$\eta = \frac{C_2 - C_1}{C_0 - C_1} \times 100$$

where, C_0 (mg/mL) represents the initial concentration of Ca^{2+} in the solution. C_1 is Ca^{2+} concentration in the blank after precipitation and C_2 (mg/mL) denote the concentrations of Ca^{2+} in the absence and presence of the scale inhibitor, respectively [16].

2.1 Characterization

FTIR and EDX analyses are crucial for characterizing PASP-Zn nanoparticles (NPs) as scale inhibitors. FTIR helps identify functional groups and chemical bonding, confirming successful functionalization, which is essential for understanding how these groups interact with scale-forming ions. The functionalization of ZnO was confirmed using Fourier Transform Infrared Spectroscopy (Perkin Elmer, Frontier) in the wavenumber range of 4000–400 cm⁻¹.

EDX provides information on the elemental composition and distribution of the nanoparticles, ensuring they have the intended makeup and purity necessary for effective scale inhibition. Together, these techniques validate the synthesis process and optimize the performance of PASP-Zn NPs in preventing scale formation.

The surface morphology of carbonate scale was analyzed using Scanning Electron Microscopy (SEM, JEOL JSM-IT300LV) operated at an accelerating voltage of 15 kV. The samples were mounted on aluminum stubs using conductive carbon tape and coated with a thin layer of platinum using a sputter coater to improve electrical conductivity.

3.0 RESULTS AND DISCUSSION

3.1 Energy-Dispersive X-ray (EDX)

The EDX analysis of PASP (Figure 3) and PASP-Zn (Figure 4) shows differences in elemental composition, confirming the incorporation of Zn in PASP-Zn and the elemental percentage is shown in Table 1. The PASP spectrum mainly consists of oxygen (44.1 wt%), carbon (33.5 wt%), sodium (13.7 wt%), and nitrogen (8.7 wt%). In contrast, PASP-Zn exhibits a significant Zn peak at 17.6 wt%, with reduced oxygen (32.3 wt%) and sodium (9.9 wt%). Additionally, chlorine (2.7 wt%) and fluorine (0.3 wt%) appear in PASP-Zn, likely from the synthesis process. The reduction in nitrogen (5.8 wt%) suggests possible interactions between PASP and Zn.

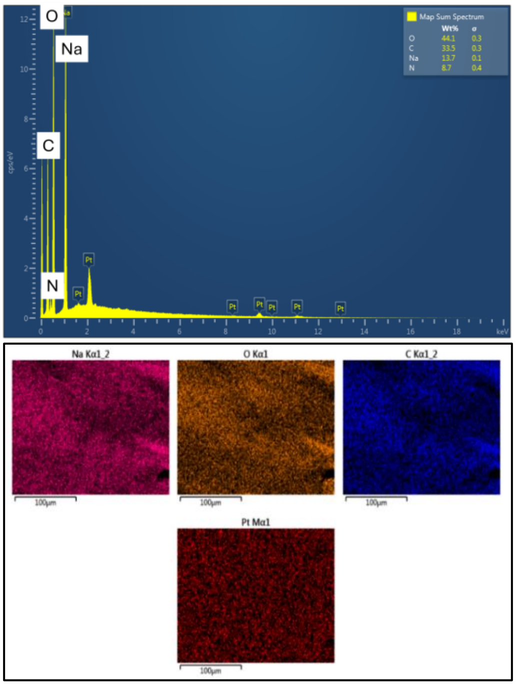

Figure 3 EDX spectrum and mapping for PASP

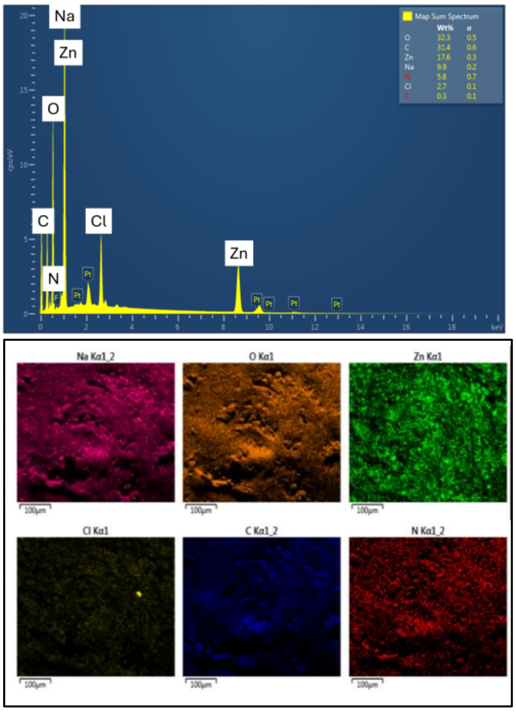

Figure 4 EDX spectrum and mapping for PASP-Zn

Table 1 Quantitative calculations for EDX analysis

Element	PASP (wt%)	PASP-Zn (wt%)
O	44.1	32.3
C	33.5	31.4
Na	13.7	9.9
N	8.7	5.8
Zn	-	17.6
Cl	-	2.7
F	-	0.3

3.2 Fourier-Transform Infrared Spectroscopy (FTIR)

Figure 5 shows the FTIR spectra of pristine PASP and PASP after ZnO incorporation. The asymmetric stretching band of the carboxylate group (–COO⁻) shifts from 1565 cm^{-1} in PASP to 1571 cm^{-1} in PASP–Zn, confirming the coordination of Zn^{2+} ions with the carboxyl functional sites of PASP [23]. This shift to a higher wavenumber indicates Zn–carboxylate complex formation, consistent with metal–ligand bonding observed in PASP–metal hybrids [1].

New absorption bands at 1044, 902, and 716 cm^{-1} appear in PASP–Zn, corresponding to Zn–O stretching and Zn–O–C linkages, verifying the incorporation of ZnO and formation of polymer–metal coordination bonds [2]. The slight shift in the broad O–H/N–H stretching band from 3274 to 3276 cm^{-1} further supports hydrogen-bond reorganization induced by ZnO interaction [10].

Collectively, these peak shifts and new Zn–O vibrations confirm that Zn^{2+} is effectively chelated within PASP's carboxylate matrix, evidencing successful bonding and hybrid formation between PASP and ZnO.

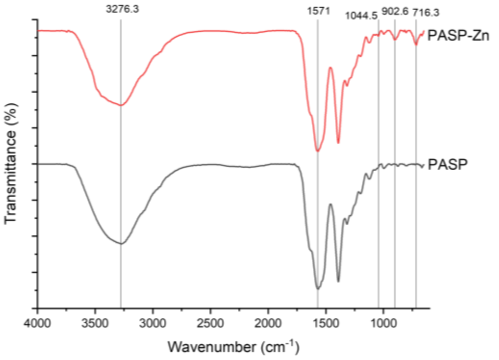

Figure 5 FTIR spectrum for PASP and PASP-Zn

3.3 Effect of Inhibitor Against CaCO3

The performance of scale inhibitors at different concentrations was evaluated using EDTA titration. From the EDTA titration results in Figure 6, both PASP and PASP-Zn was successfully mitigate calcium carbonate scaling by maintaining free Ca^{2+} in solution. PASP alone exhibits its highest inhibition roughly around ~42 % – 43% at a relatively low dosage of 10 ppm, after which inhibition decreases slightly, suggesting a potential optimal dosing window or variability under test conditions. In contrast, PASP-Zn, though starting with slightly lower inhibition at 10 ppm, demonstrates a more stable and gradually improving performance with increasing concentration, reaching its highest inhibition roughly around ~44.85% at 50 ppm.

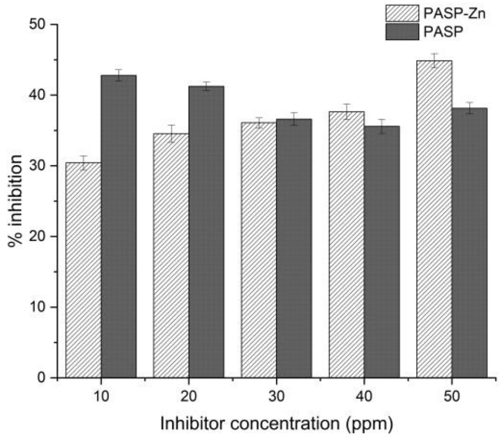

Figure 6 Percentage inhibition against concentration

The results of this study demonstrate that the incorporation of Zn^{2+} ions significantly enhance the inhibitory performance of poly(aspartic acid) (PASP) against calcium carbonate ($CaCO_3$) deposition, particularly at higher concentrations, due to the synergistic interaction between Zn and the carboxyl functional groups of PASP. This observation aligns with earlier findings reported by Cheng *et al.* (2021) and Jalab *et al.* (2021) [7 , 11], who highlighted that PASP effectively suppresses $CaCO_3$ nucleation via carboxylate adsorption on active crystal growth sites, while the addition of metal ions such as Zn^{2+} increases the number of coordination sites and enhances the affinity toward Ca^{2+}, resulting in improved inhibition efficiency. Similarly, Kan *et al.* (2009) observed that Zn^{2+} can form stable complexes with organic inhibitors, improving their retention and stability under varying water chemistries, which supports the consistency of inhibition observed in the PASP–Zn system [12]. Furthermore, Goldie (2008) and Wang *et al.* (2012) demonstrated that Zn^{2+} ions independently modify $CaCO_3$ crystallization by favoring the formation of less adherent aragonite phases over calcite, thereby reducing overall scaling propensity [9, 21]. The synergistic mechanism between Zn^{2+} and organic inhibitors has also been discussed extensively in the review by Mazumder *et al.* (2020), who emphasized that transition-metal ions, particularly Zn^{2+}, interact with carboxyl-rich polymers to stabilize crystal growth inhibition and improve long-term performance [15]. Comparable findings were reported by Chen *et al.* (2019), showing that PASP-based composite inhibitors containing transition metals exhibited superior efficiency compared to PASP alone, due to enhanced electrostatic and coordination interactions with Ca^{2+} [4]. Collectively, these results confirm that Zn incorporation strengthens PASP's inhibition capability by facilitating complexation with Ca^{2+} and altering the crystal growth pathway, making PASP–Zn a more reliable and robust green inhibitor for scale control applications.

Scanning electron microscopic (SEM) analysis was done on the precipitated calcium carbonate ($CaCO_3$) crystals, both in the absence of inhibitor and after treatment with 50 ppm PASP and PASP–Zn. Based on Figure 7**,** it highlights how the presence of inhibitors alters the morphology of calcium carbonate crystals. In Figure 7a the $CaCO_3$ crystals appear as relatively large, well-defined rhombohedral or blocky structures, characteristic of uninhibited calcium carbonate precipitation. Meanwhile, in Figure 7b after the addition of 50 ppm PASP, the crystal surfaces become more irregular and less uniformly shaped, indicating that PASP disrupts the usual crystal growth pathways. Finally, after addition of 50 ppm PASP–Zn, the distortion of the crystal habit is even more pronounced as shown in Figure 7c, with structures appearing more agglomerated or “flower-like.” This suggests that ZnO, in conjunction with PASP, further alters the crystal growth mechanism, leading to smaller or more distorted particles that are less prone to forming dense, adherent scale [22].

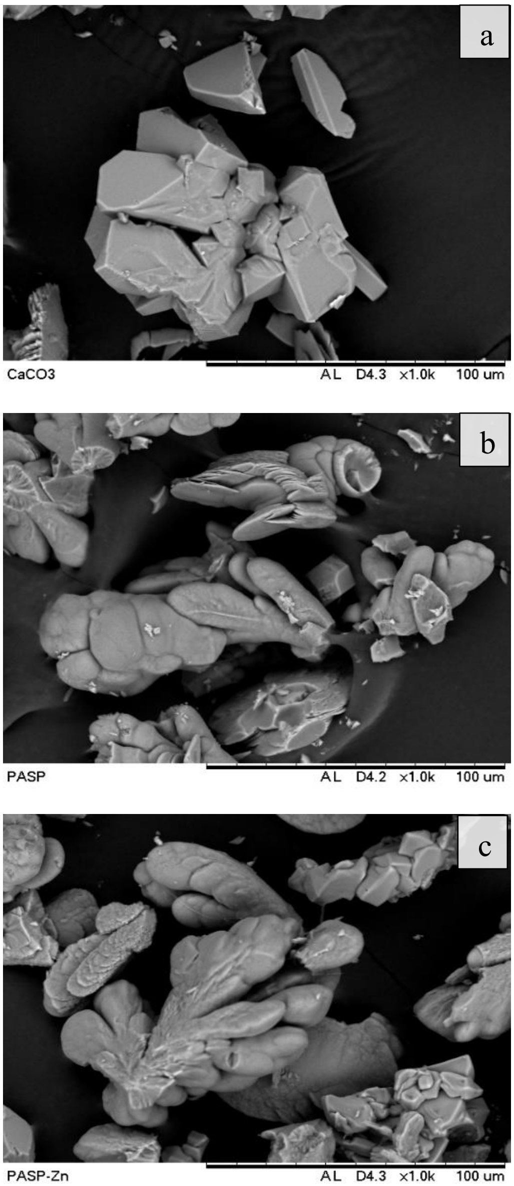

Figure 7 SEM image of (a) $CaCO_3$, (b) $CaCO_3$ after addition of PASP; (c) $CaCO_3$ after PASP-Zn addition

4.0 CONCLUSION

This study successfully synthesized and evaluated a PASP–ZnO composite as an eco-friendly inhibitor for calcium carbonate ($CaCO_3$) scale, with characterization analyses confirming the successful fabrication of the composite. The modification was verified by EDX analysis, which revealed a distinct Zn signal at 17.6 wt%, and FTIR spectra showing clear Zn^{2+} coordination and Zn–O bonding peaks, validating the structural integration of ZnO into the PASP matrix and the enhancement of its chemical interaction potential. The inhibition performance, evaluated via EDTA titration, demonstrated that PASP–Zn exhibited a more stable and superior inhibition trend, achieving a maximum inhibition efficiency of 44.85 % at 50 ppm, compared to unmodified PASP, which showed fluctuating results. Furthermore, SEM micrographs confirmed that PASP–Zn effectively altered $CaCO_3$ crystal morphology, producing highly irregular and loosely packed structures that are less adherent, thereby mitigating scale deposition on surfaces. These findings confirm that ZnO modification strengthens PASP’s scale inhibition capability by influencing nucleation and crystal growth mechanisms while improving stability across concentration ranges.

For future work, further optimization of Zn loading ratios, pH effects, and temperature tolerance is recommended to get better understanding on how PASP perform under different conditions. Advanced kinetic and thermodynamic studies such as isothermal titration calorimetry or molecular dynamics simulations may also be performed to elucidate the coordination mechanism between Zn^{2+}, carboxyl groups, and Ca^{2+} in a solution. Additionally, the application of PASP–ZnO composite into membrane technology is seen the highly beneficial. Embedding or surface-coating the PASP–ZnO on the polymeric or ceramic membranes could significantly mitigate scaling and biofouling during desalination, wastewater treatment, and produced-water filtration processes. The hydrophilicity, carboxyl functionality, and antimicrobial properties of PASP–ZnO make it a viable additive for anti-scaling and self-cleaning membrane systems, potentially extending the membrane lifespan and reducing maintenance costs. Future studies should therefore assess its long-term stability, leaching resistance, and compatibility with membrane fabrication techniques such as phase inversion or electrospinning. Overall, the PASP–ZnO composite presents a sustainable and versatile platform not only for scale inhibition in industrial water systems but also for next-generation green membrane coatings in advanced separation and water purification technologies.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest regarding the publication of this paper.

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