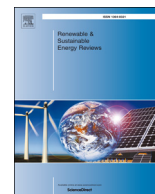




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Microwave assisted preparation of activated carbon from biomass: A review

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ABSTRACT

Activated carbon has been widely used as adsorbents, catalyst support and energy storage materials. Microwave heating is a promising technique for thermochemical treatment and activation of activated carbons due to its fast, selective, uniform and volumetric heating. Previous reviews mainly focused on specific feedstocks (e.g. agricultural biomass or oil palm wastes) or ignored important parameters for microwave activation (e.g. mass of activated carbon precursor). Characteristics of activated carbons prepared from conventional and microwave treatment were not compared and economics of microwave activation were not described in previous studies. This paper presents a state of the art review of activation under microwave irradiation for various biomass sources (e.g. agricultural wastes, woody biomass and sewage sludge). Characteristics of microwave heating, main activation parameters, research progress and technology status of activated carbon preparation and regeneration under microwave irradiation are investigated with key challenges and economics being addressed. Characteristics of activated carbons made from microwave irradiation and conventional activation are compared and the advantages of microwave-assisted preparation are highlighted. It shows that the BET (Brunauer–Emmett–Teller) surface area, iodine number, methylene blue adsorption capacity and the yield of activated carbons under microwave irradiation are up to 2500–3000 m²/g, 1800–2200 mg/g, 500–700 mg/g and 13–96 wt%, respectively, competitive with conventional methods on the quality of activated carbons and process economics. Potential applications and future perspectives are described with pertinent information on activated carbon preparation under microwave irradiation.

1. Introduction

Activated carbon (AC) has been proven to be an effective adsorbent for removing a wide variety of organic and inorganic pollutants, polar and non-polar compounds in the aqueous phase or from gaseous environment [1,2], and has also been used in energy storage fields [3]. AC has large porous surface area, tunable pore structure and surface chemistry, good thermostability at high temperatures in inert or reduction atmospheres, low acid-base reactivity and receives much attention owing to its superior and efficient ability in air pollution control [4–6], solvent recovery [7], food processing [8], wastewater treatment (e.g. dyes, heavy metals, detergents, herbicides, pesticides and polyaromatic hydrocarbons) [4,9–12], metal recovery [13], catalysis [14–16], electrode materials for supercapacitors, porous matrix to host active substances for cathodes [17], as well as improving odor and taste [18,19]. AC is generally produced from woody biomass, agricultural wastes and coal by pyrolysis and activation at elevated temperatures,

such as sawdust [20], coconut shells, fruit stones and peels [21–26], peat [27] and bituminous coal [28].

AC consists of functional groups bonded to fused-aromatic rings, which would be expected to possess chemical properties similar to those in the aromatic hydrocarbons. Surface functional groups on the carbon matrix can be manipulated and adjusted by thermal, hydrothermal or chemical treatments for particular functions. Pyrolysing hydrothermal char in the presence of KOH increased the surface area from 10 m²/g to > 1200 m²/g, due mainly to removal of acidic side chains that would block pore access [29]. Hydrothermal chars have higher oxygen content than pyrolysis chars and have shown promises for adsorption of metal cations from aqueous solutions (e.g. Cu and Cd), [29]. However, ACs prepared from hydrothermal chars were not systematically investigated in previous studies. The sorption performance and pore characteristics of ACs depend on the physical and chemical properties of precursors as well as activation techniques [30]. Various carbonaceous solids, either natural or synthetic, can be used as

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| Nomenclature | | DS | Date stones |
|--------------|---|-----|--|
| AC | Activated carbon | MB | Methylene blue |
| BET | Brunauer–Emmett–Teller | MSW | Municipal solid waste |
| BUCT | Beijing University of Chemical Technology | MW | Microwave |
| C-C JCBERI | The China-Canada Joint Centre for BioEnergy Research & Innovation | MWA | Microwave-assisted absorbers |
| DLTP | Dielectric loss tangent parameter | MWP | Microwave-assisted pyrolysis |
| | | NG | Natural gas |
| | | UBC | University of British Columbia, Canada |

precursors for preparation of ACs. The choice of precursor is largely dependent on its availability, cost, purity, manufacturing processes and intended application of the product. Agricultural residues, forestry wastes and sewage sludge are considered as important precursors because they are cheap, renewable and available in large quantities [31]. In addition, agricultural and forestry wastes have high carbon and low ash contents (0.2–10 wt%) [32–34].

Physical and chemical activations are common techniques to prepare ACs. Chemical activation is usually preferred due to the simplicity, shorter activation time, higher yield, lower temperature and better development of the porous structure. Recently, microwave (MW) activation becomes a viable alternative to conventional methods in preparation of ACs, due mainly to its unique characteristics such as fast, selective, uniform and volumetric heating, instant and accurate control, non-direct contact between the heating source and heated materials. The equipment size is reduced owing to the fast reaction rate under MW irradiation [35]. MW heating increases the carbon yield, improves the AC quality, provides high energy efficiency, minimizes formations and emissions of hazardous materials, thereby rendering the technique environmentally friendly [36–39].

Detailed comparisons of characteristics of ACs made from MW activation and conventional techniques are not provided and economics of MW activation are not addressed in previous studies. Previous reviews either focuses on specific feedstocks (e.g. agricultural biomass or oil palm wastes) or ignores important parameters of MW activation (e.g. mass of AC precursor activated in the reactor under MW irradiation). This paper presents a comprehensive review of AC activation under MW irradiation for different biomass sources (e.g. agricultural wastes, woody biomass and sewage sludge), and summarizes research progress and technology status of AC preparation under MW irradiation. The review compares characteristics of ACs made from conventional methods and MW activation and the advantages of MW-assisted preparation of ACs are highlighted. The MW heating characteristics, main activation parameters, key challenges and future perspectives are described, providing the readers with pertinent information on MW-assisted preparation of ACs.

2. Activated carbon from conventional methods and the drawbacks

2.1. Conventional methods for activated carbon preparation

Carbon materials with low oxygen content, basic properties, highly hydrophobic characteristics and resistance to aging can be obtained by appropriate thermal treatments in different gaseous environments [40–42], which is usually achieved by convective, conductive and radiative heating of the AC precursor in fixed bed (e.g. tube reactor and vertical shaft kiln), moving bed (e.g. rotary kiln and multiple hearth furnace) or fluidized bed reactors. Conventional AC preparation methods are mainly physical activation, chemical activation and combinations of the above techniques (Table 1). The temperature, reaction time and impregnation ratio are all important variables for the extent of carbonization reactions, and the textures and properties of the produced ACs [43]. Selection of appropriate activation techniques and optimization of parameters could improve AC properties via

development of porosity and favorable surface functionalities [44]. Conventional activation techniques, kinetic models for pyrolysis and activation, applications and uses of agricultural residues based ACs are reviewed [45]. However, the textural properties and characteristics of ACs (e.g. specific surface area) are not linked to the corresponding process parameters. The modification of AC surface chemistry has been frequently the target of a variety of AC beneficiation treatments. Oxidation is one of the most conventional modifications used for ACs. Oxidation methods involve the utilization of oxidizing gases or oxidizing solutions (i.e. nitric acid, hydrogen peroxide, chlorine water, etc.) to introduce carbon–oxygen surface groups in ACs [46]. Research aiming at modifying the surface chemistry of ACs should pay much attention to a parallel study of the porous structure since porous structure considerably affects the behavior and performance of ACs. Textural characteristics, surface chemistry and oxidation of ACs have been reviewed and interpreted by Daud and Houshamnd [47]. It was reported that the changes in surface chemistry and textural characteristics of ACs treated by a specified oxidant were not similar, and in some cases, were in conflict. The inconsistencies were possibly attributed to the diversity of used ACs, oxidants, oxidation methods, treating procedures, as well as analysis methods [47].

2.2. Heating rate and non-uniformity of heating

The heating techniques strongly affect the physical and chemical characteristics of ACs. For conventional heating (e.g. electric, steam and flue gas heating), the heat source heats the carbon bed from outer surface to its interior through convection, conduction, and radiation mechanisms, and results in surface heating of the bed and different temperature distribution for particles of different shapes and sizes, generating a temperature gradient from the hot surface to the interior [81]. Some authors provided information of particle size, shape and moisture content of AC precursors, others didn't even mention these parameters [82]. Particle size, shape and moisture content affect heating rate and temperature profile of bulk solids and the individual particles. The surfaces, edges and corners are generally hotter than the inside of the particle. Small particle size (e.g. < 0.2 mm) and dry AC precursors are beneficial to avoid uneven heating. However, size reduction and drying are energy intensive processes and increase the total cost of ACs production. Gas-solid contacting characteristics of fixed bed and peculiar properties of AC precursors always lead to non-uniform heating of bulk solids and individual particles.

To avoid this thermal gradient, a slower heating rate at intermediate final temperatures with isothermal holding is generally used, increasing the duration of the preparation process, thereby resulting in greater energy consumption and higher expenses [81,83,84]. The thermal gradient impedes the release of pyrolysis gases to the surroundings. Hence some volatile components may remain inside the particles, giving rise to carbon deposition problems. The deposited carbon is possible to obstruct the microporous network, leading to distortion and inhomogeneous microstructure, low values of total pore volume and BET surface area [19,85–87]. Consequently, the quality of AC products is frequently inferior to the desired result [35,85]. Fast firing is also a disadvantage of the conventional heating method, depending on different AC precursors, process configurations, reactor types and

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