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UTILIZATION OF AVOCADO SEED WASTE IN THE SYNTHESIS OF SULFONATED CARBON CATALYSTS USING CHLOROSULFONIC ACID FOR BIODIESEL PRODUCTION

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ABSTRACT

A series of sulfonated avocado seed carbon catalysts for biodiesel production was successfully synthesized. The carbonization of avocado seed has been varied at 300°C, 400°C, 500°C, and 600°C using nitrogen gas purging followed by sulfonation applying HSO_3Cl (chlorosulfonic acid) in chloroform solvent. The designed catalyst has been characterized using FTIR, XRD, and TPD-NH_3. The results show strong absorption peaks in FTIR spectra at the wave numbers of 1140 and 1030 cm^{-1}, which indicates an O-S-O bonding vibration in asymmetric and symmetric stretching. Meanwhile, the XRD patterns show two main peaks, namely the broad peak at 2θ 10°-30° corresponding with the C (002) plane and the sharp peak at 2θ 40°-45° corresponding with the C (101) plane. The TPD-NH_3 reveals that the catalyst with the highest acid site number showed for sulfonated avocado seed carbon calcined at 400°C (AS400-S) sample. The catalytic activity shows the designed catalyst can convert PFAD to form biodiesel until 64.6061%.

Keywords Biodiesel, Chlorosulfonic Acid, Sulfonated Carbon Catalyst, Avocado Seed, Palm Fatty Acid Distillate

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INTRODUCTION

The increasing consumption of fossil fuels causes several problems such as depleting oil availability, air pollution, and global warming.[1,2] It is encouraging researchers to investigate energy sources from renewable materials, sustainable and ecologically aware. Among the renewable energy sources, biodiesel is one of the alternative fuels being developed in Indonesia because the raw materials and catalysts used in the reaction can be obtained from organic waste.[3] Biodiesel feedstock based on waste materials is now being developed due to its biodegradability, low carbon emission, and low production costs.[4,5] The feedstocks used can be obtained from waste containing high triglycerides such as used oil or those containing high free fatty acids such as animal fat and palm fatty acid distillate (PFAD) from the residue of crude palm oil (CPO) process.[6,7] As the world's largest producer of palm oil, Indonesia has discovered PFAD (palm fatty acid distillate) in a large amount of organic waste produced by CPO processing.[8] The organic waste contains FFA > 85% so it can be converted to form FAME (Fatty Acid Methyl Ester) through an esterification reaction.[9] The esterification reaction is a slow equilibrium reaction that needs a catalyst to induce its reaction rate and increase its production.[10] For a feedstock with high FFA content, such as PFAD, the eligible catalyst is a catalyst with acid properties because it is not affected by the water released from the esterification reaction.[11] Sulfonated carbon catalyst is a solid acid catalyst that has many uses in biodiesel production.[1213] This catalyst can also be synthesized from organic waste containing starch, cellulose, hemicellulose, and lignocellulose.[14] Several wastes, like empty palm bunches[15], corncobs[16], palm shells[17], and sugarcane[18], were reported in the synthesis of sulfonated carbon-based solid acid catalysts as carbon sources.[19]

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The avocado seed is a solid organic waste released from food commercial processes having economic value. This solid waste has been applied as starch resources, animal feeding, and absorbent.[18] Based on the composition, avocado seed has about 60-80% starch, which makes this waste a potential alternative carbohydrate resource (especially starch).[19,20] Due to the high starch content in avocado seed, therefore, it has the potential to be used as an alternative carbon source of sulfonated carbon catalysts. However, there has not been found the use of carbon from avocado seeds in the synthesis of sulfonated carbon-based solid acid catalysts until now.[19] The purpose of this study was to apply carbon material produced from the calcination of avocado seeds in the synthesis of sulfonated carbon catalyst for biodiesel production from waste cooking oil. The sulfonation process has been conducted by mixing chlorosulfonic acid in a chloroform solution.[20] Chlorosulfonic acid can be used as an alternative source of sulfonic acid besides sulfuric acid because it can be used in the same amount as the amount of carbon sample to be sulfonated.[21] The sulfonation process can take place at lower temperatures and the amount of water used is less for the sample neutralization process. The synthesized catalyst is characterized by FTIR, XRD, and TPD-NH_3, and applied for biodiesel production from PFAD. The catalytic activity of the synthesized catalysts was determined by analyzing the density and acid number as well as calculating the percent conversion of the biodiesel produced.

EXPERIMENTAL

Material and Methods

The chemicals used for this work were divided into 2 parts including materials for the synthesis of catalyst and biodiesel production. The materials used for the synthesis of the catalyst consisted of avocado seed, HSO_3Cl (Merck, 97%), chloroform (Merck, 99.5%), and aquadest. The materials used for biodiesel production included PFAD, methanol, and catalyst. An avocado seed was obtained from the fruit market, while PFAD was collected from Wilmar Company. To get the mole of PFAD, this study used the molecular weight of PFAD obtained from its saponification number.

General Procedure

Preparation of Catalyst

As much as 100g of avocado seeds that had been dried and mashed were calcined under nitrogen flow in a cylindrical furnace with varied temperatures (300°C, 400°C, 500°C, and 600°C) for 2h. The resulting carbon was then mixed with HSO_3Cl in a 1:1 ratio. The mixture was added into 50 ml of chloroform in a three-neck bottle and continued by refluxing the mixture at 70°C for 4h. Furthermore, the sulfonated carbon sample was washed to remove the unreacted HSO_3Cl with hot water and then oven dried at 80°C for 24h and produced a sulfonated carbon from avocado seeds. The sulfonated carbon obtained was characterized by FTIR, XRD, and TPD-NH_3 and afterward applied for biodiesel production.

Application of Sulfonated Avocado Seed Carbon

Biodiesel produced from the reaction between FFA in PFAD and methanol with the addition of the sulfonated avocado seeds carbon catalyst was conducted in a 250 ml three-neck bottle provided with a thermometer (360°C), condenser, heater, magnetic stirrer, and water bath. The mole ratio of methanol and PFAD was made as 6:1 and the catalyst loading were 3% of PFAD as well as 25g PFAD. A catalyst 3% and 6:1 mole ratio of PFAD to methanol was mixed in a three-neck bottle and then agitated at 100 rpm for 3 min and afterward, 25g PFAD was added slowly into the three-neck bottle. The mixture was agitated at 300 rpm at 65°C for 2h. After the reaction was completed, the product would separate with centrifuges for 10 min at 3000 rpm. This step is to separate the product from the catalyst used. The product consisted of biodiesel and methanol. Then the product was heated at 70°C to remove excess methanol in biodiesel production. The acid number and density of biodiesel products were analyzed to determine the catalytic activity of the synthesized catalyst.[19]

Characterization and Analysis of the Sample

The characterization of the sample was conducted with FTIR (Fourier Transform Infrared Thermo Fisher Scientific), XRD (X-Ray Diffraction PHILIPS PW 3040/60 MPD X'PERT HIGH PRO ANALYTICAL), and TPD-NH_3 (Temperature Programmed Desorption (TPD) Thermo Finnigan, TPDRO 1100 series equipped with thermal conductivity detector).[22]

Equation of Properties of the Biodiesel:

Density

$$\rho = \frac{m\ (gram)}{v\ (ml)} \quad (1)$$

Acid number (AN) [23]

$$AN = \frac{v\ KOH\ (ml)\ x\ M\ KOH\ x\ Mw\ KOH}{m\ sample\ (gram)} \quad (2)$$

Saponification number (SN) [23]

$$SN = \frac{[(vol\ HCl\ Blank - vol\ HCl\ sample\ x\ N\ KOH\ x\ Mw\ KOH}{m\ sample\ (gram)} \quad (3)$$

$$Mr = \frac{56{,}1\ x\ 1000}{SN}$$

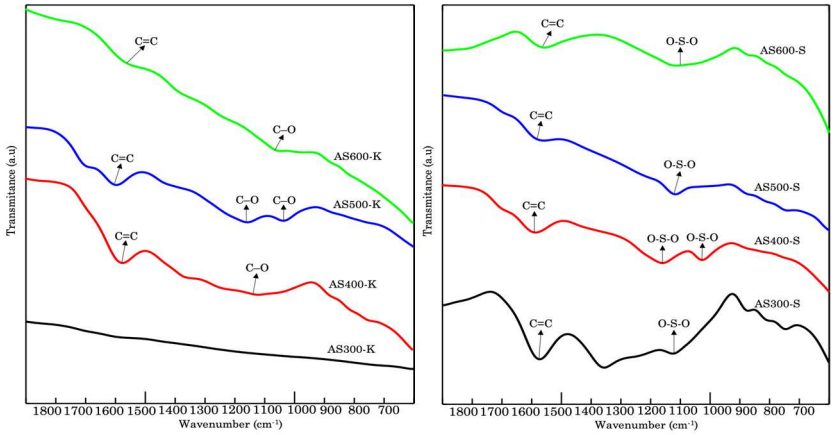
Table-1: The Samples Applied in This Work are as Follows

Calcination temperature	Carbon	Sulfonated carbon
300ºC	AS300-K	AS300-S
400ºC	AS400-K	AS400-S
500ºC	AS500-K	AS500-S
600ºC	AS600-K	AS600-S

RESULTS AND DISCUSSION

FTIR Spectra

An FTIR spectrophotometer was used to characterize the avocado seed samples after calcination and continued with sulfonation. All samples were characterized at the wavenumber of 1900-600 cm^{-1} because it is sensitive to the functional groups of starch and fatty acid in avocado seeds such as C=O, C=C, and C-O bonds [24]. The existence of functional groups in the avocado seeds carbon obtained by calcination in FTIR spectra is presented in Fig.-1. The wave numbers around 1600-1500, 1100-1000, and 1300-1030 cm^{-1} were detected as the main absorption bands of calcined and sulfonated samples in the FTIR spectra.[25] The absorption bands at wavenumber 1600-1500 cm^{-1} show a vibration of C=C bond indicating the existence of an aromatic ring structure in the polyaromatic structure of calcined avocado seed carbon.[26] A similar result is also shown for calcinated sulfonated avocado seed carbon catalyst as shown by absorption peak at a wave number of 1100-1000 cm^{-1} due to C-O bond from functional group C-O-H in tannin and C-O-C bond of glycoside in starch.[27,28] The presence of absorption peaks of calcinated avocado seed carbon shows that the calcination process yields incomplete carbonization.[29] Therefore, the sulfonation process can be conducted for calcinated avocado seed carbon.[26]



(A) (B)

Fig.-1: FTIR Spectra of Calcined (a) Carbon and (b) Sulfonated Avocado Seed Carbon Catalyst. Calcination 300, 400, 500, and 600ºC. HSO_3Cl. N_2 Purging. Chloroform Solvent.

The presence of a sulfonic group in all sulfonated calcined avocado seed carbons is observed by a strong absorption peak at wave number 1140 cm^{-1}. This absorption peak is due to the asymmetric vibration of the O-S-O group.[30,31] The AS400-S sample shows an additional strong absorption peak at wave number 1030

cm^{-1} related to the existence of symmetric vibration of the O-S-O group.[32,33] This finding shows that the -SO_3H group covalently bonded to calcined carbon.[34]

XRD Pattern

Moreover, all sulfonated avocado seed carbons were analyzed by XRD using 2θ 10-60° with a speed of 2°/min. The result of XRD characterization is illustrated in Fig.-2. As seen in Fig.-2 all diffraction patterns in Fig.-2 have two main peaks, i.e. a broader peak at 2θ 10-30° related to C (002) plane and a sharp peak at 2θ 40-50° related to C (101) plane.[35,36] The broader peak at around 2θ 10-30° shows typical characteristics of amorphous carbon containing layers of aromatic carbon. At the moment, at 2θ 40-50° shows a sharp peak of crystalline sulfonated avocado seed carbon catalyst.[27,37] Both peaks show the existence of an amorphous carbon structure consisting of carbon rings of polycyclic aromatic with random orientation.[38,39] The C (101) plane is due to the presence of graphite.[40,41] This finding shows that the sample consisted of greater carbon layer stacks that affected catalytic action during the esterification reaction process. The greater carbon layer gives opportunities for pasting more SO_3H groups. The carbonization process at higher temperatures is proportional to the sulfonation process using strong acid yielding a reduction of C-O-C bonding in carbon sample.[10,25] This process yields more rigid and amorphous sulfonated carbon due to increasing retardation in carbon layers.

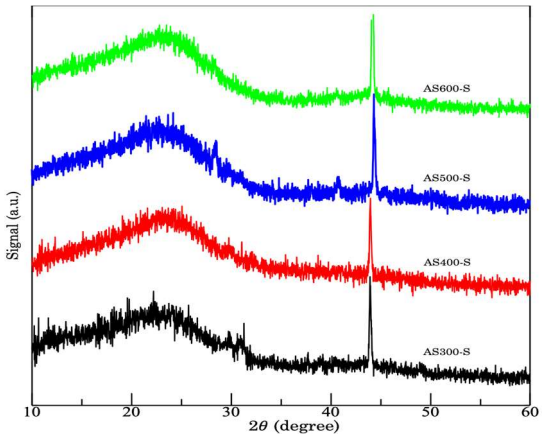


Fig.-2: XRD Spectra of Sulfonated Avocado Seed Carbon. Calcination 300, 400, 500, and 600°C. HSO_3Cl. N_2 Purging. Chloroform Solvent

TPD-NH_3

The TPD-NH_3 examination is used to determine the acidity on the catalyst surface.[42] The temperature of desorption is a measure of active acid sites while the region of the desorption band is a measure of acid site number on the catalyst surface[4344] The desorption band lower than 200˚C shows weak acid sites and the desorption band between 200 and 400°C is related to medium acid sites. While the desorption band higher than 450°C is related to strong acid sites.[45]

Figure-3 shows the characterization of sulfonated avocado seed carbon catalyst applying TPD-NH_3. The desorption band NH_3 observed for all catalysts at 100-200°C related to the existence of weak Brønsted acid sites.[46,39] This finding is due to the presence of free fatty acids in an avocado seed.[47] The second peak observed in the region around 200-450°C is related to the presence of medium Brønsted acid. This second peak is related to the existence of a tannin compound in avocado seed. The third peak in the region of 450-900˚C is related to the presence of strong Brønsted acid sites.[48] This third peak is due to the existence of the SO_3H group pasted to the carbon layer of polycyclic aromatic in sulfonated avocado seed carbon.

Table-2: Active Acid Site of Sulfonated Avocado Seed Carbon.

Sample name	Total acid (μmol/g)
AS300-S	4616.81862
AS400-S	9344.26222
AS500-S	5004.32914
AS600-S	4567.88573

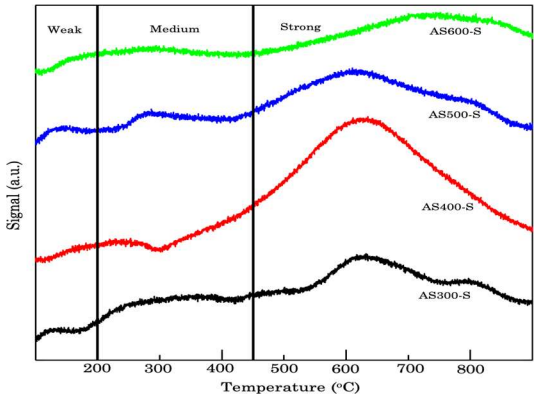


Fig.-3: (a)TPD-NH_3 Spectra of Acid Site Number (-SO_3H) in Sulfonated Avocado Seed Carbon. Calcination 300, 400, 500, and 600°C. HSO_3Cl. N_2 Purging. Chloroform Solvent

The analysis data of TPD-NH_3 reveals that the highest acid site number is shown by the AS400-S sample. This finding agreed with the graphic pattern related to high intensity at a higher temperature than 450°C showing the presence of strong acid in the AS400-S sample.

Biodiesel Production Applying PFAD

All synthesized catalysts have been applied for biodiesel products yielded from the reaction of PFAD and methanol. The products have been analyzed to examine the catalyst activity. The tests of density and acid number have been conducted to examine the catalytic activity of those catalysts. The physicochemical properties of catalyst products strongly influenced the biodiesel product, particularly in the presence of SO_3H acid site number formed at the catalyst surface.

Examination of Biodiesel Properties

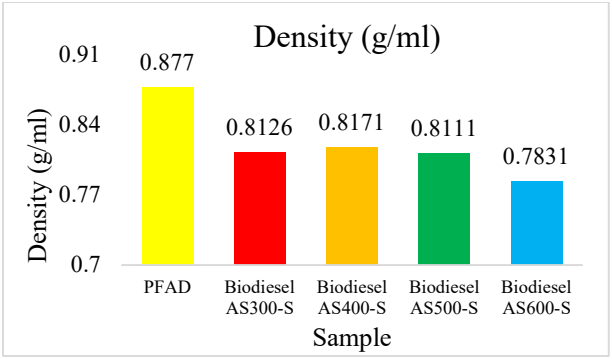
The examination of biodiesel properties included density and acid number. The result data of density and acid number examinations are shown in Table-3. Based on the examination the results show that the density and an acid number of biodiesel products are lower than that of PFAD. The reduction shows the existence of catalyst activity.

Table-3: The Properties of PFAD and Biodiesel Applying Sulfonated Avocado Seed Carbon Catalyst

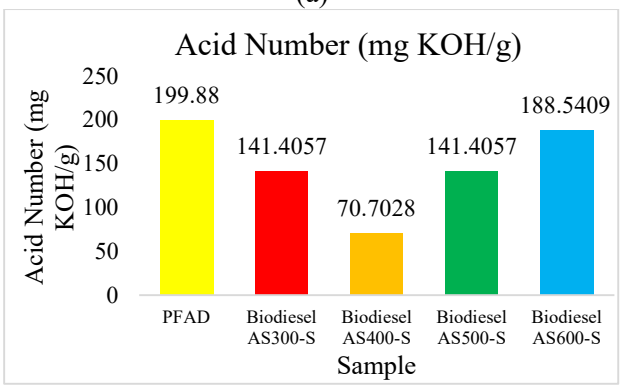
Sample	Examination of biodiesel properties		
	Density (g/ml)	Acid number (mgKOH/g)	FFA Convertion (%)
PFAD	0.877	199.88	-
Biodiesel AS300-S	0.8126	141.4057	29.2122
Biodiesel AS400-S	0.8171	70.7028	64.6061
Biodiesel AS500-S	0.8111	141.4057	29.2122
Biodiesel AS600-S	0.7831	188.5409	5.6163

The biodiesel density is compared to the standard value. According to ASTM D5002, the standard density of biodiesel using PFAD is recorded as 870 ± 0.01 kg/m^3. The value of biodiesel density obtained shows the nearest value to the standard value, that is the biodiesel product applying AS400-S catalyst. The catalytic activity is influenced by the active acid site number of the catalyst, which is illustrated in Fig.-4. The examination of acid number is to determine the percentage of FFA conversion from PFAD to biodiesel. The lower acid number yielded a higher production of biodiesel. Based on the examination result of acid number it shows that the lower acid number of biodiesel products is related to the higher active acid site number of catalysts applied. The lowest acid number of biodiesel products applied was the AS400-S catalyst. The biodiesel with an acid number higher than the standard value causes corrosion problems in the engine system. It can be deduced that an increased active acid site in the catalyst yields a lower acid number of biodiesel products. The percentage of FFA conversion is calculated based on the difference in acid number between the feedstock and the product. Based on % FFA conversion, it shows an elevation of catalytic activity of samples until the AS400-S sample. After that, the catalytic activity reduced which

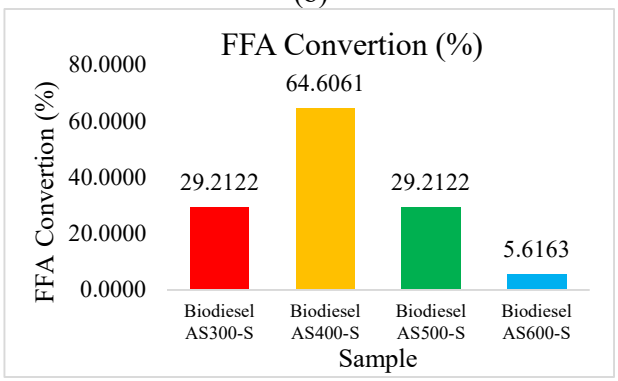
showed for AS400-S until AS600-S. This finding is related to the active acid site number of AS400-S being higher than that of AS300-S, AS500-S, and AS600-S samples. The existence of active acid sites may increase the catalytic activity of sulfonated avocado seed carbon catalysts.



(a)



(b)



(c)

Fig.-4: Correlation between Active Acid Site Number (-SO_3H) of Sulfonated Avocado Seed Carbon Catalyst and (a) Density, (b) Acid Number, and (c) % FFA Conversion. Calcinations 300°C, 400°C, 500°C, and 600°C. HSO_3Cl. N_2 Purging. Chloroform Solvent.

CONCLUSION

The solid acid catalyst as the sulfonated avocado seed carbon catalyst can be synthesized through calcinations with nitrogen purging followed by a sulfonation process applying HSO_3Cl in chloroform solvent. The characterization of the designed catalyst is determined by applying FTIR showing a strong absorption band at wave numbers 1140 cm^{-1} and 1030 cm^{-1}. The XRD characterization showed the existence of amorphous and crystalline regions. Moreover, the TPD-NH_3 characterization showed that AS400-S had the highest active acid site number. The sulfonated avocado seed carbon as a solid acid catalyst can be used for biodiesel production. The catalyst can convert PFAD to form biodiesel until 64.6061%. The density and acid number of biodiesel products have undergone a reduction compared to that of PFAD feedstock.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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