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Comparison of activated carbon and iron/cerium modified activated carbon to remove methylene blue from wastewater

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ABSTRACT

The methylene blue (MB) removal abilities of raw activated carbon and iron/cerium modified raw activated carbon (Fe–Ce-AC) by adsorption were researched and compared. The characteristics of Fe–Ce-AC were examined by N_2 adsorption, zeta potential measurement, FTIR, Raman, XRD, XPS, SEM and EDS. After modification, the following phenomena occurred: The BET surface area, average pore diameter and total pore volume decreased; the degree of graphitization also decreased. Moreover, the presence of Fe_3O_4 led to Fe–Ce-AC having magnetic properties, which makes it easy to separate from dye wastewater in an external magnetic field and subsequently recycle. In addition, the equilibrium isotherms and kinetics of MB adsorption on raw activated carbon and Fe–Ce-AC were systematically examined. The equilibrium adsorption data indicated that the adsorption behavior followed the Langmuir isotherm, and the pseudo-second-order model matched the kinetic data well. Compared with raw activated carbon, the maximum monolayer adsorption capacity of Fe–Ce-AC increased by 27.31%. According to the experimental results, Fe–Ce-AC can be used as an effective adsorbent for the removal of MB from dye wastewater.

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Introduction

Dyes are coloring agents used by a variety of industries, including textile, plastics, paper and pulp, to colorize their final products. A total of 30% of the world production of dyes may be lost during the dyeing process (Nethaji and Sivasamy, 2011). However, most dyes are toxic. The uncontrolled release of dye residues as industrial wastewater into receiving water streams has led to a number of negative impacts, including a decrease in light penetration as well as carcinogenic and mutagenic changes to organisms (Ghaedi et al., 2015). The dye wastewater can also cause allergies, dermatitis, and skin irritation, and induce cancer and mutations in humans (Ghaedi et al., 2013a; Mirzaei et al., 2013; Rosenkranz et al., 2007). Therefore, wastewaters with large dye contents must be treated before discharge into the environment, so as to minimize health hazards and the threat to the environment (Ghaedi et al., 2013a; Mirzaei et al., 2013). However, dyes are relatively difficult to biodegrade and remove from dye wastewater due to their high solubility in water and complex aromatic molecular structures (Kayan et al., 2010; Yao et al., 2010). Providing an effective method to control and manage dye wastewater is thus of great urgency.

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There are many dye wastewater treatment methods available including coagulation (Tunay et al., 1996), reverse osmosis (Forgacs et al., 2004), photo-degradation (Zhai et al., 2011), chemical oxidation (Tehrani-Bagha et al., 2010), membrane filtration methods (Alventosa-deLara et al., 2012), ozonation (Robinson et al., 2001), and biosorption (Ghaedi et al., 2013b). However, the abovementioned methods are generally ineffective in dye wastewater treatment or have high operating costs. Adsorption has attracted our attention owe to its many advantages such as high efficiency, environmental friendliness and low cost compared to other treatment methods for dye wastewater. Activated carbon, a material having an abundant porous structure and strong adsorption capacity, is widely used in many different industries, including in separation, removal of dyes and pollutants from wastewater, and as a catalyst support (Auta and Hameed, 2013; Ge et al., 2016). It is capable of adsorbing many kinds of dyes, but its adsorption efficiency and adsorption capacity is low. These disadvantages restrict its extensive application.

To overcome these disadvantages, more and more attention has been paid to the modification of activated carbons and synthesis of high performance adsorbents to treat dye wastewater. A popular method is using metal oxides to modify activated carbon. Modifying activated carbon with metal oxides may provide a structure with enhanced porosity, which can be varied in terms of pore structure and surface functional groups (Ge et al., 2016). Therefore, its adsorption properties are altered. According to literature reports, metal oxides such as aluminum oxide, iron oxide, zinc oxide, manganese oxide, copper oxide and vanadium oxide have been used to impregnate activated carbon to enhance its adsorption capability or improve its catalytic oxidation capability for dye wastewater (Chen et al., 2014; Fang et al., 2013; Yurum et al., 2014). Roosta et al. (2015) successfully synthesized a kind of modified carbon material ($Zn(OH)_2$-NP-AC), which has good performance in the removal of sunset yellow from aqueous solutions. Shah et al. (2014) also prepared a FeAC material *via* iron doping of activated carbon. The results show that FeAC is capable of up to 98% methylene blue (MB) removal from aqueous media. Goscianska et al. (2015) reported ordered mesoporous carbons modified with cerium as effective adsorbents for azo dye removal. Though many studies have been devoted to preparing and synthesizing various kinds of highly effective adsorbents, adsorbents based on single metal oxides have some disadvantages. Recently, composite adsorbents impregnated with two or more different metal oxides have attracted considerable research attention. The reason is that they could possess the advantages of multiple kinds of metal oxides as compared to adsorbents prepared with a single metal oxide (Su et al., 2015). What is more, they could show synergistic effects that greatly improve their adsorption performance (Ren et al., 2012).

Various rare earth compounds have been extensively developed as adsorbents for hazardous anions from aqueous solutions and have shown promising results (Deng and Yu, 2012; Liu et al., 2002). Iron oxides are also regarded as promising materials in dye wastewater treatment owing to Fe_3O_4 having magnetic properties (Xu et al., 2012; Gong et al., 2009). This means that adsorbents including Fe_3O_4 can be easily separated from dye wastewater in an external magnetic field. However, to the best of our knowledge, there have been a limited number of studies regarding synthesis of Fe–Ce composite modified activated carbon, based on the advantages of both types of oxides. In this work, we present a technical route whereby the raw activated carbon is treated with ultrasound and impregnated with $Fe(NO_3)_3$ and $Ce(NO_3)_3$, forming a kind of composite adsorbent (Fe–Ce-AC) by microwave heating. The cationic organic dye MB is a basic dye widely used in the abovementioned industrial applications. Therefore, MB was selected as the model pollutant to test the removal performance of the Fe–Ce-AC composite. In addition, we also thoroughly studied the equilibrium isotherm and kinetics of the adsorption process. Both the raw activated carbon and Fe–Ce-AC were characterized by using a number of characterization techniques.

1. Experimental

1.1. Materials and methods

The raw activated carbon was purchased from the Chengdu Kelong Chemical Reagent Factory of China. Chemical reagents including $Fe(NO_3)_3$ (China, Tianjin Reagent Chemicals Co., LTD), $Ce(NO_3)$ (China, Tianjin Reagent Chemicals Co., LTD) and MB (China, Tianjin Standard Technology Company) were all of analytical grade. The raw activated carbon was treated by ultrasound for 30 min (ultrasound-activated carbon) to clean off the impurities on the surface of the activated carbon by ultrasonic cavitation, to prepare for impregnation. Afterwards, 15 g of dry ultrasound-activated carbon was placed in 150 mL of an aqueous solution with $Fe(NO_3)_3$/$Ce(NO_3)$ mole ratio of 4/2 (using a mixture of 0.1 mol/L of $Fe(NO_3)_3$ and $Ce(NO_3)_3$ solution) for 3 hr. Then, the suspension was filtered and the solid was dried at 105°C for 24 hr. Finally, the sample was heated in a microwave furnace with microwave power of 700 W, temperature of 700°C and heating time of 25 min. The microwave furnace utilized multi-mode continuously controllable microwave power for the experiments. Table 1 lists the industrial analysis of the raw activated carbon and ultrasound-activated carbon. As shown in Table 1, the fixed carbon of the ultrasound-activated carbon is increased and other components are decreased because of ultrasonic cavitation, facilitating $Fe(NO_3)_3$ and $Ce(NO_3)_3$ impregnation.

1.2. Characterization methods

The N_2 adsorption measurement of Fe–Ce-AC was carried out at 77 K using an automatic adsorption apparatus (Autosorb-1-C, USA) with the relative pressure (P/P_0) range from 10^{-7} to 1. The total pore volumes were estimated as the equivalent liquid volume of the adsorbate (N_2) at a relative pressure of 0.99. The Brunauer–Emmett–Teller (BET) surface area of the samples was

Table 1 – Industry analysis on raw activated carbon and ultrasound-activated carbon.

	Ash (%)	Volatile (%)	Moisture (%)	Fixed carbon (%)
Raw activated carbon	3.29	3.99	1.9	92.72
Ultrasound-activated carbon	2.28	3.65	1.5	94.06

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