

Research Article

Preparation and Aromatic Hydrocarbon Removal Performance of Potassium Ferrate

Wei Guan, Zhigang Xie, and Jia Zhang

Chongqing Key Laboratory of Environmental Materials & Remediation Technologies, Chongqing University of Arts and Sciences, Chongqing 402160, China

Correspondence should be addressed to Zhigang Xie; xinghu2200@163.com

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This experiment adopts the hypochlorite oxidation method to constantly synthesize potassium ferrate. Under the condition of micropolluted source water pH and on the basis of naphthalene, phenanthrene, and pyrene as research objects, the effects of different systems to remove aromatic hydrocarbons were studied. Various oxidation systems to remove phenanthrene intermediate are analyzed and the detailed mechanisms for removal of phenanthrene are discussed. The study found that the main intermediate of potassium ferrate system to transform phenanthrene is 9,10-phenanthraquinone and its area percentage reached 82.66%; that is, 9,10-phenanthraquinone is the key entity to remove phenanthrene.

1. Introduction

Ferrate is an ideal water treatment reagent. In acidic medium, ferrate redox potential is very high; in a wide pH range, its oxidation is strong, and it has strong deactivating effect on microorganisms. Ferrate in sewage purification shows excellent oxidation, adsorption, and flocculation to remove contaminants, and it has stronger sterilization effect than chlorine department oxidant [1–3]. Ferrate is the best oxidant replacement for chlorine source as water purification agent. In addition, ferrate as the positive active material of the green power supply has high electrode potential and capacitance [4–6]. This unique feature makes ferrates environmentally friendly green oxidation reagents with dual function.

The ferrate crystal belongs to orthogonal crystal system. For potassium ferrate (K_2FeO_4), ferrate has regular tetrahedron structure. Fe atom is at the center of the tetrahedron, with four oxygen atoms in the four corners of tetrahedron, presenting a slightly distorted tetrahedron structure. Ferrate is a deep purple solid, its solution has specific purple color, and maximum absorption wavelength is 510 nm. At the same time, ferrate ion has an absorption peak at 786 nm, which is also its characteristic absorption peak. The main compound in ferrate is potassium ferrate and in solid state it is dark

purple powder, and melting point is 198°C. When ferrate is in dry conditions, it starts to break down at 230°C [7–9].

Iron (VI) ions in potassium ferrate, located in the highest valence state of iron, have strong oxidativity; also they have selectivity. They can oxidize plenty of organics. Their oxidativity is significantly stronger than conventional water treatment oxidant, such as potassium permanganate, ozone, and hypochlorite. Under acid medium their standard electrode potential is 2.20 V; however, it is 0.72 V in alkaline condition. We can modify the ferrate cations' structure and adjust pH value to control the oxidation activity, so as to achieve high selectivity [10–14].

Heavy metal pollutants in the micropolluted water, such as cadmium and manganese, can be removed by potassium ferrate, and the removal rate reached 80%. In addition, potassium ferrate has good ability to remove organic compounds, such as phenols, alcohols, phenols, organic acid, organic nitrogen, and amino acid, lipid compounds containing sulfur and benzene, and some refractory compounds.

At present, much domestic and foreign source water is polluted by aromatic hydrocarbon. The concentration of aromatic hydrocarbon in water is very low, but the removal efficiency of conventional water treatment technology is not ideal. Due to the special stability of the benzene ring

TABLE 1: Main reagents and their specifications.

| Number | Name | Chemical level | Manufacturer |
|--------|----------------------|----------------|--------------|
| 1 | Caustic potash | AR | ACROS |
| 2 | Calcium hypochlorite | AR | ACROS |
| 3 | Potassium nitrate | AR | ACROS |
| 4 | Sodium sulfite | AR | ACROS |
| 5 | Cyanoacrylate | AR | ACROS |
| 6 | Butanol | AR | ACROS |
| 7 | Glacial acetic acid | AR | ACROS |
| 8 | Anhydrous ethanol | AR | ACROS |
| 9 | Cupric nitrate | AR | ACROS |
| 10 | Naphthalene | AR | ACROS |
| 11 | Phenanthrene | AR | ACROS |
| 12 | Pyrene | AR | ACROS |

structure, aromatic hydrocarbon in oxidation might generate series of intermediates. There are some potential environmental risks; it is difficult to guarantee the safety of drinking water. This paper completes the transformation and removal of aromatic hydrocarbons in micropolluted water, in view of the refractory aromatic hydrocarbon in micropolluted source water and in view of potassium ferrate treatment technology.

2. Experiment Methods

2.1. Experiment Materials and Reagents. The main reagents and specifications used in the experiment are shown in Table 1.

2.2. Instruments and Equipment. The main instruments and related information used in the experiment are shown in Table 2. The infrared spectrum of potassium ferrate by Fourier infrared spectrometer (FTIR) is drawn, and the infrared characteristic peaks of potassium ferrate crystal are measured. Fluorescence spectrophotometer determines the degradation rate of the sample. Gas chromatography-mass spectrometry instrument determines the intermediate of sample transformation and removal.

Some small instruments used in experiments consist of the beaker, 1000 W long arc xenon lamp, magnetic stirrer, electronic balance, separatory funnel, vacuum pump, the suction filter device, and so forth.

2.3. The Preparation of Potassium Ferrate. Proper 3 mol/L and 13 mol/L saturated potassium hydroxide solutions, cooling to room temperature, are prepared, respectively, for subsequent experiments. 15 g calcium hypochlorite is weighed in a small beaker; 25 mL of 13 mol/L potassium hydroxide solution is added in slight agitation with a glass rod. The resulting solution is sucked with 800-mesh filter cloth to leach impurities such as potassium chloride, collecting the yellow-green filtrate. 20 mL cooling saturated potassium hydroxide solution is added with batch into the yellow-green filtrate. Impurities are removed by careful control of temperature, sufficient mixing, and air pump filtration, and then alkaline

saturated potassium hypochlorite solution is obtained. 8 g $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ is weighed and is ground to powder and then is added slowly into the alkaline saturated potassium hypochlorite solution in batch with vigorous agitation. This reaction is exothermic with the ice-bath to control reaction temperature and stirring. The oxidation reaction is fast; soon the solution turns into purple. After 1-hour sufficient reaction, 90 mL cooling saturated potassium hydroxide solution was added into purple-black solution. The mixture is continuously stirred for 5 min and then is allowed to stand to cool in ice-bath for 30–40 min, followed by quickly sucking filtration with air pump. The liquor is abandoned and the resulting filtered cake, a crude potassium ferrate product, survives. The crude potassium ferrate product is washed 3 times with 3 mol/L potassium hydroxide solution, 5 mL for each washing. The resulting potassium ferrate is redissolved, and filtration is done with air pump sucking. Filter cake is withdrawn, whereas the liquor is retained. The liquor is poured into distillation flask in ice-water bath, with addition of 40 mL cooling saturated potassium hydroxide solution. Recrystallization for 20–30 minutes is done, followed by sucking filtration with air pump. The resulting filter cake is the potassium ferrate, leached with n-hexane (4×25 mL), with pentane (4×25 mL), with methanol (4×10 mL), and with ether (2×10 mL), respectively. The final product potassium ferrate crystal (purple-black), dried at 65°C for 2 hours, weighed, and kept in the drier, is prepared for subsequent use.

2.4. The Experiment of Potassium Ferrate to Remove Aromatic Hydrocarbon. Self-made potassium ferrate transforms aromatic hydrocarbon. In the micropolluted water medium (pH = 7.1) with naphthalene, phenanthrene, and pyrene, the ability of potassium ferrate to transform aromatic hydrocarbons is explored on the basis of reaction time, potassium ferrate amounts, and removal of aromatic hydrocarbon.

On account of water quality of Chongqing Water Plant, polluted water by aromatic hydrocarbon is simulated by artificial addition of 200 $\mu\text{g/L}$ aromatic hydrocarbon. The pH of the water is 7.1. 1000 mL of the polluted water is taken into a beaker with addition of different quantity of potassium ferrate, carrying out oxidation reaction with magnetic stirrer. At a specified time point 20 mL of the said solution is taken out of reaction system, followed by addition of sodium sulfite as termination agent. The content of aromatics remaining in the solution is determined by fluorescence spectrophotometer.

2.5. The Main Testing Index and Method. The removal of aromatic hydrocarbon is quantitatively determined by fluorescence spectrophotometer. The exciting wavelength and emission wavelength of three materials are as follows: naphthalene EX/EM is 218/332 nm, phenanthrene EX/EM is 250/362 nm, and pyrene EX/EM is 238/372 nm, respectively. Excitation and emission slit are 5 nm; scanning speed is 2400 nm/min.

The properties of phenanthrene oxidation intermediate are determined by gas chromatography-mass spectrometry. After 30 min reaction to attain constant state, 200 mL solution is taken out from phenanthrene reaction system, followed

TABLE 2: Main equipment and the respective specifications.

| Number | Instrument name | Instrument model | Manufacturer |
|--------|---|------------------|-----------------|
| 1 | X-ray diffractometer | XD-2 | SHIMADZU, Japan |
| 2 | UV spectrometer | UV-3010 | SHIMADZU, Japan |
| 3 | Fourier transform infrared spectrometer | IRPrestige-21 | SHIMADZU, Japan |
| 4 | Fluorescence spectrophotometer | F-7000 | SHIMADZU, Japan |
| 5 | Gas chromatograph-mass spectrometer (GC-MS) | 6890N GC/5973 MS | SHIMADZU, Japan |

by introduction into 400 mL separatory funnel. 6 g NaCl is added into the filtered solution. When completely dissolved, 10 mL CH_2Cl_2 is poured. The said solution is shaken for 10 min, and then it was allowed to stand for 5 min. CH_2Cl_2 layer is transferred to the pear-shaped flask. Additional 10 mL CH_2Cl_2 is put into the separative funnel twice, 5 mL for each. The solution was allowed to stand until organic layer survives. Then, the organic layer is merged and transferred. Anhydrous sodium sulfate is added into CH_2Cl_2 layer for drying. CH_2Cl_2 layer is concentrated at low temperature in the rotary evaporator; highly pure nitrogen is bubbled into the said layer until only 1 mL of the said solution is left. GC-MS analysis is applied.

Gas chromatography-mass spectrometry, Agilent 6890 Net GC-Agilent 5973 Net MS series, is used; chromatographic column for HP-5 MS quartz capillary column (25 m \times 0.25 mm \times 0.25 μm). The pattern of input sample is branch-stream-free system, carrier gas is helium gas, and the flow rate is 1.0 mL/min. Temperature raising procedure is 40~290°C (10°C/min). Ionization mass spectrometry conditions are as follows: voltage is 70 eV and the scanning range is of 30~400 m/z .

Fourier transformed infrared spectrometer and ultraviolet-visible spectrometer are used to qualitatively characterize potassium ferrate.

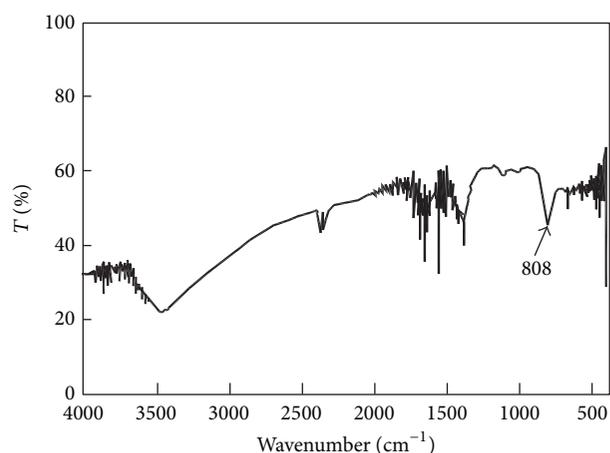
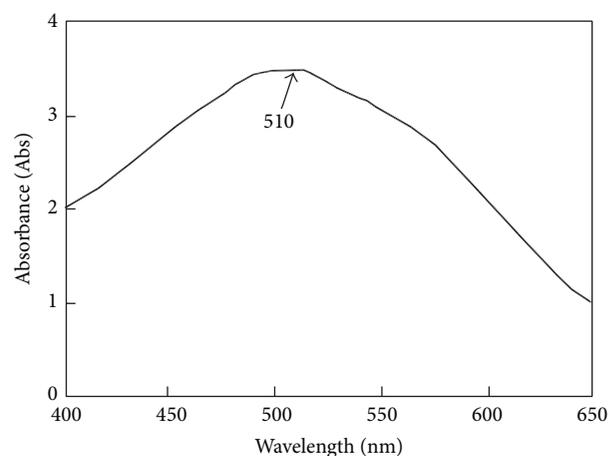
3. The Results and Discussion

3.1. The Characterization of Potassium Ferrate Crystal. Fourier infrared spectrometer determined the purple crystal infrared spectrum at experimental conditions, as shown in Figure 1.

As can be seen in the figure, there is an absorption peak at 808 cm^{-1} , which is potassium ferrate crystal Fe–O bond stretching vibration characteristic peak. The peak is considered as the infrared characteristic peak of potassium ferrate. Other impurity peaks in the infrared spectrogram should be water vapor occurring in the instrument and the characteristics of absorption peak of the organic solvent used in the purification elution [15, 16].

Potassium Ferrate Ultraviolet-Visible Absorption Spectrum Characteristics. 0.0934 g purple crystal is weighed to prepare 100 mL of potassium ferrate solution in volumetric flask. Ultraviolet-visible spectrometer is used to test the ultraviolet-visible absorption spectrum, as shown in Figure 2.

As can be seen in the figure, there is an obvious characteristic absorption peak at 510 nm, which is ultraviolet-visible

FIGURE 1: FTIR spectra of K_2FeO_4 .FIGURE 2: UV-Vis DRS of K_2FeO_4 .

characteristic absorption peak of potassium ferrate. The peak is also considered as the quantitative analysis wavelength.

3.2. The Influence of Reaction Time on Aromatic Hydrocarbon Removal. When the oxidant concentration of potassium ferrate is at 6 mg/L, the removal percentage of three kinds of aromatic hydrocarbon along with the change of time is shown in Figure 3. As can be seen in the figure, the transformation oxidation process of potassium ferrate for three kinds of aromatic hydrocarbon mainly occurred in the initial 5–10 minutes when the removal percentage of

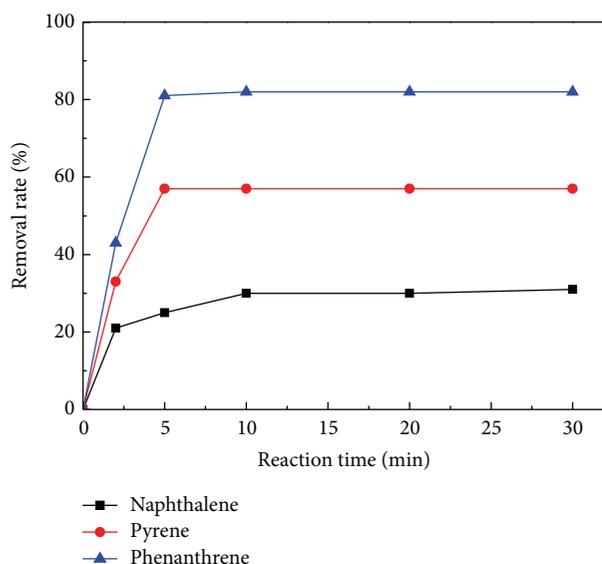


FIGURE 3: Influence of reaction time on aromatic hydrocarbons conversion and removal.

aromatic hydrocarbon increased rapidly as time increased. In five minutes later, the removal percentage of aromatic hydrocarbon only increased slightly. The result is consistent with the degradation rule of potassium ferrate oxidation degrading BPA [17] and arsenic [18]. Furthermore, it can be seen that over 80% final removal percentage of phenanthrene, 37% that of naphthalene, and 58% that of pyrene are developed, respectively. This shows that potassium ferrate can effectively transform phenanthrene in a relatively short time. These results are consistent with potassium ferrate high activity and instability. By comparison of three kinds of aromatic hydrocarbon removal percentage it is found that potassium ferrate has high oxidation, but to different samples its oxidation ability is not the same. Relatively, phenanthrene is most likely to be transformed, and naphthalene is more stable. The experiment results consisted of the three substances' delocalization. According to the experimental results, the oxidation reaction time of potassium ferrate to the three substances is preliminarily identified to be 20 min.

3.3. The Influence of the Amount on Aromatic Hydrocarbon Removal Effect. The influence of potassium ferrate amount on aromatic hydrocarbon removal ability is shown in Figure 4, derived from measurement of samples in 20 min. As can be seen in Figure 4, as potassium ferrate amount increases, three kinds of aromatic hydrocarbon removal percentage gradually increased. Relatively, at the same reagent amount, phenanthrene has the highest removal percentage. When the potassium ferrate concentration is 10 mg/L, phenanthrene has the highest removal percentage, reaching 98%; pyrene and naphthalene were 84% and 61%, respectively. Further improvement of amount of oxidant potassium ferrate cannot obviously improve the removal percentage for the three substances. Removal of the aromatic hydrocarbons in micropolluted source water, therefore, has the most appropriate 10 mg/L potassium ferrate amount.

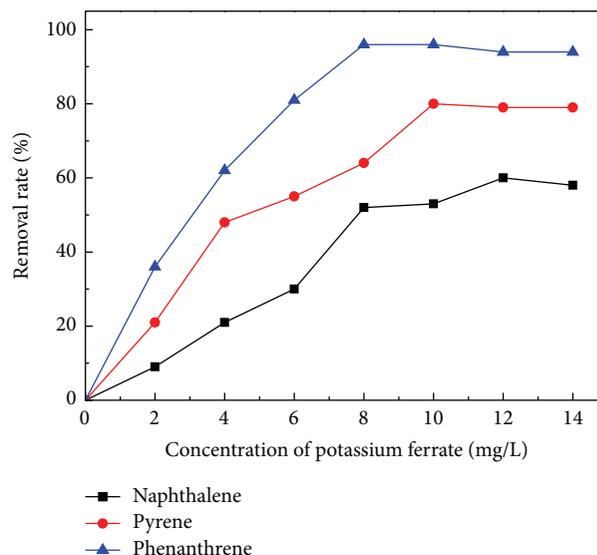


FIGURE 4: Influence of K_2FeO_4 concentration on aromatic hydrocarbons conversion and removal.

Potassium ferrate has strong oxidation, regardless of being in acidic medium or alkaline medium. The electrode potential is significantly higher than potassium permanganate and potassium dichromate; it can oxidize most organic substances. In hydrolysis process potassium ferrate produces strong oxidation hydroxyl free radicals [19]; at the same time, the unstable intermediate pentavalent iron and tetravalent iron also have a strong oxidizing ability [20]. The final reduction product is ferric iron. Potassium ferrate removal of aromatic hydrocarbon is the outcome of concerted action of these strong oxidizing substances. Low concentration potassium ferrate can transform removal of aromatic hydrocarbon. When the reaction time is 20 min, potassium ferrate concentrations are 12, 8, and 10 mg/L; naphthalene, phenanthrene, and pyrene removal percentage reached the maximum value, that is, 67%, 98%, and 84%, respectively.

3.4. Analysis of the Intermediate of Removing Phenanthrene. Due to the special stability of benzene ring structure, intermediate products can be generated during the oxidation process of aromatic substances. Thus, the removal process of aromatic substances has a potential environmental risk. Therefore, we studied the concrete structure of intermediate by GC-MS analysis.

Following are the GC of potassium ferrate system and mass spectrogram of some intermediates to transform phenanthrene.

Figure 5 is the GC of potassium ferrate system to transform phenanthrene.

As can be seen in the figure, the retention time of phenanthrene and main potassium ferrate reaction products was 12.443, 13.375, 13.575, and 18.139 min, respectively. In terms of corresponding mass spectrum, the peak retention time at 12.453 min is the undegraded phenanthrene peak. Compared

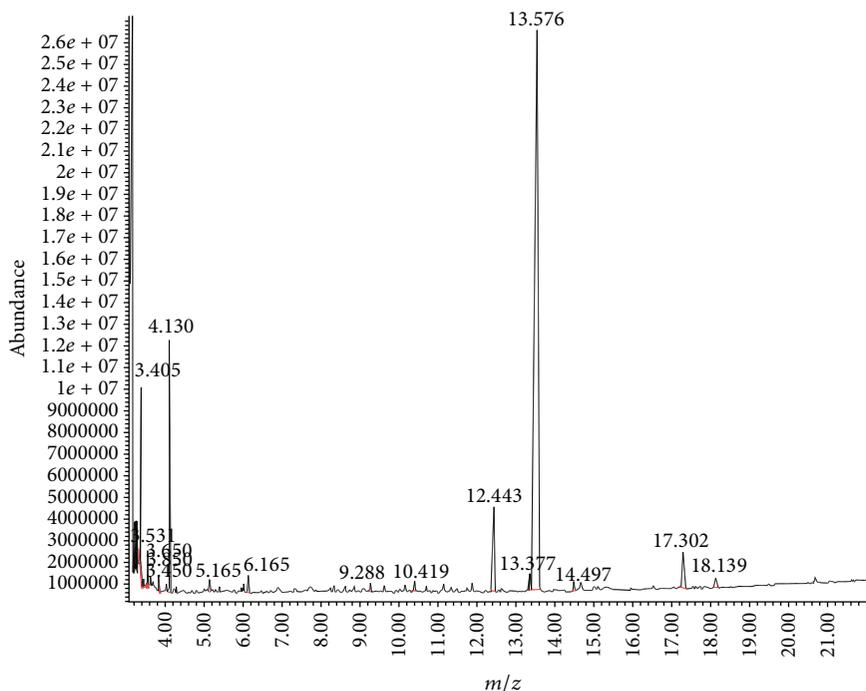


FIGURE 5: The GC map of phenanthrene conversion and removal by potassium ferrate system.

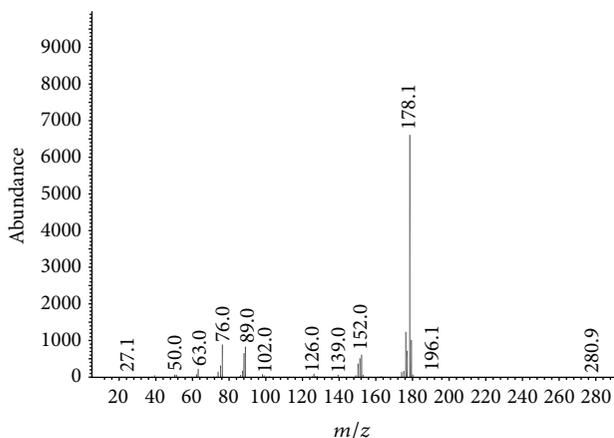


FIGURE 6: The mass spectrum of 12.443 min.

with mass spectrometry retrieve library standard spectrogram, other intermediates mainly include butyl phthalate, 9,10-phenanthraquinone, and diphenyl-2,2'-biformaldehyde. In addition, there are some significant intermediates, such as 9-fluorenone and dimethylbenzenal. And the peak of the main production prepared by phenanthrene reaction with potassium ferrate is at 13.575 min; the component area percentage accounted for 82.66%. Specific mass spectrum and structural formula of main products are as follows.

- (1) The mass spectrum peak diagram in GC of retention time at 12.443 min is as shown in Figure 6.

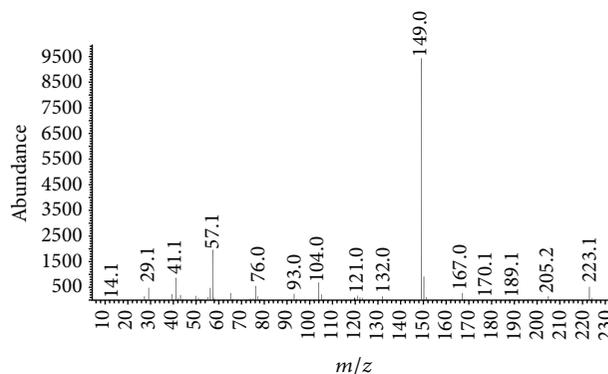
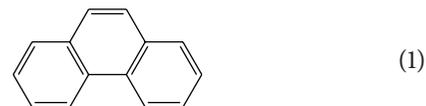


FIGURE 7: The mass spectrum of 13.375 min.

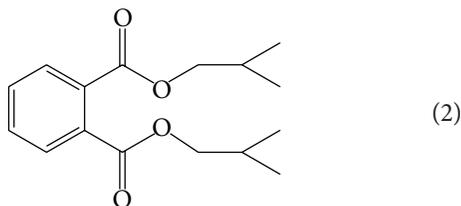
According to comparison with the spectral library spectrum standard substance, phenanthrene residue is identified in original solution, with 96% matching degree with the literatures, as the following structural formula:



- (2) The peak mass spectrum diagram in GC as of retention time at 13.375 min is as shown in Figure 7.

According to comparison with the spectral library spectrum standard substance, this material is identified as butyl

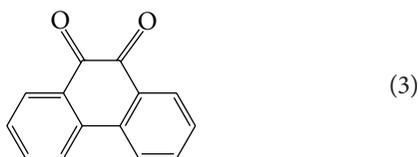
phthalate, with 86% matching degree, and its formula is as follows:



This material area percentage is only 1.32%, to show that this material accounts for a very small proportion in the product. This kind of material can be the intermediate produced by complex reaction of oxidation.

(3) The peak mass spectrum diagram in GC of retention time at 13.575 min is as shown in Figure 8.

According to comparison with the spectral library spectrum standard substance, this material is identified as 9,10-phenanthraquinone, with 92% matching degree, and its formula is as follows:



This material area percentage is 82.66%, to show that this material accounts for a very high proportion in the product. It is the main product of potassium ferrate in transformation of phenanthrene. Phenanthrene is made up of three benzene rings, where 9,10-position has strong chemical reactivity, prone to oxidation. In potassium ferrate oxidation system, phenanthrene gives Fe (VI) electron to be oxidized. In addition, phenanthrene can get 98% transformation in ferrate oxidation system, but it cannot completely inorganic, but is turned into 9,10-phenanthrenequinone. According to the research results by Lawton et al. [21], quinone structure is a kind of functional group more biophile than benzene ring. Its relatively weak biological resistance is advantageous over the biochemical conversion process. It changes aromatic hydrocarbon environmental durability characteristics and, to some extent, reduces the environmental risk.

(4) The peak mass spectrum diagram in GC of retention time at 18.139 min is as shown in Figure 9.

According to comparison with the spectral library spectrum standard substance, this material is identified as diphenyl-2,2'-biformaldehyde, with 70% matching degree, and its formula is as follows:

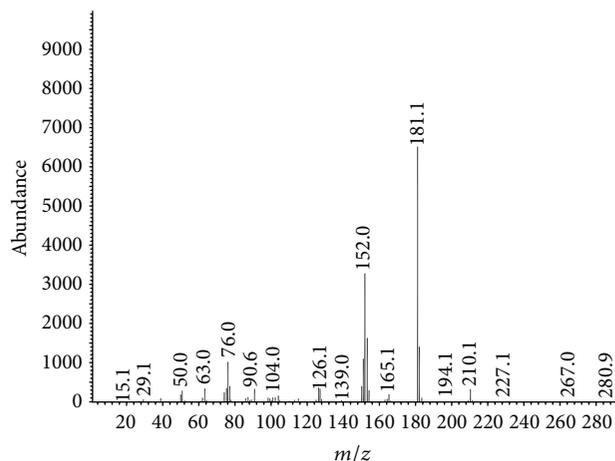
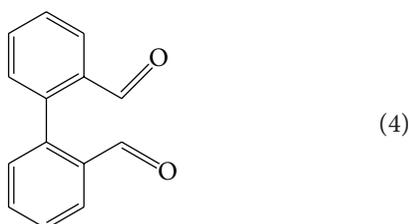


FIGURE 8: The mass spectrum of 13.575 min.

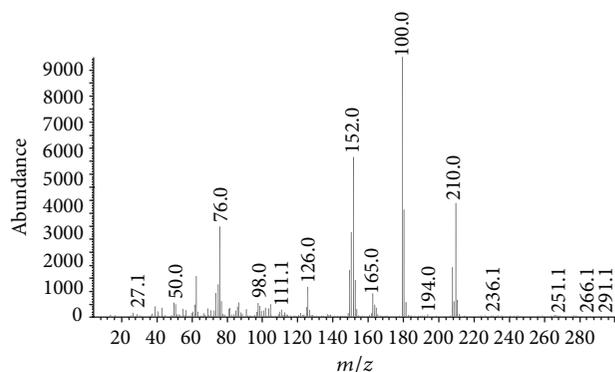


FIGURE 9: The mass spectrum of 18.139 min.

This material area percentage is only 1.05%, showing that this material accounts for a very small proportion in the product. In the process of potassium ferrate oxidation of phenanthrene, Fe (VI) to be reduced generates Fe (V), Fe (IV), and Fe (III) [22, 23] through single electron transfer steps. Phenanthrene 9,10-position has strong chemical reactivity, prone to oxidation. In potassium ferrate oxidation system, phenanthrene gives Fe (VI) electron to be oxidized. Since this is an oxidation system, diphenyl-2,2'-biformaldehyde is easy to be oxidized by potassium ferrate, so its proportion in products accounted for quite a few.

3.5. Primary Analysis of Oxidation System Mechanism to Transform Phenanthrene. In the process of potassium ferrate to oxidize phenanthrene, Fe (VI) to be reduced generates Fe (V), Fe (IV), and Fe (III) [24, 25] through single electron transfer steps. Phenanthrene is made up of three benzene rings, of which 9,10-position has strong chemical reactivity prone to oxidation. In potassium ferrate oxidation system, phenanthrene gives Fe (VI) electron to be oxidized. According to the single electron transfer mechanism, the possible reaction of potassium ferrate and phenanthrene is shown in Figure 10.

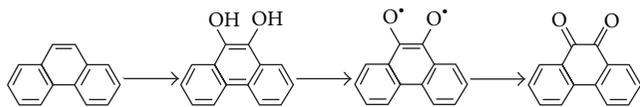


FIGURE 10: The possible way of reaction of potassium ferrate with phenanthrene.

4. Conclusions

This experiment adopts the hypochlorite oxidation approach to constantly synthesize potassium ferrate. The transformation oxidation process of potassium ferrate for aromatic hydrocarbons mainly occurred in the initial 5–10 minutes. Compared with aromatic hydrocarbon transformation removal percentage, it was found that potassium ferrate has high oxidation, but to different samples its transformation oxidation ability is not the same. Relatively, phenanthrene is most likely to be transformed, and naphthalene is more stable. In potassium ferrate, to remove phenanthrene, in GC and in the intermediate mass spectrum analysis, phenanthrene gives electron to Fe (VI) of potassium ferrate oxidation system, so that phenanthrene oxidation occurs. Thus possible reaction path is proposed: the quinone structure is a kind of functional group more biophile than benzene ring. Because of its biologically weak resistance, it is advantageous to the biochemical conversion process, to conversion of aromatic hydrocarbon's environmental durability characteristics, and, to some extent, to reduction of the environmental risk.

Conflict of Interests

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

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