Preparation of side chain liquid crystal polyether chelating resin and its adsorption properties for Cu^{2+} in water

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Abstract

With epoxycyclohexane as critical modifying monomer, the copolyether was obtained by cationic ring-opening polymerization of epichlorohydrin and epoxycyclohexane. Then biphenyl was used as the mesogen, and diglycolamidic acid was used as the terminal chelating group, a new type of side chain liquid crystal polyether chelating resin was prepared at last. The adsorption influence factors, reuse performance, adsorption model and adsorption kinetics of the chelated resin on Cu^{2+} in water were studied systematically. The results showed that the resin has good adsorption and reuse performance for the treatment of Cu^{2+} in water, and the adsorption of Cu^{2+} is Langmuir monolayer adsorption, and the adsorption process conforms to a quasi-second-order kinetic model. The adsorption performances of the chelated resin has great potential for recovery of copper resource from non-ferrous smelting industry.

Introduction

With the rapid development of non-ferrous smelting industry, the discharge of wastewater containing metal ions has become a more sensitive environmental topic. The smelting wastewater may contain some harmful metal ions such as lead, mercury, arsenic, and may also contain copper, zinc, silver, gold and even rare metal elements. On the one hand, the direct emissions will cause serious pollution to the ecological environment. On the other hand, it will also cause a huge waste of resources, especially rare resources [1]. Therefore, the effective treatment of wastewater has become an indispensable treatment, which has also been extensively studied by many scientific and technological workers. Currently, the main technologies are mainly focused on reduction precipitation method, electrodialysis method, solvent extraction method, membrane separation method, coagulation method and adsorption method, etc. [2-6]. Among these methods, the chelating resin adsorption method has attracted particularly attention not only the unique characteristics of large adsorption capacity, fast adsorption rate and good adsorption selectivity, but also the advantages of simple operation and reusability[7]. In recent years, it has become one of the research hotspots for the treatment of wastewater.

Side chain liquid crystal polyether chelating resin is a kind of chelating resin with mesogen groups laterally attached to polyether main chain. In particularly, the chelating properties of the resin could be finely tuned by the side chain structure and chelating groups at the end of the primitive, so that the molecular structure of the resin is portably designable and the adsorption performance can also be regulated. So it is expected to play an important role in many fields such as wastewater treatment, rare metal recovery, reaction catalysis, and preparation of new liquid crystal materials[8-11]. The polyether chelating resins developed at present mostly are belong to open-chain epoxy compounds such as ethylene oxide and epoxy (chloro) propane as monomers. Due to the strong random motion ability of the backbone, the ordered arrangement of the mesocrystalline groups in the side chain will be hindered, so that these ordered side groups tend to succumb to the random motion of the backbone, which is unfavorable to improve the absorption selectivity because of losing the external induction of mesogen groups. Moreover, due to the small molecular weight and polarity of the resin molecules, the lateral interaction force between the molecules is weak, resulting in poor mechanical properties of the resin[12].

The polyether polymers have attracted great attention owing their high flexibility[13]. However, the service life of polyethers resin is often short, which seriously restricts the further application and promotion. Expect for ethylene oxide and propane epoxide, epoxy-cyclohexane is another important epoxy compound [14]. Consideration its unique six-member ring structure, the rigidity and polarity of polyether chain could be finely tuned by structure modification, thus their liquid crystal properties, mechanical properties and even absorption performance of the polyether resin could be improved than its predecessors. Based on these consideration, a new type of side chain liquid crystal polyether chelating resin was elaborately designed with epoxycyclohexane as the modifying monomer. The adsorption performances of the chelated resin on Cu^{2+} in water were systematically studied, aiming at recovery of copper resource from non-ferrous smelting industry. In addition, the adsorption mechanism and adsorption kinetics were also further discussed.

Experimental

Instruments and materials

DFY-5/25 low-temperature constant temperature reaction bath, RE-52C rotary evaporator and DZF-6020 vacuum drying box, Gongyi yuhua instrument co., LTD. FD⁻¹B-50 vacuum freeze-drying oven, Beijing boyikang experimental instrument co, LTD. DMF, methylene chloride, ethylene glycol, epichlorohydrin and phthalic anhydride were all analytical reagent and purchased from Comio chemical reagent co. LTD. Analytical pure p-hydroxybiphenyl and boron trifluoridether were purchased from Alighting reagent (Shanghai) co., LTD. All reagents were purified before use. Chemical pure epoxycyclohexane was purchased from Shenma Nylon Chemical Co. Ltd.

Preparation process of the chelating resin

40 mL dichloromethane, ethylene glycol (0.01mol, 0.621 g) and boron trifluoride ether (0.01 mol, 1.278 g) were added into a flask and stirred mechanically at 0 °C for 0.5 h. After mixing epoxycyclohexane (CHO, 0.2 mol, 19.629 g) with epichlorohydrin (ECH, 0.3 mol, 27.756 g), the mixture was slowly added to the reaction bottle for 1 h and kept their temperature at 0 °C for 5.5 h. When the reaction was finished, a large amount of water added into the mixed solution and then stirred vigorously for 5 min in order to separate the oil phase. The crude product was washed repeatedly for three times, then dissolved with dichloromethane. The water, catalyst remained in product and most of dichloromethane were removed slowly by atmospheric distillation, and then distillation was carried under reduced pressure. The ECH-CHO copolyether(Compound I), a colorless and transparent gum was obtained after the residual dichloromethane was removed completely.

The p-hydroxybiphenyls were salted with sodium hydroxide in advance, then 1.06 g of co-polyether and 2.02 g of sodium salt were mixed with 25 mL anhydrous DMF. The reflux reaction was performed at 110 °C for 20 h. After filtration, the filtrate cake was immersed, separated and refined to obtain copolymerized ether grafts(Compound II).

Compound II(2.7g,1mmol) , paraformaldehyde(2.4g,80mmol) and anhydrous ZnCl_2 (1.86g, 25mmol) were added into the reactor successively and dissolved in 20mL of cyclohexane under stirring. Then HCl gas was passed into the reactor at 55 °C for 12 h and cooled down to room temperature. After filtration, the filtrate cake was washed to neutral by cyclohexane and water to obtain a white powder(Compound III).

Weighing 1.25 g of compound III and 0.58 g of diethyl iminodiacetate to react at 80 °C for 20 h, to obtain the white product. After refluxing with saturated KOH alcohol solution at 90 °C for 3 h, the product was washed to be neutral. Acidizing with $2 \text{ mol} \cdot \text{L}^{-1}$ hydrochloric acid at room temperature with magnetic stirring for 2 h, then adjusted the solution to be neutral, and the product was isolated as pale yellow powder (the average particle size is about 1.25 μm). Wrapped with filter paper and extracted with ethanol Soxhlet for 48 h, and the side-chain liquid crystal polyether chelating resin(Compound IV) was obtained after drying.

Analytical Methods

Chemical Analysis

The molecular weight of five polyether samples measured by the hydroxyl value titration method were 3172.5, 3347.4, 3294.3, 3162.9 and 3109.8 respectively. The average molecular weight of polyether was calculated to be about 3217.4. The ratio of ECH-CHO was determined by analysing the content of epoxy cyclohexane units in the copolyether. So, the high temperature melting process [15] was used to determine the chlorine content of five polyether samples. Respectively, the measurement results were 21.92%, 20.63%, 23.21%, and 23.21%, so the average chlorine content of the copolyether was 22.2%. From the above data, the molar ratio of epichlorohydrin and epoxycyclohexane in the copolyether is about 1.4 : 1.

FT-IR Analysis

The structure of target chelating resin was determined by comparing the infrared spectrum of the chelating resin and its intermediates. Their infrared spectrum were recorded with Bomem MB154S spectrometer and demonstrated in Fig. 2.

It can be seen from the infrared spectrum of the chelating resin(IV) that a bending vibration peak of N-H appears at 1650 cm^{-1} , and at 1725 cm^{-1} , an obvious C=O stretching vibration absorption peak in -COOH appears. The carbonyl peak is significantly enhanced (because the benzene ring is conjugated with C=O, the absorption peak of the carbonyl group shifts to a low frequency). An wide and strong stretching vibration peak of -OH appeared at 3090 cm^{-1} , and hydroxyl peak increased significantly, while absorption peak of C-Cl is significantly weakened, which demonstrated the grafting rate of chelating group is almost completed. In addition, the critical changes from compound I to compound IV, such as the formation and disappearing of the C-Cl bond, can be found by comparing the change of corresponding absorption peak.

Thermogravimetric Analysis

Thermogravimetric Analysis was employed to test the chelating resin to determine the thermal stability of the chelating resin (Fig.3). Thermogravimetric curve was analyzed by a Netzsch STA 2500 thermal analyzer, and the rate of temperature rise and fall is $5\text{ }^{\circ}\text{C}\cdot\text{min}^{-1}$; the range of temperature is from $20\text{ }^{\circ}\text{C}$ to $500\text{ }^{\circ}\text{C}$ and the gaseous atmosphere is air.

As shown in Fig.3, in the first stage, the weight loss rate was about 4.7% at around $25\text{--}170\text{ }^{\circ}\text{C}$. It could be interpreted that the chelating resin loses the free water adsorbed between the molecules as the temperature increases. When temperature raised to above $190\text{ }^{\circ}\text{C}$, the chelating resin started to lose weight authentically. During the process of $190\text{--}375\text{ }^{\circ}\text{C}$, the weight loss rate was 24.9%, indicating that the resin begins to decompose at about $190\text{ }^{\circ}\text{C}$. In the third stage, and the weight loss rate is about 17.6% from $380\text{ }^{\circ}\text{C}$ to $530\text{ }^{\circ}\text{C}$. In the final stage, the weight loss rate was 6.3%, which occurred at $530\text{ }^{\circ}\text{C}$, and the residual amount of the final resin was 46.5%. All these results suggested that the chelating resin has good thermal stability and can be long-time used below 100°C conservatively.

Differential Scanning Calorimetry(DSC)

The phase structure of the side chain liquid crystal polyether chelate resin was tested in Fig.4 under different temperature by differential scanning calorimetry (DSC, STA 449 F3 Jupiter), and the rate of temperature rise and fall is $10\text{ }^{\circ}\text{C}\cdot\text{min}^{-1}$, the range of temperature is from $-30\text{ }^{\circ}\text{C}$ to $200\text{ }^{\circ}\text{C}$ and the gaseous atmosphere is nitrogen.

It can be seen that there are three endothermic peaks during the whole process of the temperature rising, and a small endothermic peak appears at 9.7°C (T_g) (Fig.4), which reflects the glass transition behavior of the sample, indicating that the product contains a small amount of amorphous ingredients. With the continuous temperature incensement, there is a wide melting peak appears at $86.8\text{ }^{\circ}\text{C}$ (T_m), and a significant change is observed in specific heat. When the resin is in the liquid crystal state and the temperature rises up to $232\text{ }^{\circ}\text{C}$ (T_i), the liquid crystal state exhibits an isotropic transition peak, indicating that the resin is in the liquid crystal state between $86.8\text{ }^{\circ}\text{C}$ and $232\text{ }^{\circ}\text{C}$, and the liquid crystal state temperature range is $145.2\text{ }^{\circ}\text{C}$. The large temperature range of the liquid crystal phase reveals that the resin can maintain stable liquid crystal performance in a wide temperature range.

Adsorption operation for Cu^{2+} in water

100 mg chelate resin and 400 mL of Cu^{2+} water solution ($0.05\text{ mol}\cdot\text{L}^{-1}$, normal Cu^{2+} content in smelting waste water) were added into the conical flask, and the solution was agitated slightly for 5 h at room temperature($25\text{ }^{\circ}\text{C}$). Then the chelating resin was separated via an inorganic filter membrane, and 2.0 mL 1-(2-Pyridylazo)-2-naphthol(PAN) as indicator was added. The titration solution is changed from purple to yellow green with EDTA standard solution, which shows the end point of titration. The calculation method for the static adsorption capacity is based on the literature [16].

Results And Discussion

Influencing factors on the adsorption performance for Cu^{2+}

The effects of adsorption time, pH and initial concentration of Cu^{2+} on the adsorption performance of resin was investigated. The adsorption capacity of resin at 30, 90, 150, 180, 240, and 300 min are shown in Fig.5. With the increase of time, the adsorption amount of the chelating resins firstly increase and then remains basically unchanged (Fig.5). When the absorbing time is 4 h, the adsorption capacity of chelating resin reaches the maximum of $20.3 \text{ mmol}\cdot\text{g}^{-1}$. It can be attributed that the process of chelating resin forming coordination bond with Cu^{2+} is basically completed within 4 h and reaches the equilibrium of adsorption. The time of reaching equilibrium adsorption is short and proper, which has the potential of industrial application.

The adsorption capacity of resin at pH 4, 5, 6, 8 and 10 of Cu^{2+} solution are shown in Fig. 6. With the increase of pH, the adsorption capacity of resin first increases and then decreases. When pH is 6, the maximum adsorption capacity is $19.72 \text{ mmol}\cdot\text{g}^{-1}$. When pH is < 6 , excessive acidity could cause the protonation of N and block the formation of coordination bond between N and Cu^{2+} . On the other hand, if the alkalinity of solution is too strong, the complex of Cu^{2+} and resin will be decomposed, even that $\text{Cu}(\text{OH})_2$ deposit can be generated, which results in a decrease of adsorption capacity in strong acid or alkali environment.

The effect of initial Cu^{2+} concentration on adsorption capacity was investigated in Fig. 7. The adsorption capacity of resin at different Cu^{2+} concentration of 0.01, 0.03, 0.05, 0.10 and $0.15 \text{ mol}\cdot\text{L}^{-1}$ is determined respectively.

It can be obtained from Fig.7 that with the increase of the concentration of Cu^{2+} , the equilibrium adsorption amount also increases. When the concentration of Cu^{2+} is $0.15 \text{ mol}\cdot\text{L}^{-1}$, the adsorption capacity of the chelating resin reaches the maximum value of $27.4 \text{ mmol}\cdot\text{g}^{-1}$. This is because when the Cu^{2+} is at a low level concentration, the chelating resin actually adsorbs Cu^{2+} in unsaturated state. So, the adsorption amount actually reflects a low level adsorption dynamic equilibrium state. So, with the increase of concentration of Cu^{2+} , the adsorption capacity of chelating resin also increases concomitantly. When the saturated adsorption almost reaches, the adsorption capacity is basically not changed with the concentration of Cu^{2+} increasing. In fact, it is the real absorption limitation of the resin.

Reuse performance of the resin

Generally speaking, reuse performance is quite important to evaluating a absorption resin. So, reuse performance was investigated and the results were shown in Fig.8. When the adsorption of Cu^{2+} reached saturation, the resin must be desorbed by pickling before reuse. The saturated resin was filtered from water and transferred to 40 mL ($2 \text{ mmol}\cdot\text{L}^{-1}$) hydrochloric acid. After being soaked for 2 h under ultrasonic, the resin was filtered and washed to neutral by deionized water. Then, the resin was used for the next adsorption process.

As shown in Fig.8, when the resin is reused for the first time, the adsorption capacity has significant drop. It could be ascribed to incomplete desorption under this regeneration conditions, and the desorption rate is over 90%. In fact, complete desorption is almost nonexistent. When the resin is reused for next five times, the effect of incomplete desorption can be counterbalanced. So, the adsorption capacity is only slightly decreased because of wear mass loss of resin in the long course of repeat absorption and desorption operation. In general, the results demonstrates this chelating resin has a good reuseability.

Isothermal Adsorption Study

In order to explore the adsorption characteristics of Cu^{2+} on chelating resin, Langmuir equation(1) and Freundlich equation(2) were introduced to simulate its adsorption isotherm [17].

The data of Cu^{2+} adsorption by chelating resin at 25 °C are determined and linear fitted, the experimental results are shown in Fig.9 and Table1.

$$\frac{1}{Q_e} = \frac{1}{Q_m K_L} \cdot \frac{1}{C_e} + \frac{1}{Q_m} \quad (1)$$

$$\ln Q_e = \frac{1}{n} \ln C_e + \ln K_F \quad (2)$$

Where Q_e is adsorption capacity at equilibrium and Q_m is the maximum adsorption capacity. Q_t is adsorption capacity at t time, C_e is equilibrium concentration. K_L and K_F are adsorption equilibrium constant for Langmuir isotherm and adsorption equilibrium constant for Freundlich isotherm.

Table1 Isothermal adsorption fitting parameter

Langmuir isothermal adsorption model			Freundlich isothermal adsorption model		
$Q_m/[\text{mg} \cdot \text{g}^{-1}]$	$K_L/[\text{L} \cdot \text{mg}^{-1}]$	R^2	n	K_F	R^2
2591	0.17	0.9957	1.5467	396.2	0.86768

It can be obtained from Table 1 that the Langmuir adsorption model ($R^2=0.9957$) has a better linear relationship with the fitting curve than the Freundlich adsorption model ($R^2=0.86768$), indicating the behavior of adsorbing Cu^{2+} is monomolecular layer and follows the principle of Langmuir model. The reason is that the adsorption process is a short-range and quantitative chemical adsorption on resin surface, which also explains the regeneration adopt as chemical desorption method is quite appropriate.

Table 2 Adsorption kinetics fitting parameter

Linear fit result of pseudo first order dynamics			Linear fit result of pseudo second order dynamics		
Q_e	K_1	R^2	Q_e	K_2	R^2
2735.4	0.0198	0.9175	1795.3	$5.3454 \cdot 10^{-6}$	0.9902

Adsorption kinetics Study

In order to study the adsorption kinetics of Cu^{2+} on the polyether chelating resin, the pseudo first order and pseudo second order kinetics models are used to investigate the adsorption kinetics mechanism. The pseudo first order kinetic equation (3) and the pseudo second order kinetic equation (4) are as follows [18]

$$\lg(Q_e - Q_t) = \lg Q_e - \frac{K_1}{2.303} t \quad (3)$$

$$\frac{t}{Q_t} = \frac{1}{K_2 Q_e^2} + \frac{1}{Q_e} \quad (4)$$

The above experimental equations are used to fit the adsorption experiment data, and the results are shown in Fig.10 and Table 2.

Where the K_1 is pseudo first order adsorption rate constant and the K_2 is pseudo second order adsorption rate constant.

It can be seen from Table 2 that the pseudo second order adsorption kinetic model ($R^2=0.9902$) has a better linear relationship with the fitting curve than the pseudo first order adsorption kinetic model ($R^2=0.91752$), indicating that the adsorption process of chelating resin to Cu^{2+} is inclined to follow the pseudo second order kinetic model. So, the absorption capacity of this chelating resin is sensitive to concentration of Cu^{2+} , it might be more appropriate for dealing with waste water with high Cu^{2+} concentration.

Conclusions

In summary, a new type of side chain liquid crystal polyether chelating resin with good thermal and mechanical properties was prepared by using epoxycyclohexane as a modified monomer. The TG and DSC analysis showed that the resin has good thermal stability and can be used stably below 190°C . There are two endothermic peaks during the heating increase process, indicating that it has liquid crystallinity, and the liquid crystal state temperature range is about 145.2°C . The Langmuir adsorption model and the Freundlich adsorption model were utilized to study the adsorption behavior of the resin on Cu^{2+} in wastewater. All these results showed that the Langmuir adsorption model is consistent, and the adsorption process is mainly monolayer. The K_L is high, indicating that the resin has a good binding ability to Cu^{2+} and the maximum static adsorption capacity was up to $27.4 \text{ mmol}\cdot\text{g}^{-1}$. In order to study the adsorption kinetics of Cu^{2+} , the pseudo first order and pseudo second order kinetics models were also used to investigate the adsorption kinetics mechanism of Cu^{2+} on chelating resin. The result of adsorption kinetics shows that the adsorption process of Cu^{2+} by this chelating resin conforms to the pseudo second order kinetic model. We anticipated that this new type of side chain liquid crystal polyether chelating resin [19-20] could bring great application in the recovery of copper resource from non-ferrous smelting industry.

Nomenclature

Q_e adsorption capacity at equilibrium, $\text{mg}\cdot\text{g}^{-1}$,

Q_m maximum adsorption capacity, $\text{mg}\cdot\text{g}^{-1}$

Q_t adsorption capacity at t time, $\text{mg}\cdot\text{g}^{-1}$

C_e equilibrium concentration, $\text{mg}\cdot\text{L}^{-1}$

K_L adsorption equilibrium constant for Langmuir isotherm, $\text{L}\cdot\text{mg}^{-1}$

K_F adsorption equilibrium constant for Freundlich isotherm

K_1 pseudo first order adsorption rate constant, min^{-1}

K_2 pseudo second order adsorption rate constant, $\text{g}\cdot(\text{mg}\cdot\text{min})^{-1}$

Declarations

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Figures

Figure 1

Synthesis route of side chain liquid crystal polyether chelating resin

Figure 2

Infrared spectra of products and intermediate products

Figure 3

Thermogravimetric curve of side chain liquid crystal polyether chelating

Figure 4

DSC curve of side chain liquid crystal polyether chelating resin

Figure 5

Effect of adsorption time on the adsorption performance of chelating resin

Figure 6

Effect of pH value on the adsorption performance of chelating resin

Figure 7

Effect of Cu^{2+} concentration on the adsorption performance of chelating resin

Figure 8

The reusability of the chelating resin

Figure 9

Linear fitting of Cu²⁺ + Langmuir adsorption model (A) and Freundlich adsorption model (B) for chelating resin

Figure 10

Linear fit of pseudo first order adsorption (A) and second order adsorption (B) kinetic mode