



Agricultural waste/graphene oxide 3D bio-adsorbent for highly efficient removal of methylene blue from water pollution

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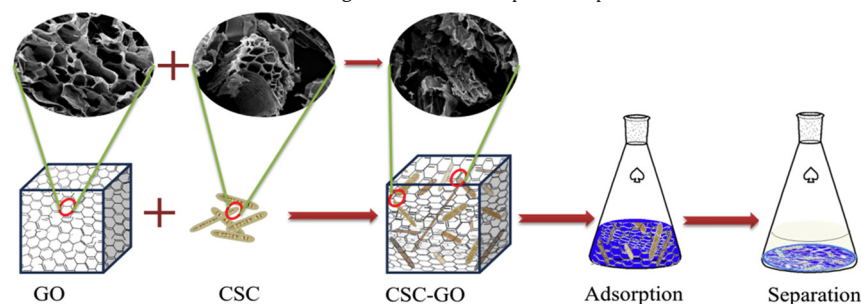
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HIGHLIGHTS

- The highly efficient 3D bio-adsorption was synthesized using a sample assembly method.
- The maximum adsorption capacity was 414.03 mg g^{-1} for MB.
- The adsorption performance obeyed the pseudo-second-order and Temkin models.
- CSC-5GO had an excellent reproducibility and environmental protection.

GRAPHICAL ABSTRACT

The schematic illustration of assembling CSC-GO and adsorption MB process.



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ABSTRACT

The objective of this study was to synthesize a novel, efficient and economical bio-adsorbent with three dimensions (3D) structure using a direct assembly method for removal of methylene blue (MB) from aqueous solution. The raw materials were corn straw core (CSC) and graphene oxide (GO). The variables that affected adsorption capacity were tested. GO loading could facilitate the removal rate. When 5 wt% GO was added (CSC-5GO), the removal rate was increased by 21.62% compared to pure CSC (64.58%). Furthermore, the adsorption by CSC-5GO fitted pseudo-second-order kinetic model ($R^2 > 0.998$) and Temkin model ($R^2 > 0.955$). The maximum adsorption capacity was 414.03 mg g^{-1} for MB at $\text{pH} = 12$, 298 K and MB concentration of 1000 mg L^{-1} . The adsorption thermodynamic test results suggested that the adsorption was a spontaneous, exothermic and randomness decrease process. Furthermore, after five cycles of adsorption-desorption test, the adsorbent removal rate was $>90\%$, which implied that CSC-5GO had an excellent reproducibility.

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1. Introduction

The modern industry promotes the development of dye industry. Dyes are widely used in textile, papermaking, construction, food processing, leather dyeing, plastic and rubber dyeing. The extensive use of dyes produces dye-containing waste water, which is one of the major harmful industrial wastewaters. In the process of material processing, an average of $>15\%$ of dye was dumped into water, which caused

water pollution and a series of problems, including water eutrophication, toxicity and water biological extinction (Tharaneedhar et al., 2017; Saleh and Gupta, 2014; Bhatti, 2015; Mittal et al., 2010a). In addition, pollutants easily enter the human body through food, water, air or absorption through skin when they come in contact with humans in agriculture, manufacturing, pharmaceutical, industrial or residential settings (Gupta and Saleh, 2013). To this end, a variety of biological and physico-chemical methods for wastewater treatment have been developed (e.g., activated carbon, reverse osmosis, and advanced oxidation) (Gupta et al., 2012a). Scientists and researchers have explored different approaches such as sedimentation, flocculation, ion exchange,

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membrane technologies, biological degradation and advanced oxidation processes (AOPs) (Saravanan et al., 2014a; Yang et al., 2017; Gupta et al., 2012b). However, most of these methods present several disadvantages like chemical requirements, low efficiency, and usually produce large amounts of sludge that can add other environmental problems (Saleh and Gupta, 2012a). Photocatalytic degradation has been considered as a green approach to removing contaminants in water. Researchers have been looking for efficient catalysts (Saravanan et al., 2013a; Saravanan et al., 2013b; Saravanan et al., 2013c; Saravanan et al., 2013d; Saravanan et al., 2014b; Saleh and Gupta, 2012b; Gupta et al., 2011a). In addition, catalysts that enable to catalyze pollutants under visible light have been reported (Rajendran et al., 2016; Saravanan et al., 2013e; Saravanan et al., 2013f; Saravanan et al., 2014c; Saravanan et al., 2015a; Saravanan et al., 2015b; Saravanan et al., 2016; Saleh and Gupta, 2011). Adsorption method has been considered as the most appropriate way to separate a variety of dye wastewater due to its low cost, operable, simple design and environmental protection (Mohammadi et al., 2011; Gupta and Nayak, 2012). Therefore, looking for an efficient, economical, pollution-free adsorbent has become the focus (Ge et al., 2016). Scientists and researchers found some high-performance adsorbents, such as modified activated carbon, which could effectively remove dyes or heavy metal ions in water compared to commercial activated carbon (Gupta et al., 1998; Khani et al., 2010; Asfaram et al., 2015).

The most conventional adsorbent is activated carbon which is often used for the purification of gas and liquid systems (Hayashi et al., 2002). Commercial activated carbon's adsorption capacity is 259.6 mg g^{-1} , about three times that of zeolite. The use of agricultural waste to prepare activated carbon has been caused for concern. A biomass activated carbon was prepared using the walnut shell as raw material (Hu and Vansant, 1995). Tian et al. (2016) made the activated charcoal after roasting to produce loose structure. The adsorption capacity of CO_2 could reach 28.6 wt% by using *Enteromorpha prolifera* as the raw material.

Straw is a kind of agro-waste material. About 300 million tons of straw is burnt or discarded a year in China, which causes serious air pollution and soil pollution (Ribeiro et al., 2010). However, straw is a valuable renewable natural resource for wood-plastic composites (WPC) and can be utilized as raw material for the preparation of reinforcing fiber (Chen et al., 2015). It also can be used as adsorbent material and electrode material (Gupta et al., 2015; Xu et al., 2016). There are many active functional groups on the surface of corncob, which can promote the adsorption capacity of dye wastewater. G.T. Li et al. (2016) and Y. L. Li et al. (2016) reported the use of sewer mud and corn stalks as raw materials to prepare adsorbents, whose maximum adsorption efficiency for methylene blue (MB) was 93.8%. Cao et al. (2017) reported the use of corn straw to prepare biomass adsorbents. The adsorption capacity for butanol vapor was 410.0 mg g^{-1} .

Graphene is composed of two-dimensional sp^2 carbon network with a honeycomb crystal structure. It has unusual properties, such as high electron mobility, high opacity due to an atomic monolayer, high

thermal conductivity, and extraordinary mechanical strength (Liu et al., 2012). Graphene oxide (GO) is the product of graphite powder stripping under strong oxidizing conditions. Many researchers have prepared adsorbents using GO as raw material and studied adsorbents' performances. Bi et al. (2012) prepared a spongy graphene (SG) by reducing GO platelets in suspension followed by shaping via moulding, showing an average absorption rate of dodecane of 0.57 g per gram of SG per second. Cheng et al. (2012) prepared a hydrophilic biocompatible 3D chitosan-graphene mesostructured adsorbent, which could remove 97.5% of reactive black 5 at initial concentration of 1.0 mg/mL. M. Zhang et al. (2012) explored the preparation and adsorption effects of the graphene-coated biochar using cotton wood as the raw material with the maximum methylene blue adsorption capacity of 174 mg g^{-1} .

In the process of preparing the above biomass adsorbents, the necessary step to form the final adsorbents was through the high temperature in inert gas, which resulted in significant energy consumption and increasing cost. So far, very few reports have used corn stalk core (CSC) and GO as raw materials for bio-adsorbent without roasting process. The aim of this study was to investigate a novel 3D structure, efficient and low-cost adsorbent by using CSC and GO as raw materials. The study further aimed at investigating the adsorption effect on MB, the adsorption kinetics and the adsorption thermodynamics.

2. Experimental section

2.1. Materials

Graphite powder (8000 meshes, 99.95%), concentrated sulfuric acid, sodium nitrate, zinc chloride and all chemical agents with analytical reagent grade were supplied by Aladdin Industrial Corporation (Shanghai, China). Corn stalk was gained from the countryside of Jinan.

2.2. Pretreatment of CSC

Firstly, CSC was stripped from sun-dried corn stalk by manual operation. The rod-shaped CSC was oven-dried at 70°C for 12 h. Then the dried CSC was smashed into powder by pulverizer, and sieved by 100 mesh sifter. Next, CSC powder (15 g) was immersed in 150 mL ZnCl_2 solution (10%). With continuous mechanical stirring, the above mixture was moved into 85°C water bath for 1 h to destroy the hydrogen bonds and exposure of hydroxyl groups (Cao et al., 2016). After forming the uniform colloid, the mixture was filtered by suction filtration. Finally, the product was successively washed by ethanol and distilled water for three times, and then dried at 70°C until constant weight for further experiment.

2.3. Preparation of CSC-GO

The GO aqueous suspension was prepared using the modified Hummers method (Chen et al., 2014). The CSC-GO was prepared using a direct assembly method. Firstly, the dried CSC was mixed with GO

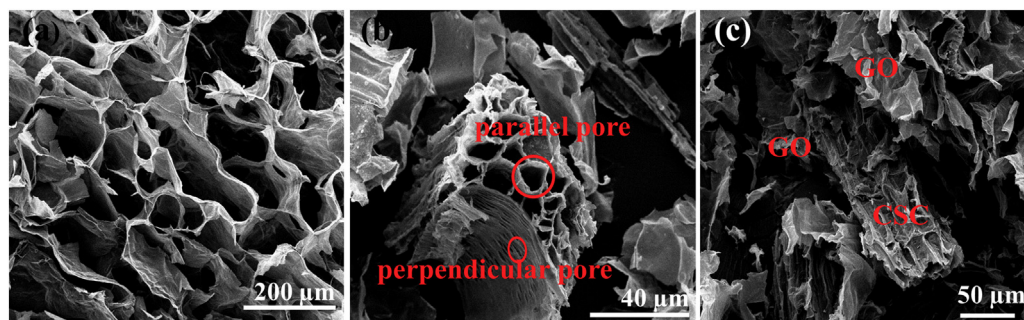


Fig. 1. Characterization of as-prepared adsorbent: (a) SEM image of freeze-dried GO; (b) ZnCl_2 treated CSC; (c) CSC-GO.

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