

## Analysis of the Ratio Catalyst Promoters BaO/CaO to Methyl Ester Content and Yield in Biodiesel Using Electrocatalytic Reactor

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DOI: <https://doi.org/10.21107/rekayasa.v15i1.13761>

### ABSTRACT

The efficiency of biodiesel production from vegetable oils needs to be developed. The process of transesterification using heterogeneous catalysts has been widely studied to replace the role of homogeneous catalysts. The use of BaO promoters into metal oxides increases the activity of heterogeneous catalysts in transesterification reactions. This study was conducted to provide information about the effect of BaO concentrations that are filtered into calcium oxide (CaO) on the transesterification reaction of tofu pulp oil into methyl ester using electrocatalytic methods. The study was conducted at room temperature with a voltage of 18.5 Volts and a reaction time of 120 minutes. Methanol ratio: tofu oil is used at 10:1 with Tetra Hydro Furan (THF) as a co-solvent. The electrocatalytic process is carried out using graphite electrodes. The research variables are the concentration of BaO promoters used at 2%, 4%, and 8%. The parameter of the test is methyl ester content obtained from the results of transesterification reactions with chromatography gas analysis (GC-MS). In this study, the BaO/CaO catalyst was impregnated by wet method and calcined at 450°C for 180 minutes. From the experiments conducted the highest methyl ester yield was obtained in BaO / CaO catalyst 2% with a yield of 99.99% and biodiesel yield of 94%.

**Key words** : biodiesel, catalysts, electrocatalytic, methyl ester, tofu oil

### INTRODUCTION

In biodiesel production processes involving transesterification reactions, the selection of catalysts has a major influence in producing methyl esters. CaO is a material that can be used as a heterogeneous catalyst in biodiesel production. CaO catalysts can be generated from thermal decomposition with materials containing CaCO<sub>3</sub>. High catalytic activity, economically low cost, reusable, has high base strength is an advantage of CaO as a catalyst because it has an active site (Liu *et al.*, 2008). To improve the performance of a CaO catalyst can be done by impregnation process as a modification of the catalyst. Impregnation is the saturation of materials by filling the material pores using active metals as buffers. CaO carried using active metals in this case BaO can increase catalyst activity (Wu *et al.*, 2012).

Widiarti (2015) conducted biodiesel production research using CaO/SrO catalysts in electrocatalytic jelantah oil transesterification reactions. Variations

in time (30, 60, 90, 120, and 150 minutes) and energy (80, 100 watts of power input) use methanol solvents. Modification of catalysts is performed using the SrNO<sub>3</sub> impregnation method on CaO catalysts. The optimum transesterification reaction occurs with the best catalyst activity at 100 watts, a reaction time of 120 minutes and the number of CaO/SrO catalysts of 4% b/b with methyl ester products of 92.86%.

Research proved that the impregnation of CaO catalysts with SrO buffers can increase the methyl ester produced, but there is a decrease in CaO crystallinity. CaO crystalline decreases as the number of SrO decreases in CaO, while SrO crystalline is increasing. The decrease in CaO crystallinity as a catalyst and the increase in SrO as a buffer have an impact on the decrease in methyl esters resulting from SrO agglomeration that closes the active CaO site and decreases the surface area of the catalyst. A decrease in surface area impacts the

#### Article History:

**Received:** Feb, 10<sup>th</sup> 2022; **Accepted:** April, 15<sup>th</sup> 2022

Rekayasa ISSN: 2502-5325 has been Accredited by Ristekdikti (Arjuna) Decree: No. 23/E/KPT/2019 August 8th, 2019 effective until 2023

#### Cite this as:

Prastyo, E., Ibrahim, P.A & Solikha, D.F. (2022). *Analysis of the Ratio Catalyst Promoters BaO/CaO to Methyl Ester Content and Yield in Biodiesel Using Electrocatalytic Reactor*. Rekayasa 15 (1). 107-113 pp.

doi: <https://doi.org/10.21107/rekayasa.v15i1.13761>

number of active sides of the catalyst decreasing and collisions between molecules decreasing (Widiarti, 2015).

Anwaristiawan (2018) conducted a research modification of BaO / Zeolit Y catalysts on the transesterification reaction of biodiesel from castor seed oil. This study aims to determine the characteristics of catalysts based on function groups, crystallinity, surface area, and catalyst activity. Research proves that the activity of Zeolite Y catalysts increases by impregnation modification using BaO as a buffer. The best catalytic activity of the transesterification reaction based on yield, obtained the optimum catalyst in BaO /Zeolite Y by 2%, the reaction time of 120 minutes produces a methyl ester yield of 94%. Analysis of the characteristics of the functional group shows the composition of the catalyst does not change. Barium Hydroxide  $Ba(OH)_2$  has been researched by (Georgogianni *et al* (2009) has strong properties to perform biodiesel synthesis reactions. Although Mazzochia *et al* (2004) confirmed that  $Ba(OH)_2$  dissolves slightly in the reactions that are sucked into biodiesel, the modification of BaO/CaO catalysts is done to improve the numbness of the catalyst used. The base properties of the catalyst can increase the active site, so that the electronegative value of conjugated metal cations increases.

Alkaline oxides, especially alkaline soils, are examples of base dopants that have high catalytic activity when compared to transition oxides. However, alkaline soil oxides have the disadvantage of having almost the same phase as transesterification products, so it is necessary to add a buffer. The addition of buffers is expected to reduce the weakness of the alkali oxide catalyst used and increase the yield yield of the transesterification reaction. Based on the preliminary description, the study was modified by bao catalyst using cao metal oxide through the process of wet impregnation. Modified catalysts are used for the process of transesterification of dreg tofu oil using electrocatalytic methods.

Previous research conducted the manufacture of modified catalysts through a combination of strong alkaline earth metals and alumina minerals. A preliminary study of biodiesel synthesis using electrocatalytic methods was conducted by Guan and Kusakabe (2009). Therefore, this study focused on the use of BaO/CaO impregnation catalysts against methyl ester content produced based on

reaction time using electrocatalytic methods. Reaction time variables and catalyst promoter ratios were studied on methyl and ethyl ester results by being analyzed by Gas Chromatography Mass Spectroscopy (GC-MS).

## METHODS

The free variable used in this study is the catalyst promoter weight ratio of 2% w/w; 4% w/w; 8% w/w. Fixed variables in the study were a reaction time of 120 minutes, a molar ratio of methanol: oil of 10:1, and a voltage used for the electrocatalytic process of 18.5 Volts.

### 1. Chemical

Tofu oil is obtained through the sokhlet extraction method using benzene solvents. Methanol as a solvent, and Tetra Hydro Furan (THF) as a co-solvent, sodium hydroxide (NaOH), Calcium Oxide (CaO), Barium Hydroxide  $Ba(OH)_2$ , graphite electrode, and aquadest.

### 2. Extration of Tofu Oil

The process of preparing the initial raw material is done by separating the tofu pulp from the water by compressing. Result pressing is further processed through drying using a 120 temperature oven C for 5 hours. Drying It aims to reduce the water content found in the tofu pulp. High water content can increase the value Free fatty acids resulting in a decrease in the quality of biodiesel. The drying process is carried out until heavy. Constant tofu dregs that indicate there is no evaporation of water in the tofu dregs. Dry tofu contains 9.96% fat that can be extracted to produce vegetable oil (Tessa, 2018). The method of extraction of refluk is used in the process of extracting vegetable oil from tofu pulp. The extraction process is carried out in batches at a temperature of 80 – 90°C as many as 15 cycles with a time span of 120 – 150 minute. Tofu pulp is used for one extraction of 50 grams with benzene solvent. Benzene used as a solvent with the ratio of the ratio between the tofu pulp to the solvent is 1:5 b/v.

### 3. Impregnation of Catalys

Catalyst impregnation is carried out by a type of wet method impregnation, in which the amount of prequel solution is increased. The active fas (CaO solution) is greater than 1.5 times the volume of the buffering pore (BaO). Impregnation of catalysts is done by inserting CaO powder into a solution of

Ba(NO<sub>3</sub>)<sub>2</sub>. Heated solution Temperature 60 – 70°C while stirring until the slurry forms for 1 hour. Slurry CaO/BaO is then dried in oven at 100°C for 2 hours. Once dry, CaO/BaO is then calcined at 550°C for 4 hours. CaO/BaO with concentrations of BaO 2, 4, and 8 % b/b, then used in synthesis process applications Biodiesel.

#### 4. Electrocatalytic Reactor

The electro-catalytic reactor is made of glassware with a volume of 500 mL consisting of electrolysis cells containing two electrode plates (10 mm × 130 mm) separated by a distance of 150 mm (Figure 1). Working electrodes (cathodes) and counter electrodes (anodes) are made of graphite. The electrolysis reaction is performed using a constant potential of 18.5 Volts and is applied between two electrodes at room temperature (28°C) and under agitation. Stirring process using magnetic stirrer at a constant speed at 100 rpm.

#### 5. Reaction Mixture

Electro catalytic reactors are filled with tofu oil and methanol using molar ratio of 1:10. Co-solvent tetra hydro furan (THF) is used to bring together tofu and methanol oil during the biodiesel synthesis process. Comparison of the THF and methanol set at 1:1 v/v. Mixing methanol with oil will It is formed in two layers because it has a phase that different so as to optimize the surface reaction between is added THF. The effect of the number of catalyst promoters was set in the study at 2%, 4%, and 8% on the methyl ester yield of biodiesel at 120 min.

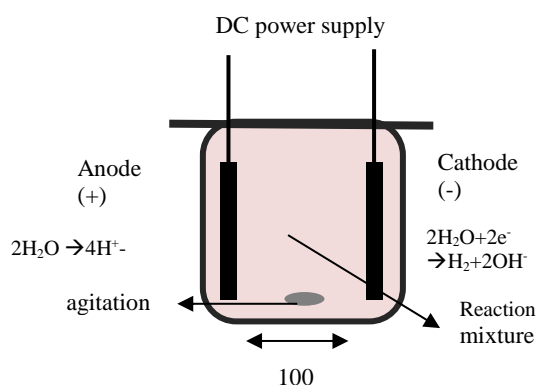


Figure 1. Schematic of Electro-Catalytic Cell And Reactions

## RESULTS AND DISCUSSION

### Characteristic of Tofu Oil

The resulting tofu pulp oil is done free fatty acid analysis to determine the next stage of the process. The results of the analysis showed the resulting tofu pulp oil had a free fatty acid content of 0.73%. The following is shown the characteristics of oil dregs know the results of extraction.

Table 1. Result of the Tofu Oil Analysis

Information	Value			Average	Standart	Unit
	P1	P2	P3			
Free Fatty Acid	0,6	0,8	0,8	0,73	<5	%
Density	0,92	0,98	0,97	0,96	0,95 – 0,97	gr/mL
Viscosity	0,07	0,09	0,09	13,60	≤40	cSt

The results of the analysis of fatty acids free of tofu oil resulting from the extraction process meet the maximum limit. Free fatty acid values >1% are not recommended to be directly used for biodiesel transesterification process because it can decrease biodiesel yield, increase of saponification reaction, and increase the viscosity value. This is in accordance with the results of the analysis which showed that when the value of free fatty acids increased, the density value and viscosity of tofu pulp oil also increased. Density values are always correlated with viscosity values, where the higher the density, the higher the viscosity (Irawati, 2018).

### Analysis of Methyl Ester Content

Identification of compounds resulting from transesterification reactions of biodiesel oil dregs tofu using CaO catalysts with BaO promoters is carried out with Gas Chromatography Mass Spectrofotometer Type QP2010S Shimadzu. Gcms testing method is done to find out the composition of the type of fatty acid that makes methyl ester from oil dregs know the transesterification results. Ningtyas et al., (2013), stated the detection of fatty acid and triglyceride types in biofuels using the gas chromatography (GC) method, followed by the analysis of mass spectroscopy (MS). The GC method is carried out for the purposes of separation, quantification, and analysis of fatty acids by first making derivatives of fatty acids, as well as MS analysis to determine the fragmentation of saturated and unsaturated fatty acids, as well as the location of double bonds of fatty acids. The results of the analysis using GC-MS are shown in the following figures.

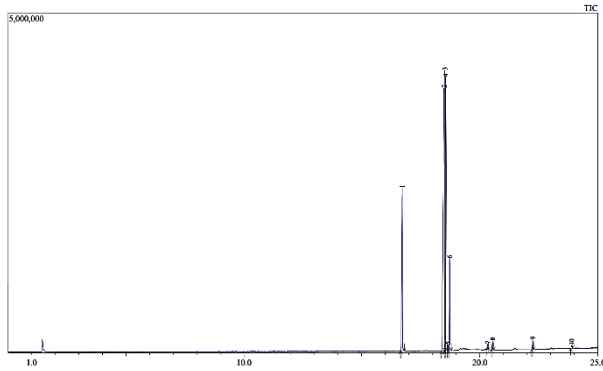


Figure 2. GC-MS result of Methyl Ester 2% BaO/CaO 120 min reaction

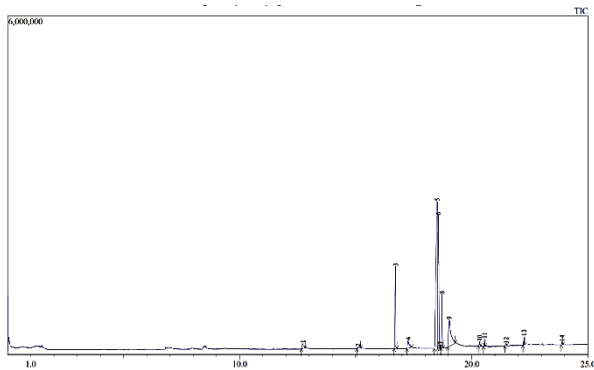


Figure 3. GC-MS result of Methyl Ester 4% BaO/CaO 120 min reaction

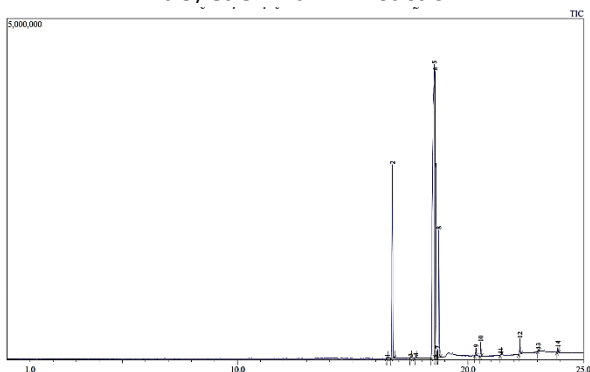


Figure 4. GC-MS result of Methyl Ester 8% BaO/CaO 120 min reaction

GC-MS analysis results at BaO catalyst promoter concentrations of 2%, 4%, and 8% indicate that the results of biodiesel synthesis using electrocatalytic methods are indeed biodiesel compounds. Catalyst promoter BaO 2% showed 10 peaks detected as methyl esters, BaO 4% showed 6 peaks detected as methyl esters with 6 other peaks as saturated and unsaturated fatty acids such as palmitic acid, lauric acid, myristic acid, and linoleic acid. Promoters of BaO 8% showed 11 peaks that belonged to the methyl ester group.

Table 2. Comparison of Methyl Ester Biodiesel Composition

Compound	Promoters					
	2% w/w		4% w/w		8% w/w	
	% Area	Peak	% Area	Peak	% Area	Peak
Methyl palmitoleate	-	-	-	-	0,05	1
Methyl Palmitat	12,35	1	10,97	3	14,11	2
Methyl heptadecanoate	-	-	-	-	0,09	4
Methyl Linoleat	30,32	2	44,45	5	54,31	5
Methyl octadecanoic	-	-	-	-	21,92	6
Methyl Oktadeka 9,12 Dienoat	19,56	3	-	-	-	-
Methyl Oleat	30,33	4	20,57	6	-	-
Methyl 9,12,15 Oktadekatrinioat	0,41	5	0,38	7	0,46	7
Methyl Stearat	5,40	6	5,68	8	6,66	8
Methyl 11 Eicosanoat	0,27	7	-	-	0,47	9
Methyl Arachidic	0,54	8	-	-	0,66	10
Methyl heneicosanoate	-	-	-	-	0,04	11
Methyl Behenat	0,61	9	0,88	-	0,78	12
Methyl Lignoserat	0,20	10	0,34	-	-	-
<b>Total</b>	<b>99,99</b>		<b>83,27</b>		<b>99,55</b>	

The three highest methyl ester compounds found in all concentrations of catalyst promoters are methyl palmitate, methyl linoleate, and methyl stearate. This is in accordance with research conducted by Rosmawaty, 2018 which states that the highest methyl ester content in biodiesel is methyl palmitat, methyl linoleate, and methyl stearate which have carbon chain bonds of 16 or more. Biodiesel has a condition one of them is the value of the number of satans produced. A high number of values can be achieved when biodiesel has an atomic compound of C of 16 or more (Kayser, 2014). Methyl palmitat has the shortest carbon chain so that its peak appears earlier than methyl linoleic, methyl oleate, and methyl stearate. While the peak of oleate appears earlier than methyl stearat because the molecular weight of methyl stearat is greater than the molecular weight of methyl oleate. In SNI 7182-2015 on Biodiesel, mentioned that the minimum methyl ester content is 96.5%, where in this study the methyl ester content reached 99.99% in BaO promoters 2%, 82.05% in BaO promoters 4%, and 99.95% in CaO promoters 8%, so that the biodiesel produced has met SNI except in the use of BaO promoters 4%.

Figure 5 shows the 4 main contents of methyl ester found in all treatment is methyl palmitate, methyl linoleate, methyl stearate, and methyl linoleic. The methyl palmitat, methyl linoleic, and methyl stearate content produced in biodiesel showed promoters of 8% were the highest compared to 2% and 4%. This suggests that the addition of a promoter of BaO catalysts to the

electrocatalytic process of tofu oil may increase the content of methyl esters (methyl palmitat, methyl linoleic, methyl stearic). Different values are found in methyl oleate compounds where promoters of 2% BaO get the highest value and 8% BaO gets the lowest value. Palmitic acid is the most important saturated fatty acid, while oleic acid is the most important unsaturated fatty acid in biodiesel (Gultom, 2001).

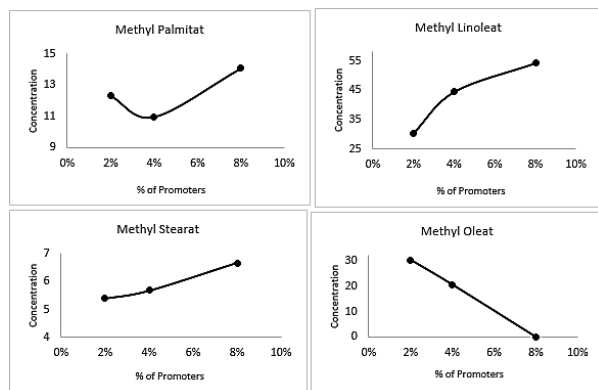


Figure 5. Trans-Esterification Curve Variations In The Number Of Catalyst Promoters

The transesterification of biodiesel using an 8% promoter indicated the absence of methyl oleic compounds or oleic acid. It is suspected that oleic acid found in tofu oil undergoes an oxidation reaction and does not convert into methyl oleic. Oleic acid is an unsaturated fatty acid that is easily oxidized compared to stearic acid and miristatic acid (Desnelli, 2009). The oxidation process occurs due to the increasing use of BaO as a catalyst promoter, there by increasing the oxide function group on the catalyst. This is reinforced from the trend of oleic acid charts that are decreasing by increasing BaO promoters on catalysts. This reaction encourages the formation of hydroperoxide that decomposes into carbonyl compounds (Rachman, 2013) which is strengthened by the emergence of Octadecadienoyl chloride compounds resulting from GC-MS analysis. The methyl ester results formed are then correlated with the content of short-chain acids based on the results of the following GC-MS analysis.

Table 3 shows the use of BaO promoters 4% has the highest short chain fatty acid content compared to the use of promoters 2% and 8%. The short-chain fatty acids found in the use of 4% of catalysts indicate that the reaction has not reached equilibrium at the reaction time of 120 minutes. This indicates that a time of 120 minutes with the use of

a 4% BaO promoter against a CaO catalyst has not been able to fully convert short-chain fatty acids into methyl esters. The decrease in catalyst performance with the addition of a 4% BaO promoter is suspected due to the closure of the catalyst's active site by the promoter. This is in accordance with research conducted by Wishnu & Putra (2017), this will cause a decrease in selectivity caused by the partial cover of the active side of the catalyst resulting in less and less methyl ester concentrations.

Table 3. Comparison of composition of short-chain fatty acid compounds

Compound	Promoters					
	2% w/w		4% w/w		8% w/w	
	Area	Peak	Area	Peak	Area	Peak
Lauric Acid	-	-	1,35	1	-	-
Myristic Acid	-	1	0,49	2	-	-
Palmitic Acid	-	-	2,08	4	-	-
Linoleic Acid	-	2	11,24	9	-	-
Octadecadienoyl chloride	-	-	-	-	0,06	3
<b>Total</b>	<b>0,00</b>		<b>15,19</b>		<b>0,06</b>	

Furthermore, excessive use of BaO can increase the saponification reaction due to the increasing caharacteristic of catalyst bases (Pahlevi et al., 2015). The increased base properties of catalysts increase the reaction of by products, namely the incognito reaction in transesterification reactions due to more reactive base catalyst reactions with free fatty acids compared to diglycerides (Pratiwi, 2018). The conversion values of triglycerides into short-chain fatty acids and methyl esters are then compared to the total methyl ester and biodiesel yield formed in the following figure.

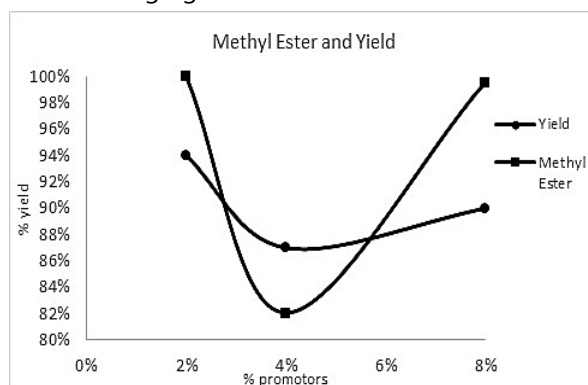


Figure 6. Methyl ester value and biodiesel yield

Figure 6 shows a percent of the product area experiencing increases and declines. Variations in the number of catalyst promoters with a concentration of 2% have the highest yield and methyl ester value compared to others, which is 94%. In the use of catalyst promoters the

concentration of 4% to 8% in general increased, but at a concentration of 4% decreased. This decrease is due to the excessive effect of BaO grazing, thus causing the formation of emulsions resulting from the inflorescence reaction (Pahlevi et al., 2015). In addition, with the closure of the pore as delivered by Vishnu & Putra (2017), this will cause a decrease in selectivity caused by the cover of part of the active side of the catalyst resulting in the concentration of methyl ester obtained getting smaller. Furthermore, the use of BaO promoters 8% shows the yield and methyl ester graph has increased when compared to BaO 4%. This happens because the conversion process from triglycerides to glycerides and FAME experiences a state of equilibrium faster than the use of 4% BaO. This is in accordance with research conducted by Anwaristiawan, 2018 which states that the addition of BaO promoters to zeolite catalysts indicates a decrease in intensity with the increasing metal oxides.

## CONCLUSIONS

Based on the results of the study it is known that the use of BaO / CaO catalysts can not increase the overall methyl ester formation reaction of tofu oil. The use of BaO as a promoter at a reaction time of 120 minutes by electrocatalytic method can affect the equilibrium state of transesterification reactions evidenced by the appearance of free fatty acid compounds that have not been converted. Methyl ester formation is highest obtained in the use of CaO catalysts with BaO promoters 2% which is 99.99% with a biodiesel yield of 94%.

Based on the results of research has been obtained catalysts that have high catalytic activity in the reaction of transesterification of oil into biodiesel using electrocatalytic methods. But research on this catalyst still needs to be developed again because it still needs to be studied the influence of catalyst particle size, the reuse of catalysts on the activity of catalysts.

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