Effect of Different Acid-Modified Coking Coals on Quinoline Adsorption

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ABSTRACT: The adsorption of quinoline from wastewater by coking coal (AC-1), HCl-modified coking coal (AC-2), HNO₃-modified coking coal (AC-3), HF-modified coking coal (AC-4), and H₂SO₄-modified coking coal (AC-5) was investigated in this paper. The effects of acid-modified concentration, modification time, and adsorption time versus quinoline removal rate were studied by batch experiments. The quinoline concentration was measured by UV spectrophotometry, the average pore size, and specific surface area of coking coal before and after modification were characterized through static nitrogen adsorption, the mineral composition of coking coal was tested by X-ray diffraction, and the surface functional groups were tested by Fourier transform infrared spectroscopy. The surface topography was tested using a scanning electron microscope. The experimental results showed that the adsorption capacity of coking coals was the best when both the modification time was 120 min and the acid-modified concentration was 0.1 mol·L⁻¹ and the quinoline removal rate reaches the highest when the adsorption time was 120 min. The specific surface area of AC-2 increased from 2.898 to 3.637 m²·g⁻¹, and the removal rate of quinoline increased from 77.64 to 90.61%. Acids reacted with inorganic mineral impurities within coking coal such as hydrogen vanadium phosphate hydrate, which caused an increase in the specific surface area. A new peak appeared in the Fourier transform infrared spectroscopy pattern at the wavenumber 2300 cm⁻¹. The surface of coking coal modified by acids was rougher than that of AC-1. The adsorption capacity of coking coal was improved after modification, and modified coking coals have the highest potential as low-cost adsorbents for quinoline removal.

1. INTRODUCTION

Quinoline is one of the higher-molecular-weight (MW) organics with a strong stench in industrial wastewater, and it is harmful to both nature and human life because it is highly toxic.1-2 Exposure to quinoline vapors for a short period of time can cause irritation of the nose, eyes, and respiratory tract and can also cause dizziness and nausea.3-4 There are several methods used for quinoline disposal, which mainly include chemical,5 biological,6 and adsorption methods.7-9 However, some of these methods have high operational and maintenance costs.9 For example, the chemical method will produce secondary pollution and generate a large amount of sludge, and the removal efficiency of the biological method is low.10 Compared with chemical and biological methods, adsorption is a traditional method, which can remove quinoline effectively. The advantage of the adsorption method is that it is easy to operate, but its disadvantage is that adsorption is a process of physical change and pollutants are not decomposed and eliminated during the disposal process.11

Among the different adsorbents available, coking coal is one of the most effective adsorbents.12 However, the adsorption capacity of raw coking coal is low; it has been previewed by researchers that the adsorption capacity of raw coking coal can be enhanced by surface modification. In this paper, the raw coking coal was processed with HCl, HNO₃, H₂SO₄, and HF acids to modify the surface properties, and batch adsorption experiments were conducted to evaluate the feasibility of quinoline removal under varying conditions. The advantage of using coking coal for quinoline disposal is that after adsorption, coking coal can be returned to produce industrial coke, burn, or prepare a coal-water slurry, where the value of coking coal was not reduced.13,14 The experimental purpose of this paper is to investigate the effect of different acid-modified coking coals on the adsorption of quinoline, and the surface property changes of raw and modified coking coals were studied also.

2. RESULTS AND DISCUSSION

2.1. Effect of Modification Times on Specific Surface Area and Pore Size Distribution. The specific surface area and adsorption average pore size of coking coal were measured, and the acid-modified concentration is 0.1 mol·L⁻¹. The results are shown in Figures 1 and 2, the specific
surface area of AC-1 is 2.898 m$^2$·g$^{-1}$, and the average pore size of AC-1 is 12.212 nm.

With the increase in the modification time, the specific surface area of coking coal increases first, then decreases, and reaches the maximum at 120 min. The sequence of specific surface areas is AC-2 (3.637 m$^2$·g$^{-1}$) > AC-3 (3.569 m$^2$·g$^{-1}$) > AC-4 (3.432 m$^2$·g$^{-1}$) > AC-5 (3.297 m$^2$·g$^{-1}$) > AC-1 (2.898 m$^2$·g$^{-1}$). The average pore size of coking coal is decreased after HCl and H$_2$SO$_4$ modification. The pore volume was decreased due to the shrinking pore size; at the same time, there are also new pore volumes generated by a dissolution reaction, but the pore volume is generally reduced. The specific surface area was increased due to the new pore channel; at the same time, the specific surface area decreased caused by the diameter reduction, and the specific surface area is generally increased. In the modification process, the specific surface area is increased, and the average pore size of AC-1 is decreased.$^{10}$

### 2.2. Effects of Modification Concentration and Time on Adsorption

The changes of the quinoline removal rate under different modification conditions were carried out. The results are shown in Figures 3 and 4.

As seen from Figure 3, in the coking coal modified with different acid amounts, the quinoline removal rate increased first and then decreased with the increase in acid concentration, and the quinoline removal rate is the highest when the acid concentration is 0.1 mol·L$^{-1}$. The change of the quinoline removal rate versus modification time is consistent with Figure 3, and the quinoline removal rate is the highest when the modification time is 120 min. With the increase in acid concentration and modification time, the dissolution reaction was more intense. At the same time, the specific surface area of coking coal samples after four different acid modifications is lower than that of raw coking coal. The larger the specific surface area, the more surface sites are available. At the optimum condition, the removal rates of quinoline by AC-1, AC-2, AC-3, AC-4, and AC-5 are 70.44, 90.61, 87.68, 87.28, and 90.61%, respectively.$^{15,16}$

### 2.3. Adsorption Kinetics of Coking Coal

The adsorption kinetics of coking coal samples was investigated. The result is shown in Figure 5.

![Figure 1. Plot of coking coal specific surface area vs modification time.](image1)

![Figure 2. Plot of average pore size vs modification time.](image2)

![Figure 3. Plot of quinoline removal rates vs acid concentration of modification.](image3)

![Figure 4. Plot of quinoline removal rates vs modification time.](image4)

![Figure 5. Plot of quinoline removal rates and adsorption capacities vs adsorption time.](image5)
As can be seen from Figure 5, the original and modified samples showed very similar kinetic behavior for quinoline adsorption where removal and adsorption capacity curves were single, smooth, and continuously leading to saturation. At the initial stage of the reaction, quinoline removal rates increase obviously and exceed 90% when the adsorption time reaches 200 min. When the adsorption time exceeds 120 min, the increase in quinoline removal rates tends to become smooth, and the adsorption process reaches equilibrium at 870 min. Xu et al.\textsuperscript{17} found that the equilibrium time needed for organic constituents from the reverse osmosis concentrate in the coal chemical industry adsorption on coking coal was almost 540 min. At the adsorption time of 120 min, the quinoline removal rates are 77.64\% for AC-1, 90.61\% for AC-2, 87.68\% for AC-3, 87.28\% for AC-4, and 90.61\% for AC-5. The results show that the adsorption capacity of coking coal after modification is higher than that of raw coking coal and the removal rate of quinoline increased from 70.44 to 90.61\%. The main reason is that the specific surface areas increase after being modified, which proves to be an effective way to enhance the adsorption capacities of coking coal. The adsorption process of coking coal follows a quasi-second-order kinetic equation well, and this conclusion can be found in another paper by Xu et al.\textsuperscript{18}

2.4. Adsorption Isotherm of Coking Coal. The adsorption isotherm is used to investigate the movement state of adsorbates on the surface properties of adsorbents and the interaction between the adsorbate and adsorbent. When the adsorption process reaches equilibrium, the adsorption capacity for gas or vapor is proportional to the mass of the adsorbent. The equilibrium adsorption capacities depend on the adsorption temperature $T$, the gas or vapor pressure $P$, and the properties of the adsorbent and adsorbate gas.

The adsorption isotherm of coking coal was tested at an acid concentration of 0.1 mol·L$^{-1}$, modification time of 120 min, and temperature of 77 K. The results are listed in Figure 6.

As shown in Figure 6, the original and modified coking coals are close to the adsorption isotherm type II. The front section of the adsorption curve rises slowly with a slightly downward concave; the rear section rises sharply and continues to rise until the relative pressure is close to 1.0. The result indicates that the capillary agglomeration occurred during the adsorption of nitrogen. In the low-pressure section ($P/P_0 = 0.0–0.3$), the first half of the curve rises slowly and has a downward dimple shape; this stage is the transition from the adsorption monolayer to the multimolecular layer. In the middle-pressure section ($P/P_0 = 0.3–0.8$), the adsorption capacities increase steadily with increasing pressure, and this stage is the multimolecular layer adsorption process. In the second half of the curve ($P/P_0 = 0.8–1.0$), the adsorption line rises sharply. Ge et al.\textsuperscript{19} found that some mesopores lead to a gradual increase in adsorption after the initial filling of the micropores followed by a more rapid increase near saturation. The saturated adsorption amount of $N_2$ increased after microwave modification, which implied an increase in the pore volume. It does not show adsorption saturation until it approaches the saturated vapor pressure. Because of capillary condensation, the large pores over volume filling, so the sample contains a certain amount of mesopore and macropore.\textsuperscript{20,21}

2.5. Effects of Different Modification Conditions on the Surface Morphology of Coking Coal. The changes in mineral composition, specific surface area, and pore structure can be more intuitively determined from the surface morphology of raw and modified coking coals. Scanning electron microscope images of coking coal samples under different modification conditions were obtained using a scanning electron microscope, which is shown in Figure 7. As can be seen from Figure 7, there is little difference in the surface morphology of the samples except for that the surface of coking coal gradually becomes rougher after modified with four different acids. As the specific surface areas of AC-X ($X = 2$–5) become bigger than AC-1, there may be some pore widening on the modified coking coal, which could have occurred due to the modification process. Moreover, Huang et al. reported that the surface of lignite raw coal is relatively flatter, and it gradually becomes rough after treatment with nitric acid; the higher the modification concentration of nitric acid, the more apparent the charge is, which fully demonstrated the corrosive effect of nitric acid on the lignite structure.\textsuperscript{22}

2.6. Infrared Spectroscopy Analysis of Raw and Modified Coking Coals. The spectral characteristics of AC-X ($X = 1$–5) are studied, the acid concentration is 0.1 mol·L$^{-1}$, and the modification time is 120 min. The results are shown in Figure 8. As seen from Figure 8, the intensity of the absorption peak for modified coking coal increases at a wavenumber of 3500 cm$^{-1}$ where $–$OH is oxidized during the modification process. For the stretching vibration and shear vibration of methyl and methylene groups, the absorption peak intensity increases at 2900 cm$^{-1}$, which are difficult to be oxidized during the modification process, so the change is not obvious. A new peak appears in the Fourier transform infrared spectroscopy pattern at the wavenumber 2300 cm$^{-1}$ and moves to a high-frequency direction. The molecule vibration can be considered belong to three-button or cumulative double-button stretching vibration at the wavenumber 2000–2500 cm$^{-1}$.\textsuperscript{23} The peak at 1600 cm$^{-1}$ belongs to the in-plane stretching vibration of aromatic hydrocarbons and carboxylic acid. During the whole modification process, the shape and strength of the peaks of unsaturated hydrocarbon bonds in aromatic hydrocarbons are basically unchanged, which indicates that aromatic hydrocarbons are difficult to be oxidized during the modification process. The peak at 1000 cm$^{-1}$ is attributed to minerals; the
peak intensity and peak shape are basically unchanged during the modification process.24 Through experimental analysis and data collation, the main absorption peaks of coking coal samples and their attributed vibrations are studied; the result is listed in Table 1.

2.7. Mineral Composition. The crystallization characteristics of raw and modified coking coals are studied by X-ray diffraction. The result is shown in Figure 9.

Coal is mainly composed of two major components: kaolinite and hydrogen vanadium phosphate hydrate (HVPH). As seen from Figure 9, the characteristic diffraction peaks of raw and modified coking coals are located at 12.36, 21.5, 24.9, 29.3, 35.6, and 38.5°. In the peak at 29.3° where the HVPH signal is weakened, the acid reacts with HVPH and the HVPH is dissolved, while the other mineral components stay unchanged. New pores are created during the dissolution process; this is the reason why specific surface area increased.

Variations of raw and modified coking coal interlamellar spacings are studied; the result is listed in Table 2.

As seen from Table 2, the diffraction peak has no change in the interlayer spacing of the kaolinite in the modified coking coal, but the interlayer spacing of the HVPH in the coking coal is increased. With the growth of the interlayer spacing, the pore volume of coal is increased, and the adsorption capacities of coking coals increased also.

### Table 1. Main Absorption Peaks and Attributed Vibrations of Coking Coal

| peak number | peak position $(\text{cm}^{-1})$ | functional group | wavenumber $(\mu m)$ | vibration |
|-------------|----------------------------------|------------------|----------------------|-----------|
| 1           | 3420                             | $-$OH            | 2170                 | stretching vibration of hydroxyl or amine groups |
| 2           | 2910                             | $-$CH$_3$        | 3143                 | methyl stretching vibration |
| 3           | 2838                             | $-$CH$_2$        | 3152                 | methylene stretching vibration |
| 4           | 1715                             | C=O              | 5183                 | stretching vibration of carboxylate |
| 5           | 1600                             | C=O              | 5192                 | stretching vibration of aromatic hydrocarbons |
| 6           | 1450                             | C=OH             | 6125                 | in-plane stretching vibration of aromatic hydrocarbons and carboxylic acid |
| 7           | 1200                             | $-$C=O           | 6204                 | C=O vibration of phenol, alcohol, ether, and ester mineral |
| 8           | 1020                             |                  | 10,185               | mineral |

The diffraction phase corresponding to the diffraction peak at other positions is kaolinite with little change; it has no effect on the adsorption performance of the coking coal.25
samples. The chemical agents were purchased from Sinopharm Chemical Reagent Co., Ltd., and the purity of agents was AR.

Four different acids (HCl, HNO₃, HF, and H₂SO₄) were used for coking coal modification, the concentrations of acids were 0.05, 0.1, 0.5, 1.0, 3.0, and 5.0 mol·L⁻¹, respectively. The amount of coking coal was 50 g, and the acids (HCl, HNO₃, HF, and H₂SO₄) were 250 mL. The modification process was performed with a magnetic stirrer (IKA RET basic, Jinzhuo, Guangzhou, China), the modification time was 120 min, the temperature was 298 K, and the speed of the magnetic stirrer was 300 rpm. The pH of modified coking coal was adjusted to 6.8–7.2 using deionized water, and the coal was then dried in a vacuum oven for ~720 min at 378 K. Coking coal samples were modified by different times were obtained at the same other conditions, except the modification times were 30, 120, 240, and 360 min, respectively.

The proximate analysis results of raw and modified coking coals are listed in Table 3.

### Table 3. Proximate Analysis of Raw and Modified Coking Coal (% by Mass)²

|     | ad | d | daf | FCd |
|-----|----|---|-----|-----|
| AC-1 | 1.28 | 6.70 | 28.61 | 63.41 |
| AC-2 | 0.83 | 2.97 | 31.67 | 64.53 |
| AC-3 | 1.09 | 3.33 | 33.12 | 62.46 |
| AC-4 | 1.11 | 3.37 | 32.23 | 63.29 |
| AC-5 | 1.05 | 3.13 | 32.11 | 63.71 |

²Note: ad; air-dried basis, d; dry basis, daf; dry-ash-free basis, M; moisture, A; ash, V; volatile matter, FC; fixed carbon.

### 3. CONCLUSIONS

The optimum acid-modified concentration is 0.1 mol·L⁻¹, and both the optimum modification time and adsorption time are 120 min. At these conditions, the specific surface areas of AC-1, AC-2, AC-3, AC-4, and AC-5 are 2.8984, 3.6366, 3.5687, 3.4315, and 3.2968 m²/g, and the quinoline removal rates by AC-1, AC-2, AC-3, AC-4, and AC-5 are 28.61, 31.67, 33.12, 32.23, and 32.11%, respectively. The surface functional groups of coking coal under different acid modification conditions were tested using a Fourier transform infrared spectroscopy analyzer (Nicolet iS10, Thermo Fisher Scientific, Waltham, MA, USA) in the range of 400–4000 cm⁻¹. The mineral composition of coking coal samples was tested by X-ray diffraction (XRD), and the scan speed is 6°/min in the range of 10°–90°. The surface morphology characteristics of coal samples under different modification conditions were characterized using a scanning electron microscope (JSM-7800F, JEOL, Tokyo, Japan) where the acceleration voltage was 15 KV and the resolution was 0.8 nm.

### 4. MATERIALS AND METHODS

#### 4.1. Raw Materials and Modified Coal Sample Preparation

The raw coking coal samples used in this paper were collected from Linhuan Coking & Chemical Co., Ltd. The raw coking coal was crushed and sieved, and then the particles with a 74 μm size were selected as the experimental samples.

#### 4.2. Quinoline Solution Preparation

An amount of 1.00 g of quinoline was weighed using an electronic analytical balance (BSA124S, Sartorius Scientific, Beijing, China), and then quinoline was added to a 1000 mL volumetric flask. Next, the solution was diluted to the volumetric flask scale line with deionized water. The standard solution was diluted to 50 mg·L⁻¹ after the volumetric flask was stirred for 6 h using a magnetic stirrer.

#### 4.3. Characterization Methods

The specific surface area of coking coals was analyzed using a nitrogen adsorption instrument (JW-BK122W, Jingwei Gaobo, Beijing, China), and the wave peak was the strongest when the wavelength was 299 nm. Quinoline solution absorbance values were measured at the wavelength 299 nm, and the concentrations of quinoline standard solutions were 5, 10, 20, 30, 40, and 60 mg·L⁻¹.

#### 4.4. Determination of Quinoline Concentration

Quinoline solution was scanned in full wavelength using a UV spectrophotometer (UV-1500, Meixi, Shanghai, China), and the wave peak was the strongest when the wavelength was 299 nm. Quinoline standard solution absorbance values were measured at the wavelength 299 nm, and the concentrations of quinoline standard solutions were 5, 10, 20, 30, 40, and 60 mg·L⁻¹.
L\(^{-1}\). Fitting the standard curve of quinoline solution, the result was shown as eq \(1\).

\[
y = 0.0259 \times x - 0.0046
\]  

(1)

where \(x\) is the absorbance value of the filtrate, and the quinoline solution concentration was calculated through the standard curve.

4.5. Batch Adsorption Experiment. 4.5.1. Adsorption Experiment by Coking Coal Modified in Different Conditions. The adsorption experiments were carried out at room temperature (298 K) in a 100 mL conical flask by mixing 2.5 g of coking coal with 50 mL of 50 mgL\(^{-1}\) quinoline solution and shaking the conical flask using a digital display water bath temperature oscillator with a speed of 120 rpm for 120 min. The test samples were filtered after shaking was completed, and the absorbance value of the filtrate was measured using a UV spectrophotometer.

4.5.2. Adsorption Kinetics Experiment. Compared with the adsorption experiment by coking coal modified in different conditions, the adsorption kinetics experiment was conducted at the same conditions, except the adsorption times were 5, 15, 45, 120, 270, 450, and 870 min. The adsorption kinetics experiment of raw coking coal was performed at the same conditions.

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The authors declare no competing financial interest.

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