

Synthesis of SnO₂-activated carbon fiber hybrid catalyst for the removal of methyl violet from water



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ABSTRACT

SnO₂/activated carbon fiber (ACF) hybrid catalyst was synthesized from kapok precursor via a two-step process involving pore-fabricating and self-assembly of SnO₂ nanoparticles. The morphology and phase structure of the obtained samples were characterized by X-ray diffraction, field emission scanning electron microscope, high resolution transmission electron microscopy and N₂ adsorption-desorption isotherm. These results demonstrated that the synthesized SnO₂/ACF retained the hollow-fiber structure of kapok fibers. SnO₂ nanoparticles dispersed uniformly over the ACF support. The obtained hybrid catalyst showed porous structure with high surface area (647–897 m²/g) and large pore volume (0.36–0.56 cm³ g⁻¹). In addition, the catalytic activities of the obtained samples for methyl violet degradation under microwave irradiation were also evaluated. It was found that the SnO₂/ACF catalyst exhibited high catalytic activity for methyl violet degradation due to the synergistic effect of microwave and SnO₂/ACF catalyst.

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1. Introduction

The increasing environmental problems caused by dye effluents have driven considerable research efforts on the degradation of dye wastewater. The microwave induced catalytic oxidation (MICO) was considered as an efficiently advanced technology to treat dye wastewater due to the following advantages [1,2]: (1) short reaction time, (2) low activation energy, and high reaction rate, (3) high yield and purity of products. These advantages are mainly due to the thermal and non-thermal effects generated by the interaction between microwave (MW) and MW-adsorbing materials, i.e. superheating, polarization, dielectric properties, hot spot formation, nuclear spin rotation and spin alignment [3]. MW-adsorbing material can be a semiconductor, ferromagnetic metal or transition metal oxides. The performance of a given MW-adsorbing catalyst could be improved by distributing the active component throughout some porous support [4]. ACF are superior to other supports due to its large surface area, porous structure, high adsorption capacity, easily recyclable and variable surface chemical composition [5].

SnO₂-based catalysts have been intensively studied as gas sensors [6,7] and catalysts for CO oxidation [8]. Recently, it was

reported that SnO₂ nanocrystal showed high MW absorption properties due to its high dielectric loss [9]. Kapok fiber is an agricultural product derived from the fruits of silk-cotton tree. It is a single cell natural cellulose fiber containing cellulose, lignin, water, ash, xylan and waxes [10]. Kapok fiber has a rather thin cell wall with a huge hollowness ratio and thus exhibits a low density and high bulkiness. Being a natural product, kapok is productive and environmental friendly. These facts inspired us to utilize kapok fiber as carbon precursor to synthesize SnO₂-ACF hybrid catalyst for dye degradation.

In this study, SnO₂/ACF hybrid catalyst was synthesized by using kapok as carbon precursor for the degradation of methyl violet (MV). The effects of various factors including the concentration of MV solution, reaction temperature and used time on the MV degradation were evaluated. Comparison of the experimental data with the generalized kinetic model was also presented.

2. Experimental

2.1. Materials

SnCl₄·5H₂O was of analytical reagent grade (Sinopharm Chemical Reagent Co., Ltd, China) and used without further purification. MV was purchased from Zhejiang Shanghai SSS Reagent Co. Ltd., China. Kapok fibers were obtained from the local market.

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2.2. Sample preparation

Natural kapok fibers were used as carbon fiber precursor for the preparation of ACF. After washing and drying, 5 g kapok fibers were heated in a tube furnace at 850 °C under a constant N₂ flow of 60 cm³ min⁻¹ and heating rate of 5 °C min⁻¹, and then followed with CO₂ activation for 20 min at 850 °C under a constant CO₂ flow of 60 cm³ min⁻¹. After that, 1 g as-synthesized ACF were impregnated in 100 mL SnCl₄·5H₂O solutions with different concentrations (0.5–1.1 M) and stirred continuously for 1 h, and then dried at 80 °C for 6 hours, during which hydrolysis and condensation reactions occurred. The impregnated ACF were calcinated at 400, 500 and 600 °C under a constant N₂ flow of 20 mL min⁻¹ for 1 h, respectively.

2.3. Sample characterization

The morphologies and chemical compositions of the obtained samples were characterized by field emission scanning electron microscope (FESEM, FEI QUANTA FEG250, USA) and high resolution transmission electron microscopy (HRTEM; TecnaiF20, Philips, Hillsboro, OR, USA). The structure and the crystal phases of the as-synthesized solid were investigated by X-ray diffraction patterns (XRD with Cu K α radiation source; Model D8-Advance, Germany) operated at 40 kV and 100 mA. The pore structure of the obtained sample was analyzed by nitrogen adsorption-desorption at 77 K on a surface area analyzer (ASAP2020M + C, Micrometrics, USA). The surface area of sample was evaluated by the Langmuir model and Brunauer Emmett Teller (BET) model, while the pore size distribution was estimated by Barrett Joyner Halenda (BJH) theory. The FT-IR spectra were obtained by Nicolet 380 FT-IR (Nicolet, USA).

2.4. Degradation of MV

The MICD process was conducted in a thermostated static MW chemistry apparatus (Fig. 1) (Huiyan MG08S-2B, Nanjing, China, 2450MHz \pm 50 MHz, 300 W). Temperature was controlled with contact Pt temperature sensor. In each experiment, 0.05 g catalyst was added into 100 mL MV solution. After MW irradiation, the suspension between catalyst and MV was sampled and centrifuged to remove catalyst at specific time intervals. The filtrates were analyzed to identify the concentration change by UV-Vis spectroscopy (U-3501, Hitachi, Japan). The change of the light absorbance at 665 nm wavelength was calibrated with different MV concentrations. The degradation ratio of MV was determined by the formula

(% degradation = [(C₀ - C)/C₀] \times 100), where C₀ and C are the initial and time *t*'s concentration of MV solution, respectively.

3. Results and discussion

3.1. Structural characterization

ACF were prepared from kapok fibers using CO₂ activation method. This material has large surface area and porous structure and we rely on the hypothesis that upon CO₂ activation it is possible to obtain ACF that not only act as supports for SnO₂ nanoparticles but also behave as catalyst with remarkable MW-induced-oxidative responses.

Fig. 2 shows the XRD patterns of impregnated ACF before and after being calcinated at different temperatures. For the impregnated sample before calcination, two weak peaks at 22.6 and 43.4° were observed, which can be attributed to the (002) and (100) peaks of graphite (PDF number: 75-1621). When impregnated sample was calcinated at 400–600 °C, four diffraction peaks at $2\theta = 26.3^\circ$, 33.9°, 52.2° and 65.1° were observed, which can be indexed as (1 10), (1 0 1), (2 1 1) and (2 2 1) of SnO₂ (JPCDS, No. 41-1445). No additional peak of another phase was observed in the XRD patterns. The average crystallite sizes of SnO₂ calcinated at 400, 500 and 600 °C were calculated to be 5.1, 9.4 and 14.2 nm, respectively, based on the Scherrer formula [11].

In order to investigate the effect of impregnation condition on the pore structure of SnO₂/ACF, SnCl₄·5H₂O solutions with different concentrations (0.5, 0.8 and 1.1 M) were used to soak ACF. The pore structures of SnO₂/ACF hybrids are characterized by the nitrogen adsorption/desorption isotherms (Fig. 3a), and the data for ACF were also shown for comparison. Table 1 shows the surface area and porosity parameters of the obtained samples. It can be seen from Fig. 3a that ACF exhibited the typical type IV hysteresis loop [12], indicating the presence of mesopores in ACF. The concentration of SnCl₄·5H₂O solution had a significantly effect on the adsorption/desorption isotherm. When the concentration of SnCl₄·5H₂O solution increased, the slope of the isotherms gradually decreased and the hysteresis loops became smaller. The isotherms of SnO₂/ACF-0.5 seem to be a mixture of Type I and IV, while the isotherms of SnO₂/ACF-0.8 and SnO₂/ACF-1.1 exhibited Type I. The corresponding pore size distributions (Fig. 3b) indicate that the pore structure of SnO₂/ACF was basically mesoporous with the average pore diameters of 2.57–3.52 nm. It can be seen from Table 1 that the surface area and mean pore size decreased with increasing concentrations of SnCl₄·5H₂O solution, approaching steady



Fig. 1. Photograph of the experimental set-up.

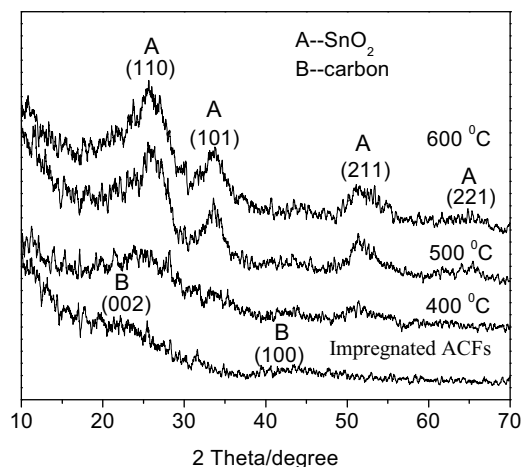


Fig. 2. XRD patterns of the impregnated ACF before and after being calcinated at different temperatures.

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