



## Synthesis of MCM-48 contains NaCl and Surfactant as Catalyst in the Esterification of Nyamplung Oil (*Calophyllum inophyllum* L.) to Biodiesel



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### Abstract

This research looked at the thermolability of the MCM-48-NaCl catalyst in the esterification of nyamplung oil. The use of various calcination temperatures was to investigate the thermolabile catalyst's optimization. The activity of the catalyst in the esterification was determined using the acid-base titration method. The active site and thermolabile state of the catalyzed were determined using XRD and FTIR characterization. In the esterification, the CTAB-MCM-48-NaCl catalyst performed 12.36% better than the conventional catalyst (H<sub>2</sub>SO<sub>4</sub>). The catalyzed achieved its optimum condition at calcination of 650 °C, with a reaction activity of 21.44%. Bronsted acid and surfactants are active sites of catalysts that play a role in the reaction. The presence of NaCl did not significantly affect the thermolabile resistance of the MCM-48-NaCl catalyst. This catalyst has the potential for esterification and hydrocracking hydrocarbon compounds of nyamplung oil suitable for biodiesel.

**Keyword:** Heterogeneous catalyst; MCM-48-NaCl; Surfactants; Esterification; Nyamplung oil

### 1. Introduction

Energy sources from fossil fuels that are depleting, non-renewable, and causing environmental problems are the result of the use of these fuel sources have caused a lot of concern in the world [1, 2]. Therefore, it is necessary to find suitable alternative energy resources to replace fossil fuels. Biodiesel has become a viable alternative energy resource in recent years because directly in diesel engines [3]: has a high flash point, a high cetane number, lubricating properties, is non-corrosive, has low environmental emissions, can be decomposed, has high levels of toxicity is negligible [4], is renewable, and sustainable [5, 6].

Palm oil is the most common source of raw material used in the manufacture of biodiesel. However, these sources are food ingredients that their use as raw materials for energy sources will compete with human food needs [7]. Therefore, non-food sources have needed for the provision of renewable energy as biodiesel. Accordingly, this research project determines the choice of seeds from the fruit of the nyamplung plant. Nyamplung is a

wild and abundant plant, also not a food plant, and data on the oil content of this fruit seed shows 15% higher than *Jatropha* [8], in addition to reports that nyamplung plants are easier to regenerate [9]. Another advantage is that the nyamplung plant can bear fruit throughout the year so that supplies for production scale can be provided [10].

The esterification step [11], which uses a homogeneous acid catalyst H<sub>2</sub>SO<sub>4</sub>, is commonly used to make biodiesel from nyamplung oil. Although this approach produces excellent results, it has a flaw. The catalyst is difficult to separate from the product and cannot be reused [12]. In addition, these catalysts can pollute the environment [13] and are not economical [14]. Heterogeneous catalysts use to address this flaw. Heterogeneous catalysts are easy to remove from the product [15], produce minimal waste [16], and maybe reused [17].

The amounts of free fatty acids (FFA) in vegetable oils are another element that affects biodiesel production; the lower the FFA concentration, the more biodiesel is produced [18]. Nyamplung oil has a high FFA content of 20-30% [19], thus attempting to lower the FFA content of

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nyamplung oil as needed. Surfactants utilize a catalyst to esterify carboxylic acids [20, 21], mesoporous silica MCM-41 to transesterify vegetable oils [22, 23], and synthesize xanthene [24]. In addition to surfactants, the mesoporous silica catalyst L-MCM-41 [25] has high activity in carboxylic acid esterification. Furthermore, Lee et al. [26] also reported that the MCM-48 catalyst in the esterification was faster to reduce the FFA content of vegetable oils when compared to the MCM-41 catalyst. MCM-48's three-dimensional pore structure, which eliminates the potential of pore-blocking [27]. According to Jiao et al. [28], to increase the hydrothermal stability of the MCM-48 catalyst, it is necessary to add NaCl salt. MCM-48, which includes surfactants and NaCl, can be utilized as a catalyst in the esterification of nyamplung oil, as demonstrated [26, 29]. As a result, MCM-48 catalyst with surfactant and NaCl salt will produce in this study, and its activity in the esterification investigated.

## 2. Experimental

### 2.1 Materials

Except For NaCl (Pa. Merck), all Specifications of materials used in Ref. [30].

### 2.2. Synthesis and Characterization of Catalysts

The CTAB-MCM-48-NaCl catalyst is a method the Taba et al. [31-33]. Calcination at temperatures of 550 °C (MCM-48-NaCl/550), 650 °C (MCM-48-NaCl/650), 750 °C (MCM-48-NaCl/550) for 5 hours was identify to determine the thermolabile resistance of the catalyst. Identification of thermolabile catalyst using XRD (Shimadzu 7000) in the range  $2\theta$  2.0-80.0 with settings 0.01 deg, 2.0 deg/min, Cu K $\alpha$ , 40.0 kV, and 30.0 mA. FTIR was used to identify CTAB and Triton X-100 in the catalyst (Shimadzu 8300).

### 2.3. Catalyst Calcination Temperature Optimization

Modifications to the esterification method [23, 34] to optimize the catalyst calcination temperature. This reaction 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%-w/w catalyst to methanol, while the ratio of nyamplung oil: methanol was 1: 9 (w/w), and the reaction time was 60 minutes. The levels of FFA the yield obtained were identified using the acid-base titration method and methyl esters using GC-MS. The oil used is the result of Soxhlet extraction and has gone through the degumming phase [30].

### 2.4. GC-MS analysis

The fatty acid methyl ester components contained in the samples were analyzed using GC-MS (QP

2010 Ultra Shimadzu). SH-Rxi-5Sil MS column (30 m x 0.25 mm) with flame ionization detector (FID) (operated in EI mode at 70 eV). The temperature and interface of the ion source are 250 °C and 280 °C. Solvent cutting time is 2.5 minutes, 35-500 m/z. The sample was identified at an of 250 °C with splitless mode, a pressure of 136.3 kPa, a flow rate of 2.48 mL.min<sup>-1</sup> with a ratio of 1:50, and column early the temperature of 40 °C. The holding time was 2 minutes and raised to 250 °C at a rate of 10 °C.min<sup>-1</sup>. The final temperature of the column was 250 °C, which lasted for 25 minutes. The abundance of each compound in the relative area (%). Qualitative identify using the Wiley7 data library [35].

## 3. Results and Discussion

### 3.1. Catalyst Characterization

The X-ray diffraction pattern of the catalyst in the 2 $\theta$  wide-high angle region (Figure 1) did not show any significant peak changes. This is because only a small amount of NaCl is added and reacts in the MCM-48 three-dimensional channel [36]. The X-ray diffraction pattern of the catalyst in the 2 $\theta$  low angle region (Figure 2) shows the specific characteristics of the CTAB-MCM-48-NaCl (Figure 2a) catalyst [37]. Likewise, the X-ray diffraction pattern for the catalysts MCM-48-NaCl/550 (Figure 2b), MCM-48-NaCl/650 (Figure 2c), and MCM-48-NaCl/750 (Figure 2d) shows the success of synthesis. The strength of the typical X-ray diffraction peaks for MCM-48 following calcination confirms this [38-40].

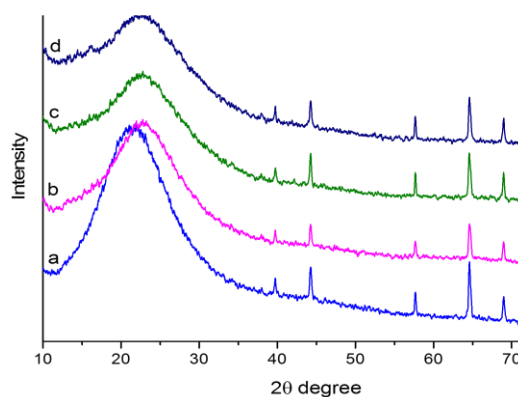


Fig. 1. X-ray diffraction pattern; a) CTAB-MCM-48-NC, b) MCM-48-NaCl/550, c) MCM-48-NaCl/650, and d) MCM-48-NaCl/750

The addition of NaCl affected the assembly of the surfactant with silica in the production of mesoporous silica, causing the intensity of these peaks to diminish [41, 42]. The effect of NaCl on surfactant and silica assembly was strengthened by FTIR absorption, which revealed a shift in the stretching asymmetric absorption of the silanol

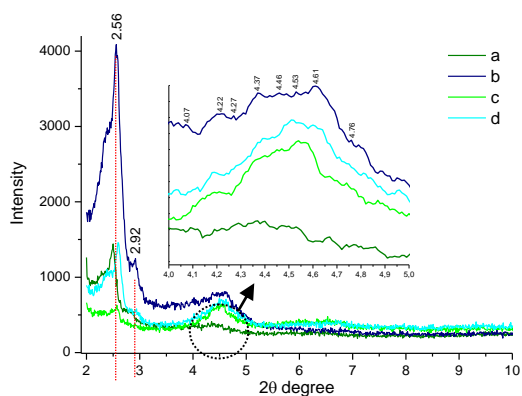


Fig. 2. X-ray diffraction pattern; a) CTAB-MCM-48-NaCl, b) MCM-48-NaCl/550, c) MCM-48-NaCl/650, and d) MCM-48-NaCl/750

group (Si-OH) to a higher wavenumber (Figure 3a, Table 1) for calcined catalysts, while it shifted to a lower wavenumber for uncalcined catalysts (Figure 3b, Table 1). Furthermore, all produced catalysts have MCM-48 FTIR spectra (Figure 3) appropriate with investigations [27, 31, 32].

### 3.2. Thermolabile Catalyst

The presence of NaCl in MCM-48 slightly increased the thermolabile resistance of the catalyst. The X-ray diffraction pattern (Figures 2b, 2c, and 2d) reveals that the distinctive peaks of the three-dimensional structure for MCM-48 may still maintain when the calcination temperature increases [42, 43].

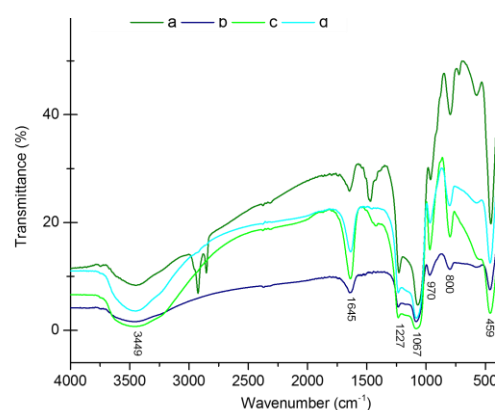


Fig. 3. FTIR Catalysts a) CTAB-MCM-48-NC, b) MCM-48-NC/550, c) MCM-48-NC/650, and d) MCM-48-NC/750

Table 1

Catalyst FTIR spectrum data interpretation

Catalyst	Group	V	$\lambda$ (cm <sup>-1</sup> )
CTAB-MCM-48-NaCl	≡Si-OH	S	3447
		B	1647
MCM-48-NaCl/550	≡Si-OH	SA	1070
		S	3451
MCM-48-NaCl/650	≡Si-OH	B	1645
		SA	1086
MCM-48-NaCl/750	≡Si-OH	S	3449
		B	1638
	-Si-O-Si-	SA	1086
		S	3451
	-Si-O-Si-	B	1638
		SA	1084

V: Vibration; SA: Stretching Asymmetric; B: Bending;  $\lambda$ : Wavenumber.

However, the increase in calcination temperature caused the collapse of the three-dimensional structural character of MCM-48. This is an indication of a decrease in the intensity of the peaks for these characters [38, 44]. The increase in the catalyst calcination temperature caused an increase in the absorption intensity of the silanol groups (3426-3480 cm<sup>-1</sup> and 1638-1647 cm<sup>-1</sup>). However, at a calcination temperature of 750 °C (MCM-48-NaCl/750), the intensity reduced. This is possible because the temperature has above the MCM-48-NaCl calcination temperature limit, causing water molecules in the gas phase to collapse into the air [45-47].

### 3.3. Optimization Of Catalyst

The optimization of the catalyst calcination in the reaction (Figure 4) yielded 36.13% for the CTAB-MCM-48/NaCl. This indicates that the presence of NaCl in CTAB-MCM-48/NaCl can increase the performance of the active catalyst sites (silanol groups and CTA<sup>+</sup>) in the reaction. This increase occurs through the role of NaCl which can increase the hydrophobic side to accelerate the meeting of active sites in the reaction system [28]. Furthermore, the presence of NaCl prevented from combining the yield with glycerol and water after the reaction [48].

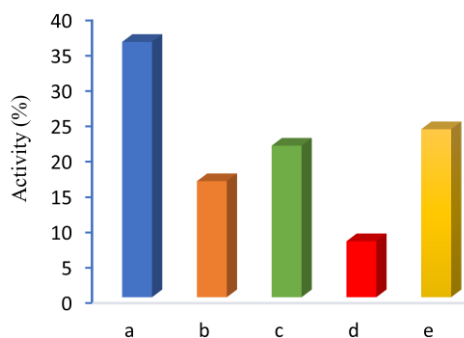


Fig. 4. Optimization of the catalyst in the esterification; a) CTAB-MCM-48-NaCl, b) MCM-48-NC/550, c) MCM-48-NaCl/650, d) MCM-48-NaCl/750, and e)  $H_2SO_4$

Increasing the calcination temperature to 650 °C (Figure 4c) increased the percentage of catalyst activity. However, on the calcination to 750 °C, the

catalyst activity decreased. The evident from the silanol group's absorption intensity (Figure 3), which is associated with the percent activity. The reduction in catalytic activity is further corroborated by the fact that the X-ray diffraction intensity of the catalyst in the low angle  $2\theta$  tends to decrease with increasing calcination temperature (Figures 2c, 2d, and 2e). The shows that the calcination temperature reaches 750 °C damage to the three-dimensional structure correlation with surface area and catalyst activity in the reaction [49, 50].

The results of identifying using GC-MS (Table 2) show two activities CTAB-MCM-48-NaCl catalyst; that is hydrocracking (Table 2A) and esterify (Table 2B) the compounds in nyamplung oil. This interesting thing about this catalyst is that the cracking results obtained belong to the range of constituent carbons for biodiesel [51-53].

Table 2: GC-MS analysis results

Treatment		A Hydrocarbons				B Fatty Acids			
Treatment	SI	Compounds*	CF	Persentase (%)	Acid (%)	SI	Compounds*	Esters (%)	SI
Before	95	Tetradecanal	$C_{14}H_{28}O$	0.01	8.64	94	Palmitic		
	90	2-trans-9-Octadecenylxyethanol	$C_{20}H_{40}O_2$	0.32	21.95	92	Linoleic		
	93	1-Tricosene	$C_{23}H_{46}$	0.29	4.26	95	Oleic		
After					3.44	90	Stearic		
	96	2-Carene	$C_{10}H_{16}$	0.10	2.01	94	Palmitic	0.19 <sup>#</sup>	96
	95	1,4(8)-P-Menthadiene	$C_{10}H_{16}$	0.17			Linoleic	1.64	96
	95	1,2,4,5-Tetramethylbenzene	$C_{10}H_{14}$	0.13	12.85	96	Oleic	3.35	96
	98	Pentadecane	$C_{15}H_{32}$	0.10			Stearic	1.98	93
	97	1-Hexadecene	$C_{16}H_{32}$	19.48			Arachidic	0.14	95
	93	1-Tetradecanamine, N,N-dimethyl	$C_{16}H_{35}N$	16.58					
	96	1-Octadecene	$C_{18}H_{36}$	0.39					
	97	1-Octadecanol	$C_{18}H_{38}O$	0.68					
	97	1-Nonadecene	$C_{19}H_{38}$	0.09					
	96	Eicosane	$C_{20}H_{42}$	0.59					
	96	Docosane	$C_{22}H_{46}$	0.17					
	97	Nonacosane	$C_{29}H_{60}$	0.22					
	96	Pentacosane	$C_{25}H_{52}$	0.58					
96	Hexacosane	$C_{26}H_{54}$	0.16						
93	Squalene	$C_{30}H_{50}$	1.97						

SI: similarity index; \*: Compounds having an SI of 90-100; CF: Chemical Formula; #: Ethyl esters

#### 4. Conclusions

The addition of NaCl to MCM-48 enhances the catalyst's thermolabile endurance and performance in the esterification of nyamplung oil. Catalyst CTAB-MCM-48/NaCl is a catalyst that gives the

most optimum activity 12.36% higher than the  $H_2SO_4$  catalyst. In addition, this catalyst also has the potential to hydrocracking hydrocarbon compounds suitable for biodiesel, has the potential to be used more than once, and the method is more economical

because it is without the removal of surfactant.

### Conflicts of interest

On behalf of all authors, the corresponding author states that there is no conflict of interest.

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