Organic Phosphorus Extraction in the Fouling of the Circulating Cooling System

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ABSTRACT: The organophosphate scale inhibitor (HEDP)-containing C−P bond is an agent widely used in the circulating cooling system to ensure the safe operation. In order to explore the effects of its decomposition and migration transformation in the system on the system security, it is necessary to carry out the quantitative and qualitative analysis of organic phosphorus in fouling. According to the Ivanoff method, with the organic phosphorus extraction rate as an indicator, the types and concentrations of extractants, extraction time, and mass of the extracted scale were optimized. When the mass of the extracted scale was 0.4 g, the extraction rates of active organic phosphorus and moderately active organic phosphorus were, respectively, 46.53 and 47.14%, and there was no inactive organic phosphorus and residual phosphorus. The total extraction rate of organic phosphorus was up to 94.32%. The optimum extraction conditions of various organic phosphorus components were determined as follows: 10 h extraction in 0.5 mol/L KHCO₃ for active organic phosphorus and 3 h extraction in 1.0 mol/L HCl solution for moderately active organic phosphorus. The optimized extraction process only involves two steps: acid extraction of active organic phosphorus and acid extraction of moderately active organic phosphorus. The optimized method is characterized by the simple operation and good extraction effect and can be applied in the detection and analysis of organic phosphorus in the fouling of the circulating cooling system.

1. INTRODUCTION

Phosphorus affects scaling, corrosion, and microbial growth in circulating cooling systems as well as the safe operation of the system.1−4 At present, the organic phosphorus scale and corrosion inhibitors are the dominant water quality stabilizers with good performance and low price. As shown in Figure 1, HEDP, ATMP, and EDTMP with −C−PO₃H₂ functional groups can protect the system for safe operation.5−10 However, the degradation or transformation of organic phosphorus agents in the system may generate various phosphorus forms, which affect the inhibition performance of the agents.11−14 Therefore, the migration mechanism of organic phosphorus in the system is the important basis for ensuring the operation safety. The extraction method of organic phosphorus in fouling is the primary prerequisite for studying the migration mechanism.

The current methods for the continuous extraction of organic phosphorus mainly include the Ivanoff method,15 the Bowman–Cole method,16 and the Golterman method.17 These methods were initially established for the extraction of organic phosphorus in the soil and then optimized and applied in the studies on the migration and transformation of phosphorus in the water–sediment interface of rivers, especially phosphorus forms in riverbed sediments.18−21 However, the extraction method of organic phosphorus in the scale of the circulating cooling system was not reported. In the study, based on the comparative analysis of various methods, according to the Ivanoff method, the extraction process was optimized based on the organic phosphorus...
Then, the conditions for the continuous extraction of organic phosphorus in the fouling, including the type and concentration of the extractant, extraction time, and mass of the extracted scale, were optimized in terms of the extraction efficiency of organic phosphorus (active organic phosphorus and moderately active organic phosphorus). The established optimization method lays a foundation for studying the migration and transformation of organic phosphorus in the system.

2. RESULTS AND DISCUSSION

2.1. Extraction Step Optimization. It can be seen from Table 1 that the extraction procedure of active organic phosphorus in the fouling of the circulating cooling system is simplified as one-step extraction with KHCO₃ because there is no biologically active organic phosphorus in the residues.

The extraction steps of moderately active organic phosphorus are significantly different. The soil phosphorus analysis involves acid extraction and alkali extraction. Acid extraction (HCl) aimed to eliminate the effect of inorganic phosphorus on the extraction of organic phosphorus. The inorganic phosphorus and moderately active organic phosphorus were extracted with HCl. The components of the fouling were minerals, and the content of inorganic phosphorus was low. After the fouling was extracted with HCl, there was no residue and alkali extraction was not required, indicating that only active organic phosphorus and moderately active organic phosphorus (extracted with acid) existed in the fouling. Pₒ-HCl is moderately active organic phosphorus (extracted with acid) without inactive organic phosphorus and residual phosphorus. The optimization method had two steps. First, KHCO₃ solution was used to extract active organic phosphorus, and HCl was used to extract moderately active organic phosphorus from the residues. However, the existence of inorganic phosphorus in the fouling reduced the apparent extraction rate of HCl.

In view of the fact that there was little inorganic phosphorus in the fouling, we attempted to extract organic phosphorus directly with NaOH. Moderately active organic phosphorus and inactive organic phosphorus were obtained. It was also found that there was no inactive organic phosphorus in the extracts, but the residues still existed. The residues were treated according to the Ivanoff method, and the total phosphorus was measured to be close to zero. Energy-dispersive spectroscopy (EDS) and X-ray diffraction (XRD) analysis indicated the presence of trace inorganic phosphorus. The analysis further verified that the organic phosphorus in the fouling only contained active organic phosphorus and moderately active organic phosphorus (extracted with alkali). The residual phosphorus was inorganic phosphorus. The optimized extraction method is shown in Figure 2.

![Formula and structures of HEDP, ATMP, and EDTMP.](image)

**Figure 1.** Formula and structures of HEDP, ATMP, and EDTMP.

**Table 1. Organic Phosphorus Extraction Optimized Method**

| organic phosphorus types | optimized method |
|-------------------------|-----------------|
| active Pₒ-La (1)        | 0.4 g of dry scale was extracted with 20 mL of 0.5 M KHCO₃ solution for 10 h and centrifuged. The difference between the total phosphorus content and inorganic phosphorus content in the extracts was the active organic phosphorus content (Pₒ-La). |
| moderately active Pₒ-M-La (2) | The residues in Step 1 were extracted with 1 M HCl and centrifuged and the difference between the total phosphorus content and the inorganic phosphorus content in the supernatant was Pₒ-HCl. |
| inactive Pₒ-non-La (3) | The residues in Step 1 were extracted with 20 mL of 1.0 M NaOH, and the obtained TP was Pₒ-HCl = Pₒ-M-La or Pₒ-fulvic. |
| residual phosphorus Pₒ-res | Pₒ-res = 0 |

![Organic phosphorus extraction optimized method.](image)

**Figure 2.** Organic phosphorus extraction optimized method.

The extraction conditions for active organic phosphorus, moderately active organic phosphorus extracted with acid, and moderately active organic phosphorus extracted with alkali were, respectively, optimized, and the optimal method was determined according to the total extraction rate of organic phosphorus.

2.2. Optimization of Active Organic Phosphorus Extraction. 2.2.1. Extractant Type. Under the experimental conditions of 0.3 g of the extracted scale, 10 h extraction, and centrifugation (8000 rpm, 10 min), the effects of the extractant type on the extraction rate of active organic phosphorus are shown in Figure 3.
The type of extractant had a great influence on the extraction rate of active organic phosphorus, and the extraction rate of the strong alkali weak acid extractant was significantly higher than that of the neutral extractant (Figure 3). When KHCO₃ was used as the extractant, the extraction rate of active organic phosphorus was the highest, 46.16%, whereas the extraction rate of H₂O was only 11.76%. Therefore, KHCO₃ was the optimal extractant for active organic phosphorus.

2.2.2. Extractant Concentration. Under the experimental conditions of 0.3 g of the extracted scale, 20 mL of KHCO₃ solution as the extractant, 10 h extraction, and centrifugation (8000 rpm, 10 min), the effects of KHCO₃ concentration on the extraction rate of active organic phosphorus are shown in Figure 4. The extraction rate increased with the increase in the concentration of extractants, Figure 4. The variation might be interpreted as follows. When the extractant concentration increased, the number of extractant molecules per unit volume was increased, and the effective collisions between extractant molecules and active organic phosphorus molecules were increased. Therefore, the extraction process was more complete. When the extractant concentration increased to 0.5 mol/L, the extraction rate tended to be stable at 45.66%. After the concentration was increased above 0.5 mol/L, the extraction rate did not increase significantly. When the extractant concentration was 1.0 mol/L, the extraction rate was only 1.32% higher than that at 0.5 mol/L. Considering the consumption of the agent and the extraction efficiency, we determined the optimum concentration of KHCO₃ as 0.5 mol/L.

2.2.3. Extraction Time. Under the experimental conditions of 0.3 g of the extracted scale, 20 mL of 0.5 mol/L KHCO₃ solution as the extractant, and centrifugation (8000 rpm, 10 min), the effects of extraction time on the extraction rate of active organic phosphorus are shown in Figure 5. The extraction rate generally increased with the extension of extraction time, but the increase was not significant (Figure 5). At the beginning of the shaking extraction stage, the extractant reacted with the scale rapidly, and a large amount of active organic phosphorus was extracted. With the extension of extraction time, the extraction agent had a higher contacting probability with active organic phosphorus, so the extraction rate continued to increase. After 10 h extraction, the extraction rate of active organic phosphorus reached 46.53% and tended to be stable, indicating that the extraction process was completed. In order to ensure that the active organic phosphorus could be fully extracted, the optimal extraction time was selected as 10 h.

2.3. Optimization of Moderately Active Organic Phosphorus Extraction. The extraction method of moderately active organic phosphorus was optimized by the acid method (HCl solution as the extractant) and alkali method (NaOH solution as the extractant).

2.3.1. Extractant Concentration. Under the experimental conditions of 20 mL of HCl/NaOH solution as the extractant, extraction time of 5 h/20 h, and centrifugation (5000 rpm, 10 min), the effects of extractant concentration on the extraction rate are shown in Figure 6. The extraction rates of the two extractants gradually increased with the increase in the extractant concentration (Figure 6). HCl solution achieved the better extraction effect of moderately active organic phosphorus than NaOH solution. When the extractant concentration was 1.0 mol/L, the extraction rate of the acid method reached 47.52%, whereas the extraction rate of the alkali method was only 36.88%. When the extractant concentration was higher than 1.0 mol/L, the extraction rate did not increase significantly and tended to be stable, indicating that the moderately active organic phosphorus had been completely extracted. Therefore, the
optimum extractant concentration of the acid or alkali method was determined as 1.0 mol/L.

2.3.2. Extraction Time. Under the experimental conditions of 20 mL of 1.0 mol/L HCl/NaOH solution as the extractant and centrifugation (5000 rpm, 10 min), the effects of extraction time on the extraction rate of moderately active organic phosphorus are shown in Figure 7.

In the acid method, the reaction between HCl and moderately active organic phosphorus was faster (Figure 7a). After 3 h, the extraction rate of moderately active organic phosphorus reached a stable value of 47.14%. In the alkali method, the extraction rate of the moderately active organic phosphorus was only 36.88% after 15 h (Figure 7b), indicating that the effect of moderately active organic phosphorus in acid extraction was better than that in the alkali method. In addition, as the extraction time increased, the extraction rate of the acid method or the alkali method did not increase significantly, indicating that the extraction process was completed. Therefore, optimum extraction time was 3 h for the acid method and 15 h for the alkali method.

2.4. Residual Residue Characterization Analysis. The residues of the alkali method were extracted with 1.0 mol/L H2SO4, and no inactive organic phosphorus was detected. The residues were characterized by EDS and XRD (Figure 8).

The residues contained trace phosphorus (Figure 8a), indicating that phosphorus-containing substances, which had not been completely extracted, still existed. Phosphorus-containing substances in the residue were inorganic phosphates (Figure 8b). Organophosphorus was the only source of phosphorus in the system, indicating that partial organic phosphorus was decomposed into inorganic phosphorus in the circulating cooling system.

2.5. Effects of the Mass of the Extracted Scale on Total Organic Phosphorus Recovery. The total extraction rates of organic phosphorus under different masses of the extracted scale are shown in Figure 9.

The total extraction rates under different masses of the extracted scale were higher (Figure 9). Under 0.2 g of the extracted scale, the total organic phosphorus extraction rate was significantly lower than that under 0.4 g of the extracted scale. When the mass of the extracted scale was 0.4 g, the extraction rate reached the highest value, 94.32%. When the mass of the extracted scale was more than 0.4 g, the total organic phosphorus extraction rate tended to decrease. The decrease might be interpreted as follows. Partial organic phosphorus was covered by the excessive scale, and the extractant could not fully contact with the internal phosphorus in the fouling, thus resulting in incomplete extraction. The optimum mass of the extracted scale was determined as 0.4 g.

3. EXPERIMENTAL MATERIALS AND METHODS

3.1. Reagents and Equipment. HEDP is an industrial product with a mass fraction of 50% and purchased from Shandong Taihe Water Treatment Co., Ltd. NaHCO3, KHCO3, HCl, and NaOH are of analytical grade and purchased from Tianjin Kemiou Reagent Co., Ltd.

The instruments used in the experiments include an ultraviolet spectrophotometer (UV-6000PC, Shanghai Yuanxi Instrument Co., Ltd.), an oscillator (KS, Changzhou Putian Instrument Manufacturing Co., Ltd.), a centrifuge (AXTG16G, Yancheng Anxin Experimental Instrument Co., Ltd.), an Energy-Dispersive Spectrometer (GENESIS, EDAX), and a X-ray diffractometer (D8 ADVANCE, Bruker, Germany).

3.2. Preparation of Fouling Samples. 1-Water bath; 2-beaker; 3-piping system; 4-circulating pump; 5-flow meter; 6-brass tube; and 7-corrosion coupon.

A simulated circulating cooling system shown in Figure 10 was established, and simulated reclaimed water was prepared according to Table 2. The flow rate was 0.6 m/s. After simulated reclaimed water evaporated until the system failed to operate, the beaker was taken out to clean the outer wall, dried, weighed, and scaled to obtain the fouling sample.

3.3. Extraction Method Optimization. 3.3.1. Active Organic Phosphorus. Scale samples were weighed in 25 mL centrifuge tubes. Then, 20 mL of extractant (different concentrations) was added, shaken for a certain time, and

![Figure 6](image_url)

Figure 6. Effects of extraction concentration on the extraction rate of moderately active organic phosphorus.

![Figure 7](image_url)

Figure 7. Effects of extraction time on the extraction rate of moderately active organic phosphorus: (a) HCl extractant; (b) NaOH extractant.
centrifuged at 8000 rpm for 10 min. Then, 20 mL of deionized water was added for 1 h extraction under the same conditions, and the extracts were combined together. The total phosphorus content and inorganic phosphorus content in the extracts were determined by ammonium molybdate spectrophotometry. The difference between the total phosphorus content and inorganic phosphorus content was the content of active organic phosphorus. Three measurements were made and averaged. The extraction rate of active organic phosphorus $E_1$ was determined according to eq 1.

3.3.2. Moderately Active Organic Phosphorus. First, 20 mL of HCl or NaOH solution (different concentrations) was added into the above residue, shaken for a certain time, and centrifuged at 5000 rpm for 10 min. Then, 20 mL of deionized water was added for 1 h extraction under the same conditions, and the extracts were combined together. The total phosphorus content and inorganic phosphorus content of the extracts were determined, and the difference between the total phosphorus content and inorganic phosphorus content was the content of the moderately active organic phosphorus. Three measurements were made and averaged. The extraction rate of moderately active organic phosphorus $E_2$ was determined according to eq 1.

3.3.3. Inactive Organic Phosphorus/Residual Phosphorus. The inactive organic phosphorus was not extracted by the alkali method, indicating that the content of inactive organic phosphorus was zero. The residues obtained after alkali extraction and burning were extracted with 1.0 mol/L $\text{H}_2\text{SO}_4$, and then the total phosphorus in the residue could not be detected, indicating that the residual phosphorus content was close to zero. Therefore, EDS and XRD were used to characterize the phosphorus form in the residues.

**Table 2. Composition of Simulated Reclaimed Water**

| components of reclaimed water | molar concentrations (mmol/L) | mass concentrations (g/L) |
|------------------------------|-------------------------------|---------------------------|
| $\text{NaHCO}_3$            | 21.90                         | 1.84                      |
| $\text{CaCl}_2$             | 7.57                          | 0.84                      |
| $\text{Na}_2\text{SO}_4$    | 2.82                          | 0.40                      |
| $\text{MgCl}_2$             | 7.18                          | 0.68                      |
| $\text{HEDP}$               | 0.01                          | 0.01                      |

Figure 8. Residue characterization results: (a) EDS; (b) XRD.

Figure 9. Effects of the mass of the extracted scale on total organic phosphorus recovery.

Figure 10. Simulated circulating cooling system.
extraction procedure, namely active organic phosphorus mass, mg; phosphorus mass in the supernatant, mg; extraction efficiency of the circulating cooling system.

The optimized extraction conditions were determined as follows: 20 mL of 1.0 mol/L NaOH and 15 h extraction, and the total organic phosphorus extraction rate determined as follows: 20 mL of 1.0 mol/L HCl and 3 h extraction, and the total organic phosphorus dosage, mg.

In this study, the organic phosphorus extraction process and conditions in the fouling of the circulating cooling system were optimized according to the Ivanoff method. The optimal extraction conditions of active organic phosphorus were determined as follows: 0.4 g of extracted scale, 20 mL of 0.5 mol/L KHCO₃ solution, and 10 h extraction. In the extraction of moderately active organic phosphorus with the acid method, the optimal extraction conditions were determined as follows: 20 mL of 1.0 mol/L HCl and 3 h extraction, and the total organic phosphorus extraction reached was 94.32%. In the extraction of moderately active organic phosphorus, the acid method realized the extraction, and the total organic phosphorus extraction rate determined as follows: 0.4 g of extracted scale, 20 mL of 0.5 mol/L NaOH and 15 h extraction, and the total organic phosphorus extraction rate reached was 94.32%. In the extraction of moderately active organic phosphorus, the acid method realized the extraction, and the total organic phosphorus extraction rate determined as follows: 0.4 g of extracted scale, 20 mL of 0.5 mol/L NaOH and 15 h extraction, and the total organic phosphorus extraction rate reached was 94.32%.

The total organic phosphorus extraction rate E was determined according to eq 2

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E = E_1 + E_2
\]

4. CONCLUSIONS

In this study, the organic phosphorus extraction process and conditions in the fouling of the circulating cooling system were optimized according to the Ivanoff method. The optimal extraction conditions of active organic phosphorus were determined as follows: 0.4 g of extracted scale, 20 mL of 0.5 mol/L KHCO₃ solution, and 10 h extraction. In the extraction of moderately active organic phosphorus with the acid method, the optimal extraction conditions were determined as follows: 20 mL of 1.0 mol/L HCl and 3 h extraction, and the total organic phosphorus extraction rate reached was 94.32%. In the extraction of moderately active organic phosphorus, the acid method realized the efficient and fast extraction, so the acid method was the optimal extraction method for the moderately active organic phosphorus. The optimal method is characterized by the simple operation and the good extraction effect and can be used as a quantitative and qualitative analysis method for organic phosphorus in the scale of the circulating cooling system.

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Notes

The authors declare no competing financial interest.

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E_1 \text{ or } E_2 = \frac{(m_1 - m_2) \times M}{S \times M} \times 100\% \quad (1)
\]

where \(m_1\) and \(m_2\) are total phosphorus mass and inorganic phosphorus mass in the supernatant, mg; \(M_1\) is the total scaling mass, mg; \(S\) is the mass of the extracted scale, mg; and \(M\) is the organic phosphorus dosage, mg.

The total organic phosphorus extraction rate \(E\) was determined according to eq 2

\[
E = E_1 + E_2
\]

E1 or E2 = (m1 − m2) × M S × M × 100% (1)

where m1 and m2 are total phosphorus mass and inorganic phosphorus mass in the supernatant, mg; M1 is the total scaling mass, mg; S is the mass of the extracted scale, mg; and M is the organic phosphorus dosage, mg.