

## Biodiesel Synthesis over Biochar-based Catalyst from Sengon (*Paraserianthes falcataria L. Nielsen*) Sawdust using Palm Fatty Acid Distillate as Low-Cost Feedstock

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**Abstract.** Biodiesel is mostly made from trans-esterification reaction using homogenous catalysts. Problems related to homogenous catalysts are the difficulty to separate the final product and the toxic that the waste contains. To prevent this negative effect, it is suggested to use heterogeneous catalysts. The heterogeneous catalysts have been considered to apply on biodiesel production associated with homogeneous catalysts limitations. Many advantages could be provided by heterogeneous catalysts including easily separated from reaction product, can be used repeatedly, generate less waste water, and environmentally friendly operations. In this paper, the Palm Fatty Acid Distillate (PFAD) esterification to form biodiesel was studied using biochar-based catalyst from sengon (*Paraserianthes falcataria L. nielsen*) sawdust as heterogeneous catalyst. Reaction parameters such as temperature, molar ratio of oil to methanol, and amount of catalyst percentage were varied to achieve the highest conversion. The highest free fatty acids (FFAs) conversion of 89.71%, was obtained by using 60°C of reaction temperature at 12:1 molar ratio of methanol to PFAD and 3% catalyst amount.

### Introduction

The important factors encouraging efforts to look for renewable energy sources are the increasing of energy demand and the concerning of global warming in order to environmental pollution, greenhouse gases emission, and depletion fossil fuels supply. The promising renewable energy that considered as an alternative substitute for diesel fuel base petroleum is biodiesel. Biodiesel is a monoalkyl fatty acid ester which has many benefits, such as using renewable sources as raw materials, biodegradable, non-toxic, and contains neither sulfur nor aromatic compounds. Biodiesel can be produced through transesterifying of triglycerides or esterifying of free fatty acids with methanol by adding acid or alkaline catalyst.

The expensive raw material and the competition between food security and energy supply are the problems that associated with the biodiesel production. The utilization of inexpensive raw materials including edible oil and used cooking oils would be reduced the production cost and avoid the problems that occur in the food and energy supply. Thus, the biodiesel synthesis using non-conventional raw materials is necessary to be intensified. Palm oil refinery generates Palm Fatty Acid Distillate (PFAD) that contains high free fatty acids as by-products. The PFAD price is only 0.25 USD/liter which is much lower than the refined palm oil. Therefore, the application of PFAD as raw material for biodiesel production could be diminish the production cost and enabled to contend economically with petroleum-based fuel.

Currently, the biodiesel has been produced commercially by using homogeneous catalysts among them alkaline catalysts (NaOH or KOH) and acids catalysts (H<sub>2</sub>SO<sub>4</sub> or HCl). Homogeneous catalysts have many shortcomings, including having to be neutralized after the reaction, difficult to separate from the product, cannot be reused, and lead to environmental problems. To solve the problems associate with the homogeneous catalysts, many researchers have started to investigate the heterogenous catalysts for biodiesel production. Usually, the high free fatty acid content on raw materials promotes the saponification reaction when using an alkaline catalyst. Therefore, the converting FFAs on PFAD to form methyl esters apply an acid catalyst.



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Different types of solid materials such as sulfated niobium oxide [1, 2], bituminous coal [3], metal oxide [4, 5], sulfonated-multiwalled carbon nanotubes [6] have been investigated as viable heterogeneous acid catalysts for esterification reaction. Carbon-based catalysts such as glucose [7], sucrose [8], carbohydrate-based catalysts [9], biomass waste [10-13], biochar [10, 13, 14], and activated carbon [15], were promisingly as as heterogenous acid catalysts in order to low cost, available abundantly, reusable, stable and have high porosity which promotes catalytic activity. The heterogenous acid catalyst which was synthesized from carbon materials is also prepared by simple procedure, including sulfonation using strong acid to attach SO<sub>3</sub>H groups on carbon surface.

Based on literatur survey, the reported study on the utilization of sawdust biochar as heterogenous acid catalyst has not been investigated yet, for biodiesel synthesis from PFAD. In this paper, the esterification of PFAD to form biodiesel was studied using biochar-based catalyst from sengon (*Paraserianthes falcataria L. nielsen*) sawdust as heterogeneous catalyst. Reaction parameters such as oil to methanol molar ratio, temperature, and catalyst amount percentage were varied to achieve the highest conversion.

## Experiment

### *Catalysts Preparation and Characterization*

The sengon (*Paraserianthes falcataria L. nielsen*) sawdust were collected from sawmills in Sleman district, D.I. Yogyakarta province. Prior to use, the sawdust precursors were screened to get uniform size with diameter less than 100 mesh. The biochar was obtained by partially carbonization of sawdust in a furnace at 400°C using heating rate of 10°C/min for 4 hours. The resulted biochar was then sulfonated according to the following procedure: (i) biochar was mixed with H<sub>2</sub>SO<sub>4</sub> p.a.; (ii) the mixture was agitated vigorously at 150°C for 12 h; (iii) after the sulfonation process was completed, the sulfonated biochar was filtered to be separated from the mixture; (iv) the sulfonated biochar was rinsed with hot distilled water and checked periodically to detect no remaining sulfate ions; and (v) the sulfonated biochar was dried in an oven at 110°C.

The measurement of N<sub>2</sub> adsorption at 77 K was carried out to analyze the porosity of characteristics. To determine the surface functional groups on solid surfaces, the FTIR spectroscopy analysis was conducted. The elements contained on materials were determined by X-Ray Fluorescence instrument. The standard acid-base back-titration was used to calculate the total acid density of the samples.

## Results and Discussion

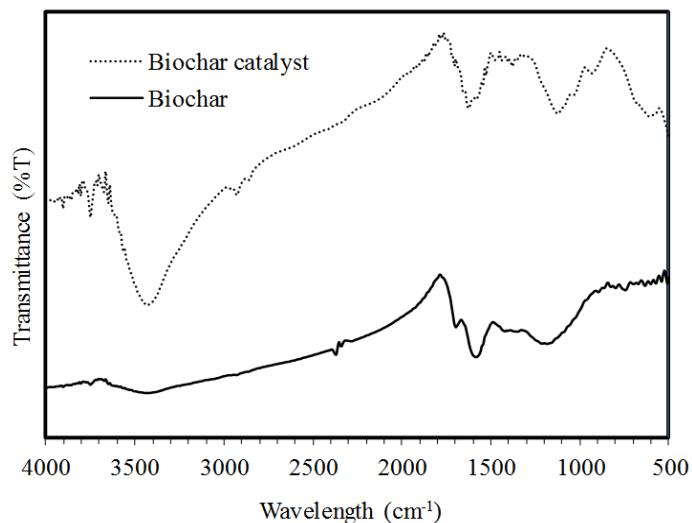
### *Catalysts Characterization*

The biochar and biochar catalyst textural characteristics were analyzed using N<sub>2</sub> adsorption desorption technique to calculate the BET surface area, total pore volume, and average pore size diameter. The biochar catalyst and biochar textural characteristics were presented on Table 1. The reduction in BET surface area from catalyst to the catalyst catalyst indicates the successful of incorporating of SO<sub>3</sub>H groups on surface. From Table 1, it also can be confirmed that the average pore size diameter was become wider after sulfonation process. Meanwhile, the total pore volume of biochar catalyst was smaller compared to the biochar. The back titration method was applied to measure the acid density of samples. The acid density of biochar catalyst dramatically increased after the sulfonation process which indicates the success of SO<sub>3</sub>H groups anchoring on the surface.

**Table 1.** The biochar and biochar catalyst textural characteristics.

| Samples         | Specific surface area (m <sup>2</sup> .g <sup>-1</sup> ) | Average pore size | Total pore volume (cm <sup>3</sup> .g <sup>-1</sup> ) | Acid Density |                   |
|-----------------|--|-------------------|---|--------------|-------------------|
|                 |  |                   |   | Total        | SO <sub>3</sub> H |
| Biochar         | 266  | 2.3               | 0.1631  | 0.19         | n.a.              |
| Bichar catalyst | 196  | 2.5               | 0.1482  | 3.24         | 2.67              |

The identification of functionalized groups for both biochar and biochar catalyst surface were presented Figure 1. The  $-\text{SO}_2-$  asymmetric and asymmetric stretching vibration bands were indicated at wavenumbers around 1035 and 1154  $\text{cm}^{-1}$ . The band at 3431  $\text{cm}^{-1}$  (biochar) and 3429  $\text{cm}^{-1}$  (biochar catalyst) were ascribed to the phenolic OH groups and O-H stretching of the  $-\text{COOH}$ . Meanwhile, a wavenumber at 1704  $\text{cm}^{-1}$  was correlated with the C=O stretching of COOH group. The content of sulfur in biochar catalyst was analyzed the Energy Dispersive X-ray (EDX) spectrometry analysis. The content of sulfur achieved 6.15% on the biochar catalyst. Based on the results, it can be confirmed that the anchoring of  $\text{SO}_3\text{H}$  groups into biochar catalyst surface was successful by sulfonation using  $\text{H}_2\text{SO}_4$  concentrated.

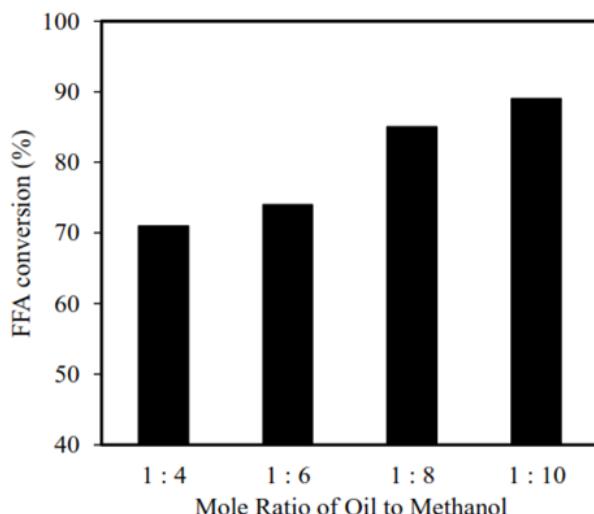


**Figure 1.** FT-IR Spectra of Biochar and Biochar Catalyst.

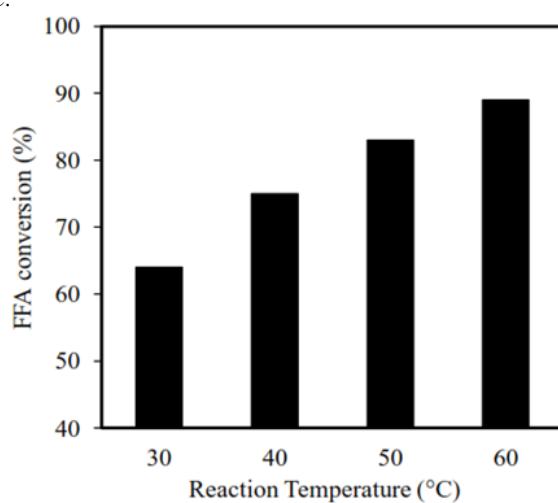
#### Activity Test

##### *Effect of Mole Ratio*

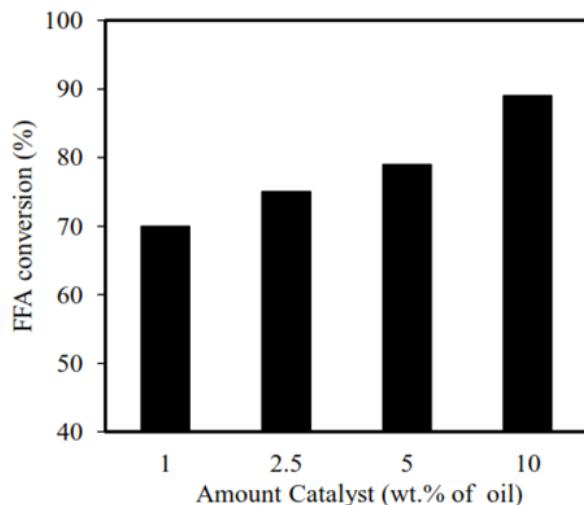
The FFA esterification reaction is limited by an equilibrium state. The equilibrium limitation should be shift to the formation of methyl esters by applying methanol excessively. One mole of methanol is needed to esterify one mole of FFAs. In this research, the mole ratio of oil to methanol was varied in the range of 1:4 to 1:10, while other reaction conditions were kept constant for 2 h at 60°C. As shown in Figure 1, the conversion of FFA was 71% at 1:4 of oil to methanol mole ratio. Furthermore, the conversion increased from 74% to 85% at 1:6 and 1:8 of oil to methanol mole ratio respectively. The maximum conversion at 89% was achieved at 1:10 of oil to methanol mole ratio.

**Figure 2.** Mole Ratio Effect on FFA Conversion.*Reaction Temperature Effect*

The investigation of reaction temperature effect on the FFAs conversion was studied at temperatures range of 30 to 60°C, while percentage of loading catalyst and the oil to methanol mole ratio were maintained stable at 10 wt. % of oil and 1:10, respectively. The reaction temperature effect on conversion was shown on Figure 2. As exhibited in Figure 2, the FFA conversion was 64% at a 30°C of reaction temperature. Subsequently, the conversion achieved 75 and 83% at reaction temperature of 40 and 50°C, respectively. The FFA conversion of 89% was obtained as maximum when the reaction temperature was 60°C.

**Figure 3.** Reaction Temperature on FFA Conversion.*Catalyst Amount Effect*

The catalyst amount effect on FFA conversion was investigated by varying the amount of catalysts within the range of 1 to 10 wt. % of oil. The effect of catalyst amount on FFA conversion is presented in Figure 2. From the results obtained as shown in Figure 2, increasing the catalyst amount results the enhancement of FFA conversion. The maximum conversion was obtained at 10 wt. % of oil catalyst amount.

**Figure 4.** Catalyst Amount Effect on FFA Conversion.

### Conclusions

The biochar-based catalyst from Sengon (*Paraserianthes falcataria* L. Nielsen) Sawdust was successfully synthesized by sulfonation process. The biochar-based catalyst was applied as heterogeneous catalyst for biodiesel production using Palm Fatty Acid Distillate. Reaction parameters such as temperature, oil to methanol molar ratio, and catalyst amount were varied to achieve the highest conversion. The highest free fatty acids (FFAs) conversion of 89.71%, was obtained by using 60°C of reaction temperature at 12:1 molar ratio of methanol to PFAD and 3% catalyst amount.

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**Simulation of Natural Gas Dispersion and Explosion in Vented Enclosure using 3D CFD FLACS Software**

Juwari, Renanto Handogo, Teguh Cahyono, Mochamad Safarudin, Rendra Panca Anugraha, Fadhilatul Aulia, Nuvies Dwi Kurniaty, Muhamad Reza Habibi and Muhammad Nuur Kharisma

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**Integrated life cycle assessment-analytic hierarchy process (LCA-AHP) with sensitivity analysis of phosphorus recovery from wastewater in Metro Manila**

Carla Mae J. Pausta, Luis F. Razon, Aileen H. Orbecido, Devendra P. Saroj and Michael Angelo B. Promentilla

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**Life Cycle Assessment of Refuse Derived Fuel (RDF) for Municipal Solid Waste (MSW) Management: Case Study Area Around Cement Industry, Cirebon, Indonesia**

T T Anasstasia, E Lestianingrum, R B Cahyono and M M Azis

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Dynamic simulation of insulin-glucose interaction in type 1 diabetes with MATLAB Simulink®

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Adsorption of Ammonium Ions Using Activated Carbon Derived from *Artocarpus odoratissimus* Skin

S M Anisuzzaman, A Y Zahrim, M Rajin, S Saalah, N M Ismail, R Duraim and D Krishnaiah

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Effect of Biocover Thickness on Methane Oxidation Rate of Landfill Gas Emission from Municipal Solid Waste Landfill in Tropical Climate Region

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Evaluation of Hydrocarbon Gas Dispersion and Explosion in a Gas Processing Plant

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Attitude and Perception on The Disposal of Pharmaceuticals and Personal Care Products In Malaysia: A Pilot Study

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Safety culture level among workers in the selected manufacturing plant

Junaidah Zakaria, Che Rosmani Che Hassan, Mahar Diana Hamid, Ezrin Hani Sukadarin and Zakaria Razak

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012154



## Adsorption of 4-Nitrophenol from wastewater using Sea Mango (*Cerbera odollam*) based Activated Carbon

Nurul Zufarhana Zulkurnai, Nor Wahidatul Azura Zainon Najib, Umi Fazara Md Ali and Tan Ru Shien

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Total phenolic content and antioxidant activity of *Pandanus amaryllifolius* by soaking and microwave-assisted extraction

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Mass transfer evaluation of substrate, intermediate products and inhibitors in membrane bioreactor on biogas production

M Hidayat, I G Kurniawan, M A Prakosa, S Dökümçü, R Millati and M N Cahyanto

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Extraction of Silica from Rice Straw Using Alkaline Hydrolysis Pretreatment

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Comparative study of Heat Pump Assisted Distillation Column and Its Application for Pressure Swing Distillation Process

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Isobaric vapor-liquid equilibrium of methanol + glycerol binary mixture at 31.3 kPa

M A Batutah, K Kuswandi and G Wibawa

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## Capacitive deionization utilizing Activated Biochar – Manganese Dioxide (AB - MD) nanocomposites for desalination applications

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Estimating species concentration in CO<sub>2</sub>-loaded monoethanolamine using Raman spectroscopy

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Effect of surface-etched modification on halloysite nanotubes (HNTs) for polysulfone mixed matrix membrane in CO<sub>2</sub>/CH<sub>4</sub> separation

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Microwave assisted extraction of lipid from *Nannochloropsis gaditana* microalgae using [EMIM]Cl

S Rezaei Motlagh, R Harun, D R A Biak and S A Hussain

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An Overview: Analysis of ultrasonic-assisted extraction's parameters and its process

Ahmad Syahir, Sarina Sulaiman, Maizirwan Mel, Maizatulnisa Othman and Siti Zubaidah Sulaiman

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Gold Leaching from Printed Circuit Boards (PCBs) as one of the Urban Mine Resources using Thiosulphate: Optimization using Response Surface Methodology (RSM)

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Study of progressive freeze concentration and eutectic freeze crystallization technique for salt recovery

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Carbon dioxide adsorption using biomass-based activated carbon functionalized with deep eutectic solvents

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Comparative Study of Total Phenolic Content and Total Flavonoid Content Extraction from Guava Juices by Progressive Freeze Concentration and Evaporation

M. A. Ahmad, N. S. M. Ghazali, S. Samsuri and M. S. H. Ruslan

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Effect of Stirring Rate and Freezing Time on the Percentage of Recovery of Residual Oil from Palm Oil Mill Effluent via a Stirred Freeze Crystallizer

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Review: A Comparison of Conditions for The Extraction of Vegetable and Essential Oils Via Microwave-Assisted Extraction

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Effect of addition of dodecyl trimethyl ammonium bromide surfactant toward recovery of bitumen from asbuton with modified hot water process method

Y N ARIQ, F A Rahmatika, M F Susanto, Susianto, A Altway and Z L Rinanda

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Assessment of patterned membrane in a tilted panel filtration system for fouling control in activated sludge filtration

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Preparation of Highly Hydrophobic PVDF-HFP Membrane with Anti-Wettability Characteristic

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Fabrication of Polythiourea-Copper Complex Composite Membrane and its Anti-fouling Property

M T Margarito, A B Beltran, MA Promentilla, A Orbecido, B Basilia, R Damalerio and U Bigol

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