Synthesis and Application of a Phosphorous-Free and Non-Nitrogen Polymer as an Environmentally Friendly Scale Inhibition and Dispersion Agent in Simulated Cooling Water Systems

Meifang Yan, Qiangqiang Tan, Zhan Liu, Haihua Li, Yuxuan Zheng, Lihui Zhang, and Zhenfa Liu*

ABSTRACT: A novel phosphorus-free and non-nitrogen scale inhibition and dispersion agent was synthesized from epoxysuccinic acid (ESA), itaconic acid (IA), and sodium methyl propylene sulfonate (SMAS). It was characterized by infrared spectroscopy (IR), nuclear magnetic resonance (NMR), and elemental analysis, and its molecular weight and distribution were determined by gel permeation chromatography (GPC). Static experimental methods were used to evaluate the scale inhibition against CaCO\textsubscript{3}, Ca\textsubscript{3}(PO\textsubscript{4})\textsubscript{2}, and CaSO\textsubscript{4}. The dispersion ability was evaluated by measuring the dispersive property of iron oxide. By observing the morphology of the CaCO\textsubscript{3} scale, the scale inhibition mechanism was discussed. The results showed that when the ratio of raw materials (ESA/IA/SMAS) was 10:10:5, the scale inhibition rate of CaCO\textsubscript{3} and CaSO\textsubscript{4} reached 100% with the dosages of 20 and 10 mg/L, respectively. In addition, the scale inhibition rate against Ca\textsubscript{3}(PO\textsubscript{4})\textsubscript{2} was up to 96.64% with a dosage of 100 mg/L. The property of dispersing iron oxide exhibited the best results with 50 mg/L of the agent. On the 21st day, the biodegradation rate reached 63.33%. This polymer was an environmentally friendly antiscale and dispersant with good scale inhibition and biodegradability.

1. INTRODUCTION

Chemical dosing is one of the most traditional and effective methods for the treatment of circulating cooling water.\textsuperscript{1−5} However, water eutrophication caused by the discharge of wastewater containing phosphorus and nitrogen has increased the strictness of wastewater discharge restrictions.\textsuperscript{6−8} To protect water resources and meet sustainable development requirements, higher demands have been put forward to produce more green water treatment agents. As such, the development of environmentally friendly water treatment agents and technologies has become the focus of many scientific studies at both home and abroad.

Polyepoxysuccinic acid (PESA), a green water treatment agent, has become a research hotspot because of its excellent scale inhibition against calcium carbonate and corrosion inhibition.\textsuperscript{9−15} However, it exhibits very poor properties of inhibiting calcium phosphate and dispersing iron oxide. To improve the abilities of inhibiting calcium phosphate and dispersing iron oxide, a sulfonic acid group was introduced into the molecular structure.\textsuperscript{16−22} Domestic and foreign scholars have synthesized modified polyepoxysuccinic acid polymers by adding monomers containing a sulfonic acid group, such as 2-acrylamido-2-methylpropanesulfonic acid (AMPS), sodium styrene sulfonate (SS), and sodium allyl sulfonate (SAS). However, at the same time, other functional groups containing phosphorus or nitrogen have been introduced into the polymers. The long-term usage of such water treatment agents can similarly result in eutrophication and pollute the water environment.

Based on the efficient utilization of water resources, environmental protection, and greenness of water treatment technologies, phosphorus-free and non-nitrogen polymers were prepared from epoxysuccinic acid (ESA), itaconic acid (IA), and sodium methallyl sulfonate (SMAS), thereby introducing sulfonate and carboxyl in the polymer molecular structure. Scale inhibition, dispersion performance, and biodegradability of the polymer were studied in this study.

2. RESULTS AND DISCUSSION

2.1. Characterization Results. The infrared spectrum indicated the absence of any C==C characteristic absorption peaks at 1640−1620 cm\textsuperscript{-1}, suggesting that the reaction...
monomers containing C=C had fully reacted. The stretching vibration peak of the open-loop ether bond C−O−C was observed at 1136 cm\(^{-1}\), and the characteristic peak of the closed epoxy ring at 1500 cm\(^{-1}\) disappeared, indicating that the epoxy ring of ESA was opened and polymerization reaction proceeded. The characteristic peaks at 3439 and 1710 cm\(^{-1}\) were attributed to the stretching vibration peaks of −OH and −COO\(^{-}\), respectively. The peaks at 1036 and 607 cm\(^{-1}\) were attributed to the stretching vibration peaks of S=O of sulfate and C=S, respectively. Based on these observations, the obtained polymer may be identified as the target product, ESA/IA/SMAS (Figure 1).

![Fourier transform infrared (FTIR) spectrum of ESA/IA/SMAS.](https://dx.doi.org/10.1021/acsomega.0c01620)

The carbon spectrum showed that the characteristic peak of C=C in the range of 110–140 ppm disappeared. This indicated that the monomers containing unsaturated double bonds C=C participated in the polymerization reaction. The −C−O−C− characteristic peak of the open ring was observed at 72 and 73 ppm, indicating that the epoxy ring was broken and the polymerization reaction occurred. The characteristic peaks between 170 and 180 ppm indicated the presence of −COO\(^{-}\) in the product (Figure 2).

As shown in Table 1, the measured value of the elemental analysis of the copolymer ESA/IA/SMAS (10:10:10) was consistent with the theoretical value, which further explained that the copolymer containing sulfonic acid group had been synthesized.

Table 2 presents the molecular weight results. The average molecular weight was within the range of 2000–3000, indicating that the polymers had low molecular weight, which was essential for efficient scale inhibition. The polydispersity index (PDI) was about 1.0000, which obviously suggested that the polymers were uniform by polymerization.\(^{26}\)

### 2.2. Performance Evaluation

#### 2.2.1. Scale Inhibition Against CaCO\(_3\)

Figure 3 presents a concordant increase between the scale inhibition rate and the concentration of the inhibitor. The scale inhibition rate of PESA reached 100% at a dosage of 20 mg/L, indicating that PESA showed excellent performance in preventing the calcium carbonate scale. The copolymer ESA/IA/SMAS (10:10:5) exhibited the best scale inhibition on calcium carbonate, and the scale inhibition rate reached 100% at a minimum dosage of 20 mg/L.

There is a great influence of polymer molecular weight on the scale inhibition performance. When the molecular weight is too low, the number of coordination groups that can be complexed is relatively small and the ability of chelating Ca\(^{2+}\) is obviously insufficient. However, when the molecular weight is too high, polymer molecules may be in a coiled state, enclosing the active group, and the reactivity is reduced and the scale inhibition is decreased.

#### 2.2.2. Scale Inhibition against CaSO\(_4\)

PESA had a general scale inhibition performance on calcium sulfate, and the maximum scale inhibition rate was only 76.35% at an agent dosage of 14 mg/L. The antiscaling effect of the copolymers with different amounts of sulfo was markedly increased, when the dosages were 6 mg/L and above. The polymer with a ratio of 10:10:5 exhibited a scale inhibition rate of 100% at a dosage of 10 mg/L, and ESA/IA/SMAS (10:10:2) presented a scale inhibition rate of 100% at a dosage of 8 mg/L. The scale inhibition rate remained constant as the dosage continued to increase. The polymers ESA/IA/SMAS (10:10:7) and ESA/IA/SMAS (10:10:10) exhibited poor performance against calcium sulfate (Figure 4).

With the increase of the proportion of sulfonic acid group in ternary polymer, the scale inhibition performance of calcium sulfate decreased on the contrary. It was mainly because carboxylate ion had a stronger ability to chelate Ca\(^{2+}\) in aqueous solution, and the increase of the proportion of sulfonic acid group led to the decrease of carboxylic acid groups in ternary polymer molecules.\(^{27}\)

#### 2.2.3. Scale Inhibition against Ca\(_3\)(PO\(_4\))\(_2\)

Figure 5 presents the poor scale inhibition of PESA against calcium phosphate (<10%). However, the antiscale performance of the polymer to calcium phosphate increased concordantly with the increase of the dosage, and a step change was observed between the dosages of 60–80 mg/L. The polymer ESA/IA/SMAS (10:10:5) exhibited the best scale inhibition effect on calcium phosphate, specifically at a scale inhibition rate of 96.64%. When the dosage was more than 100 mg/L, the scale inhibition performance of the polymer on calcium phosphate was similar to that on calcium carbonate, which indicated that the molecular weight of the polymer had a great influence on the scale inhibition performance of calcium phosphate too.

#### 2.2.4. Iron Oxide Dispersion Properties

In the experiment, the light transmittance of distilled water was taken as a reference. In general, lower light transmittances indicated better dispersion performance of the inhibitor. According to Figure 6, the light transmittance of the solution was almost the same as the blank following the addition of different PESA concentrations, suggesting that PESA does not have any dispersion properties. The sulfonic group was more likely to undergo dissociation, and the expected dispersion effect could be achieved through steric hindrance and electrostatic effect. However, an increase in the proportion of sulfonic acid group that was introduced into the terpolymer ESA/IA/SMAS did not consistently present any increase in the iron oxide dispersion properties. Among them, the ESA/IA/SMAS copolymer with a ratio of 10:10:5 presented the best dispersion effect. The polymer ESA/IA/SMAS (10:10:5) presented a transmittance of 31.9% at a dosage of 50 mg/L.

The dispersion performance of the polymer was consistent with the scale inhibition performance against calcium carbonate and calcium sulfate, indicating that the molecular weight had a significant influence on the dispersion performance. If the molecular weight was too small, the steric
hindrance was not enough to make the dispersion stable. If the molecular weight was too large, the steric hindrance effect would hinder the adsorption of the polymer on the scale-forming particles.

2.2.5. Biodegradation Property of ESA/IA/SMAS Polymer. The biodegradation of the polymer with a ratio of 10:10:5 was evaluated. Figure 7 presents a decreased inhibitor chemical oxygen demand (COD) as well as the gradual increase of the biodegradation rate with time. A degradation rate of 63.33% was observed on the 21st day. Therefore, the inhibitor was considered to be biodegradable and environmentally friendly based on the stipulations of OECD 301. Long-term use is assumed to not cause serious pollution to the soil and water environment.

2.3. Characterization of CaCO₃ Precipitate. 2.3.1. Scanning Electron Microscopy (SEM) Photographs of CaCO₃ Precipitate. A neat CaCO₃ crystal (Figure 8) was obtained in the absence of the polymer. In contrast, a very irregular and seriously distorted crystal was gathered in the presence of the polymer due to the presence of carboxylic and sulfonic groups, which adsorbed and dissociated from the polymer and...
subsequently doped onto the CaCO₃ crystal lattice. The particle surface charge density and the repulsive force between the particles increased, thus reducing the increment rate of the crystal, which prevented the formation and growth of the CaCO₃ crystal.

2.3.2. X-ray Diffraction (XRD) Analysis. Figure 9 presents the XRD patterns of the CaCO₃ precipitate. The diffraction peaks at 22.90 (012), 29.28 (104), 31.22 (006), 35.83 (110), 39.30 (113), 42.93 (202), 47.44 (018), and 48.32 (116) define the characteristic peaks of calcite. The characteristic peaks weakened in the presence of the inhibitor, indicating that the surface morphology and particle size of CaCO₃ had changed. The crystal faces reduced and the crystallinity of CaCO₃ decreased with the inhibitors. As a result, the calcium carbonate crystal loosened and hindered heat exchanger surface adhesion, which is in accordance with the SEM images.

3. CONCLUSIONS

This study successfully synthesized an environmentally friendly water treatment agent from ESA, IA, and SMAS. The polymer not only possessed excellent inhibition of CaCO₃, CaSO₄, and Ca₃(PO₄)₂ and dispersed iron oxide but also showed good biodegradability. It made up for the deficiency that PESA had no dispersing ability on Fe₂O₃ and poor scale inhibition against CaSO₄ and Ca₃(PO₄)₂.

A raw material polymer with ratio of 10:10:5 exhibited the best comprehensive performance among the products. The carboxylic acid group and sulfonic acid group in the polymer had a remarkable synergistic effect. The introduction of the sulfonic and carboxylic groups enhanced the scale inhibition and dispersion effects due to the chelating solubilization of the calcium ions and lattice distortion.

4. EXPERIMENTAL SECTION

4.1. Synthesis and Characterization of ESA/IA/SMAS Polymer. Maleic (Figure 10) anhydride and distilled water were added into a four-port flask with a condensing tube and a thermometer and stirred until dissolved. A sodium hydroxide solution with a mass fraction of 50% was then added into the
solution. The temperature of the solution was controlled below 55 °C to hydrolyze maleic anhydride into maleate. Subsequently, the water bath was adjusted to 55 °C, and the sodium tungstate catalyst was added. Hydrogen peroxide (30%) was then slowly added (about half an hour), during which the pH value was controlled within a certain range. In addition, the temperature increased up to 65 °C for epoxidation, during which an epoxysuccinic acid (ESA) solution was prepared. The ESA solution was dripped into a large amount of anhydrous ethanol to allow ESA solid precipitation. The precipitated ESA solid was then removed, washed with acetone, and dried.

A certain amount of ESA, itaconic acid (IA), and sodium methallyl sulfonate (SMAS) was weighed and added to a four-mouth flask with a certain amount of distilled water, and approximately 50% solution (mass concentration) was prepared. A certain amount of ammonium persulfate and isopropanol were then added, and the solution was heated to 96 °C under a water bath. Two injection pumps were used to simultaneously add sodium bisulfite solution (10%) and hydrogen peroxide solution (30%) at a certain speed, thereby allowing the simultaneous addition of both sodium bisulfite solution and hydrogen peroxide solution. The reaction continued for 6 h at 96 °C. The solution was reduced to room temperature after the reaction was completed, during which a light yellow transparent liquid was obtained, indicating that it was the ESA/IA/SMAS polymer aqueous solution.

**4.2. Inhibition Performance against CaCO₃.** Static scale inhibition experiments were carried out as described in GB/T 16632-2008 (China). Calcium chloride and sodium bicarbonate were dissolved by deionized water to prepare test water for the experiments. The test water, which was prepared by 600 mg/L of Ca²⁺ and 1200 mg/L of HCO₃⁻, was placed into a 0.5 L volumetric flask. The pH of the test water was adjusted to about 9.0. The water samples were heated in a water bath at 80 °C for 10 h. After that, they were cooled to room
temperature. The content of Ca²⁺ in the supernatant liquid was determined using the titration of ethylenediaminetetraacetic acid (EDTA). The scale inhibition rate of the inhibitors to CaCO₃ was calculated by the following formula 1

\[ \eta_1 = \left( \frac{V_0 - V_1}{V_2 - V_0} \right) \times 100\% \]  

where \( V_0 \) is the volume of EDTA consumed by the blank water sample after heating, mL; \( V_1 \) is the volume of EDTA consumed by the water sample with the inhibitor after heating, mL; and \( V_2 \) is the volume of EDTA consumed by the blank water sample before heating, mL.

4.3. Inhibition Performance against Ca₃(PO₄)₂.

The experiments were carried out as described in GB/T 22626-2008 (China). The water sample containing 250 mg/L Ca²⁺ and 5 mg/L PO₄³⁻ was placed into a 500 mL volumetric flask. The pH of the test water was adjusted to about 9.0. The water samples were heated in a water bath at 80 °C for 10 h. After that, the PO₄³⁻ content of the water samples was determined using a spectrophotometer (710 nm, 1 cm cuvette). The scale inhibition rate of the inhibitors to Ca₃(PO₄)₂ was calculated by the following formula 2

\[ \eta_2 = \left( \frac{C_0 - C_1}{C_2 - C_0} \right) \times 100\% \]  

where \( C_0 \) is the PO₄³⁻ concentration of the water sample without the inhibitor after heating, mg/L; \( C_1 \) is the PO₄³⁻ content of the supernatant liquid in presence of the agent after heating, mg/L; and \( C_2 \) is the content of PO₄³⁻ of the water sample without the inhibitor before heating, mg/L.

4.4. Inhibition of Performance against CaSO₄.

According to the oil and natural gas occupation standard “Evaluation method for performance of scale inhibitors used in oil field”, test water samples were prepared with CaCl₂ and Na₂SO₄ at Ca²⁺ and SO₄²⁻ mass concentrations of 5000 and 7500 mg/L, respectively. The test water samples were heated at 70 °C for 25 h in a water bath. After that, the Ca²⁺ content of the water sample was determined by titrating EDTA. The scale inhibition rate of the inhibitors against CaSO₄ was calculated by the following formula 3

\[ \eta_3 = \left( \frac{C_1 - C_0}{C_2 - C_0} \right) \times 100\% \]  

where \( C_0 \) is the Ca²⁺ content of the water sample without the inhibitor after heating, mg/L; \( C_1 \) is the Ca²⁺ content of the water sample with the inhibitor after heating, mg/L; and \( C_2 \) is the Ca²⁺ content of the water sample without the inhibitor before heating, mg/L.

4.5. Ability of Dispersing Ferric Oxide.

Test water containing 150 mg/L Ca²⁺ and 10 mg/L Fe²⁺ was placed into a 500 mL volumetric flask. The pH was adjusted to about 9.0, and then it was strongly stirred for 15 min with a magnetic stirrer. After a 50 °C water bath for 5 h, the transmittance of the supernatant of test water was measured (450 nm, 3 cm cuvette), wherein the transmittance of distilled water was 100% as a reference. In general, smaller transmittances indicated a better dispersion effect.

4.6. Test of Biodegradability.

The biodegradability was tested using a shaking-bed test. A 500 mL solution at a certain inhibitor concentration and 1.0 mL of the inoculum were added into a conical flask, which was placed on the shaking-bed. The chemical oxygen demand (COD) of the inhibitor was measured on the 1st, 7th, 14th, 21st, and 28th day at 30 °C.
The degradation rate, \( \rho \), was calculated by the following formula 4

\[
\rho = \left(1 - \frac{C_b - C_{bl}}{C_0 - C_{bl}}\right) \times 100\%
\]  

(4)

where \( C_0 \) and \( C_{bl} \) are the COD of the solution with and without the inhibitor, respectively, on the 1st, 7th, 14th, 21st, and 28th day, and \( C_b \) and \( C_{bl} \) are the initial COD of the water sample with and without the inhibitor, respectively.

4.7. Characterization. FTIR was used to detect the functional groups’ absorption spectroscopy. The wavenumber was in the range 4000–500 cm\(^{-1}\). \(^{13}\)C NMR spectrum was recorded by a AVANCE II 500 MHz spectrometer (Bruker, Germany) using D,O as the solvent. The contents of C, H, and S of the copolymer were analyzed and determined by German vario Micro cube elemental analyzer after purification and drying.

CaCO\(_3\) scale obtained from the static scale inhibition experiment was washed with distilled water and dried. The surface morphology of the scale was observed by SEM (Hitachi, Japan). The XRD patterns of CaCO\(_3\) were recorded on an Ultima IV X-ray powder diffractometer (Rigaku, Japan). The scan range of 2\(\theta\) was set at 20–60° with a scanning speed of 2°/min. The molecular weight of the polymers was measured by GPC (Waters) using a 0.1 M potassium nitrate solution as the mobile phase at 1 mL/min.

■ AUTHOR INFORMATION

Corresponding Authors
Qiangqiang Tan – State Key Laboratory of Multiphase Complex Systems, Institute of Process Engineering, Chinese Academy of Sciences, Beijing 100049, China; University of Chinese Academy of Sciences, Beijing 100049, China; Phone: +86-10-62529377; Email: qtan@ipe.ac.cn

Zhenfa Liu – Institute of Energy Resources, Hebei Academy of Science, Shijiazhuang 050081, China; Hebei Engineering Research Center for Water Saving in Industry, Shijiazhuang 050081, China; orcid.org/0000-0002-1243-4725; Phone: +86-311-83031008; Email: lz63@sohu.com

Authors
Meifang Yan – State Key Laboratory of Multiphase Complex Systems, Institute of Process Engineering, Chinese Academy of Sciences, Beijing 100049, China; University of Chinese Academy of Sciences, Beijing 100049, China; Institute of Energy Resources, Hebei Academy of Science, Shijiazhuang 050081, China; Hebei Engineering Research Center for Water Saving in Industry, Shijiazhuang 050081, China

Zhan Liu – Institute of Energy Resources, Hebei Academy of Science, Shijiazhuang 050081, China; Hebei Engineering Research Center for Water Saving in Industry, Shijiazhuang 050081, China

Haihua Li – Institute of Energy Resources, Hebei Academy of Science, Shijiazhuang 050081, China; Hebei Engineering Research Center for Water Saving in Industry, Shijiazhuang 050081, China

Yuxuan Zheng – School of Chemical Engineering and Technology, Hebei University of Technology, Tianjin 300000, China

Lihui Zhang – Institute of Energy Resources, Hebei Academy of Science, Shijiazhuang 050081, China; Hebei Engineering Research Center for Water Saving in Industry, Shijiazhuang 050081, China

Research Center for Water Saving in Industry, Shijiazhuang 050081, China

Complete contact information is available at: https://pubs.acs.org/10.1021/acsomega.0c01620

Author Contributions
*Z.L. contributed equally to this work.

Notes
The authors declare no competing financial interest.

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