

The Application of Electrochemical Impedance in the Environmentally Friendly Scale and Corrosion Inhibitor Polyepoxysuccinic ACID

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Abstract—Polyepoxysuccinic acid (PESA) is a new, non-phosphorous, biodegradable, environmentally friendly scale and corrosion inhibitor implemented in industrial water treatment. The corrosion and scale inhibition properties of PESA were investigated in the test water by using static beaker test, weight-loss method, polarization curves and electrochemical impedance spectroscopy (EIS). It is found that, PESA has better scale inhibition efficiency on CaCO_3 , BaSO_4 as well as is a phosphate-free environment-friendly inhibitor with multi-scale properties. There is a limited corrosion inhibition effect on carbon steel provided by PESA used alone at low concentration, and with the increase of PESA concentration, corrosion inhibition is gradually strengthened. The pH value has significant effect on corrosion inhibition property of PESA. There is low corrosion inhibition efficiency in acidic condition, as well as better corrosion inhibition efficiency of PESA in alkaline condition. PESA is mainly as the function of anodic corrosion inhibitor. PESA should be a non-phosphorous, biodegradable, environmentally friendly scale and corrosion inhibitor implemented in industrial water treatment.

Keywords—polyepoxysuccinic acid; scale and corrosion inhibition; polarization curve; electrochemical impedance spectroscopy

I. INTRODUCTION

The formation of mineral scales can cause a series of problems in many processes ranging from desalination to oil production, such as blockage of pipeline, damage of production system, increase of energy consumption, decrease of

productivity and unscheduled equipment shutdown^[1-10], and eventually result in unwanted increase of operating costs^[11-13]. The most practical and economical method to combat this problem is the use of chemical scale inhibitors. Recently, the impact of chemicals on the environment is an issue of increasing global importance.

From the middle 1970s to early 1990s, phosphonates had been developed and are extensively used as scale inhibitors. Phosphonates such as HEDP, ATMP, EDTMP, DTPMP, PBTCA, which contain one or more $\text{R}_3\text{C-P}(\text{O})(\text{OH})_2$ groups rather than a $\text{R}_3\text{C-O-P}(\text{O})(\text{OH})_2$, possess a number of superior qualities: high chemical stability under extremes of pH and temperature, the ability to complex metal, the ability to adsorb strongly onto metal coatings, and some dispersancy activity towards suspending matter. However, it is well known that even at moderate conditions of calcium hardness, pH, and temperature, phosphonates can react with calcium ion and precipitate as calcium-phosphonate. Furthermore, phosphonates can also cause eutrophication.

In the past three decades, many water soluble anionic polymers compositions and molecular weights have been developed as scale inhibitors. These polymers are mainly homo-, co-, or ter-polymers having acrylic or maleic as the initial monomer in conjunction with other monomers containing acrylamide, ester, sulfonic acid, phosphonic acid, etc. They possess the excellent ability as a scale inhibitor and metal ion stabilizer. Acrylic acid-based scale inhibitors such as poly-

rylic acid (PAA), acrylic acid/2-acrylamido-2-methylpropane sulfonic acid copolymer (AA/AMPS), acrylic acid/2-acrylamido-2-methylpropane sulfonic acid/2-hydroxypropyl-acrylate copolymer (AA/AMPS/HPA), although generally of low toxicity, are also unfortunately non-biodegradable.

In the 21st century, biodegradability, the capability of being broken down into simple, non-toxic materials by the action of microorganisms and fungi, becomes an important mechanism for limiting the build-up of chemicals in the environment. Polyepoxysuccinic acid (PESA) is a novel scale inhibitor first developed by Betzdearbon (U.S.). It is a representative of green scale inhibitors because of its non-nitrogenous, non-phosphorus and biodegradable features. PESA with good scale-inhibiting performances was also synthesized successfully in China^[1,2]. Versatile scale inhibition performances of PESA were analyzed as well^[3-5]. According to the advantages of less dosage, high scale inhibition efficiency in the water of high alkalinity and high solid content, as well as the performance of corrosion inhibition, PESA has become a research hotspot now^[6]. The aims of this paper are to present the corrosion and scale inhibition properties of PESA investigated by using static beaker test, weight-loss method, polarization curves and electrochemical impedance spectroscopy (EIS) in the test water.

II. EXPERIMENT SECTION

A. Main Materials

Polyepoxysuccinic acid (PESA) used in this study was obtained from Wuxi Ecolom Chemical Co. Ltd., China. The PESA was as an aqueous solution (40 wt %) in the presence of sodium polyepoxysuccinic acid. Molecular weight M_w of the PESA was approximately 2100, as shown in Figure 1.

Carbon steel specimens used in experiments were composed of the following (wt %): 0.17 ~ 0.24%C, 0.17 ~ 0.37%Si, 0.35 ~ 0.65%Mn, ≤ 0.035 P, ≤ 0.035 S, ≤ 0.025 Ni, ≤ 0.025 Cr, ≤ 0.025 Cu, and balance Fe.

B. Static Beaker Test for Calcium Carbonate Scale Inhibition Efficiency

Static beaker calcium carbonate test was conducted by using deionized water and reagent grade chemicals. The static beaker test involved the adding of an amount of the water treatment agent to a solution containing an amount of Ca^{2+} and an amount of HCO_3^- at the required pH. The beakers were incubated in a water bath for 18 hour at 80°C. After cooling, an aliquot was filtered through 0.22 μ m filter paper, the calcium concentration in the filtrate was measured by using the standard ethylenediaminetetraacetic acid (EDTA) titration method. The static scale inhibition efficiency was calculated by

$$\eta(\%) = \frac{V_1 - V_0}{V_2 - V_0} \times 100$$

where V_1 is the amount of consumed EDTA of the sample with the addition of the water treatment agent after incubation, V_2 is the amount of consumed EDTA of the sample with

the addition of the water treatment agent before incubation, V_0 is the amount of consumed EDTA of the sample without the addition of the water treatment agent after incubation.

C. Static Beaker Test for Barium Sulfate Scale Inhibition Efficiency

Static beaker barium sulfate test was conducted by adding of an amount of the water treatment agent to a solution containing an amount of Ba^{2+} and an amount of SO_4^{2-} at the required pH. The beakers were incubated in a water bath for 18 hour at 80°C. After cooling, an aliquot was filtered through 0.22 μ m filter paper; the barium concentration in the filtrate was analyzed by inductively coupled plasma atomic emission spectroscopy (ICP). Barium sulfate inhibition efficiency was calculated by

$$\eta(\%) = \frac{[Ba^{2+}]_1 - [Ba^{2+}]_0}{[Ba^{2+}]_2 - [Ba^{2+}]_0} \times 100$$

where $[Ba^{2+}]_1$ is the concentration of barium ion of the sample with the addition of the water treatment agent after incubation, $[Ba^{2+}]_2$ is the concentration of barium ion of the sample with the addition of the water treatment agent before incubation, $[Ba^{2+}]_0$ is the concentration of barium ion of the sample without the addition of the water treatment agent after incubation.

D. Static Beaker Test for Corrosion Inhibition Efficiency

Static beaker corrosion test was tested by weight loss experiment, which was conducted in a 2 liter beakers equipped with an air/CO₂ sparge. The beaker was immersed in a water bath at 45°C. The carbon steel sheets were polished with different grades of emery paper, degreased with acetone, and rinsed with distilled water. Having been dried and accurately weighed, the carbon steel specimens were immersed in the beaker with the test water in the absence and presence of inhibitors for a period of 72 hours. A typical analysis of the test water is given in Table 1. After test, the carbon steel specimens were taken out, rinsed with water thoroughly, dried and accurately weighted. Each set of experiments was repeated two times to ensure reproducibility. The corrosion inhibition efficiency was calculated by

$$R(\%) = \frac{W_0 - W_1}{W_0} \times 100$$

where R is the corrosion inhibition efficiency, W_0 and W_1 are the value of the weight loss of carbon steel immersed in test solution without and with inhibitor, respectively.

TABLE I. ANALYSIS OF THE TEST WATER.

Parameter	Value
pH	7.5
Total hardness ($\text{mg}\cdot\text{L}^{-1}$, as CaCO_3)	142
Ca^{2+} ($\text{mg}\cdot\text{L}^{-1}$, as CaCO_3)	129
Alkalinity ($\text{mg}\cdot\text{L}^{-1}$, as CaCO_3)	80
Chloride ($\text{mg}\cdot\text{L}^{-1}$)	62
Sulfate ($\text{mg}\cdot\text{L}^{-1}$)	59
Turbidity (NTU)	0.22
Total dissolved solids ($\text{mg}\cdot\text{L}^{-1}$)	324

E. Test Method of Tafel Polarization Curve

The Tafel polarization curve measurements were carried out using a computer controlled potentiostat (Autolab PGSTAT30) in a three electrode cell assembly at a scan rate of 1 mV/s. A platinum electrode and a saturated calomel electrode (SCE) were used as auxiliary and reference electrode respectively. The work electrode (WE) was a carbon steel specimen of area 1 cm². All the experiments were carried out at 30±2°C with the test water (shown in table 1) as an electrolyte under static and naturally aerated condition. The Tafel polarization curve studies were conducted in the test water containing various concentrations of inhibitor. The experiments were repeated to ensure reproducibility. The inhibition efficiency of the inhibitor for the carbon steel corrosion was calculated by

$$R(\%) = \frac{W_0 - W_1}{W_0} \times 100$$

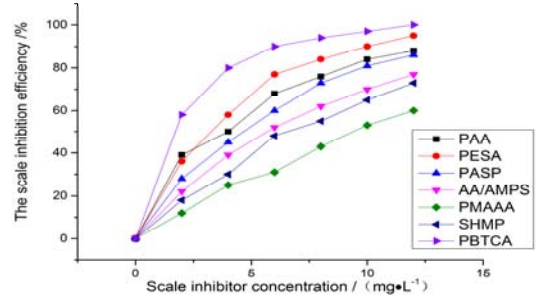
F. Electrochemical Impedance Spectroscopy (EIS)

The same instrument of the Tafel polarization curve measurements was used for electrochemical impedance measurement (Autolab PGSTAT30) at 30±2°C. Electrochemical impedance measurement was carried out in the region of 0.01 Hz to 100 KHz with the perturbation amplitude of 5 mV.

III. RESULTS AND DISCUSSION

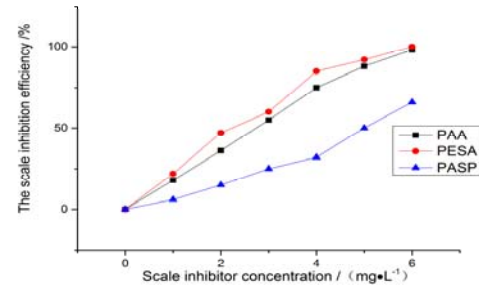
A. Calcium Carbonate Scale Inhibition Performance

In accordance with the experiment method 2.2, the additional experimental conditions are as follows: the mass concentration of Ca^{2+} and HCO_3^- are 500 $\text{mg}\cdot\text{L}^{-1}$ and 750 $\text{mg}\cdot\text{L}^{-1}$ respectively, pH value of the solution is 9.0. The comparison of static scale inhibition efficiency between PESA and other typical phosphonates is shown in Figure 1. It can be seen that although the CaCO_3 scale inhibition efficiency of PESA is a little less than PBTCA, but more than polycarboxylic acid inhibitors PAA and PMAAA, sulfonate inhibitors AA/AMPS, and polyphosphoric acid inhibitor SHMP. Therefore, PESA has good scale inhibition efficiency on CaCO_3 .

FIGURE I. INFLUENCE OF SCALE INHIBITOR CONCENTRATION ON CaCO_3 SCALE INHIBITION EFFICIENCY.

B. Barium Sulfate Scale Inhibition Performance

According to the experiment method 2.3, the additional experimental conditions are as follows: the mass concentration of Ba^{2+} and SO_4^{2-} are 20 $\text{mg}\cdot\text{L}^{-1}$ and 100 $\text{mg}\cdot\text{L}^{-1}$ respectively, pH value of the solution is 7.0. As demonstrated, PESA and SHMP have good scale inhibition effect on BaSO_4 , while the PAA, PMAAA, AA/HPA/AMPS and PBTCA still have no BaSO_4 scale inhibition effect even at the concentration of 15 $\text{mg}\cdot\text{L}^{-1}$. The impacts of PESA, SHMP and PASP concentration on BaSO_4 scale inhibition efficiency are shown in Figure 2. As demonstrated in Figure 2, the BaSO_4 scale inhibition efficiency of PESA is superior to the traditional BaSO_4 scale inhibitor SHMP; as a result, PESA has a very strong scale inhibition effect on BaSO_4 .

FIGURE II. INFLUENCE OF SCALE INHIBITOR CONCENTRATION ON BaSO_4 SCALE INHIBITION EFFICIENCY.

C. Static Beaker Test for Corrosion Inhibition

The static corrosion inhibition efficiency of PESA to carbon steel at different concentration is shown in Table 2 and Figure 3, which was tested by weight loss experiment.

Table 2 and Figure 3 illustrate that, under the condition of this water quality, the corrosion inhibition effects of PESA used alone to carbon steel at low concentrations is weak, and with the increase of PESA concentration, corrosion inhibition is gradually strengthened. Therefore, to achieve a better inhibition affect, the required quality concentration of PESA should be higher.

TABLE II. THE WEIGHT LOSS EXPERIMENT RESULTS AT DIFFERENT PESA CONCENTRATION.

PESA(mg·L ⁻¹)	Corrosion velocity(mm·a ⁻¹)	Corrosion inhibition efficiency (%)
0	1.3855	-
50	1.3524	2.39
100	1.2654	8.67
150	0.4799	65.36
200	0.3482	74.87
800	0.2454	82.29

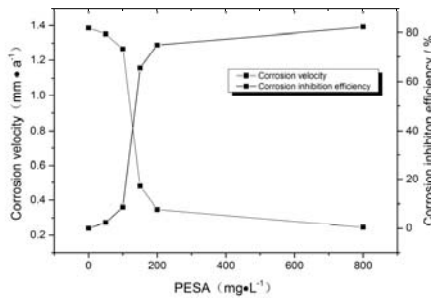


FIGURE III. PESA CONCENTRATION—CORROSION VELOCITY—CORROSION INHIBITION EFFICIENCY.

Table 3 illustrates the effect on the static corrosion inhibition efficiency of PESA to carbon steel at different pH values according to the weight loss experiment. The pH values of the test solution were adjusted to 2, 4, 9 respectively, and then dosing 200 mg · L⁻¹ of PESA to carry out weight loss experiment.

As shown in Table 2, it demonstrates that pH value has significant effect on corrosion inhibition property of PESA. There is low corrosion inhibition efficiency in acidic condition; as the pH value increases, the corrosion velocity of carbon steel obviously decreases as well as corrosion inhibition efficiency increases, namely corrosion inhibition enhances. Moreover, in alkaline condition, the corrosion inhibition efficiency of PESA is better and can reach about 80%. It is assumed that, due to the alkaline environment, PESA may dissociate, which contributes to its adsorption on the carbon steel surface.

TABLE III. THE WEIGHT LOSS EXPERIMENT RESULTS OF PESA AT DIFFERENT PH VALUES OF TEST SOLUTION.

pH	PESA(mg·L ⁻¹)	Corrosion velocity(mm·a ⁻¹)	Corrosion inhibition efficiency (%)
2	0	4.5357	-
2	200	3.9118	13.76
4	0	1.6462	-
4	200	0.6587	60.00
12	0	0.1378	-
12	200	0.0227	83.53

D. The Tafel Polarization Curve Measurement

The polarization curve experiments of carbon steel at different pH values of test solution (pH=2, 4, 9) were carried out and the corresponding results are given in Figure 4-6. As they illustrated, it is found that there are all greater impact on the anodic process of electrodes by adding PESA at different pH values of test solution; this effect amplified as the pH value increases, and the self-corrosion potential turns to the positive shift. In other words, PESA belongs to anodic corrosion inhibitor.

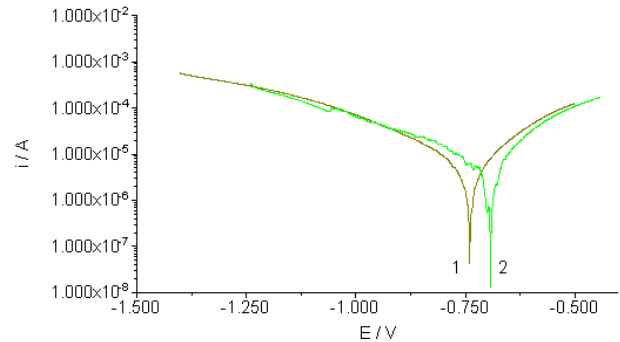


FIGURE IV. THE POLARIZATION CURVE OF PESA AT PH=2. (1-PESA, 2-BLANK)

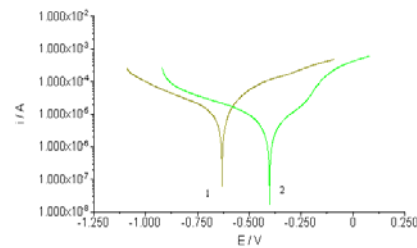


FIGURE V. THE POLARIZATION CURVE OF PESA AT PH=4 (1-PESA, 2-BLANK).

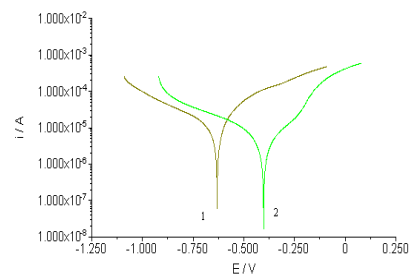


FIGURE VI. THE POLARIZATION CURVE OF PESA AT PH=9 (1-PESA; 2-BLANK.)

Figure 7 shows the results of polarization curve tests to carbon steel at different concentrations of PESA (50 mg·L⁻¹, 100 mg·L⁻¹, 150 mg·L⁻¹, 200 mg·L⁻¹ and 800 mg·L⁻¹, respectively). The parameters, such as polarization resistance (Rp), self-corrosion potential (E_{corr}), anodic Tafel Slope, cathodic Tafel slope, etc., as well as corrosion velocity, can be obtained by using the software of PGATAT30 electrochemical

workstation to carry out the Tafel extrapolation to the active part of the polarization curves in Figure 4^[7, 8], and then corrosion inhibition efficiency can be calculated according to the corrosion velocity of the blank specimen. The data is shown in Table 4.

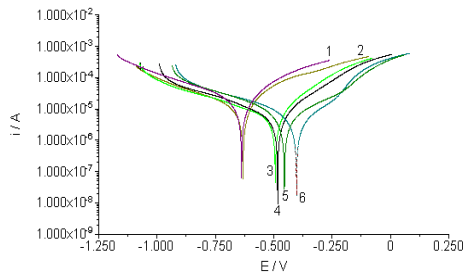


FIGURE VII. THE POLARIZATION CURVES OF PESA AT DIFFERENT CONCENTRATIONS.

(1-800mg·L⁻¹; 2-Blank; 3-50mg·L⁻¹; 4-100mg·L⁻¹; 5-200mg·L⁻¹; 6-150 mg·L⁻¹)

As it is observed in Table 4, the corrosion inhibition efficiency is better by adding more PESA in the neutral solution medium. When PESA is used alone, aiming to achieve better corrosion inhibition efficiency, the concentration of PESA should reach 200 mg · L⁻¹ or more. The OCP of PESA at different concentrations increase obviously compared to the blank, which demonstrates that PESA is mainly as the function of anodic inhibitor [9, 10]. The reason contributes to this

is that PESA adsorbs on the carbon steel surface and reacts with Fe³⁺, but also forms the membrane which hampers the proliferation of Fe²⁺ in the anodic area, so that it comes anodic polarization and anodic potential rise, and eventually leading E_{corr} to be higher.

From Figure 7, it is shown that there is no significant inhibition efficiency for the anode reaction of carbon steel by adding 100 mg · L⁻¹ of PESA, indicating that the PESA concentration is not high enough, the formed membrane has poor density and may even only covers part of the surface of carbon steel. The formed membrane by PESA, which is incomplete, makes effect on its inhibition efficiency, and thus the anode reaction cannot be effectively curbed.

Compared with the blank, the anodic Tafel Slope rises markedly with the PESA concentration increases, which illustrating that the presence of PESA controls the anode in the reaction, so as to inhibit the anodic reaction of corrosion process. Accompanying with the concentration of PESA reaching above 200 mg · L⁻¹, the corrosion potential E_{corr} decreases as well as the Tafel Slope of cathodic polarization curves rises, in accordance with which it implies that the process of cathodic reaction begins to be inhibited as the concentration of PESA increases. PESA can chelate Ca²⁺, Mg²⁺ and other ions in the test solution and then disperse in it; with the increased concentration of PESA, part of the chelate deposit in the cathode region and thus the cathodic process is suppressed [11].

TABLE IV. THE POLARIZATION PARAMETERS OF DIFFERENT CONCENTRATIONS OF PESA AT 30°C.

Mass ⁺ concentration ⁺ (mg·L ⁻¹) ⁺	-OCP ⁺ ⁺ (mV) ⁺	E _{corr} ⁺ ⁺ (mV) ⁺	R _p ⁺ ⁺ ×10 ³ oh m ⁺	B _a ⁺ ⁺ (m·dec ⁻¹) ⁺	B _c ⁺ ⁺ (m·dec ⁻¹) ⁺	Corrosion- velocity ⁺ (mm·a ⁻¹) ⁺	Corrosion-inhibi- tion efficiency ⁺ (%) ⁺
0 ⁺	-490 ⁺	-635 ⁺	4.00 ⁺	170 ⁺	368 ⁺	0.1126 ⁺	- ⁺
50 ⁺	-475 ⁺	-498 ⁺	6.91 ⁺	128 ⁺	395 ⁺	0.0702 ⁺	37.66 ⁺
100 ⁺	-391 ⁺	-480 ⁺	7.81 ⁺	204 ⁺	406 ⁺	0.0681 ⁺	39.52 ⁺
150 ⁺	-320 ⁺	-390 ⁺	1.06 ⁺	206 ⁺	492 ⁺	0.0439 ⁺	61.01 ⁺
200 ⁺	-335 ⁺	-450 ⁺	8.90 ⁺	271 ⁺	435 ⁺	0.0312 ⁺	72.29 ⁺
800 ⁺	-571 ⁺	-630 ⁺	3.01 ⁺	212 ⁺	397 ⁺	0.0209 ⁺	81.44 ⁺

As the concentration of PESA further up to 800 mg · L⁻¹, the cathode Tafel Slope of PESA polarization curve increases, it is inferred there may be a reaction between Mg²⁺ and OH⁻ generated in the cathode reaction, and the product Mg(OH)₂ deposits in the cathode region as to prevent the spread of oxygen and thus inhibit the cathode reaction [12-16].

E. Electrochemical Impedance Measurement

The EIS Nyquist plots of carbon steel electrode after the tests of dosing different concentrations of PESA in the solution have been shown in Figure 8. Since the impedance is plural, the horizontal axis is for the real part while the vertical axis is for the imaginary part. As can be seen from Figure 8, impedance spectra suffers different degrees of large semicircular features; under the condition that the dosing concentration of PE-

SA is less than 100 mg · L⁻¹, the increasing range of R_p value (charge transfer resistance) with the rising of the concentration. It demonstrates that corrosion inhibition efficiency is not very good in terms of the low dosing concentration of PESA; when the concentration to be more than 100 mg · L⁻¹, R_p value increases by a large margin. The greater R_p value indicates that the adsorbed membrane on the surface of carbon steel is thicker by the action of corrosion inhibitor, and it also comes out greater absorption area as well as the better inhibition efficiency, due to which the increase of the R_p value illuminates that PESA has significant corrosion inhibition effect to carbon steel.

The high-frequency capacitance arc appearing in the high-frequency zone of the impedance spectroscopy shows that there is a more complete adsorbed membrane forming on the surface of electrode, and the metallic corrosion reaction is in-

hibited, moreover, the charge transfer reaction plays a leading role in metal corrosion process. The chord length from semi-circle to the real axis (Z' axis) corresponds to the charge transfer resistance R_p . In terms of the corrosion inhibitor system, R_p reflects the resistance for the process of metal ionization by the covering layer of corrosion inhibitor, or reflects the rate of corrosion reaction. The greater R_p value is, indicating the greater resistance for the process of metal ionization while the smaller corrosion rate of metals.

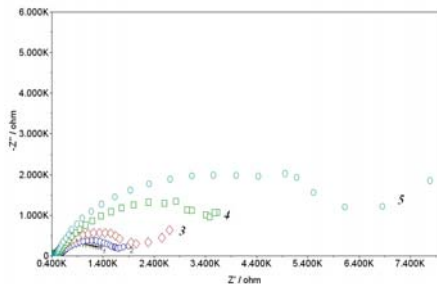


FIGURE VIII. THE EIS NYQUIST PLOTS OF CARBON STEEL ELECTRODE IN THE SOLUTIONS ADDED DIFFERENT CONCENTRATIONS OF PESA.

(1-Blank; 2-50 $\text{mg}\cdot\text{L}^{-1}$; 3-100 $\text{mg}\cdot\text{L}^{-1}$; 4-150 $\text{mg}\cdot\text{L}^{-1}$; 5-200 $\text{mg}\cdot\text{L}^{-1}$)

Table 5 shows the corrosion inhibition efficiency at different concentrations of PESA calculated by

$$\eta = (1 - R_{p0}/R_p) \times 100\%$$

where R_p and R_{p0} are the charge transfer resistance in test solution with and without corrosion inhibitor, respectively. The greater the concentration of PESA, the stronger the corrosion inhibition effect within the scope of the experiment, which is indicated in Figure 9.

TABLE V. AC IMPEDANCE AT DIFFERENT CONCENTRATIONS OF PESA.

PESA($\text{mg}\cdot\text{L}^{-1}$)	$R_p(\text{ohm})$	$\eta(100\%)$
0	4.99971×10^2	-
50	1.19138×10^3	58.03
100	1.75014×10^3	71.43
150	3.86054×10^3	87.05
200	6.62408×10^3	92.45

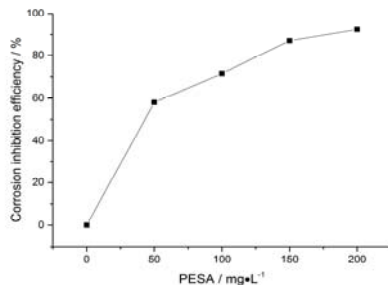


FIGURE IX. PESA CONCENTRATION—CORROSION INHIBITION EFFICIENCY.

IV. CONCLUSION

Under the experimental conditions in this paper, comparing with the scale inhibitors, such as PBTA, PAA, PMAAA and AA/AMPS, PESA and SHMP has scale inhibition effect on CaCO_3 (12 $\text{mg}\cdot\text{L}^{-1}$ of the scale inhibitor's concentration) and BaSO_4 (6 $\text{mg}\cdot\text{L}^{-1}$ of the scale inhibitor's concentration), furthermore, the scale inhibition efficiency on CaCO_3 , BaSO_4 of PESA is much better than SHMP. Meanwhile, SHMP is easy to decompose into the precipitation of calcium phosphate, reducing its scale inhibition efficiency, but also has high phosphorus content, while the PESA is a phosphate-free environment-friendly inhibitor with versatile scale inhibition properties, and thus it has a very broad application prospects.

There is limited corrosion inhibition effect on carbon steel provided by PESA used alone at low concentration ($<120 \text{ mg}\cdot\text{L}^{-1}$), and with the increase of PESA concentration, corrosion inhibition is gradually strengthened.

The pH value has significant effect on corrosion inhibition property of PESA. And PESA has corrosion inhibition effects to carbon steel by $120 \text{ mg}\cdot\text{L}^{-1}$ of the concentration to some degree. However, there is low corrosion inhibition efficiency in acidic condition; as the pH value increases, the corrosion velocity of carbon steel obviously decreases as well as corrosion inhibition efficiency increases, namely corrosion inhibition enhances. Moreover, in alkaline condition, the corrosion inhibition efficiency of PESA is better and can reach about 80%. PESA is mainly as the function of anodic corrosion inhibitor.

PESA curbs metallic corrosion process by forming the absorbed membrane on the surface of carbon steels, as well as the charge transfer reaction plays a leading role in metal corrosion process.

PESA should be a non-phosphorous, biodegradable, environmentally friendly scale and corrosion inhibitor implemented in industrial water treatment.

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