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Research article

Conversion and characterization of activated carbon fiber derived from palm empty fruit bunch waste and its kinetic study on urea adsorption

Chee-Heong Ooi [a], Wee-Keat Cheah [a], Yoke-Leng Sim [b], Swee-Yong Pung [a], Fei-Yee Yeoh [a, *]

[a] School of Materials and Mineral Resources Engineering, Transkrian Engineering Campus, Universiti Sains Malaysia, 14300 Nibong Tebal, Penang, Malaysia
[b] D115M, Department of Chemical Science, Faculty of Science, Universiti Tunku Abdul Rahman, Jalan Universiti, Bandar Barat, 31900 Kampar, Perak, Malaysia

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ABSTRACT

Urea removal is an important process in household wastewater purification and hemodialysis treatment. The efficiency of the urea removal can be improved by utilizing activated carbon fiber (ACF) for effective urea adsorption. In this study, ACF was prepared from oil palm empty fruit bunch (EFB) fiber via physicochemical activation using sulfuric acid as an activating reagent. Based on the FESEM result, ACF obtained after the carbonization and activation processes demonstrated uniform macropores with thick channel wall. ACF was found better prepared in 1.5:1 acid-to-EFB fiber ratio; where the pore size of ACF was analyzed as 1.2 nm in diameter with a predominant micropore volume of 0.39 $cm^3\ g^{-1}$ and a BET surface area of 869 $m^2\ g^{-1}$. The reaction kinetics of urea adsorption by the ACF was found to follow a pseudo-second order kinetic model. The equilibrium amount of urea adsorbed on ACF decreased from 877.907 to 134.098 mg g^{-1} as the acid-to-fiber ratio increased from 0.75 to 4. During the adsorption process, the hydroxyl (OH) groups on ACF surface were ionized and became electronegatively charged due to the weak alkalinity of urea solution, causing ionic repulsion towards partially anionic urea. The ionic repulsion force between the electronegatively charged ACF surface and urea molecules became stronger when more OH functional groups appeared on ACF prepared at higher acid impregnation ratio. The results implied that EFB fiber based ACF can be used as an efficient adsorbent for the urea removal process.

1. Introduction

Approximately, 15 million ton of empty fruit bunch (EFB) biomass generated from palm oil mills in Malaysia annually (Abdul et al., 2016). The common EFB disposal involved burning and dumping in plantation site for natural decomposition. However, both disposal methods lead to serious environmental pollution such as emission of methane and carbon dioxide (CO_2) gases (Omar et al., 2011) and create breeding habitat for pests such as rats and snakes (Lim et al., 2015). Therefore, introducing an appropriate way of handling the huge EFB biomass is crucial and it will be the highlight of this research where EFB was converted into a value-added adsorbent, activated carbon fiber (ACF). ACF exhibits large surface area and high adsorption capacity compared to conventional powdered and granular activated carbon (AC), activated alumina, mesoporous silica and zeolites (Fallou et al., 2016). Since EFB available in large quantity and inexpensive, it is an ideal raw material for the preparation of cost effective and bio-friendly ACF.

The application of activating agents during ACF synthesis and the acid impregnation ratio are the important factors for pore development in ACF. Sulfuric acid (H_2SO_4) is a popular activating agent used to improve the pore development in the carbonaceous material and promote the carbon yield (Singh et al., 2008). The increment of the acid-to-hemp based precursor ratio on the other hand resulted in the ACF with surface area to increase up to 1350 $m^2\ g^{-1}$ and mesopore volume of 1.25 $cm^3\ g^{-1}$ (Rosas et al., 2009). In brief, ACF with improved porosity and adsorption capacity can be effectively produced from EFB fiber via H_2SO_4

* Corresponding author.
E-mail address: feiyee@usm.my (F.-Y. Yeoh).

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activation.

The EFB fiber based ACF is an effective adsorbent and has been successfully used for water purification and separation applications (Lee et al., 2014). Urea is a main nitrogen containing compound found in the household wastewater. It will cause odor problem and induce the excess algae growth and diminish oxygen in the rivers, resulting in the death of aquatic lives (Liong et al., 2013). Thus, urea removal process through adsorption by the EFB fiber based ACF could be applied in the household wastewater purification to ensure the safety and quality of life. Hemodialysis treatment for patients whom suffer from renal failure involved urea removal from the patients' blood. The efficiency of the dialysis treatment could possibly be improved via urea adsorption by the ACF. In this study, ACF was prepared from EFB fiber via physicochemical activation using H_2SO_4 as an activating reagent. The effect of acid impregnation ratios during the ACF synthesis process towards the ACF properties was investigated. The EFB fiber based ACF was employed for urea removal analysis and the effectiveness of the respective ACF for urea adsorption was analyzed based on the urea removal kinetic data.

2. Experimental

2.1. Preparation of activated carbon fiber

EFB fiber was collected from United Oil Palm Industries, Penang, Malaysia. EFB was shredded into fiber in the palm oil mill. Clean EFB fiber was washed and immersed in 5% nitric acid solution for 20 min to remove the dirt attached to its surface. It was then cleaned with de-ionized (DI) water and dried overnight in an oven at 110 °C. 12 g of EFB fiber was mixed with concentrated H_2SO_4 at different acid-to-EFB fiber ratio as listed in Table 1. The acid treated EFB fiber was carbonized in a horizontal tube furnace under nitrogen (N_2) gas flow (flow rate was 100 ml min^{-1}). The acid treated EFB fiber was heated to 400 °C with a heating rate of 10 °C min^{-1} and kept for 1 h. The carbonized EFB fiber was cleaned with DI water to reduce its pH value and dried in an oven overnight at 110 °C. The carbonized EFB fiber was loaded into a tube furnace for carbon dioxide (CO_2) gas activation. The fiber was heated to 900 °C under similar N_2 gas flow at 10 °C min^{-1}. At 900 °C, the N_2 gas supply was switched to CO_2 gas flow (flow rate was 100 ml min^{-1}) for 1 h. After that, the sample was cooled down to room temperature under N_2 gas flow to yield ACF samples.

2.2. Characterization of activated carbon fiber

Field Emission Scanning electron microscope (FESEM) was used to observe the surface morphology of the EFB fiber and ACF samples. Pore characteristics of the ACF samples were determined by N_2 adsorption analysis at − 196 °C using a N_2 adsorption instrument (Quantachrome Autosorb iQ). Prior to the N_2 adsorption analysis, the ACF samples were degassed at 300 °C in a vacuum condition for 5 h. The specific surface area, micropore volume and pore size distribution (PSD) of the ACF samples were determined from the nitrogen adsorption isotherms by applying the BET equation developed by Brunauer et al. (1938), Dubinin-Radushkevich (DR) equation (1947) and Density Functional Theory (DFT) respectively. FTIR spectrometer (Perkin-Elmer Spectrum One) was used to study the functional groups present on the surfaces of EFB fiber and ACF samples using KBr technique and scanned from the range of 4000 to 400 cm^{-1}.

2.3. Urea adsorption measurement

Urea adsorption measurement was carried out to determine the urea adsorption capacity by ACF samples prepared at different acid impregnation ratio. A calibration curve was developed by measuring the UV–Vis absorbance intensity for a series of urea solutions with known concentrations at 200 nm using UV–Vis spectrophotometer (Agilent Varian Cary 50 Conc). Urea solution with concentration 38,500 μM was prepared by dissolving 0.462 g of urea powder in 200 ml of DI water. This concentration corresponds to the mean value of the urea concentration in most patients with kidney failure (Wernert et al., 2005). About 0.3 g of ACF was added into conical flask containing 200 ml urea solution. The conical flask was shaken with an orbital shaker (Lab Companion SK-300 Benchtop Shaker) to ensure even mixing of the ACF with the target urea solution. At different intervals, 3 ml of urea solution was taken using a filtered syringe. The samples were diluted with 6 ml DI water to obtain a concentration that fit into the linear range of calibration curve. Then, the urea solution was scanned by UV–Vis spectrophotometer. The concentration of urea solution was obtained by comparing the absorbance intensity to the calibration curve. The amount of urea adsorbed by ACF was calculated by the following equation:

$$q_t = \frac{(C_u - C_t) \times V}{m} \quad (1)$$

where q_t, C_u, C_t, V and m are the amount of urea absorbed by ACF (mg g^{-1}) at time t, initial concentration of urea solution without adsorbent (control, mg L^{-1}), concentration of urea solution after adsorption at time t (mg L^{-1}), total volume of urea solution (L) and mass of ACF employed in the experiment (g) respectively.

3. Results and discussion

3.1. Burn-off of activated carbon fiber

The burn-off for the ACF derived from EFB fiber was analyzed to determine the effect of acid impregnation ratio on the pore development. The burn-off of the ACF was calculated based on the following equation:

$$\text{Burn} - \text{off}\ (\%) = \frac{m_i - m_f}{m_i} \times 100\% \quad (2)$$

where m_i and m_f were the initial mass of precursor (g) and final mass of ACF (g) respectively.

The burn-off for ACF-I0.75, ACF-I1, ACF-I1.5, ACF-I2 and ACF-I4 samples are 78.5, 76.3, 82.7, 85.8 and 92.4 wt % respectively. The

Table 1
Physical properties of ACF samples with different acid impregnation ratio.

Sample	Acid-to-EFB fiber ratio (g/g)	BET surface area (m^2 g^{-1})	Micropore volume (cm^3 g^{-1})	Total pore volume (cm^3 g^{-1})
ACF-I0.75	0.75:1	654	0.27	0.28
ACF-I1	1:1	670	0.28	0.29
ACF-I1.5	1.5:1	869	0.39	0.41
ACF-I2	2:1	586	0.25	0.27
ACF-I4	4:1	568	0.24	0.27