Synthesis and Characterization of Sodium Silicate Produced from Corncobs as a Heterogeneous Catalyst in Biodiesel Production

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Abstract: *In this study, silica derived from corncobs impregnated with sodium hydroxide to obtain sodium silicate was calcined, prepared, and employed as a solid base catalyst for the conversion of oils to biodiesel. The catalyst was characterized by X-Ray Diffraction (XRD), Fourier-Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscope Energy Dispersive X-Ray Spectroscopy (SEM-EDS), and Brunauer-Emmet-Teller (BET) and Barrett-Joyner-Halenda (BJH) methods. Gas Chromatography-Mass Spectrometry (GC-MS) was used to characterize the biodiesel products. The optimum catalyst conditions were calcination temperature of 400 °C for 2 h, catalyst loading of 2%, and methanol: oil molar ratio of 12:1 at 60 °C for 60 min, that resulted in a yield of 79.49%. The final product conforms to the selected biodiesel fuel properties of European standard (EN14214) specifications. Calcined corncob-derived sodium silicate showed high potential for use as a low-cost, high-performance, simple-to-prepare solid catalyst for biodiesel synthesis.*

Keywords: *biodiesel; corncobs; silica; heterogeneous catalyst*

■ INTRODUCTION

Currently, alternative fuels for diesel engines are particularly crucial because of diminishing petroleum reserves and the environmental consequences of exhaust gases from petroleum-fueled engines. Biodiesel is a good candidate for replacing petrol-diesel fuel because it is clean, renewable, biodegradable, non-toxic, and environmentally friendly [1-2]. Biodiesel can be produced via a direct transesterification reaction between renewable vegetable oils or animal fats source and alcohol in the presence of a catalyst [3]. In the transesterification reaction, a triglyceride reacts with an alcohol in the presence of a catalyst, producing a mixture of fatty acid methyl esters (FAME) and glycerol [4]. Usually, KOH and NaOH are used as homogeneous base catalysts because of their high catalytic activity, which enables the reaction to complete in 1 h at 40–60 °C [5]. However, the use of these catalysts involves some drawbacks, such as its removal and inability to be reused or regenerated, difficult catalyst separation, soap formation, and reactor corrosion. Also, a large amount of water is required to wash the biodiesel product to eliminate

the catalyst, leading to an increase in the overall production costs and environmental problems [6].

The use of heterogeneous catalysts could be an attractive solution to these problems. Heterogeneous catalysts can be easily separated through filtration, reused and recycled several times, and requires no neutralization or washing process. In addition, they can also produce high-purity glycerol as a side product. Hence, heterogeneous catalysts are effective and efficient materials for biodiesel production [7-9]. Sodium silicate with the formula $Na_2O \cdot nSiO_2$ (commonly known as water glass or liquid glass) can be easily prepared from silica and sodium hydroxide [10-11]. It is an effective solid base catalyst for the transesterification process at operating conditions of 60 °C and 60 min. Furthermore, it can be reused at least five times without loss of activity [12-13]. Silica is widely used in various industries as catalysts [14], catalyst support materials [15-16], and used in ceramics [17]. Silica is highly stable, chemically flexible, biocompatible [18], and can be obtained from minerals and vegetable ingredients.

Corncobs are a form of agricultural waste obtained from corn plants. Corncob ash contains > 60% silica, together with a small amount of metal elements [19] and is an economical raw material for the production of silicates, silica, and silica nanoparticles. It is obtained as a fine powder after combustion and further grinding.

The focus of this research is the synthesis and characterization of calcined sodium silicate solid catalysts derived from waste corncobs via impregnation with the alkali metal sodium. The obtained material is used as a heterogeneous catalyst in biodiesel production from palm oil and methanol. The European standard method (EN 14214) for biofuels is used to evaluate the fuel properties of the obtained biodiesel after purification and treatment.

■ EXPERIMENTAL SECTION

Materials

Refined, bleached, and deodorized palm olein (RBDPO) containing 0.15% FFA and 0.06% moisture purchased from Deli Serdang, Indonesia, was used as the raw material. Other materials used were methanol from Merck, with a purity of > 99.5%; HCl 37% from Sigma-Aldrich, NaOH from Sigma-Aldrich with a purity of > 97%, and corncobs collected from Deli Serdang, washed with deionized water and dried at 110 °C overnight.

Instrumentation

Crystallinity of materials both before and after calcination was analyzed by XRD using a Bruker with Cu voltage of 40 kV and a current of 30 mA. The morphological structure and composition of the catalyst were characterized using SEM-EDS EVO-10 Zeiss and GSA Quantachrome to investigate the surface area, pore diameter, and pore volume of the catalyst. The functional groups of the catalyst were analyzed by FTIR Shimadzu IR-21 with a range of 450–4000 cm^{-1}, while GC-MS Shimadzu-2010 was used to investigate the obtained biodiesel products.

Procedure

Preparation of corncob ash (CCA)

Dry corncobs, which had been sifted with 100 mesh filters, were calcined at 700 °C for 3 h to remove organic compounds [16]. Then, 10 g of corncob ash was washed with 60 mL HCl 1 M to eliminate organic compounds and increase the silica content. The sample was then dried again in an oven at 105 °C for 2 h and cooled to room temperature to obtain the corncob ash for further experiments [15-16].

Catalyst preparation

The sodium silicate was prepared using the wet impregnation method. As an initial step, corncob ash was dissolved in water, and the NaOH solution was then slowly added to the suspension. The mixture of NaOH:CCA with a mole ratio of 1:2 was then stirred at 300 rpm and heated at 80 °C for 2 h [20]. After the impregnation process was completed, the slurry was dried at 150 °C for 1 h and calcined at 400, 500, and 600 °C for 2 h [15,21]. The reaction for sodium silicate formation from corncob ash is as follows:

$$2NaOH_{(1)} + nSiO_{2(s)} \rightarrow Na_2O \cdot nSiO_{2(s)} + H_2O_{(1)}$$
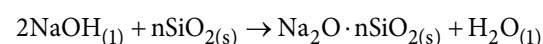
Transesterification process

The transesterification process was conducted in a three-neck flask-batch reactor system with condenser reflux equipment with a magnetic stirrer rate of 300 rpm equipped with a thermometer. The catalyst was mixed with methanol for 1 h at room temperature and added to 25 g of RBDPO. The biodiesel obtained was then filtered to separate the catalyst. The washing process was then carried out using 500 mL water, followed by heating at 105 °C to remove the water content and the remaining methanol.

Product analysis

Biodiesel products were analyzed to determine the levels of methyl ester using GC-MS Shimadzu-2010. A digital density hydrometer (ZEAL) was used to measure the density of biodiesel. Meanwhile, the viscosity of the biodiesel was measured using a Cole-Parmer rotational viscometer with stainless steel spindles and viscosity measurements ranging from 20 to 2×10^6 mPa. The biodiesel was tested according to the European standard method (EN14214) for fuels derived from vegetable oils. The yield of biodiesel is determined as follows:

$$\text{The yield of biodiesel } (\%) = \frac{\text{Weight of biodiesel}}{\text{Weight of oil}} \times 100$$

■ **RESULTS AND DISCUSSION**

FTIR Analysis

FTIR provides information on catalyst surface characteristics in terms of the structure of metal oxides, with the position of the bands or peaks showing metal-oxygen bonding. Fig. 1 presents the results of the FTIR analysis of corncob ash and the sodium silicate catalyst. In all samples, there is a Si–OH function group that appears at a wavenumber of 3151–3383 cm^{-1}. The hydroxyl group found in this sample is water vapor bound to the Si element as a good adsorbent [16]. No absorptions were found at 2800–3000 cm^{-1} in any samples, suggesting the absence of remaining organic compounds as a result of the silica purification process [15]. Furthermore, a Si–OH group with flexible vibrational mode was found in corncob ash at 1631.78 cm^{-1}. The presence of bending vibration of water molecules bound to the silica in the catalyst is observed at the wavenumber of 1662.64–1666.50 cm^{-1} [15]. The same phenomenon was reported by Attol and Mihsen in which a broadband appeared at 3200 and 3600 cm^{-1} due to the O–H stretching vibrations of the Si–OH group and water absorbed onto the silica surface of rice husk ash [22]. In addition, O–H stretching vibrations of the silanol group (Si–OH) appears at the wavenumber of 3426 cm^{-1} for silica gel from rice husk ash, as reported by Sakti et al. in 2013 [23].

In corncob ash, there is a functional group Si–O–Si (siloxane group), which can be seen at wavenumber of 1080.14 cm^{-1} and on the catalyst at 1454.33 cm^{-1}, where this siloxane group will transform into Si–O–Na group. This group can be observed between the wavenumbers of 871–983 cm^{-1} [13,15-16]. The presence of this Si–O–Na group was obtained from the wet impregnation process using alkali metal in the form of NaOH in the corncob ash, indicating a successful synthesis of sodium silicate solid catalyst. The results of the sodium silicate synthesis are compared with commercial sodium silicate, where the stretches located at the wavenumbers of ~1005, 941, and ~1400 cm^{-1} correspond to Si–O–Na and Si–O–Si stretches [24]. The results of the FTIR show that the synthesis of sodium silicate from corncob ash does not differ significantly from commercial sodium silicate, except for the –OH group absorption at the wavenumber of 3600 cm^{-1}.
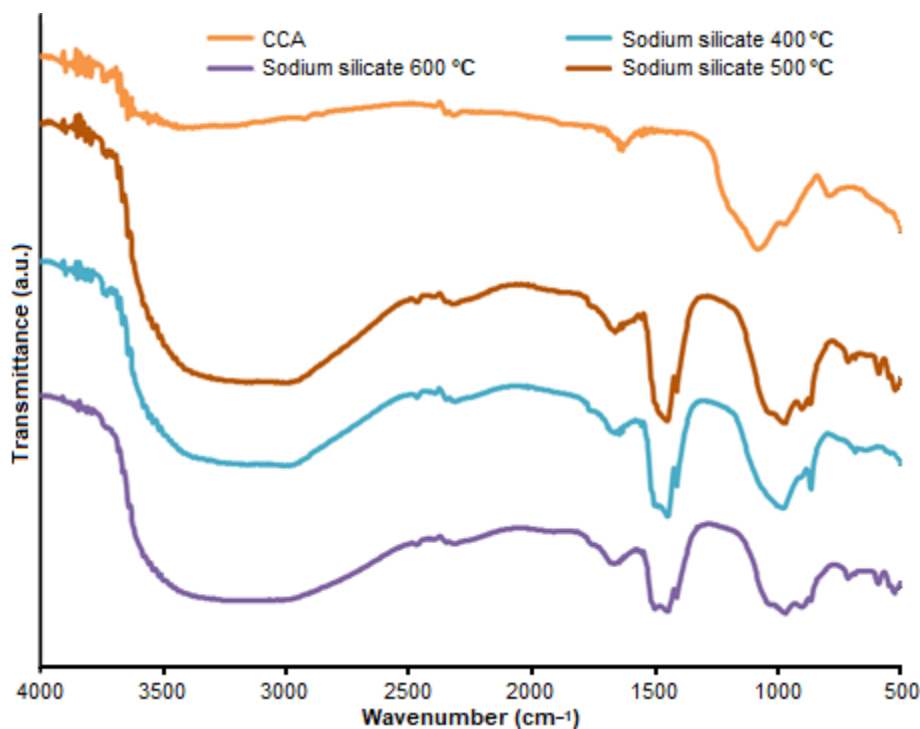

Fig 1. FTIR spectra of CCA and sodium silicate

XRD Analysis

Characterization by XRD is intended to identify catalyst bulk phase and to determine the crystallinity of the catalyst. Amorphous analysis of corncob ash and the crystalline structure of the catalyst using XRD were obtained. Fig. 2 shows the XRD results of corncob ash in the range of $2\theta = 16-40°$ with sharp peaks at $2\theta = 21.8557$, 26.6516, and 28.0366°, suggesting that the silica particles are amorphous structures [15-17]. The results indicate that corncob ash is amorphous and can be used as a catalyst buffer with alkali metals since it has a random and irregular pattern of atoms and molecules so that in various conditions, amorphous silica is more reactive in the presence of hydroxyl groups.

The effect of calcination temperature can be seen in the crystalline phase of the sodium silicate catalyst. In Fig. 2, the catalyst obtained a range of $2\theta = 17.17-67.10°$, with the strongest peak at 29.50°, indicating the formation of the crystalline phase by calcination [13,15-16]. Thus, the process of impregnation of NaOH onto calcined silica corncob ash was successfully carried out to form a crystalline phase of the sodium silicate catalyst.

BET and BJH Analysis

Generally, textures of pore structures of particles are characterized as surface area, pore size distribution, and pore shape. Configuration of the heterogeneous catalyst surface area (m^2/g) is a crucial criterion for solid catalysts because it determines the number of active sites for catalyst activity. Surface area, pore volume, and pore size distribution are also important parameters since they can control displacement phenomena and selectivity in catalytic reactions.

The BET method is used in determining the surface area of solids. This method is based on the kinetic model of the Langmuir adsorption process.

All samples were measured for the specific surface area using BET for nitrogen gas sorption at a liquid nitrogen temperature of 77.35 K. Upon impregnation and calcination of alkali metals, the BET surface area decreased as a result of the sintering effect (Table 1). The relative pressure in the range of 0.1–0.4 corresponds to the formation of the monolayer nitrogen molecule on the surface of the material, as shown by the initial linear curve in Fig. 3. The study of pore uniformity is particularly important in the synthesis of mesoporous solids because pore uniformity determines the quality of the solid as a catalyst. The level of uniformity is ascertained by looking at the distribution patterns obtained from BJH nitrogen isotherm adsorption data. Fig. 4 shows the uniformity of pores with a radius of 17–20° (Table 1). From Fig. 4, it can be seen that the pore
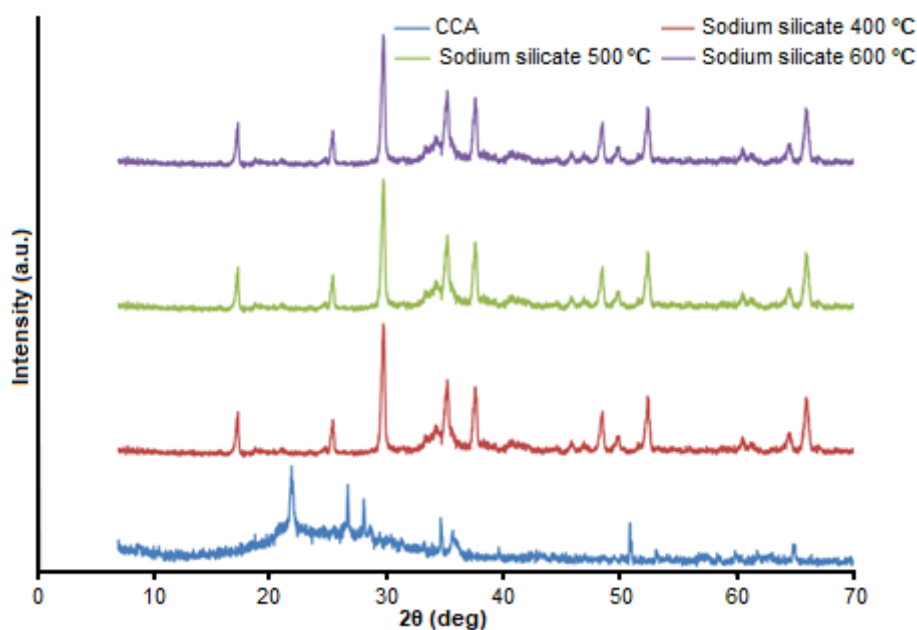

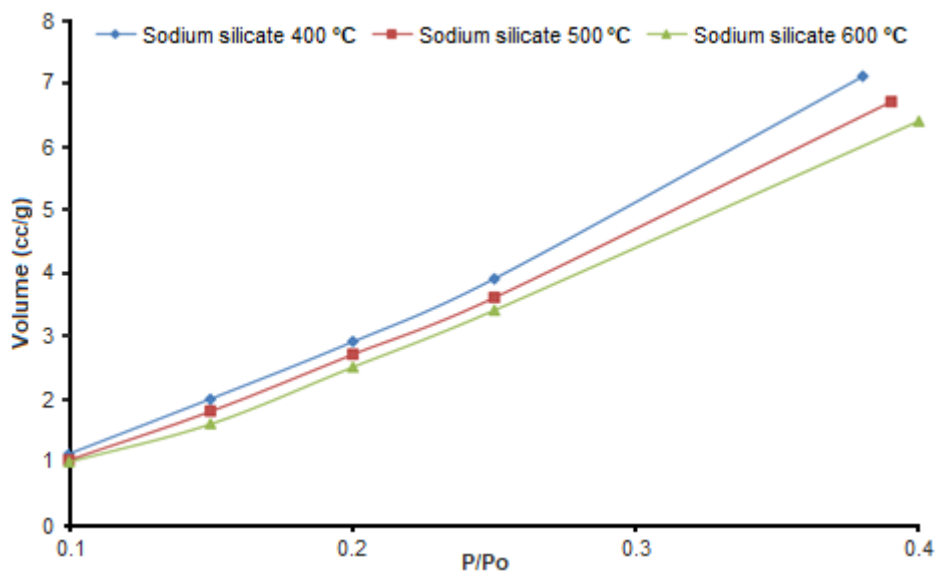**Fig 2.** XRD patterns of corncob ash and sodium silicate
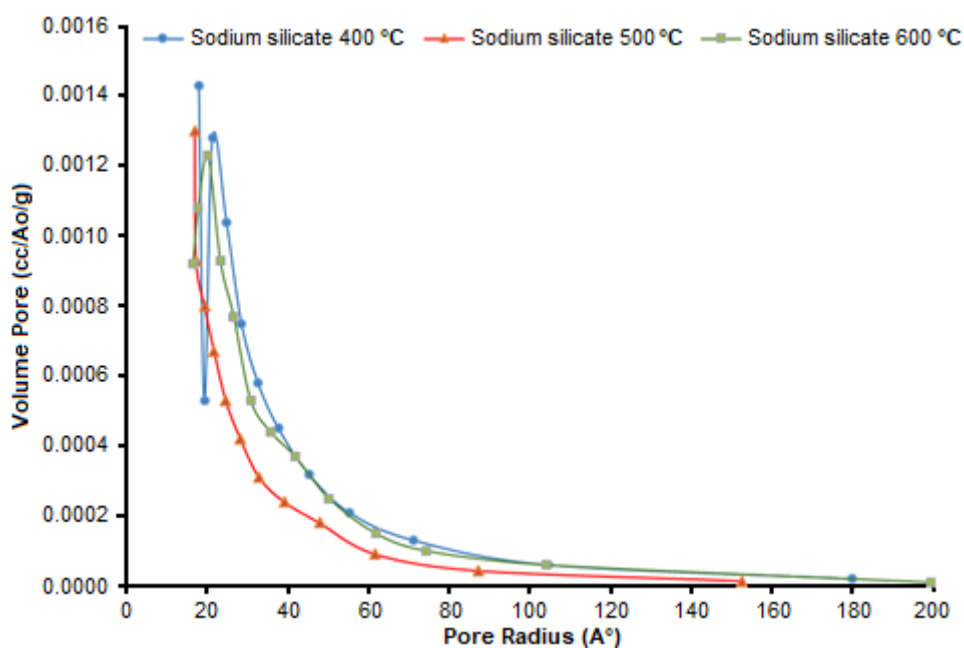
Fig 3. Multipoint sodium silicate curve

Fig 4. Sodium silicate pore distribution curve

Table 1. Surface analysis and pore structure of sodium silicate

Sample	S (BET) (m²/g)	V (BJH)(cm³/g)	D (nm) (BJH)
Sodium silicate 400 °C	72.824	0.034	30.42
Sodium silicate 500 °C	67.846	0.033	34.22
Sodium silicate 600 °C	22.868	0.021	40.16

uniformity level of sodium silicate is quite high, as indicated by the pore distribution pattern not having widened [25].

The average pore diameter of sodium silicate with different calcination temperatures is summarized in Table 1. Even though the value of the porous diameter is

almost the same, sodium silicate at 600 °C with a ratio of 1:2 has a more homogeneous pore distribution [15].

Based on Table 1, it can be seen that the sample has a surface area that shrinks with increasing calcination temperature, possibly due to the sintering process [16]. Furthermore, the pore volume decreases with increasing calcination temperature. Note that the sintering process at higher temperature resulted in a smaller surface area and pore volume, inversely proportional to the pore radius.

SEM-EDS Analysis

SEM analysis was carried out to determine the morphology of the sodium silicate catalyst. Fig. 5 shows a significant agglomeration of particles on the surface of the amorphous silica. The pores within the agglomerates were found to be smaller as silica content increased. The EDS profile of sodium silicate contains predominantly the elements listed in Table 2, which are Na, O, and Si. Both Si and O peaks correspond to the silica, and the signal of carbon is originated from carbon coating in the SEM-EDS analysis.

This indicates that the sodium content of corncobs raw material is higher than rice husk; however, the higher sodium content shows that the alkaline impregnation has been successful.

The Effect of the Calcination Temperature of the Catalyst on the Yield of the Biodiesel

Sodium silicate was applied as a catalyst in the palm oil transesterification process. The amount of catalyst was 2% (w/w oil), while the molar ratio of the methanol to oil was 12:1. The reaction was kept at 65 °C for 1 h. The raw material used was RBDPO obtained from crude palm oil purification with an FFA of 0.15% and moisture of 0.06%.

Table 2. Elemental composition sodium silicate catalysts

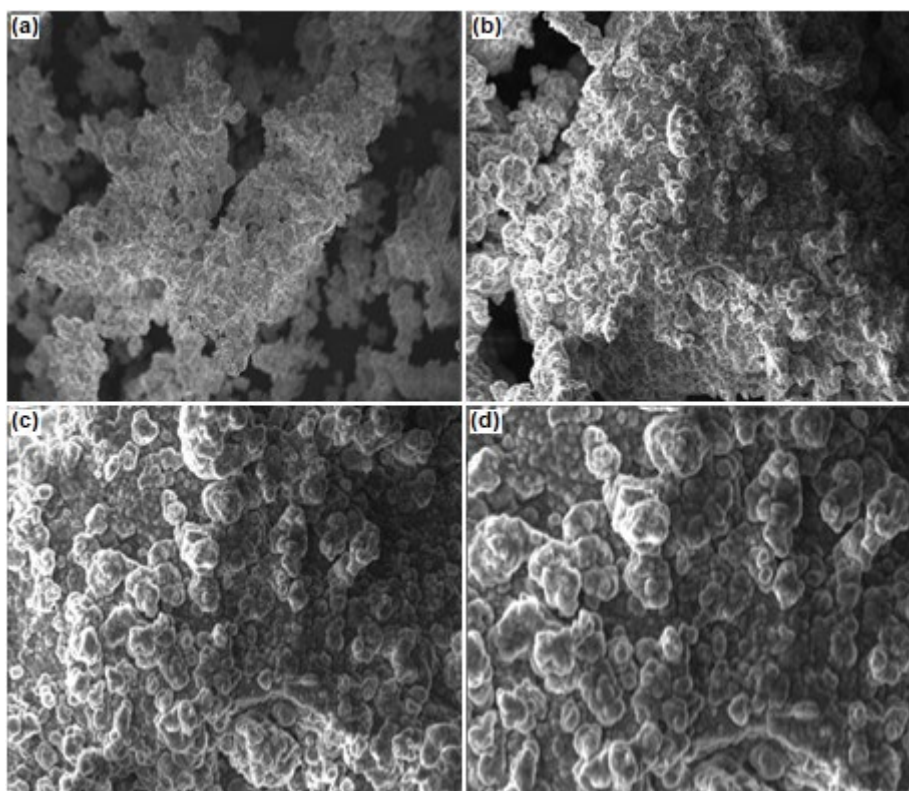| Sample	Na (%)	O (%)	Si (%)
Sodium silicate from corncob ash	43.29	36.48	20.23
Sodium silicate from rice husk [26]	22.63	71.98	5.39

Fig 5. Surface morphology: (a) corncob ash; (b) sodium silicate 400 °C; (c) sodium silicate 500 °C; (d) sodium silicate 600 °C

Biodiesel is composed of fatty acid methyl esters, and their composition is commonly determined by GC analysis. Analysis result of the current study is presented in Table 3 which shows that seven types of methyl ester compounds were found in the biodiesel produced from RBDPO. Unsaturated and saturated fractions were obtained at 98.04 and 1.3285%, respectively, while the overall level of FAMEs was 99.3685%. It can be stated that the oil was successfully transesterified to biodiesel using the sodium silicate catalyst. The performance of calcined sodium silicate for transesterification of RBDPO was compared with other reported solid catalysts using palm oil (waste cooking oil/WCO), a low grade oil, as raw material. The use of commercial sodium silicate in the production of biodiesel with waste cooking palm oil (WCO) raw material was able to provide a yield of 57.9% despite the poor quality raw material, as shown in Table 4 [23]. WCO is palm oil that has been used for cooking thus it can be ascertained that the hydrolysis reaction occurred during the cooking process due to the contact with water at high temperatures. This causes an increase in FFA and water content. With relatively high FFA and water content, the sodium silicate catalyst was able to catalyze reactions without WCO pretreatment, thus exhibiting a good catalytic performance.

Fig. 6 shows the impact of calcination temperature on the yield of biodiesel. The higher the calcination temperature, the lower the yield of biodiesel. The sintering effect caused this reduction of yield during calculations, which resulted in the lowering of surface area (Table 1). The optimum condition that resulted in a yield of 79.49% was observed at temperature of 400 °C.

Properties of Palm Oil Biodiesel

The physicochemical properties of the biodiesel was studied after going through a purification process, in order to remove impurities, and a heating process to remove residual [15]. The biodiesel was measured according to EN14214 standard.

The results shown in Table 5 indicate that the biodiesel produced in this study that was measured against six physicochemical parameters, has values that comply with EN14214 standards. Although there are still many other parameters to be looked at in analyzing biodiesel products as fuel, the above six parameters can serve as an indicator for the initial stage of such production.

Table 3. Methyl ester composition of the synthesized biodiesel

Peak	Identified Compound	Composition (%)
1	Methyl palmitate (C16:0)	0.3803
2	Methyl stearate (C18:0)	0.9482
3	Methyl oleate (C18:1)	36.9969
4	Methyl oleate (C18:1)	59.7634
5	Methyl linoleate (C18:2)	0.0670
6	Methyl linolenate (C18:3)	0.6517
7	Methyl arachidate (C20:0)	0.5610
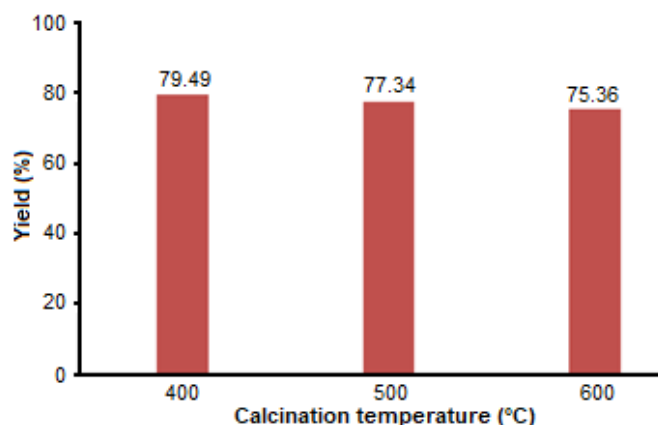
Fig 6. Effect of the temperature of calcination on biodiesel yield

Table 4. Sodium silicate in the biodiesel production using different quality of raw material

Catalyst	Type of oil	Catalyst dosage (%)	Alcohol	Ratio	Reaction temp. (°C)	Reaction time (min)	Yield (%)
Commercial sodium silicate [23]	WCO	2.5	Methanol	6:1	64	240	57.9
Synthesis sodium silicate [this study]	RBDPO	2	Methanol	9:1	65	60	79.49

Table 5. Physicochemical properties of biodiesel from palm oil

Fuel Properties	Unit	This Work	EN14214/03
Methyl ester content	%w/w	99.3685	≥ 96.5
Density	kg/m^3	868	860–900
Viscosity kinematic	mm^2/s	4.57	3.5–5.0
Monoglyceride	%	0.24	Max 0.8
Diglyceride	%	-	Max 0.2
Triglyceride	%	-	Max 0.2

■ CONCLUSION

In summary, an efficient sodium silicate solid base catalyst was synthesized using corncobs and NaOH solution. The prepared catalyst was used in the transesterification conversion of palm oil to biodiesel. Under optimized reaction conditions, the yield reached 79.50% after only 60 min at 65 °C, at catalyst content of 2% and a molar ratio of 1:12. The obtained biodiesel product after purification and treatment processes had high-quality fuel properties according to EN14214 standards, showing that low-cost sodium-silicate-derived corncob catalysts have high catalytic performance and could be used for biodiesel production at industrial scales.

■ ACKNOWLEDGMENTS

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