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Synthesis of siloxane-modified melamine-formaldehyde microsphere and its heavy metal ions adsorption by coordination effects

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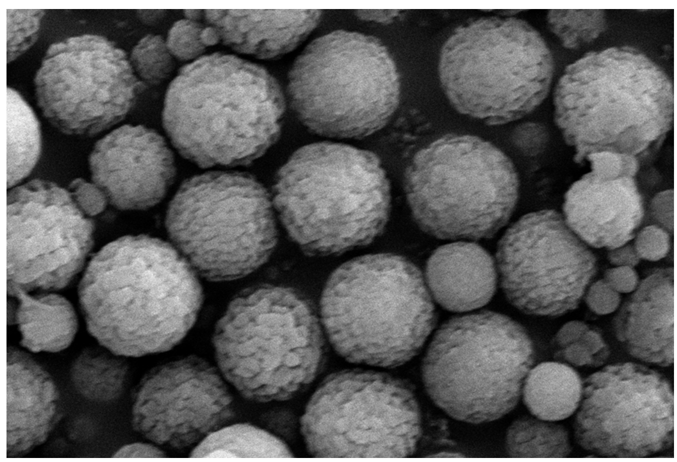
HIGHLIGHTS

- The siloxane-modified melamine-formaldehyde (Si–MF) microspheres were synthesized by an etherification reaction.
- The Si–MF microsphere surface was bumpy.
- The Si–MF microsphere showed a higher adsorption capability for metal ions.
- The Si–MF microsphere could be a potential metal ion trapping agent for Pb^{2+}.

GRAPHICAL ABSTRACT



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ABSTRACT

Microspheres could be utilized in the industrial waste water treatment due to their huge specific surface areas and surficial functional groups, which possibly adsorb heavy metal ions in the industrial waste. Melamine-formaldehyde (MF) microspheres were easily prepared as mono-dispersed micro-sized particles with a smooth surface. However, these rich amine groups in MF microspheres were frequently ignored for heavy metal ions adsorption owing to coordination effects. In this paper, a rough and bumpy siloxane-modified melamine-formaldehyde (Si–MF) microsphere was prepared using a dispersion polymerization method. Based on the experimental measurement, it is found that the particle size, surface morphology and dispersion state of Si–MF microspheres were significantly influenced by dispersing agent dosages, reaction temperature and pH. And Si–MF microspheres can be degraded in low pH. Moreover, the adsorption of the metal ions (Cu^{2+}, Pb^{2+} and Ni^{2+}) by Si–MF microspheres was also measured by an Inductive Coupled Plasma (ICP) instrument. The result indicated that the Si–MF microsphere showed a high adsorption capability for metal ions, especially Pb^{2+} as a potential metal ion trapping agent.
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1. Introduction

Heavy metal ions from the industrial waste are becoming a crucial factor in the serious environmental pollution [1]. Heavy metal ions are stable and significantly harmful to human bodies since they cannot be degraded or destroyed [2]. Many techniques have been carried out to remove such toxic metal ions, including filtration, chemical precipitation, neutralization, chelating ion exchange, and adsorption [3–7]. Among above methods, adsorption plays an appropriate strategy due to its flexibility in design and operation, superior efficiency and reversibility under certain conditions [8]. Microspheres have been widely used in many important areas, such as drug delivery and release, flexible display device, waste treatment by molecular imprinting and so on [1,9–12], owing to their huge specific surface area, good adsorptive property, adjustable structure and components, controllable particle size and shapes [13,14].

Melamine-formaldehyde (MF) resin is one kind of amino resins that are synthesized by a hydroxylation reaction and polycondensation [15,16]. The MF resin with good optical performance, high temperature resistant, low-cost raw source, has been widely used in photovoltaic materials, foam plastic, synthetic fiber, leather tanning agent, wood adhesives, etc. [17–19]. With the development of the polymerization techniques, the various kinds of MF resins, such as microspheres or microcapsules, are exploited as hollow vehicles for drug delivery [20], phase change thermal storage carriers [21], self-healing materials [22], flame retardant coating agents [23], etc. Renner prepared MF particles using several surfactants as reinforcing materials for natural rubber [24]. Hu et al. prepared a novel carboxymethyl cellulose modified melamine-formaldehyde (MF) phase change capsule with excellent encapsulation by in situ polymerization [25]. There MF microspheres have a smooth surface and rich functional amine groups [26]. However, the MF resins used as adsorbents in wastewater treatment have been rarely reported.

Siloxane (Si) has many excellent properties, such as good thermal stability, UV resistance, oxidation resistance, low surface energy, and good flexibility [27], owing to its natural structure composed of flexible Si—O bonds [28]. We envision the smooth surface of MF microspheres would be influenced by introducing siloxane components because of the interaction between rigid MF resins and flexible siloxane. Moreover, the rich amine groups from MF resins contribute to adsorb heavy metal ions due to the coordination effects between metal ions with unoccupied orbitals and amine groups with rich electrons. As a result, the Si–MF microspheres would be hoped as potential metal ion trapping agents to resolve environmental pollution coming from industrial waste water.

In this paper, we prepared Si–MF microspheres by a dispersion polymerization. The physical properties of Si–MF microspheres, including particle size, surface morphology and dispersion state, are measured by changing dispersant concentration, reaction temperature and pH during polymerization. Additionally, the adsorption of the metal ions (Cu^{2+}, Pb^{2+} and Ni^{2+}) by Si–MF microspheres in the aqueous media was measured by an Inductive Coupled Plasma (ICP) instrument.

2. Experiment

2.1. Materials

Melamine and formaldehyde aqueous solution (37.0 wt%), acting as monomers, were supplied from Aladdin Chemical Reagent Co., Ltd., China and Tianjin Insein Fine Chemical Factory Co., Ltd., China, respectively. Sodium hydroxide (NaOH) and hydrochloric acid (HCl), used to control the pH of solution, were purchased from Aladdin Chemical Reagent Co., Ltd., China. Polyvinyl alcohol (PVA, Aladdin, China) was used as stabilizers or dispersants in the polymerization reaction. Hydroxyl-terminated polydimethylsiloxane (DHPDMS) was supplied from Guangzhou Batai Chemical Co., Ltd., China. Deionized water (18.2 Ω cm, resistivity at 25 °C) was used in the experiment.

2.2. Preparation of Si–MF microspheres

The Si–MF microspheres were synthesized by a dispersion polymerization method and the preparation process included the following two-step process:

(1) Synthesis of methylol melamine prepolymer: a certain amount of formaldehyde solution was added into the reaction vessels and the pH of the solution was adjusted to 8.5–9.0 by using a 0.1 M sodium hydroxide solution. The composition of melamine and formaldehyde was set as 1:3 in molar ratio and the calculated amount of melamine was added to the solution. The mixture was stirred at 85 °C. After the melamine dissolved, the reaction was carried out for 20 min to form a prepolymer solution.

(2) Preparation of Si–MF microspheres: the polymerization reaction was performed in a 250-mL, three-necked, round-bottomed flask with a magnetic stirrer and conducted at a speed of 400 rpm at different temperature. Firstly, the calculated amounts of DI water, PVA and DHPDMS were placed into the flask and distributed by agitating magnetically at different temperature. Then, the above prepolymer was added to the reaction system. The pH of the mixture was adjusted to acidic condition (pH 4–6.5) quickly by using 1 M hydrochloric acid solution. The reaction was carried out for 30 min after the white turbidity appearing in the system, and then the sample was rapidly put into the ice water. The products were washed repeatedly with DI water to remove impurities and any remaining un-reacted monomers. The purified Si–MF products were dried by a freeze drying method. The MF microsphere was ultimately prepared by the same procedure at the same condition except without adding DHPDMS.

2.3. Chemical structure by FTIR

Fourier transform infrared spectroscopy (FTIR) was used to identify the siloxane composition of microspheres. The samples of MF and Si–MF microspheres with a weight of 0.9–1.0 mg were ground and dispersed in potassium bromide (KBr), followed by compression to form the sheet. FTIR spectra were obtained in the wave number range from 400 cm^{-1} to 4000 cm^{-1}.

2.4. Morphology observation

The surface morphologies of microspheres before and after modification were observed by using JSM-6510 scanning electron microscope (SEM). Before observation, samples were vacuum sputter coated with a thin layer of gold to provide electrical conduction. The particle size distribution was obtained by diameter analysis software based on the SEM images.

2.5. Adsorption of metal ions

In order to explore possibility of the Si–MF microsphere as a potential metal ion trapping agent, the adsorption of metal ions from the metal ion-containing aqueous solution was investigated. The Si–MF microsphere sample (0.03 g) was added into a 30 mL solution containing Cu^{2+}, Pb^{2+} or Ni^{2+} with a concentration of 50 mg/L, and the mixture solution was sealed into a 50 mL test bottle and vibrated for 2 h at room temperature using an oscillator. The concentration of metal ions absorbed by the Si–MF microspheres was evaluated by measuring the metal contents of the residual solution by using Inductive Coupled Plasma (Optima 8x00

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