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HIGH-PERFORMANCE CaO/SiO_2 COMPOSITE PREPARED FROM LIMESTONE AND PUMICE SILICA AS CATALYST FOR RUBBER SEED OIL TRANSESTERIFICATION

K.D. Pandiangan✉, H. Satria, Z. Sembiring, W. Simanjuntak, D.I. Alista, N. Bramanthio and R.N. Putra
Department of Chemistry, Faculty of Mathematics and Natural Science, Lampung University, Bandar Lampung-35145, Indonesia
✉Corresponding Author: kamisah.delilawati@fmipa.unila.ac.id

ABSTRACT

In this work, five CaO/SiO_2 composites with different mass ratios were synthesized from limestone and pumice silica and subsequently applied as catalysts for the transesterification of rubber seed oil. A series of experiments were conducted to evaluate the effect of catalyst compositions, calcination temperatures, and other reaction variables including catalyst loads, methanol to oil (M/O) ratios, and reaction times on the reaction yields. The catalyst exhibited the highest activity and was characterized using X-ray diffraction (XRD) and Scanning Electron Microscope (SEM). The results displayed promising activities of the CaO/SiO_2 composites synthesized, with the highest transesterification yield of 100% achieved using a CaO/SiO_2 catalyst with a ratio of 1:5 calcined at a temperature of 800 °C. This complete conversion of the oil into biodiesel was achieved with the use of a catalyst load of 2.5% of the mass of the oil, an M/O ratio of 6:1, and a reaction range of 6 hours. The overall results demonstrated that CaO/SiO_2 synthesized has the promising potential as a catalyst to support biodiesel production from various feedstock.

Keywords: Biodiesel, Rubber Seed Oil, CaO/SiO_2, Limestone, Rice Husk Silica.

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INTRODUCTION

Depletion of petroleum reserves and concerns over the environmental impacts associated with the massive use of fossil fuels are two compelling reasons for the recent and extensive development of renewable energy sources around the globe. In this respect, biomass-derived fuels (biofuels) are of particular importance considering the availability and renewability of biomass as sustainable feedstocks. In addition, several technologies are now available and continuously developed to improve their performance as well as the quality of biofuels produced. Of various existing conversion technologies, transesterification to convert vegetable oils or animal fats into biodiesel has reached commercial scale. At present, biodiesel has been applied as a practical fuel, although still in the form of a blend with petrochemical diesel at certain percentages. Several examples are B20, which means the blend contains 20% biodiesel and has been applied in India[1] and Indonesia[2], B30 used in Egypt[3], and B40 used in Taiwan.[4] Blending of biodiesel with multi-walled-carbon nanotubes with different proportions has also been reported by previous workers.[5] Regardless of its existence which has reached commercial status, the price of biodiesel is still higher than that of petrochemical diesel, reflecting the need for further effort to reduce the production costs of biodiesel. In this regard, the efforts have been emphasized on exploration of the feasibility of non-edible vegetable oils to replace palm oil which is still the main raw material, and development of heterogeneous catalysts to replace homogeneous catalysts. In the search for alternative feedstock, the potential of various non-edible vegetable oils has been explored, including but not limited to *Jatropha curcas* oil[5,6], rubber seed oil[7,8], *Ricinus communis* oil[9], mixtures of *Melia azedarach*, rice bran, and water hyacinth oil[10], and Moringa seed oil.[11] To support the transesterification reaction, the development of an effective heterogeneous catalyst to replace a homogeneous catalyst is another challenge. Various types of heterogeneous catalysts, primarily composite systems in which metal oxide is supported on solid, have been developed and tested. Of particular interest are supported catalysts, with different metal oxides as active sites, such as MgO/SiO_2[12], CaO/SiO_2[13], Fe_3O_4/SiO_2[14], and NiO/SiO2.[15] Although not as widely as a heterogeneous catalyst, several researchers have explored the application of lipase enzyme.[16] In this work, transesterification of rubber seed oil (RSO) was

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carried out in the presence of CaO/SiO_2 with different mass ratios. The catalysts were synthesized from the local limestone industry in West Sumatera Province and amorphous silica was extracted from pumice collected from a local source (Kalianda, South Lampung). The catalysts were then utilized in transesterification reactions of RSO with the main purpose of evaluating the feasibility of the rubber seed oil as biodiesel feedstock and the effect of the compositions of the catalyst on the yield of the reaction. Other influencing factors investigated were methanol-to-oil ratios, catalyst loads, and reaction times.

EXPERIMENTAL

Material and Methods

Limestone with a CaO purity of 98% was obtained from the limestone industry in West Sumatera Province and pumice from Suak Beach, Kalianda, South Lampung Regency, Lampung Province. Sodium hydroxide, nitric acid, hydrochloric acid, and methanol were obtained from Merck and Aldrich. Rubber seeds were collected from Way Kanan Regency, Lampung Province. The instruments used are PANanalytical Type Xpert MPD XRD instrument, Scanning Electron Microscope (SEM) type ZEISS EVO MA 10, PANalytical Epsilon 3 X-Ray Fluorescence (XRF) instrument, and Gas Chromatography-Mass Spectrometry (GC-MS) model QP2010S SHIMADZU.

General Procedure

Extraction of Amorphous Silica from Pumice

The pumice was cleaned from natural dirt by washing with running water, and then sun-dried for 3 days. The pumice was ground to a fine powder using a mortar and pestle and then sieved to obtain 150 mesh powder. The powder (350 grams) was soaked in 150 mL of 1 M HCl and left for 24 hours. The pumice was rinsed with distilled water to expel the acid, followed by oven drying at 100 °C for 12 hours. Extraction of silica from pumice was conducted using the sol-gel method adopting the procedure reported by previous workers.[17] Typically, 50 g of pulverized pumice was mixed with 750 mL of NaOH with varied molarities of 2.0; 2.5; 3.0; 3.5; and 4.0, and then subjected to refluxing treatment under 300 rpm stirring for 24 hours at 100 °C. The filtrate which contains silica (silica sol) was collected by filtration, followed by neutralization of the sol using 3 M HNO_3 solution to produce silica gel. The gel was aged for 24 hours at room temperature, followed by rinsing with warmed distilled water and completed by 24 h oven drying at 100 °C. Finally, the sample was pulverized and sieved using a 250-mesh sieve. The four samples were then characterized using XRD as a basis for selecting the silica used for the preparation of the CaO/SiO_2 catalysts.

Preparation of CaO/SiO_2 Catalysts

Preparation of the CaO/SiO_2 catalyst was conducted using the sol-gel method, with varied mass ratios of 1:1; 1:2; 1:3; 1:5; and 1:10. The specified mass of SiO_2 was dissolved in 1.5% NaOH solution (3 mL of solution for 1 gram of SiO_2), and specified mass of CaO to satisfy the composition was dissolved in concentrated HNO_3 (3.75 mL acid for 1 gram of CaO). The CaO solution was then slowly poured into silica solution under magnetic stirring until the gel was formed. The gel was filtered and then dried in an oven at 110 °C for 24 hours. The dried solid was calcined for 6 hours at 800 °C to ensure the formation of CaO/SiO_2 and finally ground into powder and sieved with 300 mesh sieves.

Extraction of Rubber Seed Oil (RSO)

To extract the RSO, the rubber seeds were peeled and the kernels were collected and cut into small pieces, then oven-dried at 80 °C for 4 hours. The oil was extracted using a screw-pressing machine.

Transesterification Reaction

A series of experiments were carried out sequentially to study the effect of reaction variables on the biodiesel yield achieved. All experiments were carried out using a mixture of 20 mL of RSO and 5 mL of coconut oil as a co-reactant and acidified with 1 mL of concentrated H_2SO_4. The inclusion of coconut oil as a co-reactant was based on the results of previous research[12] which showed that rubber seed oil was difficult to transesterification, but with the addition of coconut oil as a co-reactant, the reaction yield increased very significantly. The yield of biodiesel was calculated based on the volume of unreacted oil. The formation of biodiesel (fatty acid methyl esters, FAMEs) was confirmed by analysis of the sample using GC-MS. The first series of experiments were carried out to evaluate the effect of catalyst composition as a basis for selecting the catalyst with the highest performance to be used for subsequent experiments. For this purpose, the reaction mixture was prepared by mixing 150 mL methanol with 20 mL of RSO and 5 mL of coconut oil (M/O ratio of 6:1) and loaded with 10% catalyst (relative to the mass RSO). The transesterification reaction was set at 70 °C and allowed to proceed for 6 hours. The biodiesel yield from each of the experiments was calculated as a base to select the catalyst with the highest activity. Selected catalyst from these experiments was then used in the experiments to study the effect of M/O ratios, catalyst loads, and reaction durations. The selected catalyst was also calcined at different temperatures to evaluate their effect on the activity of the catalyst.

RESULTS AND DISCUSSION

Characterization of Pumice Silica

The samples of pumice silica obtained using NaOH with different concentrations were characterized using the XRD technique, producing diffractograms as displayed in Fig.-1. Diffractograms of the samples compiled in Fig.-1 indicate that the samples obtained using NaOH solution with molar concentrations of 2.0; 2.5; and 3.0 are amorphous, while the samples produced with the use of NaOH with molar concentrations of 3.5 and 4.0 are a mixture of amorphous and crystalline silica as suggested by the existence of sharp diffraction peaks concerning the diffractograms (Fig.-1), the silica produced using NaOH solution with a concentration of 2.5 M was selected for the preparation of CaO/SiO_2 catalysts. This particular silica was selected since its mass (11.8 grams) is not significantly different from the mass of the silica obtained using 3.0 M NaOH solution (12.1 gram), but requires less NaOH. In this regard, the use of a 2.5 M NaOH solution is considered more efficient from a cost point of view. The existence of the sample as amorphous silica is supported by the surface morphology as can be observed in the SEM image shown in Fig.-2. As can be seen, the surface of the sample is marked by particles with irregular forms without any evident shape, which is a characteristic feature of amorphous material. To study the chemical composition of the silica selected, the sample was characterized using the XRF method, and the main oxides composing the sample are shown in Table-1. The results in Table-1 show that the prominent component is silica with a contribution of 82.47% to the composition of the sample. The second abundant component is Al_2O_3. The presence of Al_2O_3 is advantageous since this compound is known as a good adsorbent. The purity of pumice silica observed in this study is higher than that of silica extracted from acidic pumice (74.26%) reported by others.[18,19]

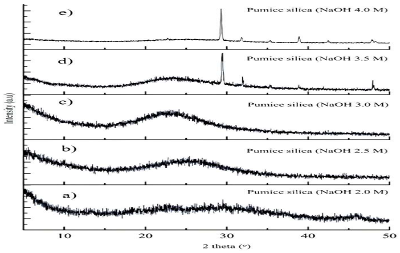

Fig.-1: Diffractograms of Pumice Silica Obtained Using NaOH with Different Concentrations, (a) 2.0 M, (b) 2.5 M, (c) 3.0 M, (d) 3.5 M, and (d) 4.0 M

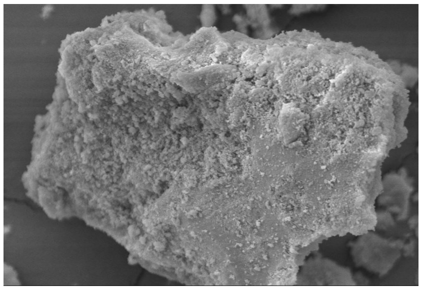

Fig-2: SEM Image of Pumice Silica Extracted Using 2.5 M NaOH Solution

Results of Transesterification Reactions

The results of transesterification experiments carried out using the CaO/SiO_2 catalysts with different compositions to obtain the catalyst with the highest performance are shown in Table-2. As stated before, the volume of oil subjected to reactions was 25 mL. The experiment presented in Table-2 demonstrates that reaction yield is strongly influenced by the composition of the catalyst. As can be observed, the practically complete conversion of the oil into biodiesel resulted from the utilization of CaO/SiO_2 of 1: 5 as a catalyst. In this respect, this catalyst was selected for the rest of the investigation. The use of a CaO/SiO_2 catalyst has also been reported by Albuquerque *et al*[20], in which the use of a catalyst with a CaO content of 15% was reported to exhibit the highest activity in the transesterification reaction of ethyl butyrate with methanol. In another study[21] application of CaO/SiO_2 with the ratio of 2: 1 was reported to achieve 93% conversion of waste cooking oil to biodiesel. Experiments to investigate the effect of methanol-to-oil ratios were then conducted using the selected catalyst and different methanol-to-oil ratios, producing the data presented in Table-3. The research results in Table-3 show that the ratio of methanol to oil is also a factor that strongly influences the reaction toward the product. This trend is by the character of the transesterification reaction which is an equilibrium reaction. In this regard, the use of methanol in excess is needed to drive the reaction towards the product (biodiesel). Based on the results in Table-3, the reactant composition with a methanol-to-oil ratio of 6 was used for the other experiments. A significant role of the M/O ratio in the transesterification of vegetable oils is also acknowledged by other workers.[22] The results of experiments undertaken to study the effect of catalyst loads are shown in Table-4. As can be observed with the use of a 2.5% catalyst, a reaction yield of 100 percent was achieved. This achievement demonstrates that the synthesized catalyst has excellent performance in the reaction investigated. Several workers have also investigated the effect of catalyst load on transesterification reaction and revealed that the catalyst load required to achieve optimum reaction yield varies depending on the types of vegetable oil processed.[23,24] Another kinetic variable that is recognized to have an influencing part of biodiesel production is the time of transesterification reaction. The experiments carried out in the current investigation to study the effect of this variable produced the results shown in Table-5. A very significant role of reaction time is displayed by the experimental results in Table-5. As can be seen, the reaction yield increases following the extension of reaction time, and reaches 100% yield with a reaction time of 6 hours. The effect of reaction times has been investigated by many workers and revealed the variation in reaction time, depends on the feedstock processed and the catalyst used.[23,25,26] The last factor investigated was the effect of the calcination temperatures of the catalyst. For this purpose, the selected catalyst (CaO/SiO_2 1:5) was calcined at different temperatures and then utilized in transesterification reactions, with the results as shown in Table-6. The results in Table-6 show that calcination temperatures also influenced the performance of the catalyst. As can be observed, practically complete conversion of the oil was gained with the use of catalysts calcined at 800 and 900 °C. These results suggest that the calcination of the catalyst at 800 °C was sufficient to activate the catalyst to work optimally. To ensure that the product resulting from the transesterification reaction is biodiesel (a mixture of FAMEs), one of the products was analyzed using GC-MS. The GC-chromatogram of the sample is shown in Fig.-3, and the chemical constituents of the sample confirmed with the aid of the MS-Library database are presented in Table-7. As can be seen in Table-7, the sample is a mixture of methyl esters of fatty acids composing RSO and coconut oil. In this regard, it can be concluded that the transesterification reaction has proceeded to produce pure biodiesel, as expected. The emergence of methyl oleate as a prominent component is consistent with the existence of linoleic acid as the constituent of rubber seed oil with the highest percentages of 47.81%[27] and 39.6%.[28]

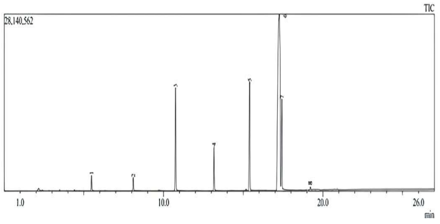

Fig-3: GC-chromatogram of the Biodiesel Representative Produced

Table-1: The Main Oxides Composing Pumice Silica Extracted Using 2.5 M NaOH

Oxide	Percentage (%)
Al_2O_3	14.33
SiO_2	82.47
P_2O_5	1.78
K_2O	0.50
CaO	0.28
Others	0.64

Table-2: The Yields of Reactions Using Different CaO/SiO_2 Catalysts

No.	CaO: SiO_2	Unreacted oil (mL)	Reaction yield (%)
1.	1:1	15	39.2
2.	1:2	8	66.4
3.	1:3	4	83.2
4.	1:5	0	100.0
5.	1:10	0.8	96.8

Table-3: Effect of Methanol to Oil (M/O) Ratios on the Yield of Transesterification

No.	M/O ratio	Unreacted oil (mL)	Reaction yield (%)
1	2	20	19.6
2	3	12	51.2
3	4	6	75.6
4	5	2.5	90.0
5	6	0	100.0

Table-4: Effect of Catalyst Loads on the Yield of Transesterification

No.	Catalyst load (%)	Unreacted oil (mL)	Reaction yield (%)
1.	2.5	0	100.0
2.	5.0	0	100.0
3.	10.0	0	100.0
4.	12.5	0.5	98.0
5.	15.0	0.6	97.6

Table-5: Effect of Reaction Times on the Yield of Transesterification

No.	Reaction time (h)	Unreacted oil (mL)	Reaction yield (%)
1.	2	13.0	48.0
2.	3	11.0	56.1
3.	4	6.5	74.1
4.	5	3.5	86.2
5.	6	0	100.0

Table-6: Reaction Yield Achieved Using Catalyst Calcined at Different Temperatures

No.	Calcination Temperature (^{0}C)	Unreacted oil (mL)	Reaction yield (%)
1	500	6.0	76.1
2	600	8.0	68.0
3	700	2.0	92.1
4	800	0	100.0
5	900	0	100.0

Table-7: Chemical Composition of Biodiesel Representative

No.	Retention time	Formula	Compound name	Relative percentage (%)
1.	5.477	$C_9H_{18}O_2$	Methyl Octanoate	1.31
2.	8.1	$C_{11}H_{22}O_2$	Methyl Caprate	1.14
3.	10.756	$C_{13}H_{26}O_2$	Methyl Laurate	10.03
4.	13.169	$C_{15}H_{30}O_2$	Methyl Myristate	3.97
5.	15.402	$C_{17}H_{34}O_2$	Methyl Palmitate	11.37

6.	17.272	$C_{19}H_{34}O_2$	Methyl Linoleate	62.66
7.	17.426	$C_{19}H_{38}O_2$	Methyl Stearate	9.20
8.	19.216	$C_{21}H_{42}O_2$	Methyl Arachidate	0.31

CONCLUSION

The results obtained in this research clearly show that the CaO/SiO_2 composites synthesized from limestone and pumice silica exhibited excellent catalytic activity for the transesterification of rubber seed oil. The results of the transesterification experiments revealed that a reaction yield of 100% was achieved using a CaO/SiO_2 catalyst with a ratio of 1:5 calcined at a temperature of 800 °C. This complete conversion of the oil into biodiesel was achieved with the use of a catalyst at 2.5% of the mass of oil used, the ratio of methanol to oil of 6:1, and a reaction time of 6 hours. The overall results demonstrated that CaO/SiO_2 synthesized has the promising potential as a catalyst to support biodiesel production from various feedstocks.

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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:

K.D. Pandiangan https://orcid.org/0000-0001-6347-2361
H. Satria https://orcid.org/0000-0003-0779-5830
Z. Sembiring http://orchid.org/0000-0001-7623-3343
W. Simanjuntak https://orcid.org/0000-0001-8152-5084
D.I. Alista http://orchid.org/0009-0002-1365-9704
N. Bramanthio http://orchid.org/0009-0007-0126-3696
R.N. Putra http://orchid.org/0009-0008-9263-0697

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