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Research Article

The Effect of MgO Loads on Catalytic Activity of MgO/SiO_2 in Coconut Oil Transesterification

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Abstract

This research aims to obtain biodiesel by transesterification of coconut oil with MgO/SiO_2 as a catalyst, with the objective to examine the effect of MgO loads on the performance of the catalysts expressed in terms of oil conversion. The MgO/SiO_2 composites with different mass ratios of 1:1, 1:2, 1:3, 1:5, and 1:10 were synthesized from $Mg(NO_3)\cdot 6H_2O$ and SiO_2 extracted from rice husk silica using sol-gel method and followed by calcination at 800 °C for 6 h. The produced catalysts were then tested in the transesterification of coconut oil to investigate the effect of catalyst composition, oil to methanol ratios, and reaction times. The experiments were carried out using a fixed catalyst load of 10% oil (w/v) and at 70 °C. The catalyst with the highest performance was then characterized by using X-ray diffraction (XRD) and scanning electron microscopy (SEM) to confirm the formation of crystalline MgO/SiO_2. The highest conversion of the transesterification product was analyzed using gas chromatography-mass spectrometry (GC-MS) technique to confirm the biodiesel production. The XRD diffractogram of the synthesized sample is characterized by the presence of sharp peaks confirming the existence of the sample as crystalline material attributed to the pyroxene-ideal (MgO_3Si) phase. The existence of the sample as crystalline material is also supported by the absence of a broad diffraction peak attributed to amorphous rice husk silica, and SEM image of the sample which is characterized by the visible existence of crystalline structure in the forms of platelet prismatic, and tetrahedral shaped structures. The results demonstrated that the highest conversion (98%) was achieved with the use of MgO/SiO_2 (1:5) as a catalyst, a ratio of oil to methanol of 1:6, and a reaction time of 4 h. The formation of methyl esters was confirmed by the results of GC-MS analysis.

Keywords: MgO/SiO_2, sol-gel, catalyst, biodiesel, transesterification

1. INTRODUCTION

Research on renewable energy has attracted attention in response to the depletion of fossil fuels and environmental danger as a consequence of their utilization. Up to present, biodiesel is the most attractive renewable fuel and has been widely used in several countries in blended forms with petrochemical diesel with gradually increased percentages [1][2]. Biodiesel is an attractive renewable fuel compared with fossil diesel. It has better combustion characteristics, lower viscosity, and higher cetane number [3][4]. In addition, biodiesel is nontoxic and biodegradable, therefore more environmentally friendly than fossil diesel. Biodiesel is produced from various raw materials, either vegetable oils or animal fats, by reacting them with simple alcohols, mostly methanol and ethanol. With the use of methanol, for instance, the biodiesel produced is a mixture of fatty acid methylesters (FAMEs) [5][6].

In biodiesel production, free fatty acids contained in the raw materials undergo an esterification reaction while the glycerides undergo transesterification. However, since the composition of vegetable oils and animal fats is dominated by glycerides, transesterification is much more important than the esterification reaction. The yield of biodiesel is acknowledged to depend on several factors, with the most influencing being catalyst, oil to alcohol ratio, and reaction time. Although non-catalytic transesterification is being developed, such as the supercritical technique [7], catalytic transesterification is still the main choice, primarily because the process is simpler and faster than the first one. The type of catalyst that has been applied since the beginning of the biodiesel industry is homogeneous catalyst, either strong acids such as H_2SO_4 [8][9], HNO_3 [9], and HCl [9][10] or strong bases, primarily NaOH [11] and KOH [8][12]. Homogenous catalysts are acknowledged to exhibit high performance leading to the production of biodiesel in high yield. However, apart from this advantageous feature, the use of homogeneous catalyst is constrained by several shortcomings that

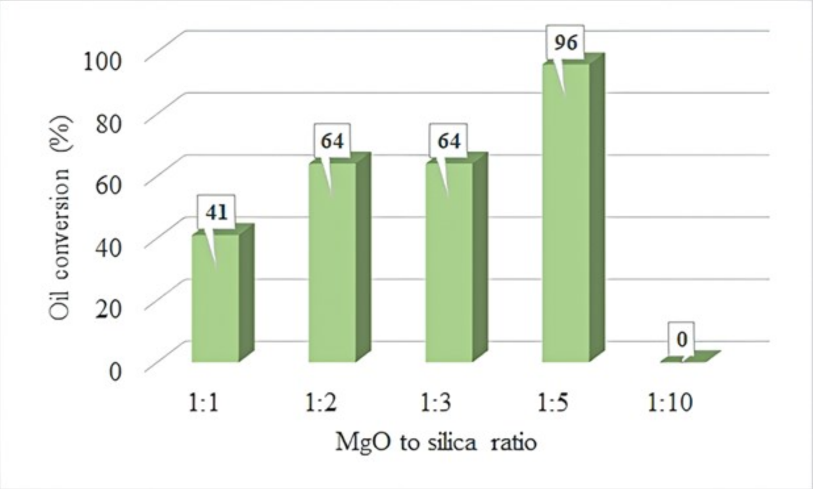

Figure 1. Oil conversions are achieved using catalysts with different compositions.

contribute to production costs. Strong acids and bases are corrosive chemicals and as a consequence, the biodiesel must be washed with a considerable quantity of water to remove the catalyst, causing a problem to the environment where the wastewater is released. In addition, reuse of homogeneous catalysts is practically impossible.

In response to the shortcomings of homogeneous catalysts, many works have been continuously devoted to the development of heterogeneous (solid) catalysts as alternatives. As a solid, heterogeneous catalyst can be separated from biodiesel simply by filtration therefore no produced wastewater and the catalyst can be reused. In addition, heterogeneous catalyst is not corrosive. In recognition of the advantages they offered, many solids have been explored including, but not limited to, metal oxides [13], supported metal oxides [2][5][14][15] and various zeolites [16]-[20].

Besides the type of catalyst, other factors acknowledged to significantly influence transesterification are catalyst composition, oil to alcohol ratio, and reaction time [15][18][21]. With respect to the role of these factors, in this study transesterification of coconut oil with methanol for biodiesel production was investigated using MgO supported on rice husk silica (MgO/SiO_2) with different compositions (mass ratio) of 1:1, 1:2, 1:3, 1:5, and 1:10 as catalyst. A number of experiments were conducted to evaluate the effect of catalyst compositions, oil to alcohol ratios, and reaction time on the reaction yield. The synthesis and application of MgO/SiO_2 composites with a considerably wide range of compositions explored in this research provides an opportunity to gain more detailed insight into the role of composition in determining the performance of the synthesized MgO/SiO_2 catalyst, is a new approach which has not been extensively explored in detail in previous investigations. To ensure whether the MgO/SiO_2 composite has been successfully formed as expected, the catalyst with the highest performance was then characterized using X-ray diffraction (XRD) and scanning electron microscopy (SEM).

Table 1. Several examples of composite catalysts and their compositions used in biodiesel production.

No.	Catalyst	Mass ratio	Raw material	Reference
1	TiO_2/SiO_2	3:1	Waste cooking oil	[23]
2	CaO/SiO_2	1:5	Coconut oil	[24]
3	ZnO/β-zeolite	9:1	Waste cooking oil	[17]
4	MgO/SiO_2	1:5	Rubber seed oil	[22]
5	Li/NaY zeolite	1:1	Castor oil	[19]

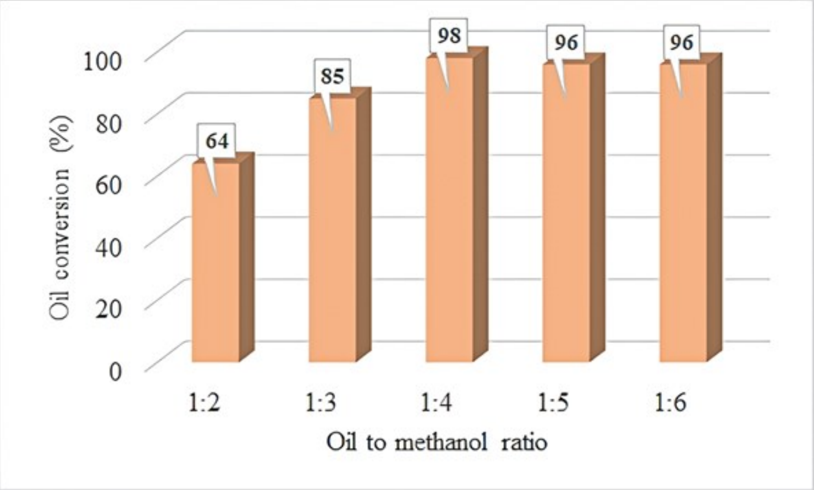

Figure 2. The experimental results demonstrating the effect of oil to methanol ratios on oil conversion.

2. MATERIALS AND METHODS

2.1. Materials

Rice husk (RH) was obtained from a local source (rice milling company in Natar, South Lampung), coconut oil was purchased from local market in Bandar Lampung, analytical grade nitric acid (HNO_3), sodium hydroxide (NaOH), magnesium nitrate hexahydrate ($Mg(NO_3)\cdot 6H_2O$), and methanol (CH_3OH) were purchased from Merck.

2.2. Methods

2.2.1. Extraction of RH Silica

Alkaline extraction using 1.5% NaOH solution was applied to obtain rice husk silica. The typical experiment was run by boiling the mixture of 50 g of husk and 600 mL of NaOH solution for 30 min and then left overnight at room temperature. The mixture was then subjected to filtration to collect the filtrate containing silica (silica sol). The sol was slowly neutralized using 10% HNO_3 until silica gel was formed and allowed to stand for 24 h for aging process. The silica gel was washed with warm water to remove organics that dissolved during the extraction step. The gel was oven-dried at 110 °C for 24 h to obtain solid silica. Finally, the silica was ground into powder and sieved using a 200 mesh sieve to obtain silica powder.

2.2.2. Catalyst Preparation and Characterization

In this work, five MgO/SiO_2 samples with the MgO to SiO_2 mass ratio of 1:1; 1:2; 1:3; 1:5, and 1:10 were prepared using sol-gel process adopting the method described in Pandiangan et al. [22]. The first step was the preparation of raw materials, by dissolving the silica in a 1.5% NaOH solution (solution A), and $Mg(NO_3)\cdot 6H_2O$ in distilled water (solution B). Both solutions were then mixed slowly under stirring to ensure the homogeneity of the mixture, followed by acidification of the mixture using 10% HNO_3 until the gel was formed. The gel formed was then filtered and dried in an oven at 110 °C for 24 h. The dry MgO/SiO_2 samples were ground into powder and then calcined at 800 °C for 6 h.

Table 2. The effect of oil to methanol ratio on conversion of different vegetable oils into biodiesel.

No.	Vegetable oil	Oil to methanol ratio	Oil conversion (%)	Reference
1	Sunflower oil	1:9	91.6	[20]
2	Waste cooking oil	1:15	96.9	[20]
3	Rubber seed oil	1:12	98.7	[25]
4	*Ricinus communis* oil	1:6	96.0	[6]
5	Castor oil	1:9	95.0	[26]

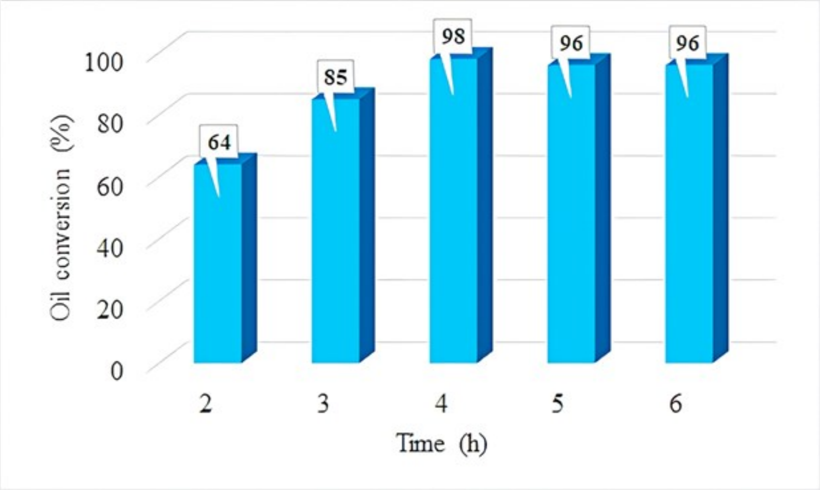

Figure 3. The experimental results show the effect of reaction time on oil conversion.

Catalyst characterization was carried out using the XRD technique on Empyrean-type PANalytical equipment, scanning the sample over a 2θ angle in the range of 5−50° with a counting time of 8 s/step. The CarlZeiss/Evo MA 10 type apparatus was used for SEM analysis. Characterization of the sample was limited to these two techniques because the main purpose is to obtain information regarding the structure and microstructure of the sample since these two properties have been acknowledged to play the most important role in governing the performance of a catalyst. However, the contribution of other physical characteristics including surface area, surface chemical composition, and basicity, should also be acknowledged. In this respect, these characteristics should be tajen into account in the future works.

2.2.3. Transesterification Reaction

Transesterification was carried out in a transesterification reactor consisting of a 500 mL round bottom flask equipped with a reflux condenser, immersed in a water bath placed on a hotplate magnetic stirrer. The first series of experiments were conducted to study the effect of catalyst compositions as a way to select the catalyst with the highest performance to be used for the rest of the experiments aimed to investigate the effect of raw material composition (oil to methanol ratios) and reaction time. The experiment was carried out at 70 °C for 4 h using 25 mL of coconut oil, 100 mL of methanol, and 2.5 g of catalyst. After the completion of the reaction, the sample was allowed to cool to room temperature and then filtered into a separatory funnel, to separate the catalyst. The sample in the separatory funnel was allowed to settle for 24 h to separate the biodiesel (upper layer) from the unreacted oil, and then the biodiesel was collected. The excess of methanol was removed by evaporation and the biodiesel volume was used to calculate the oil conversion, using the following equation 1 [6], where, V_1 is initial volume of oil

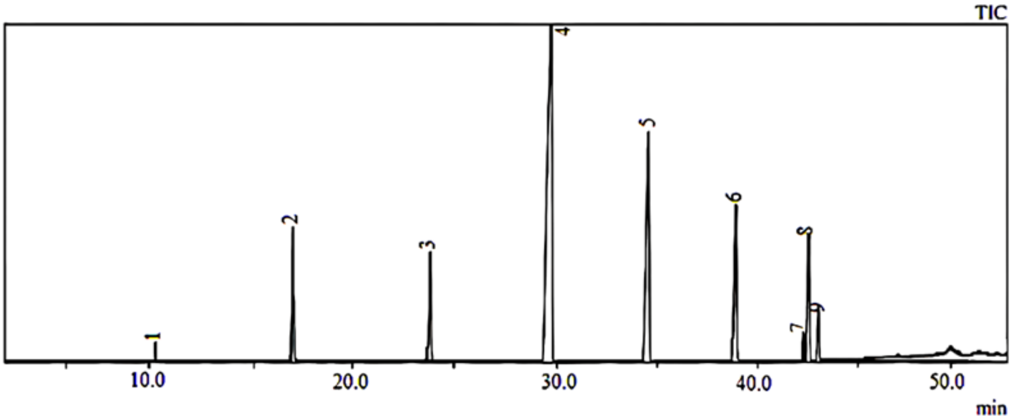

Figure 4. Chromatogram of biodiesel obtained using MgO/SiO$_2$ 1:5 as a catalyst.

Table 3. The components of the transesterification product of coconut oil with methanol.

Peak	Retention time (min)	Molecular Formula	Compound Name	Relative Percentage (%)
1	10.703	$C_7H_{14}O_2$	Methyl caproate	0.76
2	17.971	$C_9H_{18}O_2$	Methyl caprylate	9.62
3	24.242	$C_{11}H_{22}O_2$	Methyl caprate	8.84
4	29.790	$C_{13}H_{26}O_2$	Methyl laurate	39.94
5	34.617	$C_{15}H_{30}O_2$	Methyl myristate	20.76
6	38.998	$C_{17}H_{34}O_2$	Methyl palmitate	11.84
7	42.260	$C_{19}H_{34}O_2$	Methyl linoleate	2.12
8	42.435	$C_{19}H_{36}O_2$	Methyl elaidate	8.23
9	42.968	$C_{19}H_{38}O_2$	Methyl stearate	3.90

and V_2 is the volume of unreacted oil.

$$\text{Oil converison} = \frac{V_1 - V_2}{V_1} x\,100\% \qquad \textbf{(1)}$$

The reaction yields obtained with the application of MgO/SiO_2 with different compositions were used to choose the best catalyst for the next experiments. To determine the optimum oil to methanol ratio, experiments were carried out with the ratio of 1:2; 1:3; 1:4; 1:5, and 1:6 for 4 h. The optimum ratio obtained from these experiments was then applied for the determination of optimum reaction time, in which the experiments at reaction time of 2, 3, 4, 5, and 6 h were run. The transesterification product obtained from the experiment involving the optimum factors investigated was then analyzed using gas chromatography-mass spectrophotometry (GC-MS) type GM-MS QP-2010S Shimadzu. The NIST12 Library System Software was used to identify the components of the product.

3. RESULTS AND DISCUSSIONS

3.1. Effect of Catalyst Composition on Oil Conversion

As previously mentioned, the MgO/SiO_2 with the MgO to SiO_2 mass ratio of 1:1; 1:2; 1:3; 1:5, and 1:10 were prepared and then tested as catalyst for coconut oil transesterification. The five experiments were run at a fixed oil to methanol ratio of 1:4 and fixed reaction time of 4 h. The experimental results are compiled in Figure 1. It was found that with the use of a catalyst with a MgO to SiO_2 mass ratio of 1:10, practically no oil conversion was achieved.

As displayed by the experimental results in Figure 1, the effect of catalyst compositions on oil conversion is evident in which increased conversions to reach the optimum value of 96% were observed following the change in composition from 1:1 up to 1:5. These results outline that the MgO to SiO_2 ratio of 1:5 is an ideal composition reflecting that the number of MgO molecules as active sites and SiO_2 molecules reaction host function synergistically to optimize the oil conversion. In the case of the catalyst with the MgO to SiO_2 ratio of 1:10, it is most likely that the excessive numbers of SiO_2 particles covered the MgO particles, preventing them from effective interaction with the oil molecules. With respect to the results obtained, the catalyst with MgO to SiO_2 ratio of 1:5 was then selected for the rest of the experiments. The use of composite catalysts with varied compositions has also been reported in several previous works. Some examples are shown in Table 1.

3.2. Effect of Oil to Methanol Ratios on Oil Conversion

The experimental data produced from the experiments with different ratios of oil to methanol as compiled in Figure 2 display quite significant increases in oil conversion following the ratio from 1:2 up to 1:4 but for the last two ratios no significant change was observed. Concerning these results, the ratio of 1:4 was then considered as the

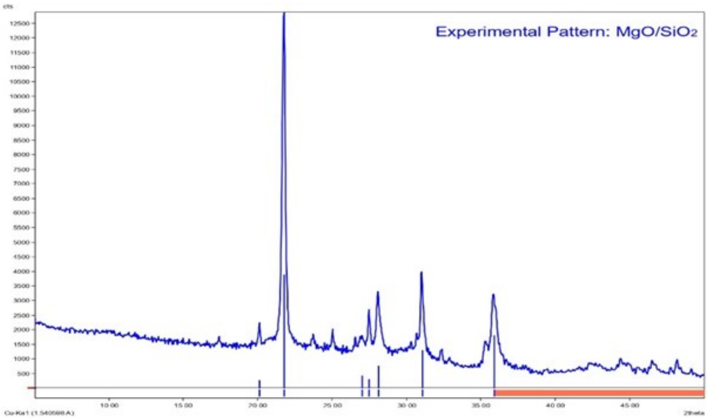

Figure 5. Diffractogram of MgO/SiO_2 with the ratio of 1:5 and calcined at 800 °C and identified phases.

optimum ratio, with an oil conversion of 98% was achieved.

In biodiesel production by transesterification, the ratio of oil to methanol is acknowledged as a prominent variable and therefore has been extensively investigated by many workers, showing that the composition of reactants to some extent depends on the types of the feedstocks treated. The results of previous investigations presented in Table 2 illustrate effect of feedstock composition (oil to methanol ratio) on conversion of several vegetable oils. Some examples of work conducted to investigate the effect of feedstock composition (oil to methanol ratio) on oil conversion are shown in Table 2.

3.3. Determination of the Optimum Reaction Time

Another factor that has been acknowledged to play very significant role in transesterification of vegetable oils is reaction time. In appreciation of this variable, the experiments at different durations of time were conducted and the results produced are presented in Figure 3. The experimental results presented in Figure 3 were obtained from the experiments conducted using the MgO/SiO_2 with MgO to SiO_2 ratio of 1:5 as a catalyst and at fixed oil to methanol ratio of 1:4. The results in Figure 3 indicate significant increases in oil conversion achieved from the experiments at 2-h, reaching the optimum value of 98% at 4-h reaction time. It is also observed that by elongation of reaction time to

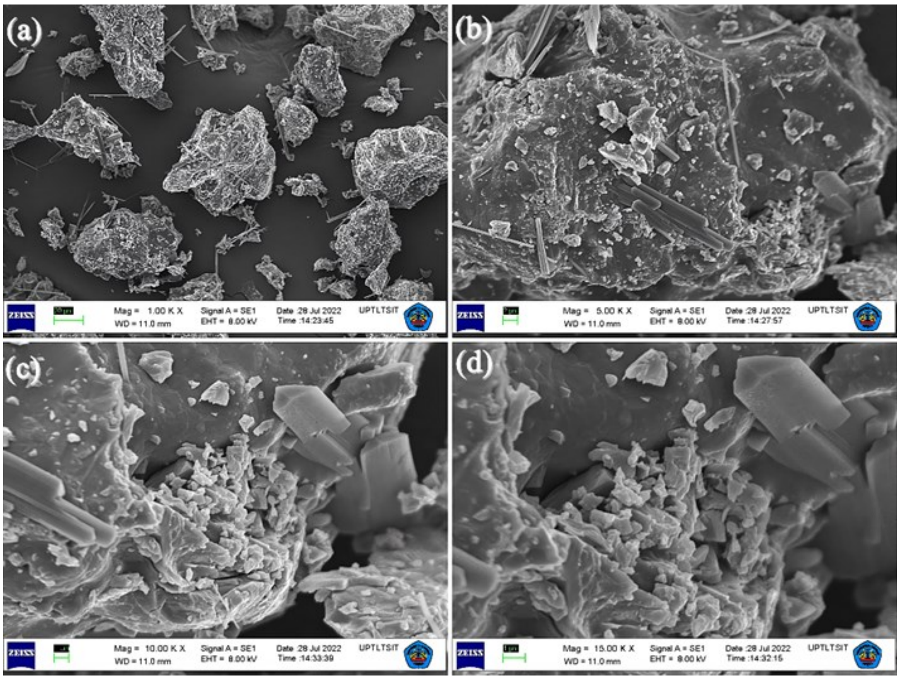

Figure 6. Micrographs of the MgO/SiO_2 1:5 catalysts at different magnifications: (a) 1,000×; (b) 5,000×; (c) 10,000×; and (d) 15,000×.

5 and 6 h led to slight decreases in oil conversion, implying that 4 h is the optimum time. The effect of reaction time on transesterification reaction has also been investigated by other workers [6][17][26], showing the existence of variation in optimum reaction time, depending on the feedstock treated, and other reaction variables implemented.

3.4. GC-MS Analysis of Transesterification Product

To identify the components contained in the transesterification product, the sample was analyzed by GC-MS, and the chromatogram is presented in Figure 4.

Figure 4 indicates that there are nine compounds composing the biodiesel, as listed in Table 3, confirming that the fatty acids contained in the oil have reacted with methanol to produce a mixture of methyl esters. The main component obtained is methyl laurate which is derived from lauric acid according to the fatty acids the main content in coconut oil [13][27].

3.5. Characterization of Selected Catalyst

The catalyst with the highest activity (selected catalyst) was then characterized by XRD and SEM techniques. The XRD diffractogram of the sample is shown in Figure 5.

The wide band in Figure 5 (2 theta = 20−30°) is due to the presence of amorphous silica [28] and the sharp peaks indicate that a crystalline phase has been formed in the sample. The XRD pattern was then analyzed using Match Program (Phase Identification from Powder Diffraction – Version 3) to identify. With the aid of this phase identification software, it was found that the sample has the molecular formula of $MgOSiO_2$ with pyroxene-ideal as the main phase. The existence of this crystalline phase is affirmed by the peaks at diffraction angles of 2θ = 32.4°, 35.7°, 36.5°, and 39.7°, which are attributed to crystalline MgO. The XRD results obtained in this study are in agreement with that reported by previous works [29]. Successful formation of $MgOSiO_2$ is also confirmed by the absence of wide diffraction band in the 2θ region of 20−30°, which is the characteristic XRD feature of amorphous silica.

The surface morphology of this particular catalyst was analyzed by SEM and the micrographs with different magnifications are presented in Figure 6. The surface morphology of the MgO/SiO_2 1:5 catalysts in Figure 6 shows the presence of particles with a prismatic or tetrahedral form, aggregates, or fibrous collections of thin plates. On the surface, scattered cavities originating from silica were detected. The surface morphology of the MgO/SiO_2 catalyst is more clearly visible under higher magnifications. As can be observed, in the image with 1,000× magnification, the crystalline phase is not yet clearly visible. The presence of particles as prismatic or tetrahedral forms, aggregates, or fibrous collections of thin plates can be observed in the image produced with 15,000× magnification.

4. CONCLUSIONS

The MgO/SiO_2 catalyst has been successfully synthesized by a sol-gel technique by varying the MgO to SiO_2 mass ratio of 1:1; 1:2; 1:3; 1:5; and 1:10. Thermal treatment by calcination at a temperature of 800 °C for 6 h was found to produce MgO/SiO_2, with the main phase being the pyroxene -ideal and amorphous silica phase according to XRD characterization. The transesterification experiments reveal the significant effect of the ratio of oil to methanol and reaction time on the conversion of the oil into biodiesel. The highest conversion (98%) was achieved using MgO/SiO_2 1:5 as a catalyst with an oil to methanol ratio of 1:4, and reaction time of 4 h. The formation of biodiesel was confirmed using GC-MS analysis.

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Conflicts of Interest

The authors declare no conflict of interest

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