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Carbon composite lignin-based adsorbents for the adsorption of dyes

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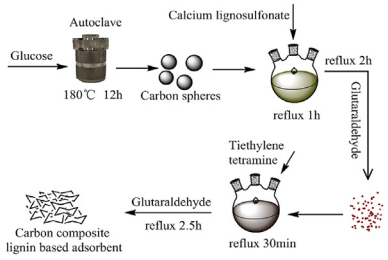
HIGHLIGHTS

- The low-cost calcium lignosulfonate waste was reused.
- The carbon composite lignin-based adsorbent (CCLA) was prepared using glucose as carbon source.
- The fragments stacking structure of CCLA has abundant pores for dye adsorption.
- The CCLA successfully acted as excellent adsorbent for the removal of anionic azo dyes.

GRAPHICAL ABSTRACT



The fabrication process of carbon composite lignin-based adsorbents

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ABSTRACT

Carbon composite lignin-based adsorbent were prepared through hydrothermal method with glucose as carbon source, calcium lignosulfonate and triethylene tetramine as raw materials, respectively. The optimum synthesis conditions were determined by investigating the addition of carbon and triethylene tetramine. The adsorbent was used for the adsorption of azo dyes Congo red and Eriochrome blue black R, and the five factors affecting the adsorption were discussed, including pH of dyes, initial concentration, adsorption time, adsorption temperature and adsorbent dosage. The corresponding adsorption mechanism such as pseudo first order kinetics, pseudo second order kinetics, intraparticle diffusion, Langmuir adsorption isotherm, Freundlich isotherm, Temkin isotherm, Dubinin-Radushkevich adsorption isotherm, thermodynamics were also studied. When the dye concentration is 40 mg L^{-1}, Congo red and Eriochrome blue black R dye removal rates reach 99%. Moreover, the adsorption process of two kinds of dyes follow the pseudo second order kinetics and the Langmuir adsorption isotherm.

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1. Introduction

Dyes are essentially colored organic compounds with very complex structures that can stain other substances (Yagub et al., 2014; Gonga et al., 2005). At present, a large amount of dyes are used in textile, paper, leather, plastics, rubber, food, pharmaceutical and many other industrial fields (Mittal et al., 2010; Wang et al., 2004; Chiou and Chuang, 2006; Hu et al., 2006; Bradder et al., 2011). It is reported that over 700,000 tons of different dyes are used in industrial production and more than 20% of effluent pollution comes from above dyes each year (Babalolac et al., 2016; Banerjee et al., 2017). The discharge of these dye wastewater not only affects the aesthetic nature but also decreases the transmittance of light in water as well as reduces the photosynthesis which leads to the destruction of water ecosystem, and substantial numbers of dyes are toxic that can cause damage to aquatic communities and human body (e.g. central nervous system, reproductive system, kidney, brain and liver) as well as bring about the

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terrible consequences such as carcinogenic, teratogenic, mutagenic and so on (Jiang et al., 2017; Kösters et al., 2018; Salleh et al., 2011; Ahmad et al., 2007; Ozcan et al., 2007; Demir et al., 2008; Abramian and Rassy, 2009). Therefore, one of the main environmental problem today is the removal of dyes from the wastewater because of its own color visibility and toxicity even at low concentrations.

Various treatment technologies are being studied such as chemical oxidation, electro catalytic degradation, reverse osmosis, membrane filtration, coagulation/flocculation, adsorption, microbial degradation for the removal of dyes in the wastewater (Gomez et al., 2007; Inyinbor et al., 2016; Wong et al., 2003; Chinoune et al., 2016; Lata et al., 2007; Li et al., 2016; Blanco et al., 2017). These methods have their own advantages and significant disadvantages, among all the above mentioned methods, adsorption is recognized to be one of the most promising method on account of its simple operation, low cost and high efficiency (Wang et al., 2017; Weng and Pan, 2007; Pala et al., 2017; Garg et al., 2003; Chakraborty et al., 2005). Thus, the adsorption is a simple and economically viable alternative method for the removal of dyes pollution from the aqueous solution. However, it has been found that most of the high efficient adsorbents are expensive for the treatment of pollutants such as the activated carbons (Gupta et al., 2006; Silva et al., 2016). Therefore, the high cost of commercial adsorbents has prompted researchers to develop efficient and environment-friendly alternative materials.

Lignosulfonate is the main by-product of wood hydrolysis industry and paper industry, but industrial lignosulfonate can be recycled from the waste of pulping black liquor. Due to its low price, biodegradability, non-toxicity, environmental friendliness and containing many potential reactive functional groups, the calcium lignosulfonate has a very large value in the development and utilization (Hao et al., 2016; Tan et al., 2012; Won and Borden, 2016; Ouyang et al., 2006a; b; Pang et al., 2008; Matsushita and Yasuda, 2005; Hao et al., 2017; Li et al., 2012; Ouyang et al., 2006a; b). Hydrothermal carbon materials, as the name suggests, is a carbon material obtained by hydrothermal carbonization. Hydrothermal carbonization is a reaction which is the thermochemical conversion that the aqueous solution of biomass undergoes (carbohydrates or natural plant materials) in a specially designed sealed reactor (autoclave) under the condition of saturated vapor pressure at 150–350 °C to obtain black carbonaceous insoluble matter (Sevilla and Fuertes, 2009; Liu et al., 2016; Hu et al., 2010). Hydrothermal carbon obtained from biomass components is inexpensive, mainly spherical, and has micron size, contains sp^2 hybrid carbon skeleton and forms a large number of oxygen-containing functional groups (Chen et al., 2011; Xiao et al., 2017; Ming et al., 2013; Fechler et al., 2013). The material with better hydrophilicity can be further functionalized and well dispersed in water because of the presence of oxygen-containing functional groups.

In this study, the carbon spheres were prepared by hydrothermal carbonization of glucose and then combined with calcium lignosulfonate and modified by amination to prepare lignin based adsorbents. The synthetic process is simple, low cost, easy to operate and less pollution. It can not only manage the environment, but also make rational use of resources and turn waste into treasure.

2. Materials and methods

2.1. Materials

Glucose (AR), glutaraldehyde (AR, 50%), triethylene tetramine (CP), Congo red (AR), Eriochrome blue black R (AR) were purchased from Shanghai Sinopharm Chemical Reagent Co., Ltd. Calcium lignosulfonate (industrial grade) was purchased from Tianjin Fu Chen. All reagents are purchased directly for use.

2.2. Preparation of carbon spheres

First, the glucose solution of 100 mL 0.55 $molL^{-1}$ was prepared and then 80 mL of glucose solution was removed into a Teflon lined autoclave. Then, the autoclave was placed in a 180 °C oven for 12 h. After the reaction, the product was filtered and washed with deionized water and then dried at 60 °C for overnight, and grinded into brown powder as the carbon spheres (Shin and Lee, 2016).

2.3. Preparation of carbon composite lignin-based adsorbent (CCLA)

A certain amount of carbon spheres, 4 g calcium lignosulfonate and 70 mL deionized water were added into the 250 mL three-necked flask, and then dispersed by ultrasound. Then, the three-necked flask was placed in a 60 °C water bath and refluxed for 1 h, after that a certain amount of glutaraldehyde was added continuously in the above solution. The reaction lasted 3 h, after the reaction, filtering and washing to get brown precipitate. Next, brown precipitation above, 6 mL triethylene tetramine, 70 mL deionized water were transferred to 250 mL three-necked flask, then ultrasonic dispersion. And then, three-necked flask was placed in a 60 °C water bath and refluxed for 30 min, after that 5 mL glutaraldehyde was added continuously in three-necked flask, reaction lasted 3 h. After the reaction, filtered and washed by deionized water, and dryed at 60 °C for overnight.

The experimental parameters for preparing carbon composite lignin-based adsorbent under different conditions are presented in Table 1A.

The adsorbents with different amounts of carbon spheres are named as follows, for example the amount of 0.1 g carbon spheres are named CCLA-0.1; the addition of 0.15 g carbon spheres and different amounts of glutaraldehyde are named as follows, for example, the amount of 0.15 g carbon spheres, 9 mL glutaraldehyde

Table 1
Experimental parameters design (A) and the experimental results (B).

A

Number	Carbon sphere/g	CLS/g	Glutaraldehyde/mL
1	0.1	4	5
2	0.15	4	5
3	0.2	4	5
4	0.25	4	5
5	0.3	4	5
6	0.15	4	1
7	0.15	4	3
8	0.15	4	7
9	0.15	4	9

B

Number	Carbon sphere/g	CLS/g	Glutaraldehyde/mL	Carbon content %	Nitrogen content %	Removal rate ([a]CR)%	([b]ER)%
1	0.1	4	5	58.32	18.05	42.13	74.05
2	0.15	4	5	61.22	17.98	98.54	97.91
3	0.2	4	5	58.72	17.66	60.88	81.30
4	0.25	4	5	60.17	17.75	54.19	77.62
5	0.3	4	5	59.54	17.51	35.17	68.88
6	0.15	4	1	60.91	18.18	39.98	69.24
7	0.15	4	3	60.11	18.41	68.46	77.83
8	0.15	4	7	61.89	18.33	80.71	85.33
9	0.15	4	9	61.75	18.72	41.80	74.60
10	–	0.01	–	43.03	0	9.51	11.02

[a] CR: Congo red.
[b] ER: Eriochrome blue black R.

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