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Determination of polyepoxysuccinic acid in circulating water by spectrometric method

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ABSTRACT

In this article, a simple and sensitive spectrometric method used for determination of polyepoxysuccinic acid (PESA) in circulating water was investigated. The availability of this method depends on the turbidity formed by PESA and dodecyl dimethyl benzyl ammonium chloride (1,227). In order to obtain the optimal experimental conditions, influencing factors such as dosage of 1,227, pH value, and stabilization time were discussed. Interferences of coexisting substances which may hamper the determination of PESA were also studied. The optimal experimental conditions were as follows: 1,227 concentration of 250 mg L^{-1}, pH range of 9–10, and stabilization time of 30 min. Under these conditions, a better linear relationship between absorbance and concentration was observed within a PESA concentration range of 0–16 mg L^{-1}. It was also demonstrated that foreign ions (i.e. PO_4^{3-}, SO_4^{2-}, Cl^-, Fe^{2+}) and water quality stabilizers (i.e. 1,2,3-benzotriazole and 1-hydroxy ethylidene-1, 1-diphosphonic acid) had no influences on the determination. In addition, interferences of Ca^{2+} and Mg^{2+} can be eliminated by adding a desired amount of ethylenediamine tetraacetic acid disodium salt. To minimize the influence of sodium hexametaphosphate, a proper amount of HCl was added into the solution, and then it was boiled after mixing. In summary, this method is very simple, concise, and accurate with a lower detection limit, which could provide a theoretical and practical guidance for the determination of PESA.

Keywords: Turbidity; Polyepoxysuccinic acid; Dodecyl dimethyl benzyl ammonium chloride; Spectrophotometry; Circulating water

1. Introduction

Water is widely used in industrial processes as a coolant to remove heat from equipment. However, with continuous circulation and evaporation losses, the concentrations of certain ions, such as Ca^{2+}, Mg^{2+}, and Ba^{2+}, are gradually enhanced. When concentrations of abovementioned ions exceed certain limits, the resulting reaction products will precipitate and deposit on the surface of equipment [1]. The presence of scales or deposits will greatly hamper the heat transfer rate and hence disturb the efficiency of the

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circulating water system. Furthermore, cleaning or removal of such scales or deposits is rather expensive due to the shutdown of system [2]. Therefore, inhibiting the formation of scales on industrial equipment is of great importance [3]. Phosphate scale inhibitors and some water-soluble polymers, including polymers derived from unsaturated carboxylates and unsaturated sulfonates or their salts, are helpful to ensure the solubility of cation–anion reaction product [4]. However, some adverse effects (e.g. eutrophication and non-biodegradability) of these additive agents have been widely reported [5–8].

Polyepoxysuccinic acid (PESA) is a non-nitrogen, non-phosphorus scale inhibitor with excellent biodegradability [9], which was firstly synthesized at American Betz laboratory [10], and has been widely used as a scale inhibitor, corrosion inhibitor, and dispersant. It has been successfully applied in high alkalinity, high hardness, and high temperature conditions [6,11]. The general formula of PESA is illustrated in Fig. 1 [12]. In the formula, M can be hydrogen or water-soluble cation such as NH_4^+, Na^+, or K^+; R can be hydrogen, C_{1-4} alkyl, or C_{1-4} substituted alkyl; n is varied from 2 to 50. The scale inhibition ability of PESA is closely related to its polymerization degree. When n is ranging from 2 to 25 and better in 2–11, superior scale inhibition effects are exhibited [13]. In addition, PESA has a higher ion tolerance in comparison with other traditional scale inhibitors [14]. It also possesses relatively higher Langelier saturation indexess in water system, which ranges from 2.5 to 3 [14]. Meanwhile, PESA could present a good synergistic effect with other inhibitors, such as inorganic phosphates, organic phosphates, and polyacrylic/polymaleic acids inhibitors [15–17]. On account of its unique property, PESA has become a research hotspot and attracted much attention.

Over the past few decades, the researches of PESA mainly focused on synthesis [14,18,19], scale inhibition [14,15,20–22], corrosion inhibition [9,23,24], and synergistic performance [9]. However, there are almost no tests focused on its own physical and chemical index.

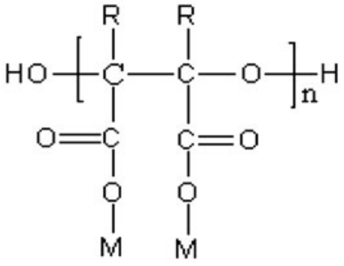

Fig. 1. Chemical structure of PESA.

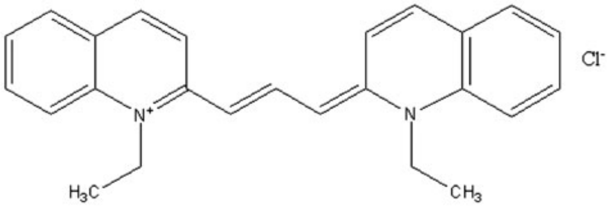

Fig. 2. Chemical structure of pinacyanol chloride.

In Chinese patent 102252985A [25], Zhang et al. proposed a colorimetric method for determination of PESA through a pinacyanol chloride dye. The structure is showed in Fig. 2. However, due to the shortbacks of instability of pinacyanol chloride in water [26], lower tolerance to foreign species and narrow measurement range, this approach is not appropriate for monitoring the concentration of PESA in practice.

More recently, a novel approach using fluorescence emission technique to determine the concentration of polymer has emerged [27–35]. The fluorescent targeting agents could be bonded to polymers covalently. However, it will lead to a secondary pollution in the aqueous system and thus needs extra treatments and increases operational cost [36]. Besides, owing to the complexity of preparatory work and high prices of fluorescent agents, it is not suitable for a large-scale promotion.

The goal of this work was to establish an accurate and effective method to monitor the concentration of PESA in cooling water system timely and accurately and achieve both economical and scale inhibiting benefits. Turbidity-based spectrometric method was employed in this research. To obtain optimal experimental conditions, some factors (i.e. dosage of dodecyl dimethyl benzyl ammonium chloride (1,227), pH, stabilization time) which may influence the determination of PESA were discussed. In addition, interferences of foreign substances and their elimination methods were also investigated.

2. Experimental section

2.1. Reagents and solutions

1,227 (44 wt.%) and PESA (40 wt.%) were supplied by Shandong Taihe water treatment Co.,Ltd (Shandong, China). Standard solution of 1,227 was prepared by diluting 10 mL of 1,227 stock solution to 100 mL, which was equal to the concentration of 44 g L^{-1}. Then, it was further diluted to obtain the 1,227 working solution (1.76 g L^{-1}). Similarly, standard solution of PESA was prepared using 1 mL of PESA stock solution to 100 mL, which was equal to concentration of

4 g L^{-1}. Then, it was further diluted to obtain the PESA working solution (200 mg L^{-1}).

1,2,3-benzotriazole (BTA, Tianjin Kermel Chemical Reagent Co., Ltd, Tianjin, China), 1-diphosphonic acid (HEDP, Shandong Taihe water treatment Co., Ltd, Zaozhuang, China), and sodium hexametaphosphate (SHMP, Tianjin Kermel Chemical Reagent Co., Ltd, Tianjin, China) were dissolved in water to obtain the standard solutions in concentration of 50, 500, and 100 mg L^{-1}, respectively. A series of solutions containing possible interfering ions were prepared with $CaCl_2$, $MgCl_2·6H_2O$, $FeSO_4·7H_2O$, $Na_3PO_4·12H_2O$, Na_2SO_4, and NaCl, respectively.

All solutions were prepared with deionized water, and all reagents used in this work were analytical grade.

2.2. Apparatus

The absorbance of solution was measured by an UV-2450 spectrophotometer (Shimadzu, Japan) with a 1 cm quartz cuvette in the range of 400–700 nm. The pH value of solution was measured by a pHS-3C pH meter (Shanghai Leici instrument, Shanghai, China) with a combined electrode. Electric furnace and electronic balance (Beijing everlasting light medical instrument factory, Beijing, China) were also used during the experiments.

2.3. Experimental principle

Under certain conditions, PESA, which is negatively charged, could react with quaternary ammonium cationic surfactants and form ion-association complex precipitation quantitatively. Within a definite concentration range, a better linear correlation between formed turbidity and concentration of PESA is obtained, which could be measured by a spectrophotometric method. Thus, the concentration of PESA can be quantitatively derived through the regression equation or standard curve.

2.4. Experimental process

Batch experiments were performed to obtain the optimum experimental conditions. Firstly, a certain amount of PESA was added into some 50 mL volumetric flasks, along with different volumes of 1,227 working solution. Next, pH values of mixing solutions were adjusted using either 0.1 M HCl or 0.1 M NaOH. The mixed solutions were then diluted to the mark with deionized water. Afterward, the solutions were stood for a certain time interval and then measured by an UV–vis spectrophotometer with a 1 cm quartz cuvette in the range of 400–700 nm.

3. Results and discussion

3.1. Analysis of influencing factors

In this section, different parameters such as of 1,227, pH value, and stabilization time were investigated to obtain the optimal experimental conditions. From a practical and ecological point of view, the optimal conditions should have the advantages of low reagent consumption and high sensitivity.

The optimum 1,227 dosage was initially investigated. In our experiments, different volumes of 1,227

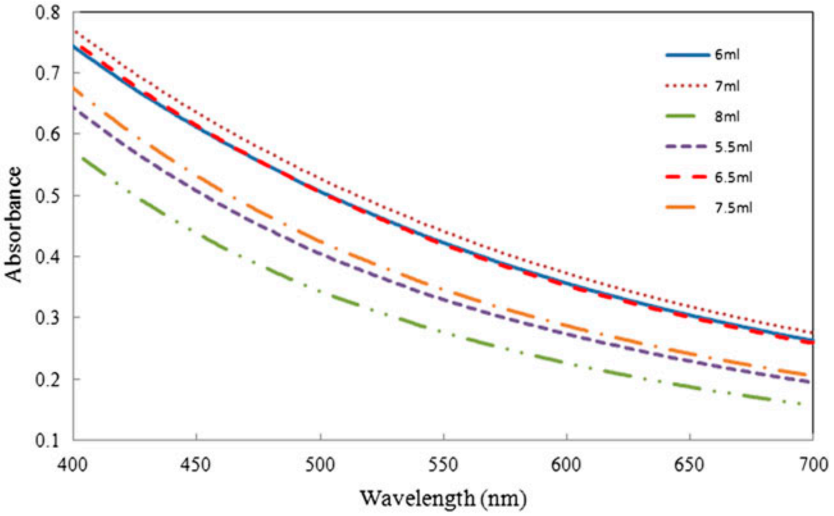

Fig. 3. Absorbance of mixed solution with different amounts of 1,227.

working solution were added to some volumetric flasks, separately. Following this, 4 mL of PESA working solution was added into above flasks separately, along with desired amount of deionized water to the total volume of 50 mL. After being mixed adequately and stood for 30 min, the absorbance of solution was measured from 400 to 700 nm with a 1 cm quartz cuvette. As shown in Fig. 3, the absorbance of solution depended highly on the dosage of 1,227 working solution. With the increase of 1,227 dosages, the absorbance of solution was first increased and then decreased with larger dosages. The absorbance could reach the peak when dosage of 1,227 working solution was 7 mL, which was equivalent to the concentration of 250 mg L^{-1}. This phenomenon is typically explained as micellar solubilization [37]. Furthermore, it should be mentioned that the concentration of PESA in this experiment, was maintained at 16 mg L^{-1}. Therefore, the optimal concentration of 1,227 solution was 250 mg L^{-1}.

In the following experiments, 420 nm was chosen as the preferable wavelength, at which the absorbances were relatively higher and the linear relationship was favorable.

Stabilization time is another important factor to be optimized. In this study, the stabilization time was varied from 0 to 50 min. The experiment was in accordance with procedures mentioned above. It should be noted that concentrations of 1,227 and PESA were maintained at 250 and 16 mg L^{-1}, respectively. From Fig. 4, it was observed that stabilization time had a great influence on the absorbance of mixed solution. With the prolongation of stabilization time, the absorbances reach their maximum value at about 30 min and then remain constant. Thus, 30 min was selected as the optimum stabilization time for subsequent experiments.

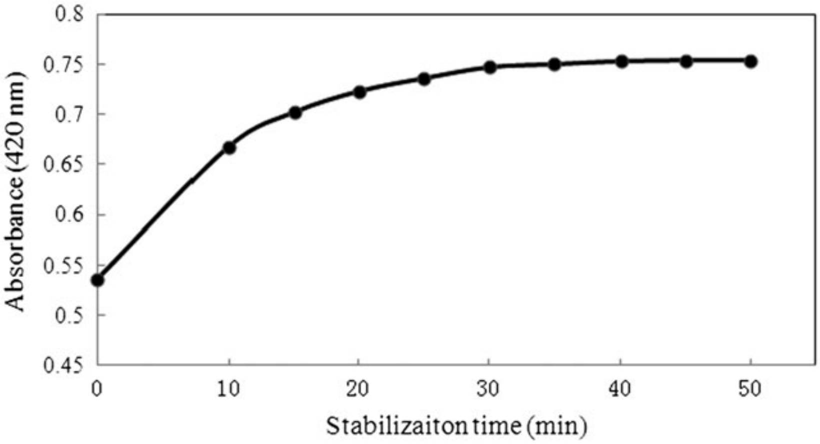

Fig. 4. Effect of stabilization time on the absorbance.

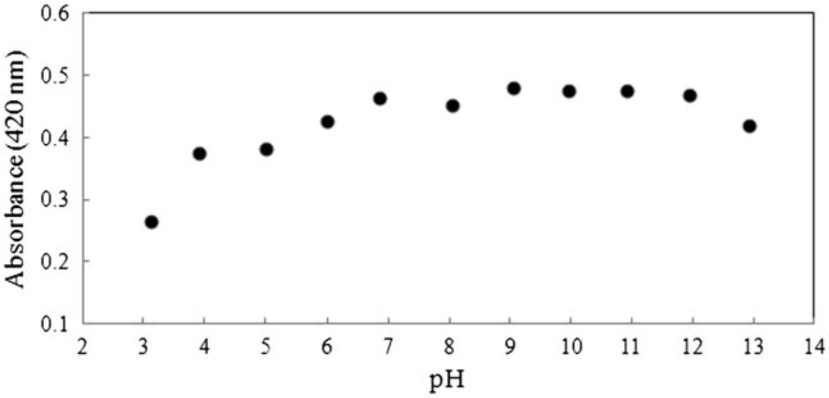

Fig. 5. Effect of pH value on the absorbance.

The pH value is an essential parameter in most turbidity-based assay. Experiments about impacts of pH values on the absorbance were carried out with pH increasing from 3 to 13. It should be noted that concentrations of 1,227 and PESA were maintained at 250 and 16 mg L^{-1}, respectively. From Fig. 5, it can be seen that pH value substantially influenced the absorbance of solution. On the other hand, when pH values ranged from 9 to 10, the absorbances were basically the same, so this pH range was appropriate for follow-up experiments.

3.2. Preparation of calibration curve

Different amounts of PESA working solution (0, 0.5, 1.0, 2.0, 3.0, and 4.0 mL) were added into some 50 mL volumetric flasks separately, along with 7 mL of 1,227 working solution and proper volume of deionized water to the total volume of 50 mL. After being mixed adequately, the solutions were stabilized at room temperature for 30 min. The pH values of solutions were adjusted (in the range of 9–10) using either 0.1 M HCl or 0.1 M NaOH. Afterward, the absorbances of these solutions were measured by an UV–vis spectrophotometer with a 1 cm quartz cuvette at 420 nm.

Under these conditions, regression analysis of experimental data for the standard Beers law plot was performed. The absorbance (Y) vs. concentrations (X) gave the equation as $Y = 0.0368X - 0.0402$ with a regression coefficient of $r = 0.983$. The concentration range of PESA in these tests was 0–16 mg L^{-1} with a detection limit of 0.215 mg L^{-1}. The relative standard deviation was 0.31% for PESA solution (8 mg L^{-1}). Recovery tests were also performed by adding different amounts of PESA working solution to three cooling water samples, with recovery rates varying from 95 to 106%.

3.3. Effect of foreign substances on PESA determination

The interferences of foreign substances which may coexist with PESA in circulating water were evaluated under optimal conditions. This was done by adding possible interfering substance to the standard solution containing 16 mg L^{-1} of PESA. Each experiment was triplicated under identical conditions, and only the mean values were given. When errors of absorbance with the interference of certain ion exceeded ±3%, it was thought that this substance had a negative influence on the determination of PESA. Table 1 summarized the tolerance limit of various substances studied.

Table 1
Effect of some interfering ions on PESA determination

Species	Tolerated limit (mg L^{-1})
Fe^{2+}	1
SO_4^{2-}, Cl^-	1,000
HEDP, PO_4^{3-}	50
BTA	2

It should be noted that all abovementioned concentrations in Table 1 are in the normal range of circulating water. Consequently, the existence of those species had no effects on the determination of PESA. On the other hand, water used in industrial cooling water system often contains high concentrations of Ca^{2+} and Mg^{2+}, which may exert a marked influence on PESA determination. From Fig. 6, it can be observed that the absorbance decreased dramatically with the increase of Ca^{2+} and Mg^{2+}. This fact could be attributed to the good chelating ability of PESA with a variety of metal ions, especially with Ca^{2+}, Mg^{2+}, and Fe^{2+} [9,24]. Due to its superior chelating ability and excellent biodegradability, PESA has also been widely used as a chelating reagent to remove heavy metals from sewage sludge or soils [38–40].

To eliminate the interference of Ca^{2+} and Mg^{2+}, ethylenediamine tetraacetic acid disodium salt (EDTA-2Na) was added into the solution. Blank experiment without Ca^{2+}, Mg^{2+}, and EDTA-2Na was also conducted. As shown in Fig. 7, EDTA-2Na implied a remarkable effect on the absorbance with Ca^{2+} and Mg^{2+}. It was noteworthy that considerable variances in absorbance with identical EDTA-2Na concentration were achieved, which should be attributed to the difference of molar mass between Ca^{2+} and Mg^{2+}. Thus, EDTA-2Na should be added with at least mole ratio of 1:1 (EDTA-2Na: Ca^{2+} or Mg^{2+}). In order to avoid a misinterpretation of data at Y-axis, it must be stressed that these data were obtained from blank experiment without Ca^{2+}, Mg^{2+}, and EDTA-2Na, which demonstrated the interferences of Ca^{2+} or Mg^{2+} could be eliminated by adding proper amounts of EDTA-2Na.

In addition, the interference of SHMP was taken into consideration. The existence of SHMP also had a pronounced influence on the absorbance because SHMP on sale in normally was prepared in Graham method [41,42]. Thereby, SHMP is not a simple hexametaphosphate with fixed composition but a long-chain compound containing 20 to 100 units of phosphate. The reaction between 1,227 and hexametaphosphate (ammonium polyphosphate) could form insoluble precipitates when the degree of polymerization is higher than 20, thus would disturb the determination of PESA. The interferences can be eliminated by adding adequate amounts of HCl to solution and

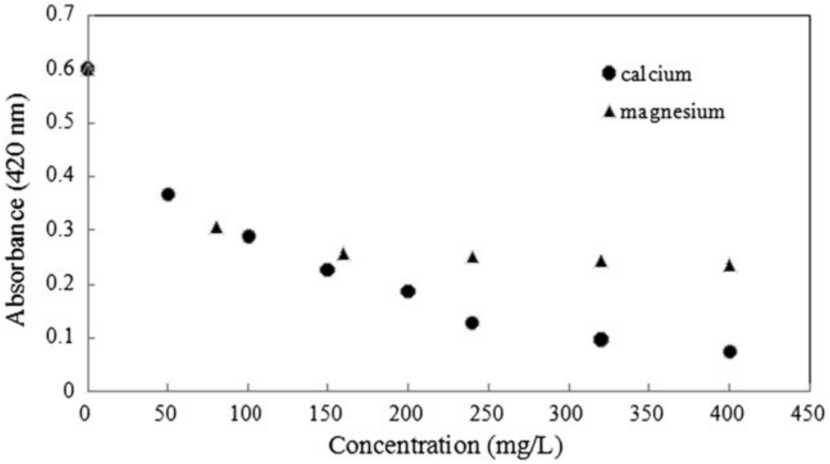

Fig. 6. Effect of Ca^{2+} and Mg^{2+} on the determination of PESA.

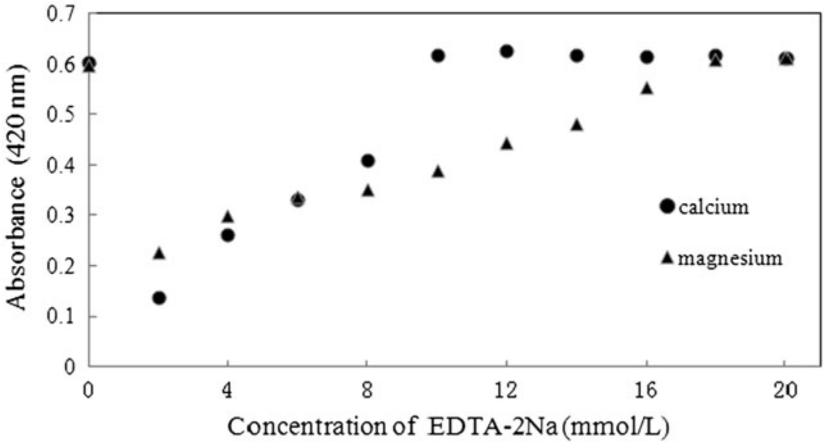

Fig. 7. Elimination of interferences of Ca^{2+} and Mg^{2+} by EDTA-2Na.

then boiling for about 10 min. This is because boiling can lead to the depolymerization of polyphosphate and thus can remove the interference of SHMP. Table 2 summarized the absorbance before and after boiling with HCl. From Table 2, it can be clearly seen that absorbances after boiling were basically the same.

Table 2
Absorbance before and after boiling with HCl

	Concentration of SHMP (mg L^{-1})				
	0	2	4	6	8
Not boiled	0.6	0.651	0.838	0.91	0.965
Boiled	0.606	0.593	0.609	0.594	0.609

4. Conclusion

This study describes a very concise and explicit technique utilized for PESA determination. The main conclusions are as follows:

- Under optimal conditions namely, 1,227 concentration of 250 mg L^{-1}, pH range of 9–10, and stabilization time of 30 min, a better linear relationship between absorbance and concentration of PESA was observed within the range of 0–16 mg L^{-1}.
- Foreign substances, such as Fe^{2+}, SO_4^{2-}, Cl^-, PO_4^{3-}, HEDP, and BTA, which may be coexisted with PESA in cooling water, have nearly no effects on the determination.
- Interferences by Ca^{2+} and Mg^{2+} could be eliminated by adding a desired amount of EDTA-2Na

into solution (mole ratio between Ca^{2+} or Mg^{2+} and EDTA-2Na was at least 1:1). Interferences of SHMP could be eliminated by adding a proper amount of HCl and being boiled thereafter.
- This technique has advantages of high reproducibility, enhanced stability, and a low detection limit of 0.215 $mg\,L^{-1}$.

All these features make this method suitable for monitoring the concentration of PESA timely and accurately, which is essential for cooling water system because the concentration of PESA must be maintained at a certain level. Furthermore, this method is employed by a spectrometry method, which allows a large number of samples to be analyzed on a day-to-day basis. Therefore, this study provides a theoretic foundation for real-time monitoring of PESA, which could boost its use in industrial application.

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