



Preparation and evaluation of adsorptive properties of micro-mesoporous activated carbon via sodium aluminate activation



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HIGHLIGHTS

- Sodium aluminate, a novel activating agent, was used to prepare activated carbon.
- The pore properties of ACs were highly affected by activation temperature.
- The obtained ACs owned high surface area and large pore volume.
- The obtained ACs showed a high adsorption capacity towards GR of 1000 mg g⁻¹.

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ABSTRACT

A novel activating agent, sodium aluminate (Na[Al(OH)₄]), was successfully employed to prepare activated carbon with microporous and mesoporous structure from marine *Enteromorpha prolifera* biomass waste. The effects of impregnation ratio, activation temperature and activation time during the preparation process, on the properties of produced activated carbon, including BET surface area, micro-mesopore surface area and average pore size, were investigated in detail. In addition, the pyrolysis behavior during the activation process was simulated by thermo-gravimetric analysis. The BET surface area and pore volume of the produced carbon could reach as high as 1374.3 m² g⁻¹ and 1.150 cm³ g⁻¹, respectively. With the acid brilliant scarlet as model adsorbate, adsorption behavior of the obtained activated carbon was studied, and it was found that, the adsorption isothermal data were better fitted by Langmuir model and the maximum monolayer adsorption capacity towards GR was 1000 mg g⁻¹. This study confirms the potential of the sodium aluminate as a successful activating agent, which could expand the synthesis methods of activated carbon.

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1. Introduction

Large-scale application of synthetic dyes in textile, cosmetic, pharmaceutical, food and other industries have led to serious environmental problems [1]. It is estimated that over 35,000 tons of dyes are discharged into the aquatic ecosystem annually [2–4]. These dyes could result in serious damages to human health, including central nervous system, kidney, brain and liver diseases [1]. Most of dyes are composed of complex aromatic structures, which cause huge difficulty in biodegradable removal of these contaminants. Therefore, many methods, such as coagulation, advanced oxidation, membrane filtration, biological treatment and adsorption, have been used to treat dye wastewater. Among these approaches, adsorption has been proved to be more efficient and favorable due to its low cost, simple design and easy operation [1,3,5].

Activated carbons (ACs) is one of the most promising and widely used adsorbent as a result of their large surface area, good thermal stability and high adsorption capacity. Adsorption properties of ACs depend on raw material, manufacturing method as well as activating agent [1,6]. In fact, wide varieties of waste materials such as hazelnut shell [7], rice husk [8], waste tires [9], fruit stones [10] and municipal waste [11] have been found to be low-cost precursors for the synthesis of ACs. *Enteromorpha prolifera* (EP), as a kind of common alga, is widely abandoned without proper utilization in Asia. The high content of carbon endows this waste with good potential for the preparation of ACs. On the other hand, the selection of an appropriate activating agent plays an important role in physical and chemical characteristics of the produced carbon materials. Generally, physical and chemical methods are used for the preparation of ACs. During the physical process, the raw material is carbonized in the presence of steam, carbon dioxide or air, in which the high temperature and long operation time are required [12,13]. As for chemical process, H₃PO₄ [14,15], NaOH [16,17], KOH

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[18,19], H₂SO₄ [20,21] and ZnCl₂ [22] are usually used as the activating agents, which can produce ACs with higher yield and better developed pore structure while the operation temperature and time are lower than those in a physical process [23]. However, these strong acidic or alkaline chemical agents can corrode the apparatus and shorten their lifetime, which further increase the production cost and limit the development of ACs. Therefore, more researches should be undertaken to explore other chemicals with relatively low corrosiveness as activating agents.

As a common assistant chemical in the water treatment, paper, petrochemical, rubber and textile industries, sodium aluminate (Na[Al(OH)₄]) is usually made of bauxite mineral. To the best of our knowledge, there are no reports concerning the usage of sodium aluminate as an activating agent for the synthesis of ACs. Hence, it is necessary to perform some additional studies to explore more details about this new activating agent. The main purpose of this work was to study the influences of some main preparation parameters, including impregnation ratio, activation temperature and activation time on pore structure of the produced ACs. In addition, the adsorption efficiency of the prepared ACs was demonstrated by using acid brilliant scarlet (GR) as a model adsorbate. The adsorption kinetic and thermodynamic experiments of GR onto the ACs synthesized under the optimum conditions were performed.

2. Experimental

2.1. Chemicals and materials

E. prolifera was collected from the seashore of the First Bathing Beach in Qingdao City (Shandong province, China) and used as raw precursor. The primary properties of EP can be found in some previous studies [13,24–26]. All chemicals were analytical grade, purchased from Sinopharm Chemical Reagent Corporation and used without any further treatment. Distilled water was utilized for preparing all solutions throughout the experiments.

2.2. Preparation of activated carbon

Prior to synthesis, the raw material was ground and sieved to get powder with a particle size lower than 0.425 mm (40 mesh). Initially, EP was carbonized under an oxygen-deficient condition using a sealed chamber type electric resistance furnace (KSY-4D-16, Longkou Electric Furnace Manufactory). The carbonization temperature was ramped from room temperature to 500 °C with a heating rate of 15 °C min⁻¹. The carbonization process was held for 90 min to obtain the char. Then, 10 g of the char was thoroughly mixed with sodium aluminate in different proportions in a least volume of water and stirred unremittingly until the formation of a homogenous slurry. The weight ratio of Na[Al(OH)₄]/char was 1:1, 2:1, 3:1, 4:1 and 5:1. The prepared mixture was dried at 110 °C until constant weight. Next, the mixture was transferred in a nickel boat to the center of a tube furnace (SKQ-3-10, Longkou Electric Furnace Manufactory) [25] and heated to different target activation temperatures (500, 600, 700, 800 and 900 °C) under nitrogen, followed by a heat soak at various duration time (0.5, 1.0, 2.0, 3.0 and 4.0 h). Finally, the resultant product was washed with hydrochloric acid and hot distilled water several times to eliminate the residuals. For convenience, the ACs obtained by sodium aluminate activation was named as SAAC.

2.3. Characterization methods

The thermal decomposition behavior of char after sodium aluminate impregnated was determined by thermogravimetric

analysis (TGA) and differential thermal analysis (DTG) using a TGA analyzer (SHI-MADZU, TGA-50). The sample was heated from room temperature to 800 °C with a heating rate of 15 °C min⁻¹ and held for 240 min. The textural properties of the ACs were performed via nitrogen sorption/desorption isotherms on a JW-BK122W surface area apparatus. The total pore volume was estimated from liquid volume of nitrogen adsorbed at the saturation of relative pressure. The micropore volume and surface area were calculated via *t*-plot method. The mesopore volume and surface area were determined by BJH method. The surface area was derived from the Brunauer–Emmett–Teller method. The pore size distribution was given from the adsorption branch by BJH method.

2.4. Batch adsorption experiments

The effects of contact time and initial dye concentration on the adsorption were investigated. Accurately weighed activated carbons (25 mg) were mixed with 50 mL of dye solution in 250 mL Erlenmeyer flasks. For the kinetic studies, the initial GR concentrations were set at 194, 404 and 592 mg L⁻¹. The sample was withdrawn at predetermined intervals. For the thermodynamic studies, the GR solutions with various initial concentrations in the range of 200–800 mg L⁻¹ were prepared. The samples were equilibrated for 48 h in a water bath shaker (SHA-B, Shanghai, China), filtered, and analyzed by a UV analyzer (UV-754, Shanghai) at 510 nm. The adsorption capacity was calculated by the following equation:

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

where C_0 and C_e are the dye concentration at initial and equilibrium, respectively (mg L⁻¹); q_e is the adsorption capacity at equilibrium (mg g⁻¹); V is the volume of dye solution (L); W is the mass of adsorbent used (g).

3. Results and discussion

3.1. Effect of processing parameters on the pore structure

3.1.1. Effect of impregnation ratio

The effects of impregnation ratio of sodium aluminate to char on the surface area and average pore size of the produced activated carbon were evaluated with the conditions of activation temperature of 800 °C and activation time of 1 h. As observed from Fig. 1a, sodium aluminate is an efficient activating agent to produce activated carbon with high surface area. Like other activating agents, sodium aluminate played a role of dehydration agent on the raw materials during the activation process, which can prevent the removal of large amount of organic carbon in the form of CO, CO₂, CH₄ and inhibit the accumulation of tars on carbon surface, resulting in the development of new pore network [27,28].

It is interesting to find that the impregnation ratio could affect the pore development greatly, with the impregnation ratio increased from 1:1 to 3:1, the BET surface area of the prepared activated carbon increased from 888 to 1445 m² g⁻¹, and then decreased slightly when the impregnation ratio was higher than 3:1. The maximum BET surface area of 1445 m² g⁻¹ was obtained at the impregnation ratio of 3:1. Therefore, the ratio of 3:1 was chosen for further experiments in this study. This result is consistent with some other reports, in which the activating agents, such as potassium hydroxide, pyrophosphoric acid and zinc chloride were used for preparing activated carbons [22,23,29].

More activating agent diffused into the precursor matrix could intensively accelerate the reaction, which could develop a great

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