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Synthesis and Characterization of Calcium Oxide Impregnated on Silica from Duck Egg Shells and Rice Husks as Heterogeneous Catalysts for Biodiesel Synthesis

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Abstract:

Biodiesel can be prepared from various types of vegetable oils or animal fats with the aid of a catalyst. Calcium oxide (CaO) is one of the prospective heterogeneous catalysts for biodiesel synthesis. Modification of CaO by impregnation on silica (SiO_2) can improve the performance of CaO as catalyst. Egg shells and rice husks as biomass waste can be used as raw materials for the preparation of the silica modified CaO catalyst. The present study was directed to synthesize and characterize CaO impregnated SiO_2 catalyst from biomass waste and apply it as catalyst in biodiesel synthesis. The catalyst was synthesized by wet impregnation method and characterized by x-ray diffraction, x-ray fluorescence, nitrogen adsorption-desorption, and basicity density. The activity of the catalyst in biodiesel synthesis was assayed at different molar ratios of palm oil to methanol ranging from 1:6, 1:9, 1:12 and 1:15. The biodiesel composition was determined by gas chromatography-mass spectroscopy and the properties of the biodiesel were also characterized. The results showed that the CaO impregnated SiO_2 catalyst was successfully synthesized based on confirmation by XRD and XRF. The catalyst has a surface area, average pore diameter, total pore volume, and basicity density of 19.38 m^2/g, 3.22 nm, 0.0122 cm^3/g, and 3.4 mmol/g, respectively. The catalyst activity assay indicates that the molar ratio of palm oil to methanol of 1:12 is the optimum condition for biodiesel synthesis. At this molar ratio, 81.4% biodiesel yield was achieved, and it met the quality standards according to ASTM D 6751.

Keywords: Biodiesel, Duck egg shell, Calcium oxide, Rice husk, Silica.

Introduction:

The world's energy consumption, especially in developing countries, is currently still dependent on the type of energy from oil and gas. For example, in Indonesia the energy consumption was 989.9 million Barrel Oil Equivalent (BOE), of which 42%, 17%, and 10% was met by gasoline, coal, and gas, respectively, with an average growth rate of fuel oil demand is 2.8% per year[1]. The greenhouse effect and global climate change as a result of fuel combustion are critical issues that affect the energy industry, public policy makers, and society. The increase in global warming and the impact of other environmental hazards have prompted almost all countries to reduce dependence on fossil fuel[2].

Biomass is one of the most important renewable and sustainable energy sources[3]. The processing of this biomass will produce one type of renewable fuel, for example biodiesel. Biodiesel in general is a methyl ester of long chain fatty acids so it is often known as fatty acid methyl ester (FAME). Biodiesel can be made from oil extracted from fungi, algae, vegetable oil, algae, animal fat, or other sources such as waste cooking oil[4-7].

Application of homogeneous catalysts in transesterification stage during biodiesel production has potency to cause several problems, including

the occurrence of saponification reactions, increased viscosity, non-optimal yield of biodiesel, and separation of the catalyst at the end of the reaction is relatively difficult[8]. The main disadvantage of homogeneous catalysts is that they cannot be reused or regenerated[9]. Therefore, the application of a heterogeneous catalyst can be considered to reduce these problems. Calcium oxide (CaO) has received much attention as a solid heterogeneous catalyst for the synthesis of biodiesel because it requires mild reaction conditions, relatively inexpensive, and has less negative impact on the environment[10].

Calcium oxide without modification from various sources has been prepared with different characteristics and applied as heterogeneous catalyst in the synthesis of biodiesel[11,12]. Improvement of the characteristics of solid heterogeneous catalysts can be carried out with various efforts. One of them is by expanding its specific surface by impregnation on silica (SiO_2). Lani *et al*. succeeded in improving the characteristics of the CaO catalyst through the impregnation method on SiO_2 from rice husk[13].

CaO impregnated on SiO_2 (CaO/SiO_2) catalyst can be synthesized from various types of biomass. CaO can be prepared from egg shell calcination, while rice husk is a potential biomass as silica source. Rice husk has a high content of amorphous SiO_2[14]. Heterogeneous catalysts synthesized from biomass have varied characteristics, depending on various factors, such as: the type and characteristics of the biomass, as well as the method and conditions of catalyst preparation. The optimal performance of heterogeneous catalytic reactions in biodiesel synthesis is influenced by the type and characteristics of the catalyst, reaction conditions, and physico-chemical properties of the oil[15].

Therefore, the present study was directed to synthesize and characterize CaO/SiO_2 from biomass waste commodities, egg shells and rice husks, and evaluate its performance as catalysts in the transesterification of palm oil into biodiesel.

Materials and Methods:

Preparation of Catalyst

Calcium oxide as the active ingredient of the catalyst was prepared from the shells of Java duck eggs. The egg shells were washed with demineralized water, dried, ground with a mortar, and sieved to obtain a 50 mesh size powder. Egg shell powder was calcined at a temperature of 900°C for 1 hour with a temperature rise rate of 10°C/minute.

Silica as the support of calcium oxide was prepared from rice husks. Rice husks were obtained from the milling process of IR-64 rice varieties from Sumedang, West Java. Rice husks were washed with demineralized water to remove impurities, and then dried in an oven at 60°C for 24 hours. Furthermore, rice husks were soaked in 1 N hydrochloric acid solution for 1 hour, washed with distilled water until neutral pH was reached and dried. Rice husks were ashed in a kiln at 600°C for 6 hours. Rice husk ash was suspended in 0.5 N sodium hydroxide solution with an ash to solution ratio of 1:100 (g/mL) while heated and stirred at 100°C for 4 hours to extract silica in the form of sodium silicate. The extracted mixture was filtered through Whatman filter paper no. 41 to take the filtrate. Then 10% sulfuric acid was added to the filtrate until the pH reached 7, and sodium silicate was converted to silica gel. The reaction was carried out for 24 hours, followed by silica gel precipitation stage for 48 hours. The silica gel was filtered, washed with distilled water, and dried at 105°C for 24 hours.

Calcium oxide impregnated on silica catalyst was prepared using the wet impregnation method. 5.0 g of calcium oxide is dissolved in 100 mL of distilled water. Then 3% (w/w) silica was mixed with calcium oxide solution, and refluxed for 4 hours at 80°C. The reflux product was filtered, and the residue was dried at 60°C for 24 hours. The dry residue from the impregnation was then it is calcined in a furnace at 800°C for 3 hours.

Characterization of CaO/SiO_2

The x-ray diffraction (XRD) pattern was collected using a Philips Analytical Diffractometer by using a Cu-Kα radiation. The elemental composition in the form of metal oxides of CaO/SiO_2 was collected using fluorescent x-ray (XRF) method. NOVA 3200e Quantachrome TouchWin v1.0 was used to obtain the surface area, total pore volume, and pore size distribution of the catalyst through the adsorption-desorption of nitrogen gas method using the Brunauer-Emmet-Teller (BET) and Barret-Joyner-Hallenda (BJH) equations. While the basicity density of the catalyst was determined by the acid-base titration method according to Kim *et al*.[16].

Synthesis and Characterization of Biodiesel

A mixture of methanol and 3% (w/w) catalyst was heated in the reactor at 50°C for 30 minutes. Furthermore, oil was added to the reactor to reach final ratio of oil to methanol of 1:6, 1:9, 1:12 and 1:15. The transesterification was carried out for 2 hours at 60°C. The reaction product was transferred to a separator funnel to separate the catalyst from the biodiesel mixture, residual methanol and

glycerol. The biodiesel and residual methanol were then separated from the glycerol by centrifugation at 7000 rpm for 15 minutes. Biodiesel was purified from methanol by distillation. The biodiesel yield was determined using Eq. 1.

$$\text{Yield of biodiesel} = \frac{\text{Weight of biodiesel}}{\text{Weight of oil}} \times 100\% \quad ...1$$

Biodiesel from the variation of the molar ratio with the optimum kinematic density and viscosity (measured at a temperature of 40°C) based on ASTM D 6751 (the American Society for Testing and Materials) was further analyzed including: acid number, iodine number, saponification number, cetane number and water content. The methyl ester composition of biodiesel was determined by gas chromatography-mass spectrometry (Shimadzu QP 2010 ULTRA). Acid number was calculated by Eq. 2 after titrating sample using KOH. Cetane number was determined by approximation as a mathematical function of the saponification number and the iodine number[17] according to Eq. 3.

$$\text{Acid number} = \frac{56.1 \times N \times V}{m} \quad ... \ 2$$

where:

V = volume of KOH solution used (mL)

N = normality of KOH solution

m = mass of biodiesel sample (g)

$$\text{Cetane number} = 46.3 + \frac{5458}{SN} - 0.255 \times IN \quad ... \ 3$$

where:

SN = saponification number in mg KOH/g biodiesel

IN = iodine number in %

Results and Discussion:

Characteristics of Calcined Duck Eggshell and Silica Extraction Results from Rice Husk

When duck eggshell is calcined, calcium carbonate ($CaCO_3$) in the shell is converted to CaO according to the reaction in Eq. 4[18].

$$CaCO_{3(s)} \rightarrow CaO_{(s)} + CO_{2(g)} \ldots \quad 4$$

During the reaction, CO_2 was released. The formation of CaO is characterized by a reduction in the mass of the calcined egg shell. The mass reduction in the form of mass lost at four times of calcination of duck egg shells is shown in Table 1, while the composition of metal oxides determined by XRF is shown in Table 2. Calcined duck egg shells contain about 98.5% of CaO and various other metal oxide impurities in relatively small levels.

Table 1. Percentage of mass lost from duck egg shell during calcination stage at 900°C for 1 hour

Calcination stage	Mass before calcination/ g	Mass after calcination / g	Mass lost/ g	Mass lost/ %
1	10.0347	7.8672	2.1675	21.6
2	10.0572	7.8647	2.1925	21.8
3	10.0590	7.8661	2.1929	21.8
4	10.0862	7.8672	2.2190	22.0
Mean	10.0593	7.8663	2.1930	**21.8**

Table 2. Duck egg shell composition from calcination at 900°C for 1 hour

Component	Content/ %	Component	Content/ %
MgO	0.7028	ZnO	0.0305
P_2O_5	0.9396	BaO	0.0176
SO_3	0.3233	Au_2O	0.0061
Cl_2O	0.0361	Al_2O_3	0.4374
K_2O	0.3758	Eu_2O_3	0.0333
Fe_2O_3	0.0786	**CaO**	**98.50**
Cu_2O	0.0111	SiO_2	0.0337

Extraction of silica from rice husk with NaOH and H_2SO_4 solution occurs based on the following serial reaction[19] Eq. 5 to 7.

$SiO_{2(s)} + 2NaOH_{(aq)} \rightarrow Na_2SiO_{3(aq)} + H_2O_{(l)} \ldots 5$

$Na_2SiO_{3(aq)} + H_2SO_{4(aq)} \rightarrow H_2SiO_{3(l)} + Na_2SO_{4(aq)} \ldots 6$

$H_2SiO_{3(l)} \rightarrow SiO_2.H_2O_{(s)} \ldots 7$

The average yield of silica gel from rice husk ash after drying is 77.31%. The silica gel yield from the three extraction stages are shown in Table 3. The silica yield obtained varies depending on the washing step with distilled water to remove sulfate salts. The relatively low silica yield is thought to be because the silica gel also escapes in the washing step. Calcined duck egg shell, silica gel, and dry silica are presented in Fig. 1.

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Table 3. SiO_2 extracted yield from rice husk ash

Extraction stage	Mass of ash/ g	Mass of SiO_2/ g	Yield of SiO_2/ %
1	10.0167	6.8111	67.10
2	10.0013	7.7518	77.51
3	10.1181	8.8352	87.32
Mean	10.0454	7.7994	**77.31**

(a) (b) (c)

Figure 1. (a) Calcined duck egg shell, (b) formation of silica gel, (c) dry silica

Characteristics of CaO Impregnated on Silica Catalyst

CaO from calcined duck eggshell after impregnation on silica from rice husk has distinctive characteristics, both surface characteristics and activity as a catalyst. The temperature factor in the wet impregnation method is one of the important factors. The dispersion of oxides (CaO and silica) as the active phase will occur if the thermal treatment of the oxide mixture is carried out at a temperature high enough to induce the mobility of the active phase[20].

Characteristics of Catalyst with XRD and XRF

The diffraction pattern of the prepared CaO/SiO_2 is presented in Fig. 2. Typical diffraction peaks of CaO were observed at 2θ of 32.2°; 37.4°; 53.9°; 64.2°; 67.4°; 79.7° and 88.6°. Meanwhile, there is a peak with a fairly high intensity at 32.0°; 32.2° and 32.6° which indicate the presence of a new phase formed, namely Ca_2SiO_4. This agrees with results described by Yamaguchi et al. who found that Ca_2SiO_4 has several forms of crystal types where in the range 2θ of 32°-33° is the crystal peak of Ca_2SiO_4 with monoclinic crystal type[21]. The presence of H_2O during the wet impregnation process between CaO and SiO_2 allows the formation of dicalcium silicate hydrate ($Ca_2SiO_4.H_2O$), which is then dehydrated to form Ca_2SiO_4 compounds. The crystal peak of Ca_3SiO_5 (hatrurite or tricalcium silicate) was observed at 2θ of 29.5°. Ca_3SiO_5 is formed from the reaction between Ca_2SiO_4 and calcium hydroxide[22]. The chemical composition of the catalyst using the XRF method showed that after CaO was impregnated on SiO_2, the CaO content in the catalyst was 72.8%, while the silica content as a buffer was 26.4% (Table 4). The more CaO content in the CaO/SiO_2 catalyst, the higher the basicity, so that the catalyst activity in the transesterification process is higher. The amount of CaO and SiO_2 in the hybrid catalyst depends on the success of the impregnation process.

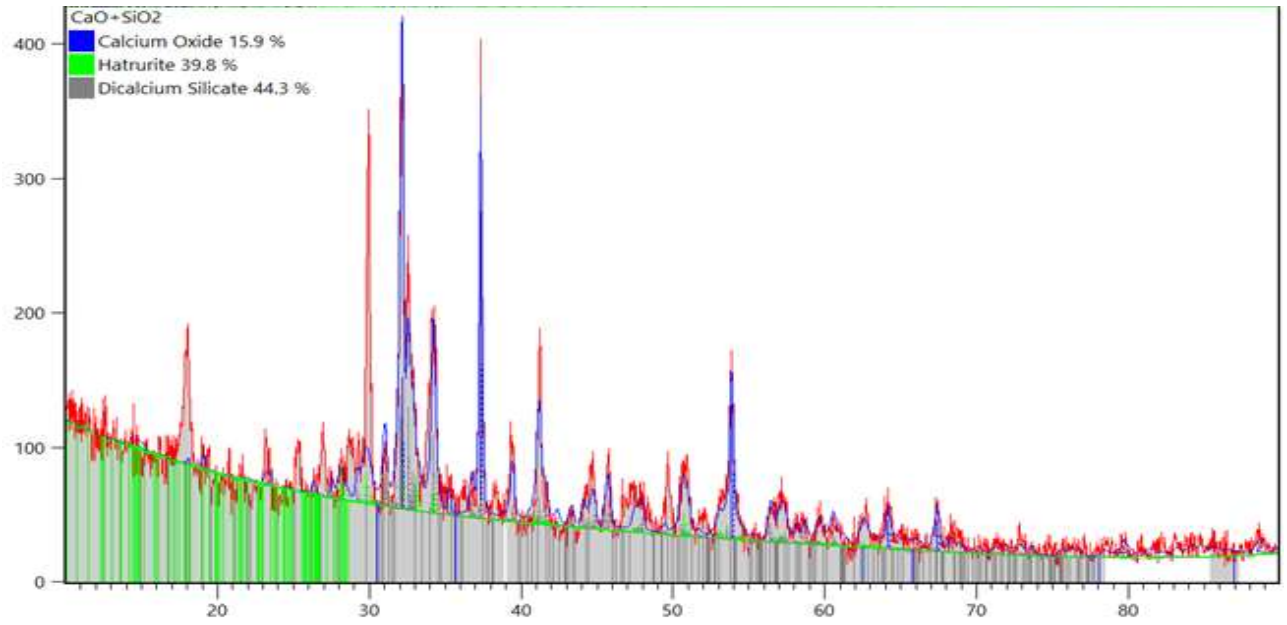

Figure 2. XRD diffraction pattern of CaO impregnated on SiO_2 catalyst

Table 4. The chemical components of the CaO/SiO_2 catalyst

Component	Content/ %	Component	Content/ %
MgO	0.5239	Fe_2O_3	0.0253
P_2O_5	0.8434	Cu_2O	0.0044
SO_3	0.2040	ZnO	0.0098
Cl_2O	0.0241	Al_2O_3	0.5356
K_2O	0.1968	**CaO**	**72.80**
TiO_2	0.0032	**SiO_2**	**26.40**

Characteristics of Surface Area, Total Pore Volume, Pore Size Distribution, and Basicity Density of the Catalyst

Table 5, shows the results of surface area, total volume, and pore diameter of Ca/SiO_2 catalysts, as well as their comparison with research results from Lani et al.[13]. The two features have relatively significant different characteristics even though they are synthesized from the same type of raw material and method. Impregnation of CaO on silica is expected to increase the activity of the catalyst by increasing the accessibility of the active site and providing sufficient attachment area[23]. In comparison, the pore diameter of CaO as catalyst 33-41 nm, is slightly larger than that of the molecular reaction, where the dimensions of glycerol and biodiesel product are about 0.10 nm and 0.64 to 1.52 nm[24], respectively. The larger average pore diameter and the interconnection between the catalyst pores will limit the molecular diffusion of the reactants, so that the molecular reactants can easily infiltrate the interior of the catalyst, and most of the active sites will be used during the transesterification reaction[25]. The result of the basicity density of the catalyst by acid-base titration was obtained at 3.4 mmol/g of catalyst.

Table 5. Surface characteristics of CaO impregnated on SiO_2

Biomass source	Surface area/ $m^2\ g^{-1}$	Total pore volume/ $cm^3\ g^{-1}$	Mean pore diameter/ nm	References
Chicken egg shell, rice husk (Perak, Malaysia)	12.29	0.0429	11.76	Lani *et al.*[13]
Duck egg shell, rice husk (IR-64, Sumedang, Indonesia)	19.38	0.0122	3.22	This study

Biodiesel Yield and Characteristics

Biodiesel is synthesized from RBDPO (refined, bleached, and deodorized palm oil) with a free fatty acid content of 0.11%, at molar ratio variation of oil to methanol 1:6, 1:9, 1:12 and 1:15, the application of 3% CaO/SiO_2 produced biodiesel and the yield are presented in Fig. 3, while the kinematic viscosity and biodiesel density values of each molar ratio variation and its comparison with quality standards according to ASTM D 6751 are presented in Table 6. Transesterification at a molar ratio of 1:12 resulted in the highest biodiesel yield,

which was 81.4%. The density of biodiesel from all oil to methanol molar ratios has met the standard of ASTM D 6751, which is 860–894 kg/m^3. Meanwhile, ASTM D 6751 requires biodiesel to have a kinematic viscosity of 1.9–6.0 mm^2/s. Therefore, the viscosity of biodiesel from transesterification with a molar ratio of oil to methanol of 1:6 does not meet the maximum limit value requirement. Based on the consideration of yield value and biodiesel characteristics in the density and viscosity parameters, a molar ratio of oil to methanol of 1:12 was decided as the optimum condition for transesterification. For comparison, Table 7, shows the characteristics of CaO-based catalysts and biodiesel yields produced by several researchers. Biodiesel yield is influenced by the characteristics of the catalyst and the conditions of the transesterification reaction.

Table 6. Density and viscosity of biodiesel from each variation in the molar ratio of oil to methanol

The molar ratio of oil to methanol	Density at 40°C/kg m^{-3}	Viscosity at 40°C/mm^2 s^{-1}
1:6	890	9.84
1:9	860	5.25
1:12	**886**	**5.57**
1:15	870	5.52
ASTM D 6751	860–894	1.9–6.0

Table 7. Comparison of biodiesel yield from palm oil with CaO based catalyst

Catalyst type	Catalyst characteristics	Transesterification performance	Ref.
CaO (chicken bone)	s = 108.74 m^2/g	T = 65°C t = 4 hours r = 1:15 K = 5% Yield = 89.33%	Farooq et al.[26]
CaO/SiO$_2$ (goat bones)	s = 11.54 m^2/g	T = 60°C t = 2 hours r = 1:15 K = 6% Yield = 94%	Lani et al.[10]
CaO/SiO$_2$	s = 12.29 m^2/g d = 11.76 nm v = 0.0429 cm^3/g	T = 60°C t = 2 hours r = 1:20 K = 3% Yield = 87.75%	Lani et al.[13]
CaO/SiO$_2$	s = 19.3819 m^2/g d = 3.8708 nm v = 0.0187 cm^3/g	T = 60°C t = 2 hours r = 1:12 K = 3% Yield = 81.4%	This study

Note: s = surface area, d = mean pore diameter, v = pore total volume, r = molar ratio of oil to mathanol, K = catalyst content

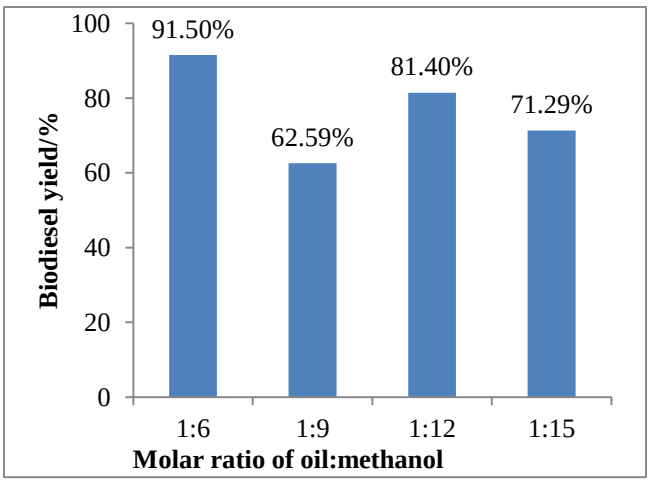

Figure 3. The relationship between the molar ratio of oil to methanol and biodiesel yield in the transesterification of RBDPO with 3% CaO/SiO$_2$ catalyst

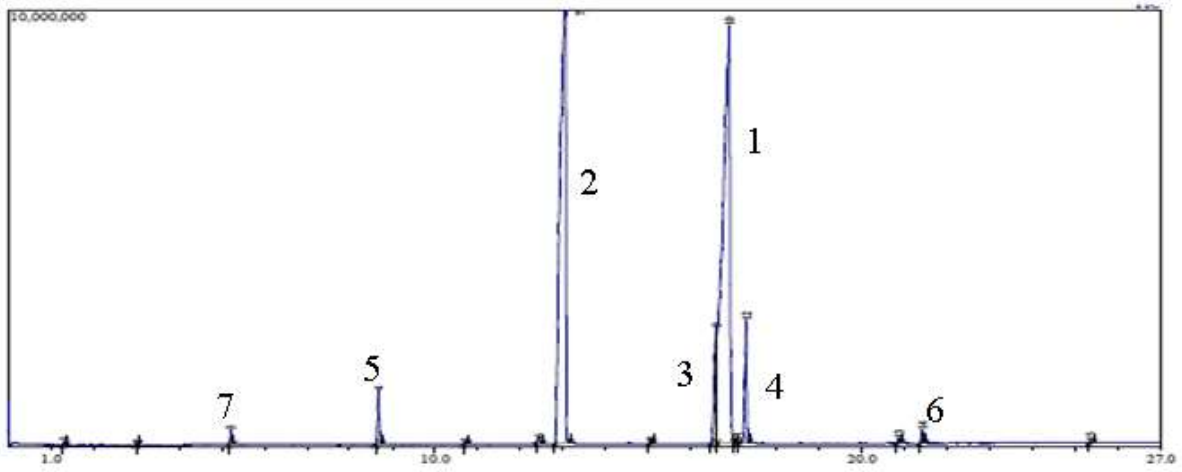

Figure 4. Chromatogram of biodiesel at optimum conditions by using GC-MS

Table 8. Characteristics of biodiesel at optimum conditions and its comparison with ASTM D 7651

Test parameters	Unit min./max.	ASTM D 7651	Results
Density at 40°C	kg/m^3	860–894	886
Viscosity at 40°C	mm^2/s (cSt)	1.9–6.0	5.57
Moisture content	%-volume, max.	0.05	0.04
Acid number	mg-KOH/g, max.	0.5	0.21
Iodine number*	%-mass, max.	-	68.88
Cetane number	min.	47	61.03

Table 9. Methyl ester content in biodiesel

No. of Peak	Types of fatty acids methyl ester	Composition/ %	Retention time/ minutes
1	Methyl oleate	47.88	16.898
2	Methyl palmitate	39.61	13.052
3	Methyl linoleate	5.16	16.597
4	Methyl stearate	4.20	17.293
5	Methyl myristate	1.52	8.675
6	Methyl arachidate	0.39	21.416
7	Methyl laurate	0.35	5.215

Further characterization of biodiesel from the optimum molar ratio obtained the results as shown in Table 8. Of all the measured biodiesel quality parameters, biodiesel from the research results fulfilled the biodiesel quality standard according to ASTM D 6751. Analysis with GC-MS showed that biodiesel or FAME is dominated by methyl oleate and methyl palmitate with composition of 47.88 and 38.61%, respectively (Table 9), while the chromatogram of biodiesel is shown in Fig. 4.

Conclusion:

CaO impregnated on SiO_2 catalyst has been successfully synthesized from local biomass waste in Indonesia in the form of duck egg shells and rice husks of IR-64 variety. The catalyst has certain characteristics based on XRD, XRD diffraction patterns, surface area, total pore volume, average pore diameter, and basicity density. The CaO/SiO_2 catalyst has optimum activity in transesterification of pure palm oil into biodiesel at a molar ratio of oil to methanol of 1:12 with biodiesel yield of 81.4% and has met the ASTM D 6751 standard.

Authors' declaration:

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are mine ours. Besides, the Figures and images, which are not mine ours, have been given the permission for re-publication attached with the manuscript.
- The author have signed an animal welfare statement.
- Ethical Clearance: The project was approved by the local ethical committee in Universitas Padjadjaran, Indonesia.

Authors' contributions statement:

This work was carried out in collaboration between all authors. H.H. designed the experiments, analyzed the data, and drafting the manuscript. S.I. performed the interpretation of the data, revise and proofread the manuscript. I.F.

performed laboratory experiment, acquisition of data, and drafting the manuscript.

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توليف وتوصيف أكسيد الكالسيوم المشرب على السيليكا من قشور بيض البط وقشور الأرز كمحفزات غير متجانسة لتخليق وقود الديزل الحيوي

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الخلاصة:

يمكن تحضير وقود الديزل الحيوي من أنواع مختلفة من الزيوت النباتية أو الدهون الحيوانية بمساعدة عامل حفاز .أكسيد الكالسيوم (CaO)هو أحد المحفزات غير المتجانسة المحتملة لتخليق وقود الديزل الحيوي .يمكن أن يؤدي تعديل CaOبالتشريب على السيليكا (SiO_2) إل تحسين أداء CaOكمحفز .يمكن استخدام قشور البيض وقشور الأرز كنفايات الكتلة الحيوية كمواد خام لإعداد محفز CaO المعدل بالسيليكا .تم توجيه الدراسة الحالية لتخليق وتوصيف محفز SiO_2 المشبع بـ CaO من نفايات الكتلة الحيوية وتطبيقه كمحفز في تخليق وقود الديزل الحيوي .تم تصنيع المحفز بطريقة التشريب الرطب وتميز بحيود الأشعة السينية، ومضان الأشعة السينية ، وامتصاص النيتروجين ، والكثافة القاعدية .تم تقييم نشاط المحفز في تخليق وقود الديزل الحيوي بنسب مولارية مختلفة لزيت النخيل للميثانول تتراوح من 6 :1، 9 :1، 12:1و .15:1تم تحديد تركيبة وقود الديزل الحيوي عن طريق التحليل الطيفي للكتلة اللونية للغاز وتم أيضًا تمييز خصائص وقود الديزل الحيوي .أظهرت النتائج أن محفز SiO_2 المشبع بـ CaOتم تصنيعه بنجاح بناءً على تأكيد بواسطة XRDو .XRFيحتوي المحفز على مساحة سطحية ومتوسط قطر مسام وحجم مسام إجمالي وكثافة قاعدية تبلغ 19.38م ^{2}/ جم و 3.22نانومتر و 0.0122سم ^{3}/ جم و 3.4 ملي مول /جم على التوالي .يشير اختبار نشاط المحفز إلى أن النسبة المولية لزيت النخيل إلى الميثانول البالغة 12:1هي الحالة المثلى لتخليق وقود الديزل الحيوي .عند هذه النسبة المولية ، تم تحقيق 81.4٪ من إنتاج الديزل الحيوي وتوافق مع معايير الجودة وفقًا لـ .ASTM D 6751

الكلمات المفتاحية: وقود الديزل الحيوي، أكسيد الكالسيوم، قشر بيض البط، قشر الأرز، السيليكا.