

# Selectivity of the New Catalyst ZnO-MCM-48-CaO in Esterification of Calophyllum inophyllum Oil

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## Research Paper

Selectivity of the New Catalyst ZnO-MCM-48-CaO in Esterification of *Calophyllum inophyllum* OilLa Kolo<sup>1,2</sup>, Firdaus<sup>3</sup>, Paulina Taba<sup>3</sup>, Muhammad Zakir<sup>3</sup>, Nunuk Hariani Soekamto<sup>3</sup><sup>1</sup>Doctoral Program of Chemistry Science, Graduate School Hasanuddin University, 90245, Indonesia<sup>2</sup>Department of Chemistry Education, Faculty of Teacher Training and Education, University of Bumi Hijrah, 97852, Indonesia<sup>3</sup>Department of Chemistry, Faculty of Mathematics and Natural Sciences, University of Hasanuddin, 90245, Indonesia✉ [nunukhariani@unhas.ac.id](mailto:nunukhariani@unhas.ac.id)🌐 <https://doi.org/10.31603/ae.6711>

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## Abstract

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The synthesis of MCM-48 containing surfactants (CTAB and Triton X-100), ZnO, and CaO aimed to find a potential heterogeneous catalyst in the esterification of *Calophyllum inophyllum* oil to biodiesel. This research is important in the production of biodiesel from vegetable oils with more than 2% free fatty acids (FFA), such as *Calophyllum inophyllum* oil using heterogeneous catalysts. Synthesis of heterogeneous catalysts, MCM-48 and ZnO-MCM-48-CaO (ZMC), using the hydrothermal method at various calcination temperatures was conducted to find the optimum calcination temperature for the reaction. The activity of the catalyst in the reaction was determined using acid-base titration methods and GC-MS. The MCM-48 catalyst calcined at 650 °C (MCM-48/650) had a catalytic activity of 35.74% and was selective for converting linoleic acid in *Calophyllum inophyllum* oil to biodiesel. In addition, this catalyst was also capable of cracking the compounds contained in *Calophyllum inophyllum* oil into suitable hydrocarbons for biodiesel. In the esterification of vegetable oils, four heterogeneous catalysts (MCM-48/550, ZMC/550, ZMC/650 and ZMC/750) had the potential to replace conventional catalysts (H<sub>2</sub>SO<sub>4</sub>), particularly in the generation of biodiesel from *Calophyllum inophyllum* oil.

**Keywords:** Free fatty acid; Heterogeneous catalyst; Mesoporous silica; Brønsted acid; Silanol

## 1. Introduction

Fossil fuels as an energy source play an essential role in human survival. However, fossil fuels are non-renewable sources of fuel. Therefore, excessive and prolonged exploration results in the reduction of these energy sources [1]. Scientists have made various efforts to overcome this problem by looking for alternative energy sources such as geothermal energy [2], solar energy [3], biogas [4], bioethanol [5], and biodiesel [6]. Among these alternative energy sources, biodiesel is a promising energy source because it is easy to produce, use, and develop [1], [7]. One of the potential sources of biodiesel is biodiesel *Calophyllum inophyllum* oil. This plant grows naturally and is widespread globally [8]. In

Indonesia, this plant is commonly found on the coast, easy to grow again, non-edible, the seeds have a high oil content (75%) [9], [10], and produce fruit throughout the year [11]. Therefore, the plant is suitable to be used as a biodiesel feedstock [12].

The high content of FFA in *Calophyllum inophyllum* oil is an obstacle to biodiesel production [13]. Although researchers prefer to use homogeneous acid catalysts in esterification to overcome this problem [14], this method is not environmentally friendly [15]. The catalyst is difficult to be separated from the product at the end of the reaction [16] and is not reusable [17]. Furthermore, the method is not economical [18], [19]. Heterogeneous catalysts can overcome these problems [20], [21].



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**Nomenclature**

CaAc/600	Calcium acetate hydrate calcined at 600 °C
CaAc	Calcium acetate hydrate (Ca(CH <sub>3</sub> COO) <sub>2</sub> ·xH <sub>2</sub> O)
ZnAc/650	Zinc acetate dihydrate calcined at 650 °C
ZnAc	Zinc acetate dihydrate (Zn(CH <sub>3</sub> COO) <sub>2</sub> ·2H <sub>2</sub> O)
CTAB	Cetyl trimethyl ammonium bromide

Several studies used heterogeneous catalysts in the esterification of *Calophyllum inophyllum* oil. The catalysts were  $\beta$ -zeolite modified by phosphoric acid [22], sulfonated microcrystalline carbon cellulose [23], MCM-48-CaO [24], Al-MCM-41 [25], and Al/Fe-graphite oxide [26]. According to research, the Al-MCM-41 catalyst provides the highest conversion rate (up to 98%). Adding aluminum to MCM-41, the catalyst's Lewis and Bronsted acid sites were enhanced, resulting in higher activity [27], [28]. Unfortunately, the method offered by this catalyst is not cost-effective (reaction time was 5 hour). Increasing the activity of the mesoporous silica catalyst in the reaction can be carried out by adding hetero-metals [29].

Hetero-metals can generate acid and base sites (bifunctionality) to catalyze esterification and transesterification simultaneously. Several studies used this bifunctional catalyst. The SrO-ZnO/Al<sub>2</sub>O<sub>3</sub> catalyst was able to convert 96% of used cooking oil (18 wt% FFA) into biodiesel [30]. The CaO-ZnO catalyst converted 96% Karanja oil (9 wt% FFA) into biodiesel and is more active than MnO<sub>2</sub>-CaO, Fe<sub>2</sub>O<sub>3</sub>-CaO, Fe<sub>2</sub>O<sub>3</sub>/CaO, Al<sub>2</sub>O<sub>3</sub>/CaO, and CaO catalysts in the reaction [31]. The W/Ti/SiO<sub>2</sub> catalyst was able to convert 98% used cooking oil (3 wt% FFA) into biodiesel [32]. The use of the catalysts reported were also uneconomical because the reaction took a long time and used a high amount of methanol. However, when applied to *Calophyllum inophyllum* oil with high level of FFA, the activity of the CaO-ZnO catalyst in esterification was better than other catalysts.

Another study also proved that heterogeneous catalyst, KIT-6, [33] was more active than MCM-41 in esterification reactions of vegetable oils. Similar to KIT-6, MCM-48 also has a three-dimensional pore structure so that the possibility of pore-clogging can be avoided [34], [35]. Other studies reported that the presence of surfactants (CTAB and Triton X-100) can increase the activity of mesoporous silica catalysts in simultaneous conversion processes of biodiesel [36]-[38].

Therefore, in this study, a new heterogeneous catalyst consisting of surfactant, MCM-48, ZnO, and CaO was prepared. This study aims to find a potential heterogeneous catalyst in the esterification reaction of *Calophyllum inophyllum* oil to biodiesel.

## 2. Materials and Methods

### 2.1. Materials

Sample *Calophyllum inophyllum* was obtained from Kalauli Hamlet, Leihitu District, Central Maluku Regency, Maluku Province, Indonesia. Phenolphthalein, Ca(CH<sub>3</sub>COO)<sub>2</sub>·xH<sub>2</sub>O, Zn(CH<sub>3</sub>COO)<sub>2</sub>·2H<sub>2</sub>O, NaOH, CH<sub>3</sub>COOH, *n*-hexane, methanol, and H<sub>3</sub>PO<sub>4</sub> were from Merck. While Ludox HS40, CTAB, and Triton X-100 were from Sigma Aldrich (all chemicals were purchased in pro analysis grades).

### 2.2. *Calophyllum inophyllum* oil

Modifying the procedure reported in reference [39] was done for degumming *Calophyllum inophyllum* oil. Extraction of *Calophyllum inophyllum* seed oil used *n*-hexane solvent in a soxhlet apparatus. After the extraction, the oil was separated from the solvent using a rotary evaporator. The extracted oil was then heated in an oven for 60 minutes at 105 °C [40].

### 2.3. Degumming

The degumming procedure of *Calophyllum inophyllum* oil used the procedure given elsewhere [41]. *Calophyllum inophyllum* oil was put in a beaker and heated to 65 °C. After that, 5% (v/v) H<sub>3</sub>PO<sub>4</sub> was added to the oil while stirring at 1000 rpm for 1 hour at 65 °C. Furthermore, the mixture was allowed to stand for 12 hours, then decanted. The oil obtained was then washed on a separatory funnel using hot distilled water (60-80 °C) until the pH of the wastewater from the washing process was neutral. The oil layer was heated in the oven for 60 minutes at 105 °C. The FFA content of the oil obtained was then determined [42].

### 2.4. Synthesis and characterization of catalysts

The procedures reported in reference [43] were modified to synthesize ZMC catalysts. In the silica source, Zn(CH<sub>3</sub>COO)<sub>2</sub>·2H<sub>2</sub>O and Ca(CH<sub>3</sub>COO)<sub>2</sub>·xH<sub>2</sub>O were added as precursors to ZnO and CaO, respectively. The composition of

ZnO and CaO in MCM-48 was 1: 1 (n/n). As-synthesized catalysts (CTAB-MCM-48 and CTAB-ZMC) and calcined for 5 hour at temperatures of 550 °C (MCM/550 and ZMC/550), 650 °C (MCM/650 and ZMC/650), and 750 °C (MCM/750 and ZMC/750). FTIR (Shimadzu 8300) was used to detect the presence of surfactants in the MCM-48 catalyst. The success of catalyst synthesis was confirmed by X-ray diffraction (XRD). XRD data for powder samples were collected using Cu K $\alpha$  radiation ( $\lambda = 0.154$  nm) in the  $2\theta$  range of 2–80° at the scanning speed of 2 °/min. Diffraction patterns were compared to references and ICDD (International Centre for Data Diffraction) data to identify the catalysts. XRF (Panalytical, Minipal 4) was used to identify metal oxides (CaO and ZnO) in the CTAB-ZMC catalyst.

### 2.5. Esterification

The esterification of *Calophyllum inophyllum* oil on accomplished using the procedure described elsewhere [44]. Esterification took place in a 100 mL three-neck flask equipped with an electric heater, a thermometer, a magnetic stirrer, and a cooler. The reaction temperature was maintained at 62 °C with a stirring rate of 1200 rpm, a 2%-wt/wt catalyst to methanol, while the ratio of oil to methanol was 1:9 (wt/wt) reaction time was 1 hour. The reaction was halted by submerging the three-neck flask in an ice chiller. Then the mixture was filtered through a Buchner funnel using filter paper. The levels of FFA in the product were then

determined using the acid-base titration technique [23]. The best conversion results were also identified using the GC-MS method (QP 2010 Ultra Shimadzu).

## 3. Results and Discussion

### 3.1. Characterization of MCM-48 and ZMC Catalysts

The XRD patterns show the success in the preparation of as-synthesized and calcined MCM-48 (Figure 1). There is a moderate intensity peak at 2.51°  $2\theta$  observed in the XRD pattern of CTAB-MCM-48 (Figure 1a). This peak is a characteristic of MCM-48 containing surfactant [45].

The XRD pattern of MCM-48/550 (Figure 1b) shows a strong peak on 2.61°  $2\theta$  and several low peaks on 4.0–5.0°  $2\theta$  (4.33, 4.48, 4.52, 4.59, and 4.68°). These peaks are specific characteristics of the three-dimensional structure of the MCM-48 catalyst after the loss of surfactant [43]. The calcination temperature of 650 °C for the MCM-48/650 catalyst improved its three-dimensional structure with increasingly intensity of (Figure 1c) peaks in the 4.0–5.0°  $2\theta$  region (4.37, 4.41, 4.44, 4.48, 4.51, 4.58, and 4.62°) [46]. The use of a calcination temperature of 750 °C revealed that the three-dimensional structure of the MCM-48/750 catalyst began to deteriorate (Figure 1d) [47]. The damage to the catalyst structure occurs caused by the calcination temperature exceeding the thermal conditions of the MCM-48 [48].

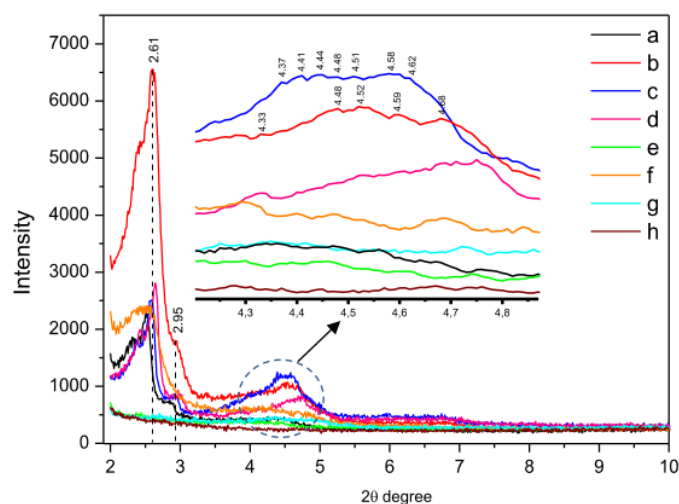
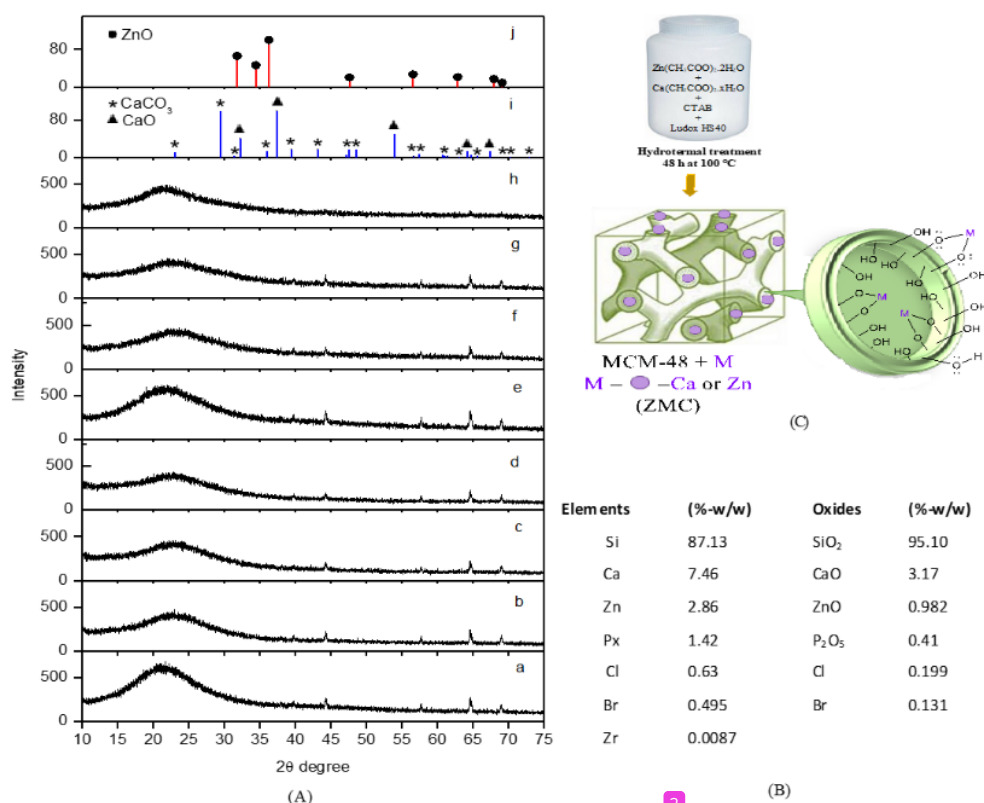


Figure 1. X-ray diffraction patterns of; a) CTAB-MCM-48, b) MCM-48/550, c) MCM-48/650, d) MCM-48/750, e) CTAB-ZMC, f) ZMC/550, g) ZMC/650, and h) ZMC/750



**Figure 2.** A) X-ray diffraction patterns for wide-angle 2 theta of a) CTAB-MCM-48, b) MCM-48/550, c) MCM-48/650, d) MCM-48/750, e) CTAB-ZMC, f) ZMC/550, g) ZMC/650, and h) ZMC/750, i) CaAc/600 (ICDD CaCO<sub>3</sub>: 00-005-0586 and CaO: 04-003-7161), j) ZnAc/650 [49], B) Composition of chemical elements contained in CTAB-ZMC, and C) Reaction of CaO and ZnO in the three-dimensional channel MCM-48

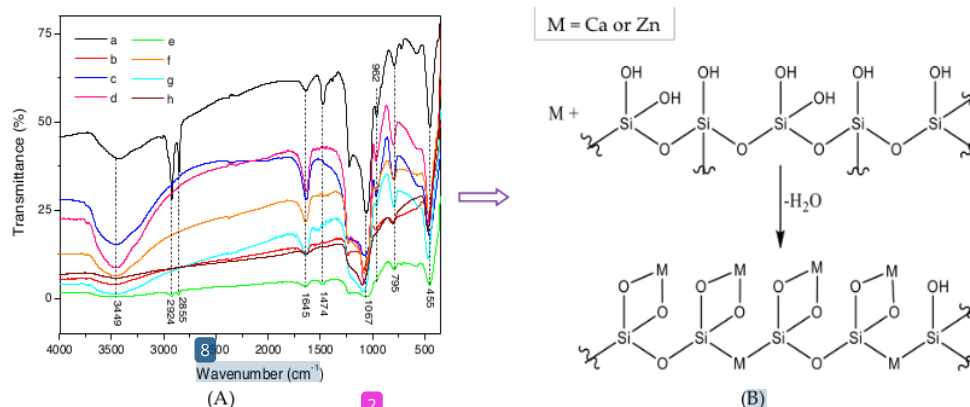
The addition of CaO and ZnO into MCM-48 destroyed the three-dimensional shape of MCM-48. Shift, decrease in intensity (Figure 1f), and loss of peaks typical for the three-dimensional structure of MCM-48 (Figure 1 e, g, and h) confirmed that matter. These character<sup>3</sup> indicate that CaO and ZnO have reacted with silanol (Si-OH) and siloxane (Si-O-Si) groups in the MCM-48 three-dimensional channel to form Si-O-Ca or Si-O-Zn (Figure 2C). The occurrence of the reaction in the three-dimensional MCM-48 channel confirmed by the results of XRF analysis (Figure 2B) and the XRD pattern for the wide-angle 2 theta of the ZMC catalyst (Figure 2A) did not show X-ray diffraction peaks for CaO and ZnO [50].

Figure 3A depicts the FTIR spectra of as-synthesized and calcined catalysts, with interpretation findings in Table 1. The loss of surfactant from the catalyst was confirmed by the FTIR analysis (Figure 3 b, c, and d), which revealed

the loss of peaks for CH stretching vibration (2853 cm<sup>-1</sup> and 2922 cm<sup>-1</sup>) and CH bending (1470 cm<sup>-1</sup>) from the CH<sub>3</sub> groups, as well as the shift of the stretching vibration peak siloxane to a larger wavenumber. The same thing happened for the CTAB-ZMC catalyst to ZMC [51], [52]. The interaction of CaO and ZnO with the silanol group of MCM-48 was supported by the findings of FTIR analysis, which revealed a drop in intensity and a shift in peaks silanol group absorption [24], [53]. In addition, the bending and stretching vibration peaks of siloxane also shifted to a higher wavenumber after the addition of ZnAc and CaAc. The reaction of ZnO and CaO with siloxane groups on the silica framework to form Si-O-Zn or Si-O-Ca confirms that matter [53], [54]. The proposed mechanism of CaO and ZnO reaction with silanol and siloxane in MCM-48 is shown in Figure 3B.

**Table 1.** FTIR analysis of synthesized catalyst

Catalyst	Group	Wavenumber (cm <sup>-1</sup> )	
		This Work	Reference
CTAB-MCM-48	Silanol	3426 and 1638	[55]
	Siloxane	1223, 1063, 795, 453	[56]
MCM-48/550	Silanol	3480 and 1644	[57]
	Siloxane	1086, 804 and 465	[58]
MCM-48/650	Silanol	3451 and 1636	[59]
	Siloxane	1082, 802, and 455	[59]
MCM-48/750	Silanol	3451 and 1641	[60]
	Siloxane	1082, 804, and 455	[59]
CTAB-ZMC	Silanol groups absorption overlaps with Si-O-Ca or Si-O-Zn groups absorption	3449 and 1645	[24]
	Siloxane group absorption overlaps with Si-O-Ca or Si-O-Zn group absorption	1227, 1067, 795 and 455	[60]
ZMC/550	Silanol groups absorption overlaps with Si-O-Ca or Si-O-Zn groups absorption	3451 and 1643	[24]
	Siloxane group absorption overlaps with Si-O-Ca or Si-O-Zn group absorption	1082, 798 and 463	[24]
ZMC/650	Silanol groups absorption overlaps with Si-O-Ca or Si-O-Zn groups absorption	3449 and 1643	[61]
	Siloxane group absorption overlaps with Si-O-Ca or Si-O-Zn group absorption	1094, 798 and 469	[62]
ZMC/750	Silanol groups absorption overlaps with Si-O-Ca or Si-O-Zn groups absorption	3443 and 1641	[63]
	Siloxane group absorption overlaps with Si-O-Ca or Si-O-Zn group absorption	1099, 810 and 471	[63]



**Figure 3.** A) FTIR Spectrum of a) CTAB-MCM-48, b) MCM-48/550, c) MCM-48/650, d) MCM-48/750, e) CTAB-ZMC, f) ZMC/550, g) ZMC/650, h) ZMC/750; and B) reaction mechanism of CaO and ZnO with silica in MCM-48

### 3.2. Catalytic Activity

In the esterification of *Calophyllum inophyllum* oil, CTAB-MCM-48 and CTAB-ZMC catalysts displayed lower activity than MCM-48 and ZMC catalysts (Figure 5). The poor activity of the catalyst is due to the surfactant's head (hydrophilic) and tail (hydrophobic) structures, which can deactivate the active group of FFA, causing the yield to mix with water and glycerol

as a by-product of the reaction, lowering the reaction yield [64]. The effect of surfactants in the CTAB-MCM-48 and CTAB-ZMC catalysts is shown in Figure 4.

Figure 5 compares the catalytic activity of the synthesized catalyst to conventional catalyst in the esterification of *Calophyllum inophyllum* oil. This catalytic activity indicates that the higher the absorption intensity of the silanol group, the

higher the activity of MCM-48 and ZMC catalysts. Despite having a higher silanol groups absorption intensity than all other catalysts, the three-dimensional structure of the MCM-48/750 catalyst has been damaged. The damage of the three-dimensional structure reduces the surface area of MCM-48/750, resulting in a lower activity of MCM-48/750 compared to that of MCM-48/650.

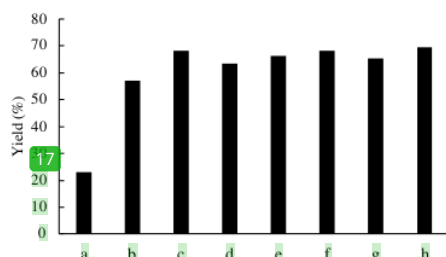


Figure 4. Profile of the effect of surfactants in the catalyst; a) CTAB-MCM-48, b) MCM-48/550, c) MCM-48/650, d) MCM-48/750, e) CTAB-ZMC, f) ZMC/550, g) ZMC/650, and h) ZMC/750 in the esterification

The addition of ZnO and CaO to MCM-48 also decreased the activity of ZMC catalyst in the esterification of *Calophyllum inophyllum* oil. Partially occluded pores with ZnO and CaO induced a decrease in the catalytic activity. However, the presence of these two metal oxides increased the thermal stability of the ZMC catalyst. The breakthrough provides a great starting point for using a catalyst in the same

process, particularly in the high-temperature synthesis of biodiesel from *Calophyllum inophyllum* oil. The optimal calcination temperature for MCM-48 was 650 °C, whereas the ZMC catalyst was 550 °C (Figure 5). The FTIR spectrum reveals that the silanol groups, as Bronsted acid, act as catalyst's active site [65], [66]. In addition, the surface area of the catalyst also affects its activity in esterification.

The results characterized using GC-MS (Table 2) show that MCM-48/650 can convert FFA of *Calophyllum inophyllum* oil (Table 2B) but it is more specific for converting linoleic fatty acids into biodiesel. In addition, this catalyst also has the activity to crack the components of the compounds contained in *Calophyllum inophyllum* oil into hydrocarbons (Table 2A). Therefore, it is suitable for producing biodiesel [67], [68].

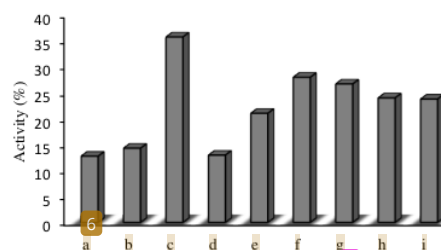


Figure 5. Catalytic activity; a) CTAB-MCM-48, b) MCM-48/550, c) MCM-48/650, d) MCM-48/750, e) CTAB-ZMC, f) ZMC/550, g) ZMC/650, h) ZMC/750, and i. H<sub>2</sub>SO<sub>4</sub> in the esterification

Table 2. MCM-48/650 catalytic activity in *Calophyllum inophyllum* oil esterification

Catalysts	A					B			
	Hydrocarbons		Fatty Acids						
	SI (R.T)	Compounds <sup>o</sup>	C	D <sup>y</sup> (%)	Acid <sup>y</sup> (%)	SI	Compounds <sup>o</sup>	Esters <sup>y</sup> (%)	SI
B.E <sup>o</sup>	95(27.62)	Tetradecanal	14	0.01	8.64	94	Palmitic		
	and				21.95	92	Linoleic		
	88(31.76)			0.24					
	85	14-Methyl-8-hexadecyn-1-ol	17	0.19	4.23	95	Oleic		
	87	1-Nonadecene	19	0.03	3.44	90	Stearic		
	90	2-(9-octadecenyloxy)-Ethanol	20	0.32					
	89	9-Eicosyne	20	0.47					
	85	2-mono-Linolein	21	0.37					
	85	di-(9-Octadecenoyl)-Glycerol	39	19.97					
	87	2-mono-Olein	21	2.88					
	93	1-Tricosene	23	0.29					
	87	9,12-Octadecadien-1-ol	18	0.10					

Catalysts	A					B			
	Hydrocarbons					Fatty Acids			
	SI (R.T)	Compounds <sup>o</sup>	C	D <sup>y</sup> (%)	Acid <sup>y</sup> (%)	SI	Compounds <sup>o</sup>	Esters <sup>y</sup> (%)	SI
MCM-48/650	94	1,3-p-Menthadiene <sup>o</sup>	10	0.31	12.42	94	Palmitic	0.84	96
								0.41 <sup>*</sup>	97
	96	9 Furan, tetrahydro-2,2-dimethyl-5-(1-methyl-9-propenyl) <sup>o</sup>	10	0.22			Linoleic	0.76	96
								29.01	87
	95	9 1,4-Cyclohexadiene, 1-methyl-4-(1-methylethyl) <sup>o</sup>	10	0.25	20.48	95	Oleic	1.42	95
	95	2-Carene <sup>o</sup>	10	0.45	5.30	95	Stearic	1.30	95
								0.24 <sup>*</sup>	96
	96	Cyclohexene, 1-methyl-4-(1-methylethylidene) <sup>o</sup>	10	0.61					
	96(10.49) and 95(11.42)	1,2,4,5-Tetramethylbenzene <sup>o</sup>	10	0.21	and				
					0.43				
	89	1,1,3,3-tetramethoxypropane	7	0.19					
	97	7 7-4-Decadienal	10	0.52					
	85	1H-3a,7-Methanoazulene, 2,3,6,7,8,8a-hexahydro-1,4,9,9-tetramethyl-, (1 $\alpha$ ,3 $\alpha$ , $\alpha$ ,7 $\alpha$ ,8 $\alpha$ , $\beta$ ) <sup>o</sup>	15	0.52					
	86	Tricyclo[4.3.0(3,6,9)]non-3-ene, 2,2,5,5,8,8-hexamethyl-(1 $\alpha$ , 6 $\beta$ , 7 $\alpha$ , 9 $\alpha$ ) <sup>o</sup>	15	0.24					
	86	Longifolene <sup>o</sup>	15	0.29					
	86	3,4,5-Trimethylphenol <sup>o</sup>	9	2.67					
	86	Cedrene-V6 <sup>o</sup>	15	1.78					
	88(17.85) and 89(19.19)	$\alpha$ -Chamigrene <sup>o</sup>	15	0.46	and				
					0.38				
	98	Pentadecane	15	0.23					
	97	Oktadecane	18	0.29					
	97	Nonadecane	19	0.29					
	94	2-Nonadecanone	19	0.29					
	97(35.43) and 94(40.29)	Docosane	22	0.27	and				
					0.28				
	97(31.82), 97(37.11) and 97(38.73)	Eicosane	20	0.27,					
				0.36	and				
				0.27					
88	18 9,12-Octadecadienoic acid (Z,Z)-, 2-hydroxy-1-(hydroxymethyl)ethyl ester	21	0.89						
87	di-(9-Octadecenoyl)-Glycerol	39	1.41						
97	1-Hexadecene	16	0.43						
93	Squalene	30	1.25						

**Table 3.** Comparison of synthesized catalytic activity with previous studies

Catalyst	Esterification-transesterification						Reference
	Methanol: Oil (n/n)	Temperature (°C)	Time (h)	FFA (%)	Feedstock	Conversion (%)	
W/Ti/SiO <sub>2</sub>	30:1	65	4	3	Waste oil	98	[32]
	30:1	65	4	8	Waste oil	49	[32]
ZnO-CaO	12:1	65	1	6	Jatropha	98	[31]
	12:1	65	1.5	9	Karanja	96	[31]
Al-MCM-41	3:1	60	5	10	<i>Calophyllum inophyllum</i>	98	[25]
ZMC/550	9:1 <sup>s</sup>	62	1	20*	<i>Calophyllum inophyllum</i>	41*	This work
MCM-48/650	9:1 <sup>s</sup>	62	1	20*	<i>Calophyllum inophyllum</i>	52*	This work
	9:1 <sup>s</sup>	62	1	22 <sup>f</sup>	<i>Calophyllum inophyllum</i>	100 <sup>g</sup>	This work

h (hour), n (molar), <sup>s</sup>wt/wt, \*Acid-base titration, <sup>f</sup>Linoleic acid (GC-MS), <sup>g</sup>linoleic acid methyl ester (GC-MS)

**Table 3** shows that increasing the FFA content of vegetable oils can decrease the activity of the catalyst in the reaction. This finding shows that the catalysts synthesized in this study (ZMC/550 and MCM-48/650) have better activity than the catalysts in the literature, especially Al-MCM-41 in the esterification of *Calophyllum inophyllum* oil.

#### 4. Conclusion

The addition of CaO and ZnO into MCM-48 caused the damage of the three-dimensional structure of the ZMC catalyst. The hydrophobic and hydrophilic sides of the surfactant decreased the catalytic activity. The Brønsted acid site of the silanol group and the surface area of the catalyst were the active sites of the catalyst that play a role in this reaction. The synthesized catalyst has higher activity than conventional catalysts in the reaction. In addition to provide the best activity in the reaction, MCM/650 catalyst was also selective for converting linoleic acid into biodiesel. Furthermore, this catalyst was also capable of cracking the compounds contained in *Calophyllum inophyllum* oil to make it suitable for biodiesel. The successful synthesis of this heterogeneous catalyst provides an opportunity to simplify the reaction system and minimize environmental hazards arising from conventional catalysts in the biodiesel production process from vegetable oils, particularly *Calophyllum inophyllum* oil.

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#### Author's Declaration

**Authors' contributions and responsibility** The authors made substantial contributions to the conception and design of the study. The authors took responsibility for data analysis, interpretation and discussion of results. The authors read and approved the final manuscript.

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#### Availability of data and materials

All data are available from the authors.

#### Competing interests

The authors declare no competing interest.

#### Additional information

No additional information from the authors.

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