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Characterization of activated carbon prepared from oil palm empty fruit bunch using BET and FT-IR techniques

A.R. Hidayu[a]*, N.F. Mohamad[a], S. Matali[a], A.S.A.K. Sharifah[a]

[a]*Faculty of Chemical Engineering,Universiti Teknologi MARA, Shah Alam 40450, Malaysia*

Abstract

Activated carbon has been known as an excellent adsorbent and is widely used due to its unique characteristics and large adsorption capacity. In this study, activated carbon produced from oil palm empty fruit bunch by steam activation was used. The activated carbons were analyzed using nitrogen adsorption isotherm as BET for specific surface area and Fourier transform infrared (FT-IR) spectroscopy. The results showed that the activated carbon at these optimum conditions; 765^{o}C activation temperature and 77 min activation time, possesses a large apparent surface area (S_{BET} = 720 m^2/g), total pore volume (0.341cm^3/g) with average pore size diameter of 18.99 Å. FT-IR results indicate that all the oil palm empty fruit bunch was successfully converted to carbon.

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Keywords: Oil palm empty fruit bunch; Physical activation; Activated carbon; Physical-chemical properties

1. Introduction

Activated carbon is one of the most effective, versatile and useful adsorbents for the removal of pollutants from polluted gas and liquid streams because of their large adsorption capacities, extremely high surface areas, well developed porous structures, fast adsorption kinetics and good mechanical properties [1-3]. The most precursors used for the preparation of activated carbons are organic materials that are rich in carbon [4]. Therefore, the development of methods to reuse waste materials as activated carbons is greatly desired and offers a promising future. Agricultural wastes, such as jathropha, corn cob, coconut shell, oil palm fiber, date stone, wood sawdust and etc are of interest as activated carbons because of their high mechanical strength and hardness. These properties can be explained by high lignin, high carbon and low ash content [3, 5]. In addition, conversion of agricultural wastes to value added product such as activated carbon is a sufficient method to solve environmental problem.

* Corresponding author. Tel.: +6-013-993-3790; fax: +6-035-543-6300
E-mail address: hidayu_rani@yahoo.com

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The global production of palm oil increased more than nine-fold since 1980 to 45.1 million tons in 2009 where Malaysia and Indonesia leading as producers contributed about 85 % of the world palm oil production [6]. Malaysia accounts 41.3% of global palm oil production and exporting about 44% throughout the world in 2010 [7]. Due to the growth of palm oil production in Malaysia and despite high economic return to the country, this industry also generates a huge amount of waste, such as empty fruit bunches (EFB) which have negative impact to environment. Since the EFB is high carbon content and rich in lignin, it has the potential to be a good precursor for the production of activated carbon, which is the most popular and suitable adsorbent used in the industries for a variety of purification and environmental purposes such as recovery of solvent, separation of gases, sewage treatment, and removal of organic pollutants [8].

The process for producing activated carbons involves two stages, which are carbonization and followed by activation. The first stage is to enrich the carbon content in order to create an initial porosity in the char. Meanwhile, activation process helps in developing the pore structure. In activation process, activated carbon can be either a physical or chemical activations. In physical activation, the prepared char is reacted with oxidizing gases, generally CO_2 or steam at high temperature. In chemical activation, the starting material is impregnated with a chemical reagent and is heated in an inert atmosphere [9, 10].

Hence, the goal of this study was to analyze the activated carbon produced from oil palm EFB by physical steam activation at optimum conditions; activation temperature of 765^{o}C and activation time of 77 min that was obtained using response surface methodology (RSM) technique.

2. Material and methods

2.1. Raw material

The oil palm empty fruit bunches (EFB) was supplied by the Meru Palm Oil Mill Sdn Bhd in Klang, Selangor. The materials were cleaned with distilled water several times to remove dust and impurities. Then, EFB samples were dried in oven at 110^{o}C for 24h to remove any surface moisture. The dried EFB were crushed with a grinder and sieved to a small particle sizes.

2.2. Activated carbon preparation

The activated carbon from oil palm EFB was prepared by physical steam activation in a vertical stainless steel reactor. The superheated steam was injected at the flow rate of 120ml/hr, activation temperature of 765^{o}C and activation time of 77 min.

2.3. Characterizations of activated carbon

In this experiment, the physico-chemical characteristics of the activated carbon from EFB prepared under the optimum conditions were determined. Proximate analysis was performed according to ASTM D7582-10 and the results are showed in terms of moisture, volatile content, fixed carbon and ash contents. Ultimate analysis were carried out using CHNS-O Analyzer model FlashEA, 1112 Series to determine carbon, oxygen, hydrogen, and nitrogen contents of the samples.

In order to determine surface area, automated gas adsorption analyzer, AUTOSORB-1 (Quanta Chrome Instruments, USA) is used with adsorption-desorption isotherms of nitrogen at -196^{o}C. For each analysis, 0.2g of sample was used. The samples were degassed at 300^{0}C under nitrogen for at least 3h. The specific surface areas of sample was calculated by the BET (Brunauer, Emmett, and Teller) method while volume of micropore was estimated using the Dubinin Radushkevich (DR) equation. For pore volume, it was directly calculated from the volume of nitrogen held at the highest relative pressure (P/Po = 0.99) [3].

The surface functional groups of raw EFB and activated carbon EFB were ananalyzed using Fourier Transform Infrared (FT-IR) spectroscopy (Perkin Elmer Spectrum One model Spectrometer, England). Firstly, the samples were mixed with potassium bromide (KBr) and the mixture was pressed as a pellet prior to analysis. The IR spectrum was obtained at a resolution of 4cm^{-1} over the range of 500-4000 cm^{-1}.

3. Results and discussions

3.1. Proximate and ultimate analysis of raw sample and activated carbon

The results of the proximate and ultimate analysis of raw EFB and activated carbon EFB which physically activated under the optimum preparation conditions are given in Table 1. According to De et al. (2013), the high fixed carbon and volatile contents of EFB make this material a good precursor for production of activated carbon [11]. During physical activation, the fixed carbon content increased from 18.67 wt% to 67.66 wt%. This is because at high temperature, volatile matter content was released [3]. Plus, physical (steam) activation also increases the carbon content from 48.48 wt% to 68.32 wt% due to the increased release of volatile matter.

Table 1. Proximate and ultimate analysis of empty fruit bunch (EFB) under optimum conditions

Sample	Proximate analysis (wt%)				Ultimate analysis (wt%)			
	Moisture	Volatile	Fixed carbon	Ash	C	H	N	O*
Raw EFB	2.44	73.63	18.67	5.26	48.48	7.14	0.64	43.74
Activated carbon EFB	7.53	15.23	67.66	9.58	68.32	3.12	2.12	26.44

* Oxygen by difference

3.2. N_2 adsorption isotherm in activated carbon

The N_2 adsorption isotherms of the activated carbon is shown in Fig.1. From this figure, activated carbon display type I isotherms and according to International Union of Pure and Applied Chemistry (IUPAC) classification, it indicates that the activated carbon is microporous. Usually, Type I isotherm display a convex curve and the platform of this type come out horizontal or virtually horizontal, and the adsorption isotherm directly intersects with the line P/Po = 1 [8, 12]. As can be seen in initial stage, the volumes adsorbed increase sharply at low relative pressure region (P/Po < 0.2). This means that nitrogen molecules are adsorbed mainly in the microporous structure [13].

Table 2 present the BET surface area, total pore volume, micropore volume and average pore diameter of raw EFB and activated carbon EFB that also obtained from N_2 adsorption isotherms. The sample show a high proportion of micropore volume (about 90% of total pore volume) and means that activated EFB carbon indicates a higher volume of wide micropores and presence of small mesopores.

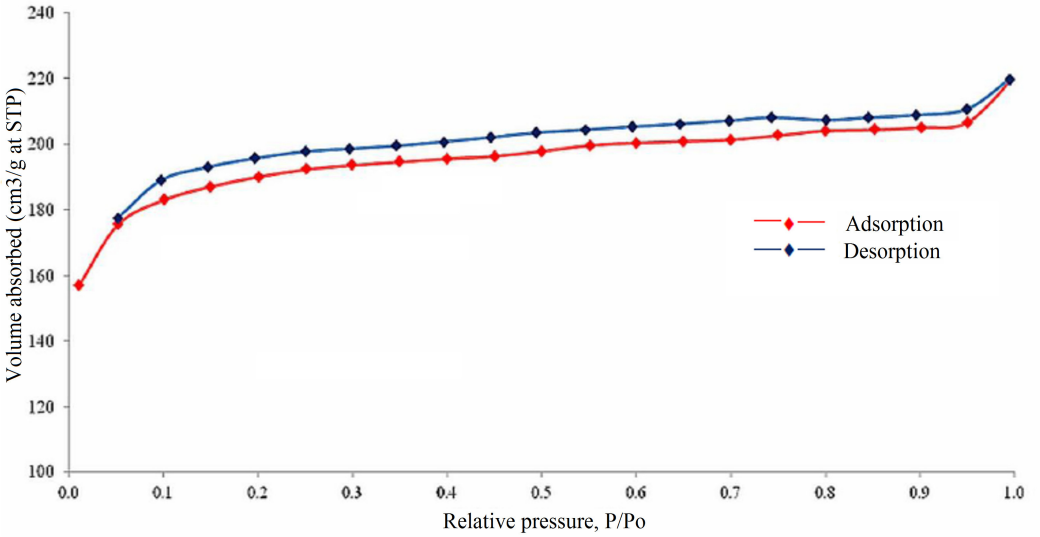

Fig. 1. N_2 adsorption-desorption isotherms of activated carbon

Table 2. BET surface area, total pore volume, micropore volume and average pore diameter of activated carbon

Sample	BET surface area (m^2/g)	Total pore volume (cm^3/g)	Micropore volume (cm^3/g)	Average pore diameter (Å)
Raw EFB	2.005	0.013	0.001	265.400
Activated carbon EFB	720.000	0.341	0.304	18.890

3.3. Fourier Transform Infrared (FT-IR) Spectroscopy

The FT-IR spectra of the raw material EFB and activated carbon EFB (AC-EFB) were given in Fig. 2a and 2b respectively. Based on the figure, raw EFB (Fig. 2a) shows the most complicated and clear spectrum. It is observed a broad adsorption peak of raw EFB at 3302 cm^{-1} which attributed to O-H stretching functional group and this indicates the presence of bonded hydroxide in the raw EFB. The bands at 1739 cm^{-1} was corresponding to C=O functional group. The EFB sample also shows two important adsorption peaks at 1216 cm^{-1} and 1032 cm^{-1} which refers to C-O stretching functional group.

For activated carbon EFB (Fig. 2b), obviously, during the carbonization and activation process most of the adsorption peaks of functional groups were disappeared. This is because the functional groups from the raw material spectrum were vaporized as volatile materials when heat was supplied to the sample [14]. This proved that the activation process has taken place successfully. In fact to confirm the statement, the commercial activated carbon (commercial AC) spectrum (Fig. 2c) was used as the reference sample. As a result, the trend of the activated carbon EFB was almost similar with the commercial activated carbon. Thus, this shows that the prepared activated carbon was successfully converted into carbon.

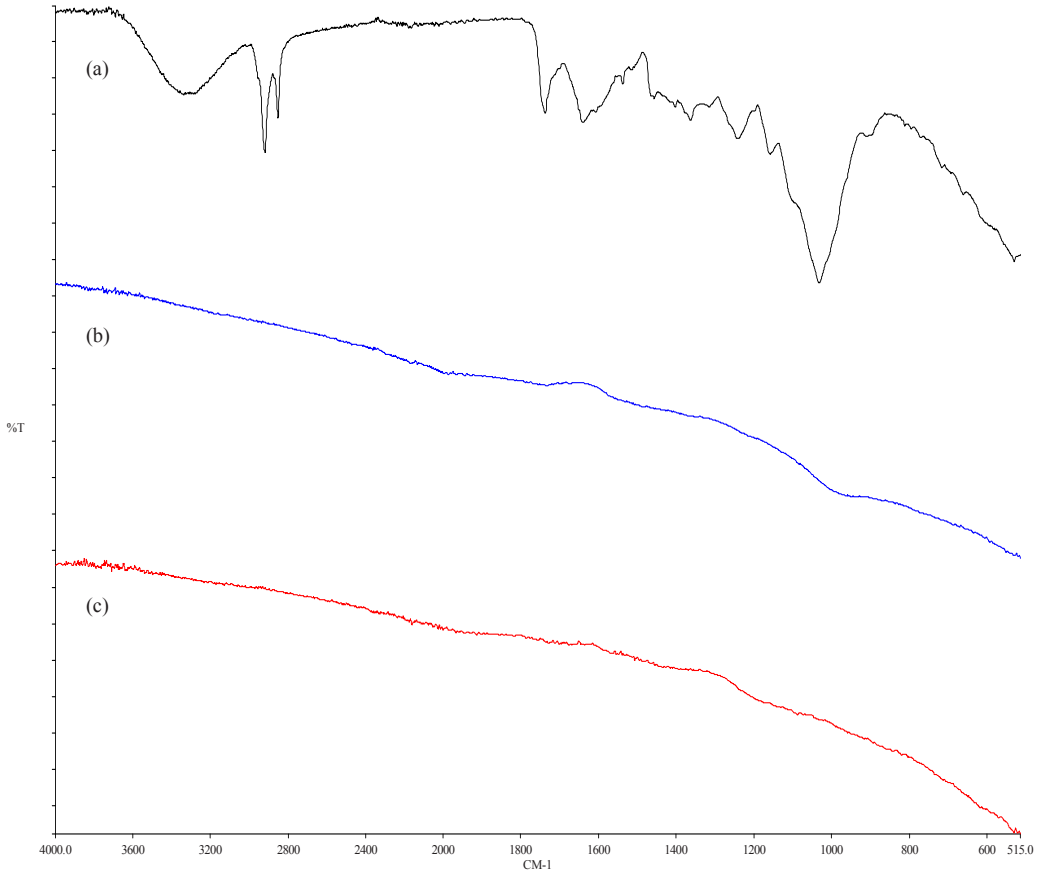

Fig. 2. FT-IR spectra of (a) raw EFB, (b) AC-EFB and (c) commercial AC

4. Conclusions

The characterizations of activated carbon prepared under optimum conditions from oil palm empty fruit bunch (EFB) was analyzed in terms of proximate analysis, ultimate analysis, BET surface area and FT-IR analysis. The BET surface area of activated carbon obtained was significantly high at 720 m^2/g and it is within the acceptable range of commercial activated carbon (500-1500 m^2/g). FT-IR results indicated that all the raw EFB were successfully converted into carbon and it is proven by the spectra of commercial activated carbon. As a conclusion, oil palm EFB can be utilized as a cheap raw material for the production of activated carbon.

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