

Kapok Derived Activated Carbon Catalyst Assisted in Biodiesel Production from Waste Cooking Oil

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ABSTRACT

The present study aims to synthesize green carbon-based catalyst from kapok (Ceiba pentandra) using two different activating agents: KOH (CB1) and K₂CO₂ (CB2) with 1:1.0 (raw material: activating agent), at activation temperature of 400 °C and impregnation time of only 15 min. *The synthesized catalysts were evaluated in the transesterification of waste* cooking oil (WCO) into biodiesel. CB1 registered higher iodine number and percentage yield (1446.30 mg/g, 62.60 %) compared to 1200.23 mg/g and 53.50% obtained for CB2. Several physico-chemical characterizations were subjected for kapok and the carbon catalysts such as Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscope (SEM) and CHNS/O Analyzer. FTIR investigation showed the disappearance or reduction in peak intensity of several peaks at 1512-1200 cm⁻¹ in the carbon catalyst compared to raw kapok, arising due to carbonization and activation processes. CHNSO analysis verified that both CB1 and *CB2 registered high carbon content of 63.93 % and 62.86 %, respectively* compared to the raw kapok (43.54 %). Morphological studies by SEM analysis showed appearance of cylindrical tube for all the samples. The biodiesel synthesis from WCO at 0.2 wt.% catalyst loading, methanol to oil (molar ratio of 3:1), reaction temperature of 60 °C for 1 h resulted in high catalysis over CB1 (89.57%), followed by CB2 (87.46%) and without catalyst (35.46%). Large iodine number and high carbon content exhibited by CB1 was the probable reasoning for the accelerated activity of CB1 in the transesterification of WCO. To conclude, the present work showed a successful conversion of waste biomass into promising carbon catalyst for



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green synthesis of biodiesel from WCO.

Keywords: Kapok; Activated Carbon; Biodiesel; Waste Cooking Oil; Catalyst

INTRODUCTION

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The exponential growth in human population and urbanization is anticipated to rise the global energy consumption to 623-710 EJ in the year 2035 compared to 567 EJ, recorded in 2013 [1]. This scenario creates a huge dependence on non-renewable fossil fuels that eventually will experience serious and rapid depletion in the coming years. In addition, the current situation had witnessed a great incline in the greenhouse gases (GHGs) emission, resulting from the burning of fossil fuels. Nearly thirty-percent of total GHGs is contributed by fossil fuels burning [2]. This severe environmental threat and stringent legislation by developing countries such as Malaysia had triggered an active exploration for alternative renewable oil namely biodiesel. This act is urgently required to safeguard the sustainability of the available resources and reducing carbon footprint. Hence, biodiesel comes into the picture as a savior owing to its desirable characteristics such as low-cost, free of aromatic hydrocarbon, eco-friendly nature, biodegradable and exhibits better emission profile [1].

Biodiesel is produced via transesterification of triglycerides with alcohols (Scheme 1) in the presence or absence of catalyst, producing fatty acid alkyl esters (or known as biodiesel) and glycerol as by-product [3].

Triglyceride	Alcohol	Alkyl Ester (Biodiesel)	Glycerol
0 II CH ₂ - 0 - C - R			СН ₂ – ОН
0 CH - O - C - R	с + 3R'ОН 4	atalyst → 3R' - O - C - R	+ СН – ОН
$CH_2 = O = C = R$			CH ₂ – OH



Nevertheless, the major concern in the process is the high cost of the feedstock, that contributes to 70-95 % of the overall production cost [5]. Therefore, utilizing a cheap feedstock such as waste cooking oil (WCO) is an ideal practice, that can reduce nearly 50 % of biodiesel price upon transforming WCO into biodiesel [6]. Approximately 16.5 million tons of WCO is produced worldwide and ended up either in landfill, drain, sewage or palatable water [7]. Thus, conversion of WCO into biodiesel appears as the best solution for the waste management and to curb high demand in fuel industry.

Similarly, catalyst is also a crucial factor in controlling the biodiesel production costs and promoting the synthesis of high quality bio-fuel. Since years, converting biomass into catalyst is becoming a considerable interest as it reduces the dependence of non-renewable energy sources and lessens the total biodiesel cost. In this context, Malaysia is truly blessed with abundance of agricultural solid waste that can be converted into active and cheap catalyst for the production of high-quality bio-oil, for example, rice husk [8], palm kernel shell [9], mango peel [10] and empty fruit bunch [11]. On the other hand, Ceiba pentandra (kapok) is a tropical tree extensively spread in Southeast Asia and from the Family of Bombacaceae [12]. The main composition of kapok is cellulose, lignin, polysaccharide, and a waxy coating [13]. Rich surface oxygen functionalities of kapok, benefits its efficiency as great adsorbent of metal ions [14]. Since years, kapok fibers have been traditionally used as a stuffing and packing material in pillow, soft toys and quilts [15], however, to date, the potential of kapok as a carbon precursor owing to its high carbon content has not been widely explored [16]. Only few studies have been published on the synthesis of activated carbon from kapok for the adsorption of methylene blue [12] and the fabrication of kapok derived biochar for sulfamethoxazole degradation [13].

To note, series of publications have emerged on the use of various catalysts for biodiesel conversion from WCO, however, most of the reported catalytic system requires prolonged reaction period, high methanol/oil ratio, large catalyst loading and tedious preparation route. For example, in a recent work, CaO catalyst derived from snail shells and modified with ZnO and TiO₂ had produced maximum yield of 90 % and 95 % biodiesel from WCO, respectively, however after 3 h of reaction [17]. In a recent work, NaOH/ bentonite catalyst requires nearly 4 h to yield 91.2 % of biodiesel using 9:1

methanol/oil molar ratio [18]. In addition, Al-Hamamre et al. [19] reported complete conversion of biodiesel, nevertheless, in the presence of high methanol:oil ratio of 45:1. Thus, to address the aforementioned drawbacks in the existing studies, a green approach was made to produce activated carbon from waste biomass, *Ceiba pentandra* using two different activating agents and evaluated in the transesterification of WCO into biodiesel at benign condition.

EXPERIMENTAL METHODOLOGY

Reagents and Waste Cooking Oil (WCO) sample collection

Methanol (CH₃OH, Fisher, 99 %), sodium thiosulphate-5-hydrate (Na₂S₂O₃.5H₂O, HmBG, 99 %), potassium carbonate (K₂CO₃, HmBG), potassium hydroxide (KOH, HmBG), potassium iodide (KI, HmBG, 99.87 %), iodine pearl (I2, Fisher, 99 %), starch (C₆H₁₀O₅, HmBG) and hydrochloric acid (HCl, Fisher, 37 %) were of analytical grade and used as received, without any purification. WCO was collected from nearby cafeterias in UiTM Arau Campus, and filtered to remove any food residues.

Preparation of kapok fibers

Ceiba pentandra (kapok) was gained from neighboring areas in Arau, Perlis, Malaysia and used as the raw material. The preparation of raw Kapok for the carbonization process is illustrated in Figure 1.

Accordingly, the raw kapok was cut into desired sizes, subsequently washed with copious amount of tap water and finally, with distilled water to remove any unwanted particles and impurities. Later, the cleaned kapok was oven dried for 12 h at 70 °C. Lastly, it was carbonized in a muffle furnace at 500 °C for 30 min and stored in an air-tight container for further use.



Figure 1: Carbonization preparation route of raw Kapok.

Synthesis of CB1 and CB2

About 10 g of the carbonized kapok was transferred into a beaker containing 50 wt.% of KOH solution in a 1:1 w/w (Kapok : Activating agent). Impregnation was done under stirring (200 rpm) for only 15 min. Later, it was subjected for filtration, recovered and heated in oven at 80 °C for 24 h and subsequently activated in the furnace at 400 °C at a heating rate of 3 °Cmin⁻¹ and hold for 30 min. Similar experiment was conducted using K_2CO_3 as the activating agent. The sample treated with KOH and K_2CO_3 was designated as CB1 and CB2, respectively.

Material Characterizations

The infrared spectra of the raw kapok and synthesized activated carbon samples were recorded on a Fourier Transform Infrared Spectroscopy (Perkin Elmer), scanned in the range of 400-4000 cm⁻¹. The samples were prepared by KBr pressed method using 1:10 (Sample: KBr w/w). The elemental composition was obtained by CHNS/O Analyzer (CHNS-O Analyzer Flash 2000) while the morphological view of the samples was investigated by Scanning Electron Microscope (SEM: JEOL, JSM-6460LA). As a preliminary analysis, CB1 and CB2 were subjected for Iodine Number analysis to quantify the surface area whereas the percentage yield was calculated according to Equation (1) and (2) respectively.

$$\frac{x}{m} = (A - (DF * B * S))/M \tag{1}$$

(1)

Where,

 $\frac{x}{m} =$ iodine absorbed per gram of carbon, mg/g

DF =dilution factor, (I+H)/F

- I =Volume of iodine, mL, H = Volume of 5 % hydrochloric acid, mL,
- F =Volume of the filtrate, mL, N_1 = Normality of sodium thiosulfate, N: 0.1 N

 N_2 =Normality of iodine, N: 0.1 N, B = N_1 * 126.93: 0.1*126.93=12.693 A = N_2 * 126: 0.1* 12693= 1269.3, S = Volume of sodium thiosulfate, mL M = Mass of carbon used, g

% yield =
$$\frac{\text{mass of carbon based}}{\text{mass of raw}} \times 100\%$$
 (2)

Pretreatment of Waste Cooking oil (WCO)

WCO was pre-treated to reduce the free fatty acid (FFA) content by adding 50 mL of methanol and 0.1 mL concentrated H_2SO_4 at 65 °C for 1 h [5]. High FFA content results in lowering biodiesel yield arising from saponification process [20]. In general, water content can promote oil hydrolysis, leading to an increase in FFA content [20]. Thus, in the present study, the filtered WCO was repeatedly washed with hot water to remove any impurities and then, was treated with anhydrous NaSO4 for the removal of moisture.

Biodiesel Synthesis and Product Identification

In brief, a two-neck 250 mL round bottom flask containing known quantity of pre-treated WCO and equipped with a stirring bar, a reflux condenser was set on a hot plate at 60 °C. Throughout the experiment, the reaction temperature was monitored by an attached thermocouple. Once the temperature was stable, a mixture containing methanol (3:1 molar ratio of methanol to triglyceride) and 0.2 wt.% of catalyst was charged into the round bottom flask containing WCO. The transesterification was allowed to proceed for 1 h and later, the reaction mixture was kept to cool and subsequently transferred into a separating funnel. Biodiesel (methyl ester, upper layer), glycerol (lower layer) and the catalyst was separated. The biodiesel layer was then added with water, shaken gently and left to settle for 15 min. The process was repeated for twice and then, the remaining solution was added with anhydrous Na₂SO₄ and traces of water was removed. Similar experiment was conducted in the absence of catalyst. Finally, the recovered methyl ester was analyzed by GC/MS (Agilent Technology 6980 N) for the composition studies according to the isothermal program summarized in Table 1.

Table 1: GCMS isothermal program for the identification of methyl esters in the synthesized biodiesel

Flow rate	5 mL/min
Sample injection (Volume)	Volume: 2 µL at 300 °C, split ratio (10:1)
Initial Oven Temperature	190 °C (Hold time: 2 min)
Ramp 1	10 °C/min
Final Oven Temperature	280 °C (Hold time: 9 min)
Ramp 2	3 °C/min

The crude yield was calculated using Equation (3).

$$Yield \% = \left(\frac{weight of oil (g)}{weight of sample (g)}\right) \times 100\%$$
(3)

RESULTS AND DISCUSSION

Iodine Number and Percentage Yield

CB1 registered a high surface area of 1446.30 mg/g compared to 1200.23 mg/g recorded over CB2. Significant percentage yield of 62.60 % was obtained by CB1 while CB2 experienced a reduction of ~14.54 % compared to those registered by CB1. Evidently, KOH appears as the best activating agent under the studied synthesizing condition. In a different work, porous carbon prepared from waste kapok flower exhibited SBET of 1231 m²/g using KOH activation at a higher temperature of 700 °C [21]. This clearly evidences the facile method employed in this present study could produce higher surface area activated carbons even at lower activation temperature of 400 °C.

FTIR Fingerprint Identification

The functional group properties of kapok fiber and the prepared carbon catalysts are compared in Figure 2.



Figure 2: FTIR spectra of the synthesized materials.

The raw kapok fibre showed the presence of assorted peaks in the region of $1512 - 1200 \text{ cm}^{-1}$ that was clearly absent in the IR spectra of CB1 and CB2. This indicates the successful breakdown of organic molecules during the carbonization and alkaline activation. A similar observation was reported by Mani et al. [22], for raw groundnut shell compared to the KOH activated groundnut shell samples. In the IR spectrum of raw kapok, the sharp intense peak at 1736 cm⁻¹ is evidence of C=O due to carboxyl and carbonyl groups [22] while the band around 1512 cm⁻¹ is associated with C=C stretching vibration of aromatic rings [22]. Meanwhile, the absorption peak at 1249 cm⁻¹ is attributed with C–N stretching or O-C-O bonds [23] while the sharp and intense peak at 1384 cm⁻¹ demonstrates the C-H bending vibration of lignin [24].

Nevertheless, the observed IR peaks at 3238-3442 cm⁻¹ are principally identical for all the samples. This peak is assigned as O-H stretching vibration, that relates to hemicellulose, cellulose and lignin [23]. The band at 1648 cm⁻¹, 1630 cm⁻¹ and 1599 cm⁻¹ appeared for Kapok, CB1 and CB2, respectively corresponds to the C-O indicating ester or aldehyde groups [25]. The shoulder peak at ~ 2929 and 2919 cm⁻¹ for Kapok and CB1 correspond to -CH₂ stretching vibration, was missing in the case of CB2. Likewise, Deng et al. [26] registered a similar IR pattern over activated carbons prepared from cotton stalk using KOH and K2CO3 activation. Referring Figure 2, the peak intensity in the region 2899-2929 cm⁻¹ was less for CB2, suggesting the effectiveness of carbonization and activation with K₂CO₃ as the activator, compared to KOH. Furthermore, the absorption band due to stretching vibration of C-O single bonds in ethers, esters, alcohol and phenol [25] was noticed to experience blue shift from ~1100 cm⁻¹ (kapok) to 1090 and 1082 cm⁻¹ for CB1 and CB2, respectively. The peaks at ~609-622 cm⁻¹ corresponds to the alkyls and C-X vibration [27]. To conclude, the FTIR analysis proved the effectiveness of carbonization and activation process during the synthesis of the activated carbons.

Surface Morphology Studies

Figure 3 compares the SEM images of the raw kapok, CB1 and CB2 carbon samples. In Fig. 3(a), a smoother surface was observed unlikely to rough surface captured for CB1 and CB2. The result was in line with a SEM image reported by Wang and co-researchers [13] for raw kapok. All

the samples have a hollow cylindrical microtube. Nevertheless, after the alkaline activation, porosity was clearly developed for both CB1 and CB2, as shown in Figs. 3(d-e), that exhibited larger pore opening compared to the raw kapok fiber. This clearly evidenced the promotion of porosity by the activation treatment.



Figure 3: SEM images of a) raw kapok fibre, b) CB1 and c) CB2 at x1400 and d) CB1 and e) CB2 at x45

CHNSO Elemental Analysis

Table 2 compares the elemental composition of raw kapok and the activated carbon catalysts. Accordingly, carbon and oxygen appear to be the dominant element for all the samples. As expected, carbon content increased tremendously about 46.8 % and 44.4 % for the carbon catalysts, CB1 and CB2, respectively in comparison to the raw kapok.

Samples	Element (wt.%)				
	Carbon	Hydrogen	Nitrogen	Sulphur	Oxygen
Raw Kapok	43.54	9.72	1.09	4.10	43.78
CB1	63.93	3.09	1.16	0.89	30.93
CB2	62.86	2.68	1.17	0.69	32.60

Table 2: Elemental composition of Ceiba pentandra and the activated carbons

This arises due to decomposition of carbon in raw after carbonization and activation processes [28]. In general, a rise in carbon content leads to an increase in the porosity, which was in great agreement with the SEM images of CB1 and CB2. On contrary, the hydrogen and oxygen content reduced significantly for the carbon catalysts compared to the raw kapok, due to the elimination of volatiles matter during the carbonization process [29]. An eco-friendly activated carbon must exhibit low content of sulphur and this is in accordance with the sulphur content registered for both CB1 and CB2 compared to that of the raw kapok.

Biodiesel synthesis via transesterification

Table 3 summarizes the biodiesel yield synthesized using methanol: oil molar ratio (3:1), catalyst amount of 0.2 % and reaction temperature of 60 $^{\circ}$ C after 1 h constant stirring.

Samples	% yield
Without catalyst	35.46
CB1	89.57
CB2	87.46

Table 3: The % yield of the biodiesel

Referring Table 3, transesterification of WCO resulted in 35.46 % yield in the absence of catalyst that was later enhanced in the presence of either CB1 (89.57 %) and CB2 (87.46 %) catalysts. Theoretically, the transesterification reaction was highly accelerated by the availability of active sites in the catalysts. CB1 showed significant improvement in the biodiesel yield, presumably due to higher surface area of (1446.3 mg/g compared to CB2 (1200.23 mg/g) that has provided more active sites for the reaction to occur. Furthermore, the enhanced catalysis of CB1 could

be also due to higher carbon content registered (CHNS/O analysis), that might have promoted the porosity of CB1. In general, high surface area and well-defined porosity appear to be the most deciding factor in controlling the catalysis. In a work reported by Ahmad Farid et al., [30], 98 % biodiesel yield was recorded in the presence of 5 wt.% catalyst prepared from palm empty fruit bunch (OPEFB) treated with KOH. Nevertheless, the reaction requires 4 h to achieve the maximum yield, while in this present study, only 1 h was required to convert WCO into nearly 90 % yield. Similarly, in a recent study, 3 h was needed to transform WCO into 75.2 % biodiesel using 1.5 g catalyst dosage [31]. Thus, the present study shows the capability of the current biowaste catalyst for the transesterification of WCO and undoubtedly has a great potential to be commercialized into a green catalyst. Table 4-6 summarizes the methyl ester compositions of the synthesized biodiesel and is in accordance with the FAME analysis of WCO over eggshells catalyst [31].

Retention time (min)	Identified compounds
28.270	9-Hexadecenoic acid methyl ester
28.698	Hexadecanoic acid methyl ester
28.932	Pentadecanoic acid, 14-methyl-methyl ester
32.911	11-Octadecenoic acid methyl ester
33.285	9-Octadecenoic acid methyl ester
35.408	Octadecanoic acid methyl ester

Table 4: FAMEs composition of the synthesized biodiesel without catalyst.

Table 5: FAMEs composition of the synthesized biodiesel with CB1.

Retention time (min)	Identified compounds
23.224	Dodecanoic acid methyl ester
25.584	Tridecenoic acid, 12-methyl-methyl ester
27.813	9-Hexadecenoic acid methyl ester
28.350	Hexadecanoic acid methyl ester
28.373	Pentadecanoic acid, 14-methyl-methyl ester
32.105	9-Octadecenoic acid methyl ester
32.762	Octadecanoic acid methyl ester

Retention time (min)	Identified compounds
25.801	Tridecenoic acid, 12-methyl-methyl ester
28.150	9-Hexadecenoic acid methyl ester
28.648	Hexadecanoic acid methyl ester
28.722	Pentadecanoic acid, 14-methyl-methyl ester
32.711	9-Octadecenoic acid methyl ester
32.785	Octadecanoic acid methyl ester

Table 6: FAMEs composition of the synthesized biodiesel with CB2.

CONCLUSION

The study was focused on the facile conversion of waste kapok fibre into activated carbon using alkaline activating agents. The synthesized catalysts were active in the transformation of waste cooking oil into biodiesel, reaching maximum yield of 89.57 % over CB1 catalyst. FTIR spectroscopic analysis verified the successful of activation and carbonization treatment, arising from the disappearance of several peaks in the IR spectrum of the activated carbon compared to raw kapok. CHNSO analysis confirmed on the increase in carbon and reduction in sulphur content for the carbon catalysts, leading to a promising biomaterial for the transesterification of waste cooking oil. High surface area and great porosity observed for the synthesized catalysts proved that kapok fibre acts as a potential biomass for the conversion into benign material.

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