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RESEARCH ARTICLE

Synthesis and performance assessment of coconut fiber solid adsorbent for waste cooking oil purification as biodiesel feedstock

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Abstract

Waste cooking oil can be considered as an alternative biodiesel feedstock for replacing edible oils. However, this feedstock can not be directly used since it contains much impurities and high Free Fatty Acid (FFA) content. Thus, pre-treatment process is required to enhance the feedstock quality. Adsorption using activated carbon is one of various methods that can be applied to reduce FFA content which is relatively easy and cheap. Coconut fiber is biomass waste that can be utilized in activated carbon production. This work has successfully synthesized activated carbon from coconut fiber with activator medium of H_3PO_4 10% weight and carbonization temperature of 600 °C, indicated from yield, water content, ash content, and methylene blue adsorption capacity. The yield of carbonization process developed in this work reached 40% while the yield for water content, ash content, and methylene blue adsorption capacity were 2.5%, 2.3% and 1646.1 mg/g carbon, respectively which complied with SNI 06-3730-1995. This adsorbent was tested on fixed bed adsorption column with FFA reduction reached up to 93% at waste cooking oil flowrate of 3 ml/min for 45 minutes operation time.

Keywords: Activated carbon, adsorption, coconut fiber, waste cooking oil

INTRODUCTION

Biodiesel has been gaining a tremendous attention as a promising alternative energy for replacing fossil fuel and optimizing renewable energy utilization in Indonesia, as requested by government policy due to its beneficial properties such as low sulphur content, lack of aromatics, higher lubricity and very high cetane number. Vegetable oil is comprised 95% of biodiesel feedstock (Sanjid *et al*., 2016). This will give impacts on biodiesel price and food feedstock competition problem (Sanjid *et al*., 2014), resulting in the increase in the proportion of operational cost in biodiesel production process up to reach 75-90% (Bonassa *et al*., 2016). Thus, finding an alternative feedstock for biodiesel production will solve problems encountered by vegetable oil. Non-edible oil can be considered as an alternative biodiesel feedstock since it can decrease operating cost until 60% (Marchetti, *et al*., 2008), which can enhance the economic viability of biodiesel production.

Waste cooking oil has a considerable potential to be converted into an environmentally friendly biodiesel which can overcome the problematic waste. However, during frying process, vegetable oil can undergo various chemical reactions such as polymerization, hydrolysis and oxidation (Ullah, *et al*., 2015). Those reactions will cause decomposition of triglyceride into other compounds such as free fatty acids (Canakçi & Ozsezen, 2010). It can degrade the quality of oil, making it to be impractical to convert it directly into biodiesel due to its high content of fatty acid (up to 15%) and impurities (Knothe, *et al*., 2010), which can be considered as persistent organic pollutants which are responsible for various lethal diseases and environmental problems (Alharbi, et al., 2018).

Adsorption process is one of favorable processes that can be employed for impurities removal due to its simplicity, cost-effectiveness and ~~its~~ efficiency (Saleh and Gupta, 2014). This process has been widely used in removal of hazardous dyes in aqueous solution to improve the quality of water. Mittal, et al. (2010) utilized bottom ash in removing and recovering Chrysoidine Y from aqueous solution. The Chemical Oxygen Demand (COD) of water was successfully decreased using adsorption process with TiO_2 solid adsorbent as catalyst (Gupta, et al. 2011) and it was redeveloped by combining TiO_2 with multi-walled carbon nanotubes as composite solid adsorbent to degrade methyl orange (Saleh and Gupta, 2012). This carbon nanotube was successfully used as a super selectivity sensor for monitoring of mercury ion(II) in water before it was employed as composite solid adsorbent (Khani, et al., 2010).

The development of solid adsorbent for waste treatment especially for dye removal has been made. Some researchers successfully developed metal oxide-based solid adsorbent coupled with photocatalytic activity for dye removal (Saravanan, et al., 2014a; Saravanan, et al., 2014b; Ali, et al., 2017; Khan, et al., 2017; Saravanan, et al., 2016a; Saravanan, et al., 2016b; Saravanan, et al., 2015a; Devaraj, et al., 2016; Saleh and Gupta, 2011; Ghaedi, et al., 2015 Saravanan, et al., 2015b; Saravanan, et al., 2013a; Saravanan, et al., 2013b; Saravanan, et al., 2013c; Saravanan, et al., 2013d; Saravanan, et al., 2013e; Saravanan, et al., 2013f;).

Besides metal oxide-based adsorbent, carbon-based adsorbent has been gaining tremendous attention for waste treatment application due to its low-cost and wide application (Ahamruzzaman and Gupta, 2011; Mohammadi, et al., 2011; Gupta and Saleh, 2013; Gupta, et al., 2014; Asfaram, et al., 2015). Activated carbon has been receiving its popularity among other adsorbent materials. It is commonly used for adsorption process (Rashidi, et al., 2013).

Activated carbon can be synthesized easily from biomass (Rashidi and Yusup, 2015) for example sugarcane bagasse (Seixas, et al., 2017), waste materials (Gupta, et al., 2014; Gupta, et al., 2013) and coconut

fiber. Coconut fiber can be considered as an appropriate feedstock for activated carbon production since it contains 64% of cellulose and 22% of lignin (Phan et al., 2006). High content of cellulose makes it possible to be synthesized as solid adsorbent (Suhas et al., 2016). Activated carbon from coconut fiber can reduce FFA content of waste cooking oil in batch process. Adsorption process using solid adsorbent (combination of activated carbon and silica) was tested and it reduced FFA content up to 39% (Sonkaew & Chaisena, 2012). To the best of our knowledge, some previous researches only focused on batch process. Very few research on continuous adsorption process have been performed. This study was focused on solid adsorbent synthesis and continuous adsorption process of waste cooking oil to reduce FFA content, which can be used as reference for biodiesel industry, especially in pre-treatment of biodiesel feedstock.

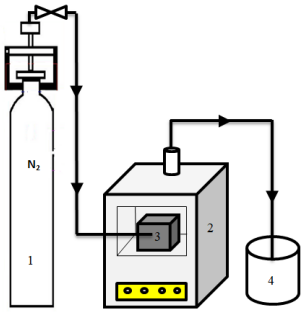
EXPERIMENTAL

Materials

Coconut fiber as primary material for solid adsorbent synthesis in this study was supplied by local supplier of carpet-home industry in Malang, Indonesia. This material was pre-treated to reduce its moisture content. Waste-cooking oil was supplied by local food-home industry in Malang, Indonesia with FFA content of less than 5%. H_3PO_4 for adsorbent activation process was purchased from Sigma Aldrich analysis grade. NaOH for FFA content measurement was purchased from Sigma Aldrich with pro analysis grade of 99% purity.

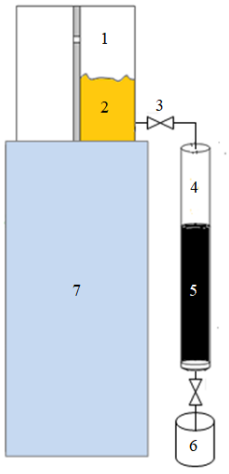
Apparatus

Apparatus used in this study consists of activated carbon solid adsorbent synthesis apparatus and adsorption column, as shown by Fig. 1 and 2, respectively. Activated carbon solid adsorbent synthesis apparatus consists of furnace 'carbolite' and stainless steel carbonization reactor with dimension of length: 13 cm, width: 15 cm, and height: 13 cm, as depicted in Fig. 1. While adsorption column was made from acrylic with dimension of ID: 2.5 cm and height: 48 cm, as shown in Fig. 2.



1. Nitrogen gas cylinder
2. Furnace
3. Carbonization reactor
4. Nitrogen gas trap

Fig. 1 Activated carbon solid adsorbent synthesis apparatus.



1. Oil reservoir
2. Waste cooking oil
3. Valve
4. Adsorption column
5. Activated carbon
6. Oil trap
7. Oil reservoir deck

Fig. 2 Continuous adsorption column apparatus.

Synthesis of activated carbon

Coconut fiber was fed into carbonization reactor inserted furnace at temperature of 600 °C for 1 hour. It was then cooled until it reached room temperature. There was nitrogen gas flow in continuous flow rate to remove oxygen in reactor. This process was called carbonization process.

After carbonization process, carbon was formed. It was then immersed and activated with H_3PO_4 with concentration of 10% for 24 hours before it was separated from its solution using filtration. Carbon produced from previous step was then washed using ultrasonic cleanser and dried in oven at temperature of 120 °C for 1 hour. Activated carbon was characterized and tested for its water content, ash content, and methylene blue adsorption capacity using Indonesian Standard Method number 06-3730-1995.

Performance test of activated carbon on adsorption column

Waste cooking oil used in this study as raw material was characterized before it was used for adsorption performance test for its FFA content using Indonesian Standard Method number 01-3555-1998. The FFA content of raw material should not exceed 3%.

Continuous adsorption process of waste cooking oil was performed by flowing it into adsorption column with flow rate of 3, 5, and 7 ml/min containing activated carbon with constant amount of 25 grams. After adsorption process was completely done, the waste-cooking oil output sample was obtained and then analyzed for its various characteristics such as FFA content, acid value, and peroxide value to evaluate the performance of activated carbon in continuous adsorption process.

RESULTS AND DISCUSSION

Activated carbon synthesis from coconut fiber

From this study, we obtained activated carbon with particle size of 50 mesh and yield of 40%. The characterization of activated carbon produced from in this study is presented in Table 1. Moreover, the functional groups of chemical compounds contained in activated carbon developed in this study were also analysed using Fourier Transform Infrared (FT-IR), as displayed in Fig. 3. This analysis was done for carbon before and after activation process.

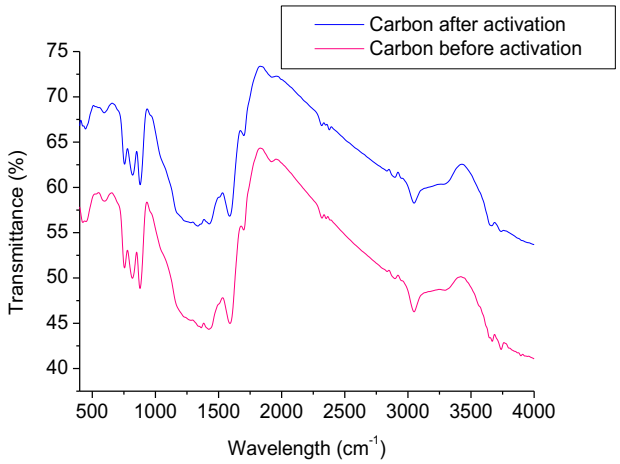


Fig. 3 FTIR spectra of carbon before and after activation.

Table 1 Activated carbon characterization results.

No	Parameters	Carbon before activation	Carbon after activation	Standardized carbon (SNI No. 06-3730-1995)
1	Water content (%)	4.1	2.5	Max. 15
2	Ash content (%)	2.9	2.3	Max. 10
3	Methylene blue adsorption capacity (mg/g)	568.4	1646.1	Min. 120

It is obvious from Table 1 that activated carbon produced from this study complied with all parameters stated in Indonesian Standard Method number 06-3730-1995, meaning that activated carbon produced from this study contained high content of cellulose.

As a solid adsorbent, one of important parameters is pore size which can be represented by methylene blue adsorption capacity. This parameter can represent macro pore and mesoporous size of solid adsorbent. Activation process could increase its pore up to 3 (three) times as can be seen from Table 1.

It can be seen from Fig. 3 that activation process developed in this study did not give impact on the change of functional group for carbon before and after activation process. Table 2 summarizes the list of functional groups detected from FTIR analysis. The alkene group (= C-H) appeared at the peaks of waves in range of 675-995 and 3010-3095 cm-1, which might be formed by breaking off the molecular structure of water at a temperature of 150-240 °C. The aromatic groups (C = C) appeared at the wavelength of 690-900 cm-1 and 1500-1600 cm-1 while the aromatic groups (CH) appeared at the wavelength of 690-900 and 3010-3100 cm-1 might be formed due to aromatization process during carbonization process at temperature of 400-600 °C. While the alkane (C-H) group appeared at the peak of 3200-3600 cm-1 could be formed by thermal degradation at temperature of 240-400 °C. FTIR analysis results proved that activated carbon produced from this study was non-polar adsorbent having an ability to adsorb free fatty acid due to the presence of non-polar C-H functional group. However, this activation process did not change the chemical properties of activated carbon. Activation process only enhanced the physical properties of activated carbon shown by its increasing adsorption capacity on methylene blue.

Table 2 FTIR functional groups analysis of carbon.

Functional Groups	Functional groups name	Wavelength
=C-H	Alkene	675-995 ; 3010-3095
C-H aromatic	Aromatic	690-900 ; 3010-3100
C=C aromatic	Aromatic	1500-1600
C-H	Alkane	2850-2970 2850-2970 ; 1340-1470

The effect of waste cooking oil flow rate on continuous adsorption process

In this study, the adsorption performance of activated carbon in adsorption column was evaluated based on free fatty acid (FFA) reduction. Fig. 4 shows the profile of relationship between FFA reduction and adsorption operating time for various waste cooking oil flow rates.

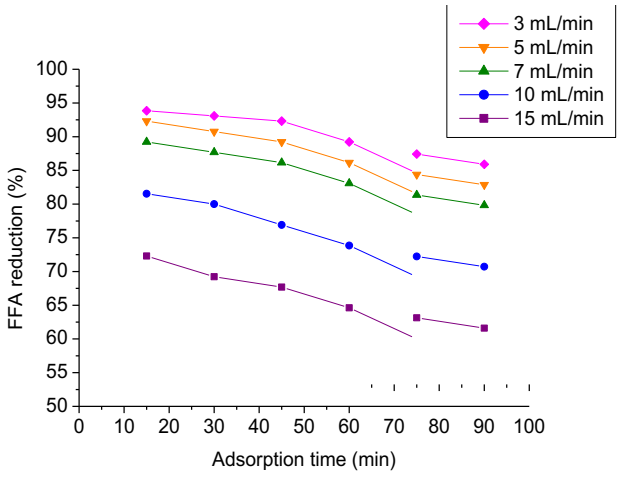


Fig. 4 Relationship between FFA reduction (%) and adsorption operating time (minute).

Fig. 4 reveals that the longer adsorption operating time, the FFA reduction became lower as well as when waste cooking oil flow rates were raised. This indicated that the adsorption capacity of activated carbon produced in this study was decreased as increasing of adsorption operating time.

Activated carbon produced in this study could achieve FFA reduction of waste cooking oil feed until 93% at waste cooking oil flow rate of 3 ml/min for not longer than 45 mins. It meant that with FFA content of 3% in the feed, this activated carbon could reduce FFA content until 0.3%, which complied with Indonesian standard method number 01-3741-2013 as biodiesel feedstock. However, for this flow rate, the product that could be obtained from this product was 135 ml. But for other flow rate variables, they could not achieve 90% of FFA reduction.

Table 3 Adsorption process product characterization.

No	Parameters	Waste cooking oil before adsorption	Waste cooking oil after adsorption	Standardized cooking oil (SNI No. 01-3741-2013)
1	Water content (%)	3.33 ± 0.02	0.2 ± 0.02	Max. 0.3
2	Density (g/ml)	0.928 ± 0.005	0.876 ± 0.002	Max. 0.9
3	Free fatty acid content (%)	3.83 ± 0.03	0.294 ± 0.05	Max. 0.3
4	Acid value (mgNaOH/g)	0.57 ± 0.00	0.444 ± 0.011	Max. 2
5	Peroxyde value (mg eq/kg)	3.33 ± 0.02	1.00 ± 0.02	Max. 2

Moreover, oil obtained from adsorption product was characterized in terms of several parameters including water content, density, FFA content, acid number, and peroxide number. Table 3 reveals that activated carbon produced from this study has a good ability to reduce those parameters to meet Indonesian standard number 01-3741-2013 requirement as biodiesel feedstock.

CONCLUSION

This study has successfully synthesized activated carbon from coconut fiber with yield of 40% and characteristics which complied with Indonesian Standard Number 06-3730-1995 in terms of water content, ash content, and methylene blue adsorption capacity with the value of 2.5%, 2.3% and 1646.1 mg/g carbon, respectively.

Activated carbon produced in this study was successfully applied for continuous adsorption process to reduce FFA content of waste cooking oil. This activated carbon reduced the FFA content of waste cooking oil up to 93% for adsorption operating time of 45 minutes and produced waste cooking oil with FFA content of 0.3% which complied with Indonesian standard method number 01-3741-2013 as biodiesel feedstock.

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