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Plam oil empty fruit bunch based magnetic biochar composite comparison for synthesis by microwave-assisted and conventional heating

N.M. Mubarak[a,*], J.N. Sahu[b,c,d,*], E.C. Abdullah[e], N.S. Jayakumar[f]

[a] Department of Chemical Engineering, Faculty of Engineering and Science, Curtin University, 98009 Sarawak, Malaysia
[b] Department for Management of Science and Technology Development, Ton Duc Thang University, Ho Chi Minh City, Vietnam
[c] Faculty of Applied Sciences, Ton Duc Thang University, Ho Chi Minh City, Vietnam
[d] Petroleum and Chemical Engineering Programme Area, Faculty of Engineering, Universiti Teknologi Brunei, TungkuGadong, P.O. Box 2909, Brunei Darussalam
[e] Malaysia – Japan International Institute of Technology (MJIIT), Universiti Teknologi Malaysia, Jalan Semarak, 54100 Kuala Lumpur, Malaysia
[f] Department of Chemical Engineering, Faculty of Engineering, University of Malaya, 50603 Kuala Lumpur, Malaysia

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ABSTRACT

One of the most abundant residues is empty fruit bunch (EFB), which are left behind after removal of oil palm fruits in the oil refining process. In this study, we investigated the potential of converting palm oil residue into magnetic biochar composite (MBC) using microwave-assisted and conventional heating. The effect of process parameters for the production of MBC using microwave-assisted and the conventional method are compared. The results revealed that magnetic biochar composite exhibited excellent ferromagnetic property with a saturation magnetization of 8.16 and 4.20 emu/g using microwave and conventional heating respectively. Microwave-assisted instead of the conventional heating in the muffle furnace can be applied to reduce production cost of magnetic pyrolytic char preparation. Finding the optimal operating conditions to prepare magnetic pyrolytic char, preparation time 11 min against 2–5 h of reactivation for conventional method. Microwave-assisted offers several advantages over conventional heating, as it is often more controllable energy and cost efficient and therefore in many cases may offer a potentially attractive alternative to “conventional” pyrolysis. Furthermore, textural properties were investigated using nitrogen adsorption, and found microwave-assisted MBC is higher than conventional heating. The novelty is that the MBC can be directly produced using microwave-assisted by single stage of activation compared to the conventional method which requires multiple stage heating.

1. Introduction

Oil palm production is a major agricultural industry in Malaysia. Currently there are more than three million hectares of oil palm plantations. In total, about 90 million metric tons of renewable biomass trunks, fronds, shell, palm press fiber and empty fruit bunch (EFB) [42]. Among these EFB 12.4 million ton/year [42] and palm shell 2.4 million ton/year [35] waste are produced. A small quantity is used as fuel for the boilers in the oil palm mills while most are unused and disposed in landfills. The burning of biomass caused emission of hazardous and toxic chemicals such as dioxins. However, due to limited landfill sites as well as the additional treatment of the leachate from the landfill, studies of effective utilization and recovery of the EFB have stimulated interest in converting waste materials into commercially valuable products.

Biochar, such as wood charcoals and crop residue-derived chars, refer to the carbon-rich residues from pyrolysis or incomplete combustion of biomass [8]. Agricultural wastes or residues including corncob, elephant grass and rapeseed oil cake, are wide available low-cost raw material to produce biochar, as well as bio oil and gases [45]. In the removal of hazardous contaminants from industrial waste waters from, among others, metal finishing, electroplating, plastics, pigments and mining industries, there is the distinct tendency to replace the costly activated carbons (ACs) and ion exchangers (IXs) with various low cost adsorbents, which have metal binding capacities [49]. Increasingly recognized as a multifunctional material, biochar has been explored for agricultural and environmental applications [9]. Biochar amendments can improve soil fertility for crop production by enhancing the retention of fer-

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[*] Corresponding author. Tel.: +60 85443939x3833; fax: +085 443837.
E-mail addresses: mubarak.yaseen@gmail.com, mubarak.mujawar@curtin.edu.my (N.M. Mubarak), jayanarayansahu@tdt.edu.vn, jay_sahu@yahoo.co.in (J.N. Sahu).

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tilizers and encouraging the host of beneficial microorganism [9]. Due to its capability for carbon storage and reducing or suppressing CO_2, CH_4 and N_2O production in soil [23], biochar may contribute to the atmospheric greenhouse gas reduction. Furthermore, Biochar in recent year has been recognized as low cost suitable material for environmental applications [39,9,34,7]. However, powdered biochar/activated carbon has a higher removal efficiency, but in practical industrial application, it is very difficult to be separated from the aqueous solution [24]. In spite of its high adsorption capacity, the use of AC on a large scale is limited by process engineering difficulties such as the dispersion of the AC powder and the cost of its regeneration [24].

An innovative technology developed recently to remove various pollutants, including phosphate, heavy metals, and organic compounds, from aqueous solutions [50,51]. Introducing magnetic biochar, to the commercial sorbent (e.g., active carbon and carbon nanotubes) by chemical co-precipitation is an efficient method, to enable the sorbent to be effectively separated by magnetic separating technique [54]. Furthermore, the combined magnetic medium offer a potential to add the functions of bulk magnetic sorbent [46] such as the strong sorption affinity of phosphate [26] selenium [30] and organic arsenic [52] with magnetic iron oxide. However, the traditional loading-process of magnetic medium increased the cost of the sorbent [53].

In recent years, microwave irradiation has attracted the attention of chemists due to its capability of molecular level heating, which leads to homogeneous and quick thermal reactions [1,29,40]. Microwave heating offers several advantages over conventional heating, as it is often more controllable [21] energy [17] and cost [31] efficient and therefore in many cases may offer a potentially attractive alternative to "conventional" pyrolysis systems. The thermal conversion of the bio material requires a uniform heating to maintain the overall quality of the biochar. The heating process should also be fast to reduce the production cost. Microwave heating can be used to heat the bio-materials fast and uniformly throughout the bulk. In addition, microwave technology allowed the carbon to be recycled and reused for a large number of times. This technique does not damage the carbon; rather, it increases the surface area allowing more contaminates to adhere, thereby increasing the value [2]. The main difference between microwave devices and conventional heating systems is in the way the heat is generated [3]. In the former approaches, thermal regeneration is conventionally performed in rotary kilns or vertical furnaces, the heat source is located outside the carbon bed, and the bed is heated by conduction and/or convection. A temperature gradient is established in the material until conditions of steady state are reached. The microwave penetrates the material and the microwave energy is converted to heat energy. In this way heat is generated throughout the bulk of the material. This can reduce the processing time and the overall quality is improved [5,20,4]. To our knowledge, no study with magnetic biochar production using agricultural waste biomass into valuable product has not yet investigated reported.

In this study, magnetic biochar composite (MBC) was produced using discarded material such as EFB by impregnating with $FeCl_3$ and then thermal pyrolysis of the impregnated EFB with the help of microwave and conventional heating technique was compared. The effect of process parameters was compared to produce the high BET surface area and high yield of MBC.

2. Material and methods

2.1. Raw material

The empty Fruit bunch was obtained from the Sime Darby in Dengkil, Selangor, Malaysia and stored at 4 °C. The EFB sample was first washed several times using tap water and finally with distilled water. The EFB sample dried at 105 °C for 24 h in oven for dehydration until a constant weight was obtained. Ferric chloride hexahydrate ($FeCl_3{\cdot}6H_2O$) were purchased from Merck and used as received.

2.2. Production of MBC using microwave-assisted

The dried biomass were crushed and sieved to a particle size of less than 150 μm. The impregnation ratio (IR) of ferric chloride hexahydrate to biomass for different amount of the sample was prepared. The mixing was performed in a thermal shaker at controlled temperature (30 °C) for a period of 3 h at 2.5 Hz using 500 mL beaker. After mixing, the slurry was subjected to vacuum drying at the 100 °C for 24 h. The pyrolysis of the biomass was carried out in a HAMiab-C1500 microwave muffle system oven. About 20 g of prepared sample was placed inside the quartz tube (35 mm OD, 38 mm ID and 500 mm length). The effect of the process parameters such as reaction time, microwave power and impregnation ratio, with the gas flow rate of nitrogen is 0.2 mL/min was optimized to produce high BET surface area and high yield of magnetic biochar. After the reaction sample was cooled down to room temperature and final weight taken to determine the yield of the product. The product was washed with distilled water until the pH becomes neutral. Finally the sample was stored in a tightly closed bottle.

2.3. Production of MBC using conventional heating furnace

The dried biomass were crushed and sieved to a particle size of less than 150 μm. It was then impregnated at ratios of 0.2–0.8 of ferric chloride hexahydrate to biomass for different amount of the sample was prepared, for 3 h at 2.5 Hz using 500 mL beaker. After mixing, the slurry was subjected to vacuum drying at the 100 °C for 24 hThe EFB were carbonized and chemical activated in a high temperature horizontal furnace (Carbolite, UK) in which the inside temperature varied from 100 to 1200 °C. About 20 g of prepared sample was placed in the middle of a quartz tube (OD 75 mm, ID 70 mm, and 1000 mm length). The tube was inserted in the horizontal furnace and its two ends were sealed by inlet and outlet metal enclosures. The effect of the process parameters such as reaction time, carbonization temperature and impregnation ratio, with the gas flow rate of nitrogen is 0.2 mL/min were optimized to produce high BET surface area and high yield of magnetic biochar. The magnetic brochure samples produced were taken out of the quartz tube, after the reaction sample was cooled down to room temperature and final weight taken to determine the yield of the product. The product was washed with distilled water until the pH becomes neutral. Finally the sample was stored in a tightly closed bottle.

2.4. Characterization of MBC

2.4.1. FTIR and BET analysis of magnetic biochar under optimum conditions

The magnetic biochar at optimal production conditions was examined by Fourier Transform Infrared (FTIR) spectroscope (Brand: Bruker, Model: IFS66v/S) was used to analyze the magnetic biochar for determination of the surface functional groups. The chemisorbed oxygen as a different functional group influences the adsorption behavior of the magnetic biochar, as done by the porosity. Acidic as well as basic properties were present for the surface oxides [6]. Physical characterization of magnetic biochar produced at the optimum condition was analyzed with Autosorb 1 surface area analyzer by nitrogen adsorption at −77 K. Prior to analysis, the samples were degassed at the 200 °C for 3 h. The surface area was calculated by the BET (Brand: Quanta Chrome Model: Autosorb 6B) equation using the nitrogen adsorption data. The adsorbed nitro-