



# Removal of methylene blue and mechanism on magnetic $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub> nanocomposite from aqueous solution



Dong Chen<sup>a</sup>, Ziyang Zeng<sup>b</sup>, Yubin Zeng<sup>a,\*</sup>, Fan Zhang<sup>a</sup>, Mian Wang<sup>a</sup>

<sup>a</sup> Department of Water Quality Engineering, School of Power and Mechanical Engineering, Wuhan University, Wuhan 430072, China

<sup>b</sup> Department of Chemical Engineering & Applied Chemistry, University of Toronto, Toronto M5S 3E5, Canada

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## ABSTRACT

Magnetic  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub> (M- $\gamma$ FS) nanocomposite was prepared using  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> as the magnetic carriers. M- $\gamma$ FS was characterized by transmission electron microscope (TEM), X-ray diffraction (XRD), and Fourier Transform infrared spectroscopy (FTIR). The characterizations of M- $\gamma$ FS exhibit the irregular core-shell structure, and SiO<sub>2</sub> has been successfully coated on the surface of  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>. M- $\gamma$ FS has much higher adsorption capacity 116.09 mg/g calculated by coated SiO<sub>2</sub> on M- $\gamma$ FS than  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub>, and the alkaline conditions are beneficial to the adsorption of MB. Coexisting cations, ionic strength and humic acid can influence the adsorption capacity. Meanwhile, adsorption kinetics data for M- $\gamma$ FS show good fit to the pseudo-second-order kinetics model, and the Langmuir equation is more appropriate to describe the adsorption behavior of MB on M- $\gamma$ FS. Moreover, after recycling four times, M- $\gamma$ FS washed by ethanol can still keep high adsorption capacity for MB adsorption.

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## 1. Introduction

Various dyes have been used in the production processes of the textile, printing and dyeing, and papermaking industries [1,2]. A large amount of these dyes have entered water bodies and become a threat to the water environmental safety. The dyes in the effluents, even at low concentrations, produce a number of toxic products via oxidation, hydrolysis and other chemical reactions [3], which either directly or indirectly cause harm to human health, e.g., by increasing the incidence of allergies, tumors and cancers [4]. Therefore, it is necessary to find simple, highly efficient, and economical methods to treat dye wastewater.

Currently, the methods of removal of dye including adsorption [5,6], membrane treatments, advanced oxidation processes [7–11], and biological treatments [12] have been studied extensively. Among these methods proposed, due to the high efficiency, cost effectiveness, and simple operation, the adsorption method has received an increasing attention [13,14]. The unique feature of the adsorption method is that no other reagent is required to promote the reaction rate between the adsorbent and adsorbate. Additionally, adsorbents generally have large specific surface areas and high removal of dye. Therefore, the adsorption to remove dyes from wastewater displays a number of advantages, such as rapid action and strong adaptability [15].

Traditional adsorbents, such as activated carbon, clay, silica, zeolites and other porous materials, have been investigated

\* Corresponding author.

E-mail address: [zengyubin@whu.edu.cn](mailto:zengyubin@whu.edu.cn) (Y. Zeng).

widely [16–18]. Especially activated carbon is frequently used since it is effective for the removal of dissolved organic matter. Monika Wawrzkiwicz et al. used mixed silica-alumina oxide consisting of 4% SiO<sub>2</sub> and 96% Al<sub>2</sub>O<sub>3</sub> to adsorb acid, reactive and direct dyes from aqueous solutions and from wastewater and got satisfactory results [19]. Dong Wei et al. reported an aerobic granular sludge as a biosorbent to treat dye wastewater, and achieved 80.72% removal of MB [20]. New sorbent rhamnolipid-functionalized graphene oxide (RL-GO) hybrid was prepared by one-step ultra-sonication and obtained a high adsorption capacity of MB onto RL-GO [21]. Nevertheless, the powder and gel adsorbents are difficult to separate from water except through high speed centrifugation; accordingly limiting their applicability. On the other hand, the powder and gel adsorbents cannot meet the developing industrial demands because of limited adsorption capacity and tedious recovery process. Additionally, difficulties in regenerating and high processing cost limit its applications [22]. Therefore, it is a challenge to develop materials with high adsorption capacity, good chemical stability and easy solid-liquid separation for the treatment of MB wastewater.

Magnetic field assisted separation technologies have a potential to provide new opportunities. Magnetic separation based on the superparamagnetic Fe<sub>2</sub>O<sub>3</sub> has received high attention and has been widely used in wastewater treatment due to its convenience, economy and efficiency [23,24]. Hence, the aim of our study is to focus on the combination of magnetic particles  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> to overcome the disadvantage of traditional sorbents mentioned above. Based on this purpose, we prepared magnetic composites  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub> (M- $\gamma$ FS) which can not only be separated easily from liquid phase, but also possess a couple of advantages, such as high specific surface area, high adsorption capacity, strong paramagnetism, and stability in water without waste products. Moreover, M- $\gamma$ FS was characterized using TEM, XRD, FTIR, BET specific surface area, pore size distribution and magnetic saturation intensity. The adsorption capacity for dye MB on M- $\gamma$ FS and its mechanism including the influences of the initial solution pH, kinetics and adsorption isotherms were investigated. Meanwhile, regeneration of M- $\gamma$ FS was also evaluated in this study.

## 2. Materials and methods

### 2.1. Reagents and materials

All of the chemicals used were analytical grade except as noted. 2,4-DCP, tetraethyl orthosilicate (TEOS), anhydrous alcohol, methylene blue, ammonia,  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> were analytical grade, all was purchased from SigmaAldrich, USA. All solutions were prepared using deionized water.

### 2.2. Preparation of M- $\gamma$ FS nanocomposite materials

At room temperature, M- $\gamma$ FS was prepared at the optimum conditions according to the evaluation experiments on preparation parameters: 0.8 g  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> was added to a mixture of ethanol and DI water (v/v 80:20) and ultrasonically dispersed for 60 min. After dispersion, 2 mL ammonium was added and homogeneously mixed. A mixture solution of 0.4 mL TEOS and 30 mL ethanol was then added in drops and stirred at 200 r/min for 3 h. The composite materials were washed three times in ethanol and then dried at 60 °C in an oven for 12 h. After cooling, the materials were ground to obtain the orange M- $\gamma$ FS powder.

### 2.3. Adsorption experiment

An amount of M- $\gamma$ FS powder was added to 50 mL MB with initial concentrations of 15–180 mg/L. The initial solution pH was adjusted to 4.0–12.0 by adding 0.1 M HCl or 0.1 M NaOH, and the solution was then shaken at 150 r/min at temperature 298–318 K for 0.5–6 h. After that, M- $\gamma$ FS was separated from the solution with an external magnetic field, the solution absorbance was measured at 664 nm. All samples were prepared in duplicate, and average values of the replicated measurements were reported in all experiments. The adsorption capacity of MB on M- $\gamma$ FS,  $q_e$  (mg/g) was calculated using Eq. (1):

$$q_e = V \frac{C_0 - C_e}{M} \quad (1)$$

Where  $q_e$  is the adsorption capacity,  $C_0$  is the initial MB concentration,  $C_e$  is the equilibrium MB concentration,  $V$  is the volume of the solution and  $M$  is the mass of the adsorbents.

### 2.4. Instrumentation

A glass pH electrode (PHS-25, China) was used for pH measurement. Sample morphology was examined using a transmission electron microscope (TEM, JEM-100CXII, Japan). X-ray diffraction (XRD) were performed on the samples to confirm the crystal structure and identity using X-ray Diffractometer (Rigaku D/MAX-RB, Japan) with Cu K $\alpha$  radiation in the 2 $\theta$  ranges of 5°~80° at a scan rate of 1 °/min. The FTIR spectrum of SiO<sub>2</sub>,  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>, M- $\gamma$ FS before and after adsorption was measured with NICOLET 5700 FTIR Spectrometer (Thermo Fisher Scientific). The specific surface area was determined with a

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