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Biodiesel Production from Canola Oil Using TiO_2CaO as a Heterogenous Catalyst

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ABSTRACT

Biodiesel is one of the renewable energy sources that is an alternative to fossil diesel that is non-toxic and produces less CO emissions. Transesterification process is a conventional mechanism to produce biodiesel from vegetable oil with a homogeneous or heterogenous catalyst. However, heterogenous catalysts are considered as more efficient than homogenous catalysts. Recently, TiO_2/CaO has been used as a compound heterogenous catalyst to produce biodiesel produce from palm oil, waste cooking oils and algae. In this research, biodiesel was manufactured using canola oil as a feedstock and titanium dioxide / calcium oxide (TiO_2/CaO) as a catalyst. The aim of this study is to prepare the catalyst, investigate the transesterification process and measure the chemical and physical biodiesel properties. Catalyst preparation required four stages: dry mixing, wet mixing, water separation and catalyst activation where there were two temperature phases (200 °C and 600 °C). Catalyst mixed with methanol by 1:16 ratio had different mixing time phases (30 minutes, 60 minutes, and 90 minutes). The Transesterification process was by blending the catalyst-methanol mixture with canola oil under 3 phases (4 hours, 5 hours, and 6 hours). The catalyst characterization was by analysis of X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), temperature activation effects and activation time effects. The transesterification process analysis showed that the optimization conditions to produce biodiesel are 600 °C activation catalyst temperature, 90 minutes of catalyst-methanol mixing, 1.5% wt. catalyst concentration and 5 hours of transesterification time. The biodiesel yield was 96.9%. Moreover, new parameters were applied for this research (time and temperature of activation catalyst, catalyst-methanol mixing parameters and transesterification process conditions). Biodiesel properties (kinematic viscosity, flash point and water content) were measured according to ASTM D6751 standards and similarity was 98%. Therefore, biodiesel can be produced from canola oil and TiO_2/CaO, but this still needs more studies on several topics such as the blending of canola with multi feedstocks, the ethanol impact and catalyst poisoning in the case of using TiO_2-CaO as a catalyst.

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1. Introduction

Biodiesel is a non-toxic renewable energy source that is gaining attention globally owing to its direct applicability in pre-existing engines without any modification [1,2]. Fossil fuel energy sources are scarce and the continued use them release carbon pollutants into the atmosphere such as CO_2 and CO, which can cause global warming [3]. The higher production cost of biodiesel compared to the fossil fuel counterpart is one of the major drawbacks of biodiesel especially in the developing countries [4].

Recently, various types of heterogeneous catalysts and continuous reactors have been invented to produce biodiesel [5]. However, homogenous catalysts are more commonly used than heterogenous ones [6], but still heterogenous catalysts are more efficient than homogenous ones because the heterogeneous allows high yields at low reaction times [7]. Heterogeneous catalysts act dissimilarly during the phase of the transesterification reaction. This type of catalysts was widely used in industries since they possess certain impediments during the esterification and transesterification processes [8].

Recently, researchers produced biodiesel from canola oil [9-12]. Biodiesel produced from canola oil used an acid catalyst and heterogenous catalyst [13,14]. However, titanium dioxide with calcium oxide (TiO_2/CaO) were used to produce biodiesel from palm oil, waste cooking oils and algal, but that was not applied on canola oil [15-21].

The transesterification reaction between vegetable oil and methanol occurred in the presence of catalyst to produce biodiesel [22]. Catalyst used in transesterification is usually a homogeneous base or acid [23]. The transesterification rate is faster in the homogeneous catalytic approach such as CaO catalyst [24]. The transesterification process is subject to a variety of factors that can be considered to improve biodiesel yield. One of the factors is the catalyst type and concentration, which play significant roles in the transesterification of biodiesel sources [25].

In this research, biodiesel will be produced from canola oil, using TiO_2/CaO mixed with methanol. Previous studies highlighted TiO_2/CaO used with palm oil and waste cooking oils only. In this study, canola oil was used with different parameters. Biodiesel's product will be tested on more than one engine to see its effectiveness accurately and clearly. This study aims to obtain a new biodiesel product with high specifications and good efficiency. There are four objectives for this research, which are preparing TiO_2/CaO catalyst at 200 ºC and 600 ºC, investigating the transesterification reaction under different phases, producing biodiesel and measuring chemical and physical properties for the biodiesel product.

One of the contributions of this study is developing previous studies on heterogenous catalyst TiO_2-CaO. Furthermore, in this study, new parameters were designed to obtain biodiesel and calculate the yield. This study also contributed in seeing the effect of increasing alcohol ratio on biodiesel yield.

2. Methodology

In this study, chemicals were used titanium dioxide (TiO_2) with a purity of 99%, calcium oxide (CaO) with a purity of 98%, canola oil (Natural brand) and methanol with a purity of 95%. The experimental work for this study included catalyst preparation and the transesterification process. These processes are used for the production of biodiesel. To set all experimental work steps, research parameters must be designed appropriately. Research parameters were designed with the Taguchi method by using the Minitab software, and three parameters were chosen for this study shown in Table 1.

Table 1
Research Parameters Design

Parameters	Design Level		
	A+	A	A-
Transesterification Time	240 Mins.	300 Mins.	360 Mins.
Mixing Time	30 Mins.	60 Mins	90 Mins.
Catalyst Concentration	0.5	1.0	1.5

2.1 Catalyst Preparation

Catalyst preparation required four stages: dry mixing, wet mixing, water separation and catalyst activation as shown in Figure 1. Catalyst preparation parameters are also shown in Table 2. Dry mixing required TiO_2 and CaO, TiO_2 mixing with CaO at mixing ratio 1:1, which needed 27g from TiO_2 and 27g of CaO at analytical balance and mixing in Pyrex beaker. Wet mixing required H_2O to mix with dry TiO_2/CaO mixture in Pyrex beaker at mixing ratio to water of 3:50 or 60%. The water separation process required to put the mixture on hot plate for 60 minutes under 100 °C to dry water from TiO_2/CaO. The final process is to put TiO_2/CaO in the furnace at different temperature (200 °C and 600 °C) for 6 hours. The catalyst will be obtained after these steps (shown in Figure 2), and must be 54g of TiO_2/CaO to be ready for transesterification process.

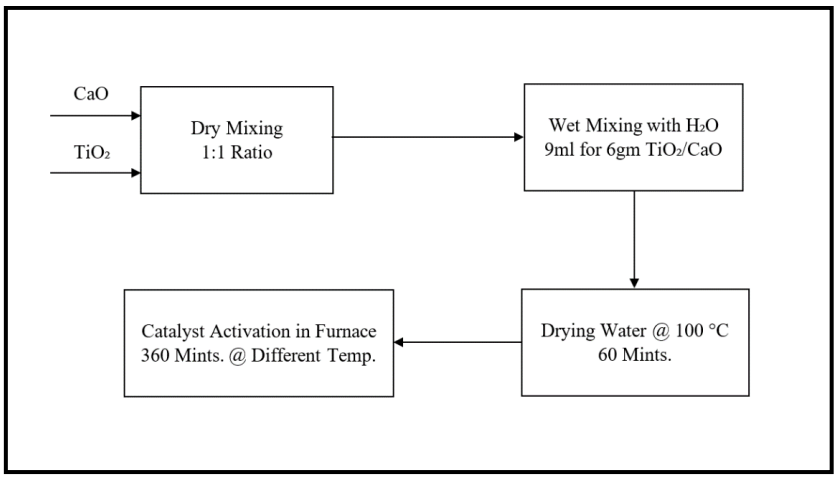

Fig. 1. Catalyst Preparation Process

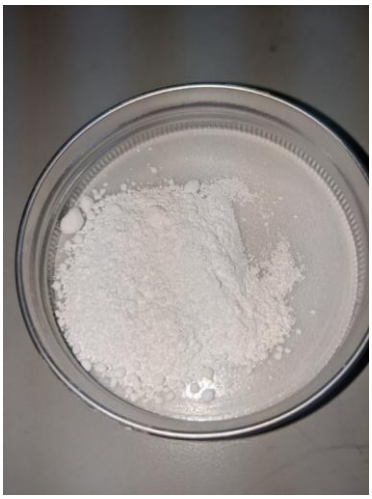

Fig. 2. Catalyst Final Product

2.2 Transesterification Process

Transesterification process to produce biodiesel require six steps, which are canola oil pre-heating, catalyst-methanol mixing, transesterification reaction, separation process, filtration process, and weight recycling catalyst. General biodiesel production is shown in Figure 3. For pre-heated canola oil, a weight of 100gm (118ml) was put in a Pyrex beaker for 60 minutes at 100 °C on a hot plate. Catalyst-Methanol mixing required weighing the catalyst by analytical balance according to research parameters, then mixing with methanol using a 3-neck glass flux at room temperature. Transesterification reactions begin after canola oil pre-heating and catalyst-methanol is finished.

The separation process started after the transesterification reaction finished and was waited on overnight to complete. Filtration process required to separate the biodiesel on glycerin, as well as collecting remined catalyst to use for another reactions. The final biodiesel products shown in Figure 4, and to calculate biodiesel yield, an equation was used for this purpose (Eq. (1) and Eq. (2)).

$$Yield\ (\text{vol.}) = \frac{Vol.Outlet}{Vol.Inlate} * 100\% \quad (1)$$

$$Yield\ (\text{wt}) = \frac{wt.Outlet}{wt.Inlate} * 100\% \quad (2)$$

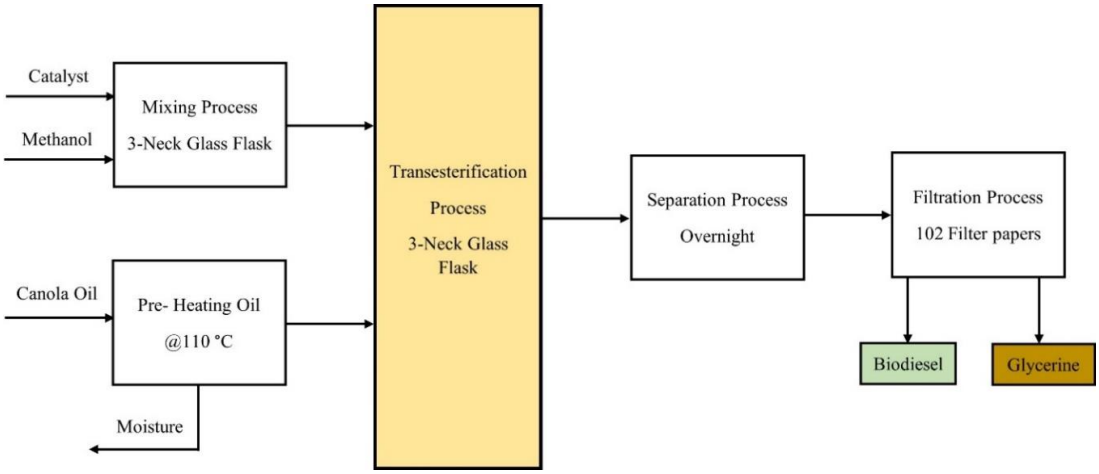

Fig. 3. General experimental work flowchart

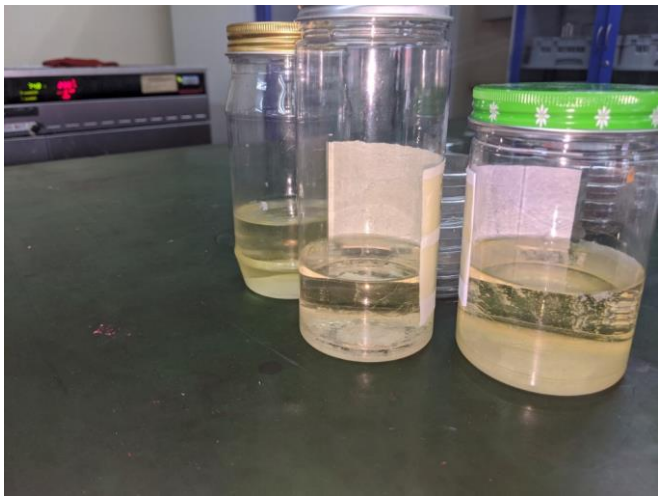

Fig. 4. Final Biodiesel Products

3. Results

Catalyst analysis tools are XRD, SEM, time effects, temperature effects, as well as how many times the catalyst will be used to produce biodiesel. Biodiesel results analysis will be based on yield, kinetic viscosity, flash point, water content, and methanol content.

3.1 Pressure Distribution

Understanding catalyst structure and mechanism used in this study provides a clear visualization of the interaction for the transesterification reaction mechanism. To characterize the catalyst, four analyses will be used to analyse the catalyst.

3.1.1 XRD analysis

X-Ray Diffraction analysis is very important to study the catalyst crystal structure. A specified amount from two samples of the catalyst is placed in an XRD device, the first sample is the catalyst at 200 °C and the second sample is the catalyst at 600 °C. The catalyst XRD analysis is shown in Figure 5 and Figure 6. There is a difference between the catalyst resulting from activation under 200 °C and 600 °C. This difference is due to the XRD scale analysis. A compound was observed in the catalyst (CaO_4) when catalyst activation occurred under 200 °C. That did not happen in the catalyst when activation occurred under 600 °C. In comparison with the most recent study, both catalysts peak at 2θ [17]. Peaks were shown for this study at 2θ = 21.1º, 38º, 48º, 63º, 70.5º and 82.8º, while the other study peaks were at 2θ = 17.9°, 28.6°, 34.1°, 46.9°, and 50.7°. Our research catalyst calcinations are 200 ºC and 600 ºC while other research catalyst calcinations were 200 ºC, 400 ºC, 600 ºC and 800 ºC. Surface areas for TiO_2-CaO compared to the other study is shown in Table 2.

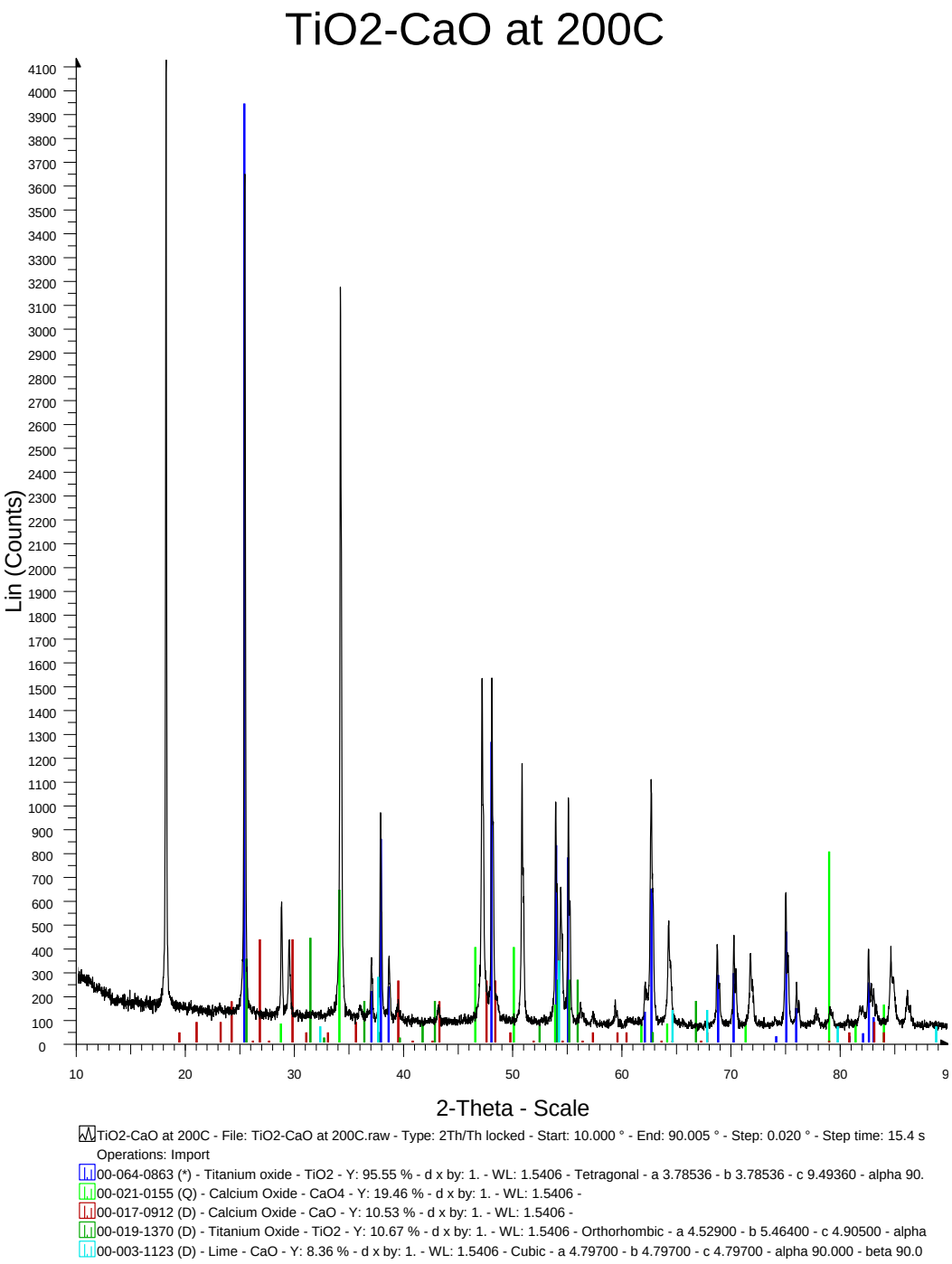

Fig. 5. XRD Analysis for Catalyst Class A

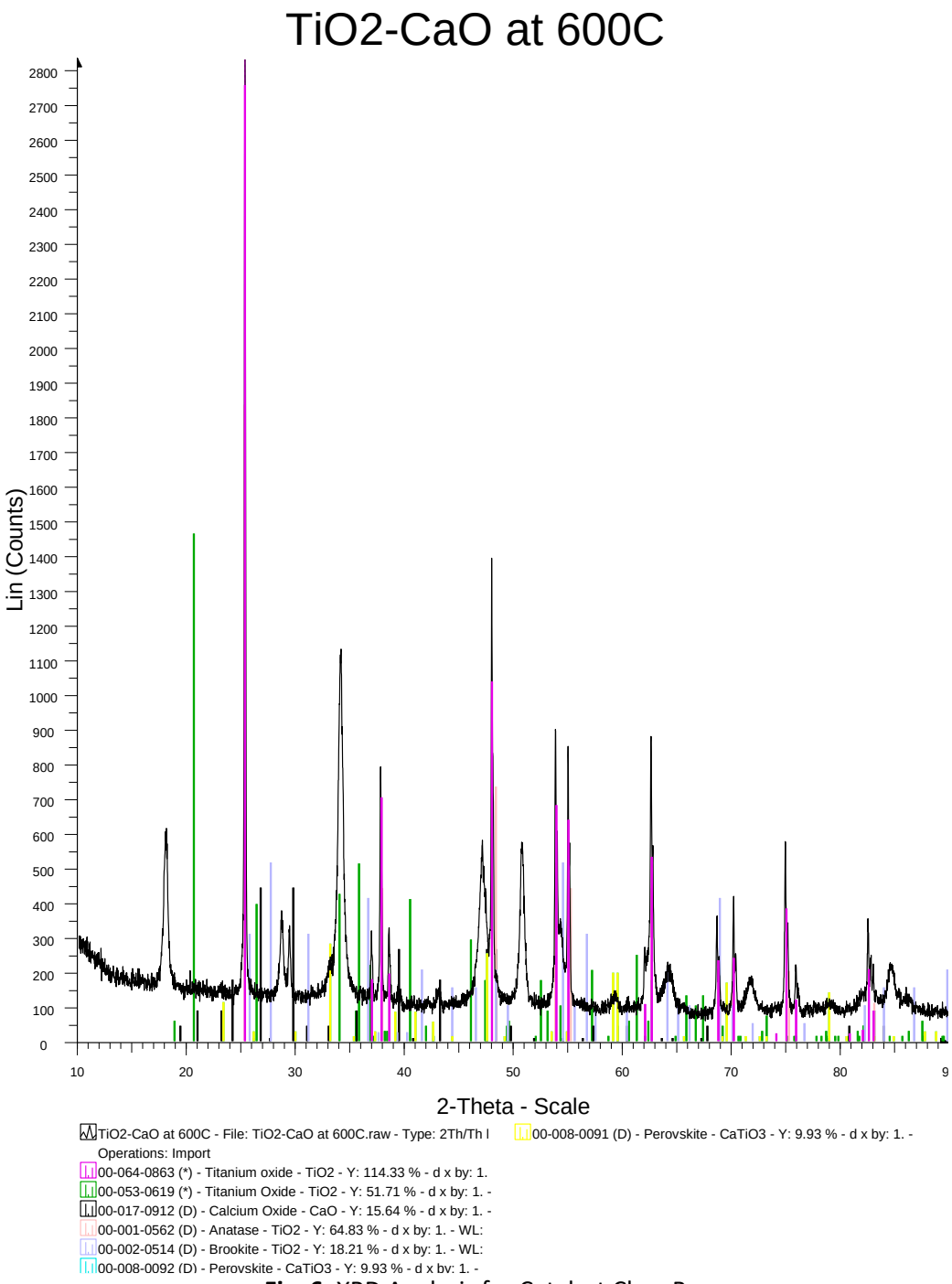

Fig. 6. XRD Analysis for Catalyst Class B

Table 2
Surface Catalyst Calcination Temperature

Catalyst Calcination Temperature	BET Area	
	Our Study	Other Study
200 ºC	9.4	14.37
400 ºC	n/a	10.56
600 ºC	26	22.96
800 ºC	n/a	15.43

3.1.2 SEM analysis

Scanning Electron Microscopy (SEM) is important to study surfaces and particles as well as targeting failure analysis of materials in catalysts. Both catalysts have nanoparticles, the pure nanoparticles were observed clearly in catalyst class B (600 ºC). Although carbon appeared in catalyst class B only, high temperature may cause the carbonization of the mixture. Moreover, CaO had more concentration in both catalysts because calcium oxide is able to dissolve, which has an effect on the component weight in catalyst crystal. To confirm catalyst formation of nanoparticles, EDX/XEM analysis was applied for each catalyst. However, both titanium dioxide and calcium oxide appear in EDX/XEM domain. In catalyst class A domain, the quantity of 72.29 of O, 22.40 of Ca and 5.31 of Ti, while in catalyst class B domain it was 69.65 of O, 15.46 of Ca, and 7.45 of Ti. Evaluating SEM analysis for catalyst (200 °C and 600 °C) showed that compounds are well mixed, and the preference goes to the catalyst activation at 600 °C. The reason is that TiO_2 required high temperature for perfect performance. Catalyst activation varying under temperature conditions will be described in temperature effects. SEM analyses are compared with those of the study shown in Figure 7 and Figure 8, which are similar with this study, and XEM analysis shown in Figure 9 and Figure 10 [17].

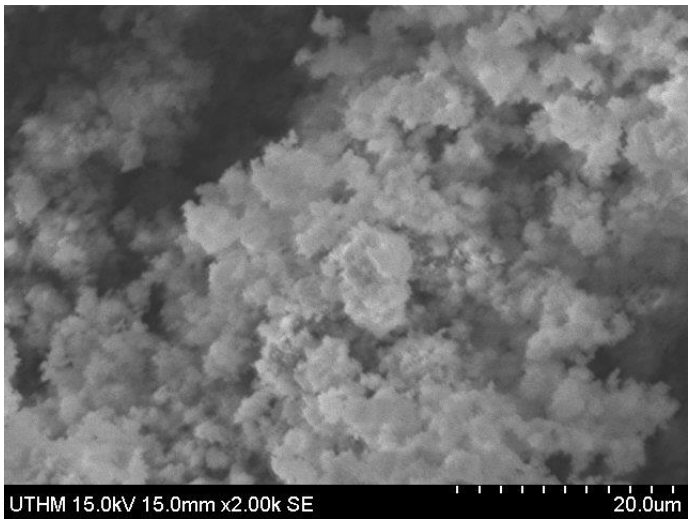

Fig. 7. SEM Analysis for Catalyst at 200 °C

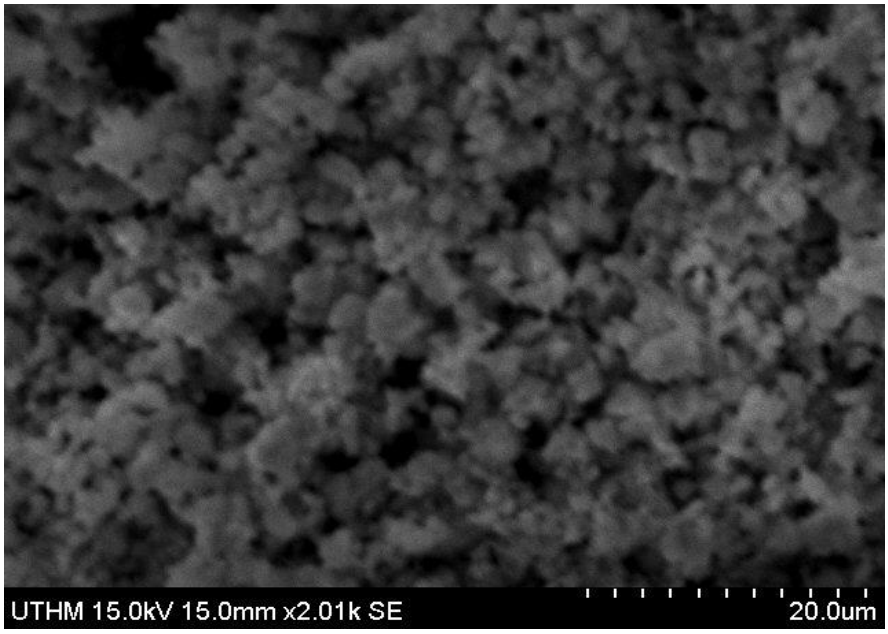

Fig. 8. SEM Analysis for Catalyst at 600 °C

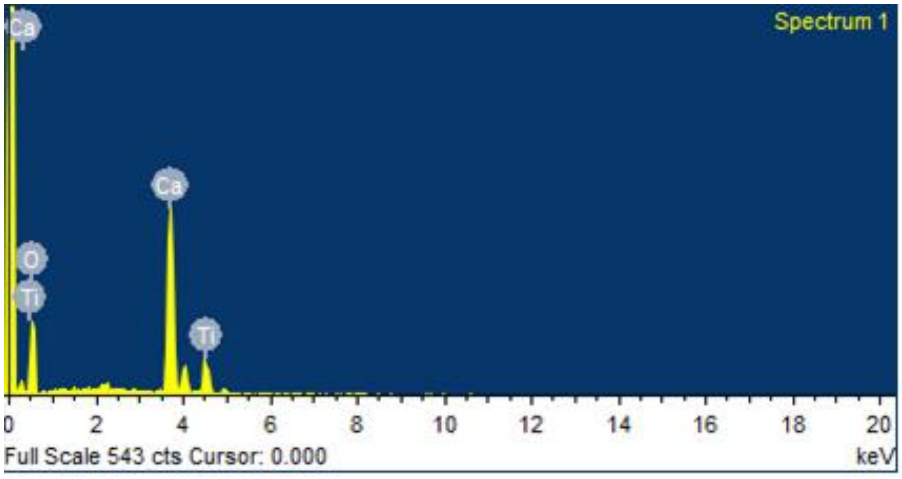

Fig. 9. XEM Analysis for Catalysts at 200 °C

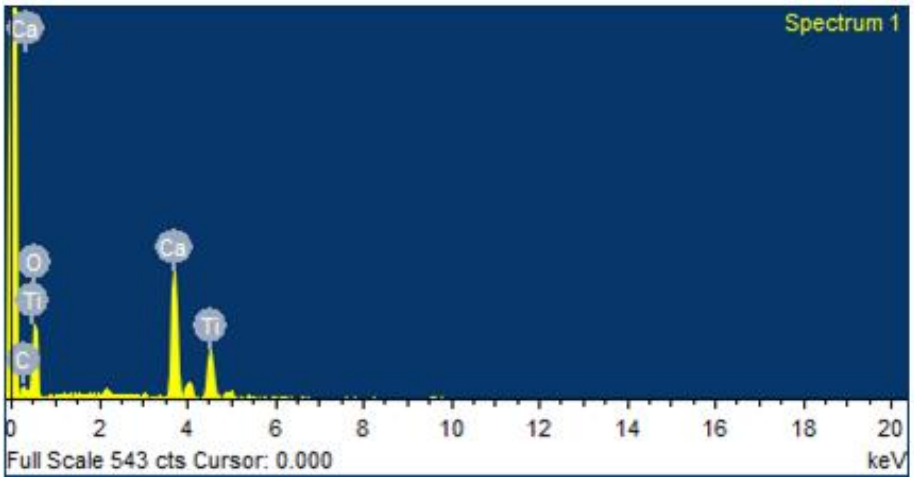

Fig. 10. XEM Analysis for Catalysts at 600 °C

3.1.3 Temperature and activation time effects

According to experimental work results based on yield, viscosity, XRD analysis, and SEM analysis, it was observed that the effect of temperature is very important in catalyst activation. This is because the two compounds (TiO_2 and CaO) differ in terms of the crystal structure, and the temperature increases catalysts' surface, which is perfectly mixed between TiO_2 and CaO. The results showed that catalyst under activation temperature of 600 °C gave better results than activation temperature of 200 °C. The reasons are that titanium dioxide and calcium oxide have different physical/chemical properties which requires a high temperature to mix them. Also, titanium dioxide is insoluble with water while calcium oxide can react with water to form calcium hydroxide. According to XED and SEM analysis, the results showed the advantage of catalyst activating temperature of 600 °C over 200 °C. The reason is that atoms and electrons interact more effectively when the activation temperature increases.

Previous studies did not mention the effect of increasing catalyst activation time. In this study, an increase of 60 minutes was made over previous studies to find out the effects of increasing catalyst activation time. According to experimental work results based on XRD and SEM analysis, time effects on peaks occurs on the catalyst structure and also to make sure that each component was mixed and activated because of different properties of titanium dioxide and calcium oxide. However, adding 60 minutes to activate the catalyst contributes to raising biodiesel yield from 90% in previous study to 96% [17].

3.2 Transesterification Process

To analyse the transesterification reactions, three models will describe the results (first model: 4 hours, second model: 5 hours, and third model: 6 hours). The optimum yields (shown in Figure 11) obtained were 88.70%, 96.9% and 90.1% under conditions of 1.5% wt. from catalyst, class B catalyst, and mixing time of 90 minutes for the first model, the second and the third, respectively

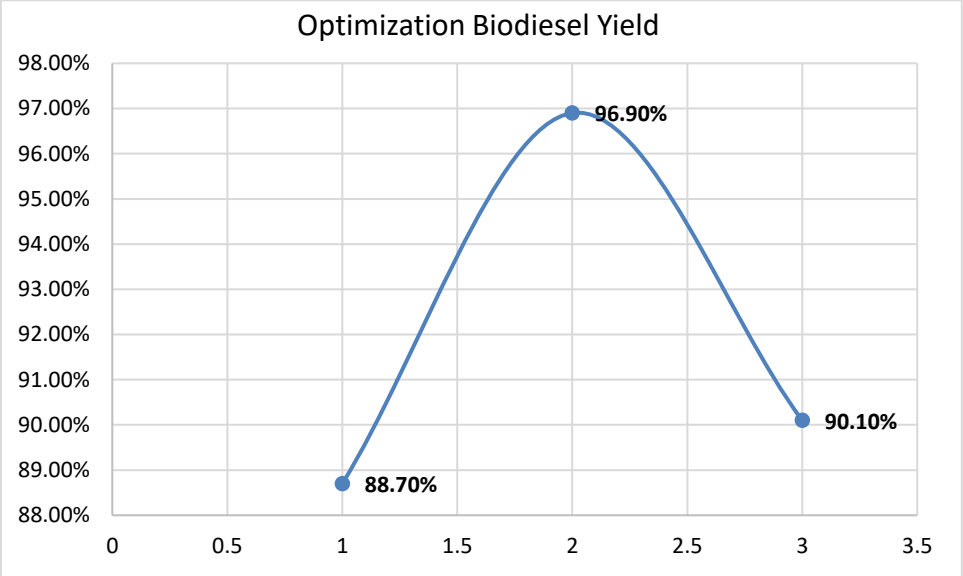

Fig. 11. Optimization Biodiesel Yield for Each Model

3.3 Biodiesel Properties

Biodiesel properties focused on viscosity, flash point and water content. It is noticed that viscosity varies from 1.9 mm²/s to 2.9 mm²/s (at catalyst class A conditions) and from 2.1 mm²/s to 3.2 mm²/s (at catalyst class B conditions). Flash points varied from 94 °C to 96 °C (at catalyst class A conditions) and from 94°C to 99 °C (at catalyst class B conditions). Water content also varied from 0.0046% vol to 0.0049% (at catalyst class A conditions) and from 0.0039% to 0.0048 % vol (at catalyst class B conditions). Biodiesel properties compare with ASTM D6751-12 standards to validate research results as shown in Table 3.

Table 3
Compassion Between ASTM D6751-12 and Research Biodiesel Specifications

Biodiesel Specifications	ASTM D6751-12	Our Research
Kinematic Viscosity	1.9-6.0 mm2/s	1.9 - 3.2 mm2/s
Flash Point	93°C	94°C - 99°C
Water Content	0.050% vol	0.0039% - 0.0049%

4. Conclusions

This study focused on producing biodiesel from canola oil and used TiO_2-CaO as a catalyst. The catalyst was prepared by mixing titanium dioxide (TiO_2) and calcium oxide (CaO) under standard parameters. The results showed that catalyst activity increased as the mixture was more perfectly mixed and as the activation temperature was raised. Catalyst's activity positively impacts biodiesel yield and viscosity.

According to research parameters, three-times transesterification reactions are designed. The results showed that the optimized reaction time is five hours as four hours of transesterification reaction negatively affected biodiesel yield and catalyst reuse time. Besides that, six hours transesterification rection negatively affected biodiesel yield even less than four hours rection. Biodiesel specifications were analyzed in this study according to the ASTM D6751-12 specification. The results showed that biodiesel properties like ASTM D6751-12 specification by 98%.

For future studies, we suggest using multiple feedstocks (Canola Oil, Sunflower Oil, and Palm Oil) with TiO_2-CaO catalyst as well as studying ethanol impact on biodiesel when using canola oil with TiO_2-CaO catalyst. It is also suggested to study catalyst poisoning for TiO_2-CaO catalyst with different oil feedstocks (canola oil, palm oil, waste cooking oil, and sunflower oil).

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