



Research paper

Influence of calcination atmosphere on adsorptive performance of composite minerals materials

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ABSTRACT

The three composite minerals materials, CMM-1, CMM-2 and CMM-3, which were prepared by four Chinese non-metallic minerals (sodium bentonite, graphite, metakaolin and rectorite) and calcinated under the atmospheres of air, vacuum and nitrogen respectively, have been studied for the removal of malachite green (MG), a cationic dye, from aqueous solution. The influences of the three calcination atmospheres on the adsorbents' adsorptive performance were evaluated by a batch study in consideration of factors, such as the adsorbent dosage, initial dye concentration, adsorption duration, varied pH and static regeneration. Experimental results suggested that the maximum capacity of CMM to adsorb MG was more than 190 mg·g⁻¹, which was calcinated under the atmosphere of vacuum. Their adsorption equilibrium and kinetics confirmed the typical pseudo-first-order, pseudo-second-order, Freundlich and Langmuir adsorption models. The thermodynamic parameters of ΔG^\ominus , ΔH^\ominus and ΔS^\ominus showed that the adsorption was an exothermic and spontaneous process without remarkable change. The spent adsorbents were regenerated five times and probable pathway for the efficient and re-utilizing adsorbent has been proposed. The results indicate that CMM could be employed as porous, low density, and large specific surface area alternatives for the removal of cation dyes from industrial wastewater. The optimum adsorption capacity of the adsorbents was discovered under the calcination condition in vacuum.

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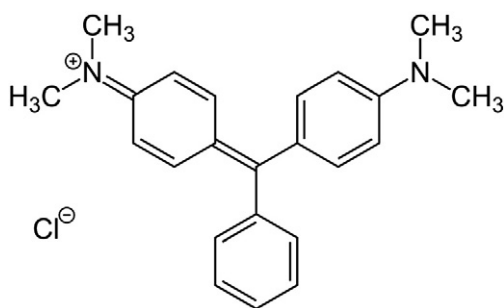
1. Introduction

The treatment of effluent containing dyes poses considerable problems in the wastewater industry. Synthetic dyes are extensively used in many industries, such as fiber, paper, plastics and other materials productions, which have discharged large volumes of dyeing wastewater. Many of them can be environmental hazard to the aquatic organisms around the industrial areas (Hameed, 2009), because these toxic organic dyes can affect plant life and thus destroy the entire ecosystem (Wu et al., 2010; Özcan and Özcan, 2004). Dyeing wastewater is commonly characterized as high in salt and organic content and low in biodegradation potential (Alinsafi et al., 2005). Hence, they cannot be completely removed by conventional biological treatment processes, such as activated sludge and anaerobic digestion (Vimonses et al., 2009a, 2009b). China is the largest country in the world where dyes are produced and applied (Chen et al., 2010). Therefore, a quite important issue is to strengthen the comprehensive management of dyeing wastewater to meet the national discharge standards.

Malachite Green (MG, N,N,N',N'-tetramethyl-4,4'-diamino-triphenylcarbenium chloride, formula weight 364.92), a triphenylmethane and water-soluble cationic dye, is commonly used for fiber and textile finishing production. Its maximum absorbance wavelength is 618 nm. MG has the structure as shown in Scheme 1.

A large number of techniques, including adsorption, ozonation, advanced oxidation, coagulation, biological treatment and membrane filtration, have been developed and used to remove the dye from wastewater (Walker et al., 2003). The treatment of adsorption has been famous as the popular process to remove dyes from aqueous solution with the advantages of simple operation, high effectiveness and easy regeneration. Many clay minerals, such as bentonite (Vimonses et al., 2009a, 2009b), rectorite (Ming et al., 2014), kaolinite (Vimonses et al., 2009a, 2009b; Harris et al., 2001), montmorillonite (Peng et al., 2003), vermiculite (Gong et al., 2010) and sepiolite (Bingol et al., 2010; Santos and Boaventura, 2008), have been studied to remove dyes from wastewater as commercially available adsorbents due to their advantages such as non-toxicity, low-cost, high chemical stability, large specific surface area, abundant availability, variety of structural and surface properties, and potential for adsorption and ion exchange. Expanded graphite shows high efficiency for the removal of organic and inorganic pollutants owing to its large specific surface area (Cao et al., 1996; Di, 2013). However, it is still limited as an adsorbent to

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Scheme 1. Molecular structure of Malachite Green.

remove the contaminants because of its high cost and difficulty in reutilization. So expanded graphite is granulated to make composite adsorption materials for clarifying sewage in authors' research group, and a good adsorptive property is exhibited for removing multiple metal ions from industrial wastewater (Lei et al., 2013a, 2013b).

This study is to evaluate the adsorption capacity of available composite minerals materials (CMM), including sodium bentonite, expanded graphite, rectorite, metakaolin, to remove MG from aqueous solutions. This research is focused on how to control the process operation parameters, such as the dosage of adsorbent, initial concentration of the dye, initial pH of aqueous solutions, time and temperature of reaction, to affect the adsorption capacity. The equilibrium adsorption isotherms were established by Freundlich and Langmuir models. The adsorption kinetic models were also established to analyze the kinetics and mechanisms of MG adsorption. Thermodynamics were also employed to examine the adsorptive tendency of the adsorbent.

2. Materials and methods

2.1. Materials

Four raw minerals, such as sodium bentonite, graphite, metakaolin and rectorite, were respectively obtained from Jianping County in Liaoning Province, Jixi County in Heilongjiang Province, Jinshan metakaolin Co., Ltd. of Enshi City in Hubei Province, Mingliu Rectorite Science and Technology Ltd. in Hubei Province. All the mineral materials were crushed to a particle size not more than 74 μm , and then dried in an oven at 105 $^{\circ}\text{C}$ for more than 3 h. Malachite Green and carboxymethyl-cellulose sodium (CMC-Na) were provided by Sinopharm Chemical Reagent Co., Ltd. The dye solution was prepared to the required concentration with ultrapure water.

2.2. Preparation of materials

2.2.1. Expanded graphite

The expanded graphite was obtained by burning the intercalated graphite, which consisted of graphite, potassium permanganate (KMnO_4 , AR) and perchloric acid (HClO_4 , GR), with a ratio of 10.0 g, 1.0 g and 10.0 mL respectively, at 900 $^{\circ}\text{C}$ for 10–20 s in a muffle furnace. Then the mixture underwent reaction for more than 3 h in a round-bottom flask, and was washed with ultrapure water to pH 6.0–7.0 and dried below 65 $^{\circ}\text{C}$.

2.2.2. CMM

Based on our group's previous research achievements (Lei et al., 2013a, 2013b; Wang et al., 2015), the precursor of CMM consisted of 17.5 wt.% sodium bentonite, 28.0 wt.% rectorite, 28.0 wt.% metakaolin, 14.0 wt.% expanded graphite and 12.5 wt.% CMC-Na. Appropriate amount of water was mixed with the precursor to make granulated materials (\varnothing 8 mm). Finally, CMM-1, CMM-2 and CMM-3 were prepared from the granulated materials by calcination in muffle furnace at

800 $^{\circ}\text{C}$, which was attained after heating for 4 h and then kept for 4 h under the atmospheres of air, vacuum and nitrogen, respectively.

2.3. Characterizations of the adsorbents

The phase compositions of CMM-1, CMM-2 and CMM-3 were analyzed on a rotation anode high power X-ray diffractometer (RU-200B/D/MAX-RB, Rigaku, JP) over the 2θ range of 3–70 $^{\circ}$. The morphological features of the adsorbents were obtained by scanning electron microscope (JSM-5610LV, Electronics Co., LTD, JP) at an accelerating voltage of 5 kV. Their functional groups were analyzed with Fourier transform infrared spectroscopy (IS-10, Nicolet, US) within a wavenumber range of 400–4000 cm^{-1} . Their Brunauer–Emmett–Teller specific surface area was measured by an automatic analyzer of specific surface area and porosity, and calculated by BET method from the data in a P/P $_0$ range between 0.05 and 0.2.

The experimental temperature and time for the determination of the ignition loss of sample were 950–1000 $^{\circ}\text{C}$ and 15–20 min respectively. It was evaluated by the following formula:

$$\psi = \frac{m_0 - m_1}{m_0} \times 100\% \quad (1)$$

where Ψ is the ignition loss of sample (%), m_0 is the weight of uncalcined adsorbents (g), m_1 is the weight of calcined adsorbents (g).

2.4. Adsorption experiments

The experiments of MG adsorption onto the adsorbents were carried out in a rotary shaker (HZQ-C, Harbin Donglian Electronic Technology Development Co., Ltd., CN) under the conditions of 110 rpm and 15–45 $^{\circ}\text{C}$. A quantitative amount of CMM-1, CMM-2 or CMM-3 was added to 100 mL of the known initial concentration MG. The influential factors, such as dosage of adsorbent, initial concentration of dye, initial pH of aqueous solutions, and time and temperature of reaction, were investigated to evaluate the adsorption potential. At the end of the adsorption equilibration period, the adsorption materials were separated by centrifugation (LXJ-IIB, Anting Scientific Instrument Factory of Shanghai, CN) at 5000 rpm for 15 min. The supernatant could be then filtered by using Millex VX filter (Millipore 0.45 μm) to ensure that the solution was free of granules before measuring the residual dye concentration. Finally, the solution was analyzed by UV–visible spectrophotometer (UV-3000PC, Mapada, CN) at $\lambda_{\text{max}} = 618 \text{ nm}$.

The dye concentration was determined colorimetrically by measuring the maximum absorbance at the wavelength of 618 nm. And then a calibration curve was plotted between the absorbance and the concentration of the dye solution. The removal ratio of the MG was calculated from the calibration curve.

2.5. Static regeneration experiments

The methods of static regeneration are the same as the preparation of CMM' (Section 2.2.2). Then the experimental conditions of static regeneration materials are in keeping with the adsorption experiments' (Section 2.4).

3. Results and discussion

3.1. Characteristics of adsorption materials

3.1.1. Physical characteristics of the adsorbents

From the results of the physical characterization of CMM-1, CMM-2 and CMM-3 (Table 1), positive correlations are shown among the BET surface area, pore volume, adsorption average pore width, porosity, scatter ratio and ignition loss of the adsorbents. In addition, the advantages of multi-pore, mesoporous, large specific surface area are revealed

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