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# Synthesis and Characterization of CaO-TiO$_2$ for Transesterification of Vegetable Palm Oil

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*P A P E R   I N F O*

*A B S T R A C T*

This study explores the potential of titanium oxide impregnated on calcium oxide (CaO-TiO$_2$) as catalyst in transesterification of vegetable palm oil (VPO) to produce biodiesel. The biodiesel yield increased with catalyst calcination temperature and reaction time, and the usage of CaO-TiO$_2$ led to higher yield of biodiesel production when compared to reaction catalyzed by CaO. Biodiesel yield of 93.33% was recorded when CaO-TiO$_2$ was used at optimized reaction conditions. Catalyst characterizations showed that addition of TiO$_2$ to CaO improved the catalytic property by increasing the surface area and strength of basic sites, hence increased the catalytic performance of CaO-TiO$_2$. This study demonstrates the potential of CaO-TiO$_2$ to convert VPO into biodiesel, and the potential of the catalyst in the conversion of waste cooking oil into renewable fuel.

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## NOMENCLATURE

| | | | | |
|---|---|---|---|---|
| °C | degree celcius | θ | diffration angle (in X-Ray Diffraction) | |
| λ | wavelength | wt% | weight percent | |
| ° | degree | | | |

## 1. INTRODUCTION

Increasing awareness of the detrimental effects of fossil fuels utilization on the environment has stimulated research on various sources of renewable energy [1]. Biodiesel oil become the most preferred solution compared to other alternatives as it is highly biodegradable, less toxic, contribute less to sulfites and carbon dioxide (CO$_2$) emissions, while having characteristics similar to diesel fuels [2-5].

Currently, Malaysia is focusing on production of biodiesel for various purposes. More than 95% of biodiesel is produced from edible oils since their properties are suitable to be used in diesel engine [6]. Palm oil is used as a significant biodiesel source in Malaysia because of the availability and accessibility of palm oil supply. Transesterification, also called as alcoholysis, is a reaction between animal fats or vegetable oils with an alcohol to form biodiesel (esters) and glycerol. Methanol is commonly used because of its low cost. Besides that, methanol has a lower molecular weight, lower viscosity and flash point compared to other alcohols. Transesterification process can be performed with homogeneous basic or acid catalysts, heterogeneous solid-basic or solid-acid catalysts, enzymatic catalysts, supercritical and subcritical alcohol, and microwave or ultrasound assisted transesterification. The use of heterogeneous solid-basic catalysts becomes an attractive choice as they produce high yield within short reaction time. Yaakob et al. [7] stated that heterogeneous solid-basic catalysts have higher catalytic activity and stability; also, it can be easily separated from the product, thus the material cost associated with the catalyst is reduced.

The popular solid base catalysts developed are alkaline earth metal oxides, zeolites and hydrotalcites.

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Calcium oxide (CaO) becomes the most attractive base catalyst in alkaline earth metal oxides group as it has high basic strength, causes less harm to the environment and can be synthesized from cheap sources such as limestone, eggshell, waste shell and calcium hydroxide [8, 9]. As the transesterification catalyzed by CaO suffers low reaction rate, many researchers have attempted to increase the reaction rate of CaO. However, it is noted that CaO causes irritation to eyes, skin, and upper respiratory tract due to its alkalinity. Inhalation of calcium oxide dust also leads to inflammation of the respiratory passages, ulceration and perforation of nasal septum, and pneumonia. Besides, abdominal cramps, vomiting and diarrhea might also take effect. Thus, care must be taken to minimize the inhalation or ingestion of CaO [10].

Among the transition metal oxides, titanium dioxide (TiO$_2$) has attracted researchers' attention for biodiesel production. Yoo et al. [11] found that 79% of biodiesel yield can be achieved using TiO$_2$ catalyst in rapeseed oil transesterification within temperature range of 200-270 °C. Besides that, Feyzi and Shahbazi [12] studied the use of Cs-Ca/SiO$_2$-TiO$_2$ nanocatalyst in biodiesel transesterification from refined vegetable oil. The best operational conditions were found to be 12:1 (ratio of methanol to oil), 60 °C (reaction temperature), 500 rpm (mechanical stirring) for 2 hours, which produced 98% biodiesel yield. It is proposed that the impregnation of TiO$_2$ onto CaO leads to the substitution of Ti ions on the Ca lattice, which results in defect creation and stable catalytic activity [13]. Therefore, TiO$_2$ is suitable to use in CaO modification for stable catalytic activity.

This research work aims to study the effects of catalyst calcination temperature and reaction time on biodiesel production via transesterification of vegetable palm oil over a catalyst consisting calcium oxide and titanium oxide (CaO-TiO$_2$). Detailed characterizations were also carried out to explore the properties of the catalyst.

## 2. MATERIALS AND METHODS

CaO and TiO$_2$ were purchased from Sigma-Aldrich while methanol used was analytical grade with purity of 99.8% purchased from Fluka. Vegetable plam oil (VPO) was purchased from a local departmental store in Malaysia.

The catalyst used in this study was prepared using wet impregnation method [14]. CaO and TiO$_2$ were mixed at weight ratio of 1:1 in solution state. After that, water was removed from the mixture via heating for 120 minutes at 100 °C, then activated at 600 °C for 300 minutes in a furnace. The catalyst was then cooled and stored in desiccator.

Catalytic effect of CaO-TiO$_2$ was tested in the biodiesel production via transesterification method as

shown in Figure 1 [14]. Predetermined amount of VPO was preheated to 65 °C before specific amount of methanol and catalyst were added into the reaction flask. The mixture was stirred with ±500 rpm throughout the reaction, and the reaction temperature was monitored using thermometers. Upon completion of the reaction, the solid catalyst was filtered off by a funnel with a filter paper, and the filtrate mixture was left to stand overnight in a separating funnel to form two layers. The top layer (biodiesel) was collected and washed with hot distilled water. The biodiesel mass was then weighed, and biodiesel yield was calculated using Equation 1. The experiment was repeated with different calcination temperature (200 - 800°C) and reaction times (30 - 150 min) [15] to observe the effects on the production of biodiesel. Transesterification process was also carried out over CaO to compare the efficieny of the CaO and CaO-TiO$_2$ catalysts. All the experiments were carried out in triplicate for each sample and the average data was tabulated in the result.

$$\text{Yield (\%)} = \frac{\text{Weight of biodiesel produced (mL)}}{\text{Weight of oil used (mL)}} \times 100\% \qquad (1)$$

Surface areas of CaO and CaO-TiO$_2$ calcined at different temperatures were determined using Micromeritics Gemini 2360 Surface Area Analyzer according to the method described by Wong et al. [16] in analysis of metal-impregnated zeolites. Prior to the analysis, the samples were outgassed at 90 °C for 2 hours (for removal of moisture in the samples), followed by heating at 360°C for 90 minutes (for removal of adsorbed or occluded gases in the samples). Finally, the instrument was controlled by microprocessor with dedicated data processing software, which allows user to determine the best fitting method.

It was shown that catalyst calcined at 600 °C has the highest catalytic property in transesterification (Section 3.2); therefore, compreheensive characterization was performed on the catalyst. Thermal stability of the catalyst was determined using TGA instrument (Perkin Elmer TGA 4000).
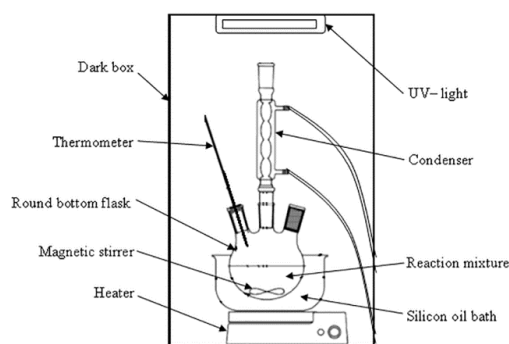


**Figure 1.** Reactor setup for biodiesel production via transesterification. Reprinted from the work [14] with permission from Elsevier

About 10 mg of sample was heated from 30 °C to 900 °C with the ramping rate of 10 °C $min^{-1}$ under nitrogen atmosphere [17]. Basicity of the catalyst was determined using Micromeritics AutoChem II 2920 Chemisorption Analyzer via temperature-programmed desorption of carbon dioxide (TPD-$CO_2$) [18]. About 0.2 g of catalyst was pretreated under helium (He) stream (30 $cm^3$ $min^{-1}$) at 600°C for 2 hours. Subsequently the catalyst was saturated with $CO_2$ (30 $cm^3$ $min^{-1}$) for 1 hour at 50°C. After flushing with helium flow for 30 min, the catalyst was heated from 50 °C to 900 °C with a ramp of 10 °C $min^{-1}$. The desorbed $CO_2$ was quantified by a thermal conductivity detector [18]. The crystallography of the catalysts was evaluated in the Bruker D8 Advance diffractometer with Cu Kα radiation (40 kV, 40 mA X-ray tube) with wavelength (λ) of 0.154 nm. The data was recorded over a 2θ range of 10°- 80° at a counting time of 10s [16]. Morphology of the catalysts was observed using JSM-6390LV Scanning Electron Microscope [19]. The functional groups of the samples were identified via Fourier transform infrared (FTIR) using IRTracer-100 spectrophotometer (Shimadzu Cooperation, Japan) coupled with attenuated total reflectance (ATR) technique [20]. The infrared spectra were recorded in the range of 400–4000 $cm^{-1}$, with 300 scans and 4 $cm^{-1}$ resolution.

## 3. RESULTS AND DISCUSSIONS

### 3. 1. Catalytic Property of CaO and CaO-TiO₂ in Transesterification

**3. 1. 1. Effect of Calcination Temperature**    The effect of catalyst calcination temperature on biodiesel yield is shown in Figure 2. It is observed that biodiesel yield increased with calcination temperature. The highest biodiesel yield was obtained at 600°C (76.67% for CaO and 88.33% for CaO-$TiO_2$). This result is in accordance with the findings from Takase et al. [21], which stated that KHC₄H₄O₆/$TiO_2$ catalysts produced the highest biodiesel yield (84.5%) when the catalyst was calcined at 600 °C. Such similarity arises due to the presence of optimum strength of the active basic sites on the catalyst surface to form the highly reactive methoxide species for the transesterification reaction [22], resulting in the highest biodiesel yield for all the catalysts in this study.

From the result of TPD-$CO_2$ (Section 3.2.3), CaO calcined at 600 °C shows stronger basic sites than CaO-$TiO_2$. However, the biodiesel yield from CaO calcined at 600 °C was slightly lower than CaO-$TiO_2$. Therefore, it is concluded  that the strongest base is not necessarily the best catalyst for biodiesel reaction, and a solid catalyst which possesses strong basic sites may hold up

the product molecules from desorbing after the reaction, which reduced the catalytic activity of the reaction and hence reducing the biodiesel yield as shown in Figure 2. On the other hand, a solid catalyst with weak basic sites may not adsorb the reactant molecules to initiate the reaction. Therefore, the optimal strength of the active basic sites is required for the transesterification reaction. This suggests that among the different catalysts, CaO-$TiO_2$ transesterification may have the most optimal active site strength for the simultaneous transesterification reactions after being calcined at 600 °C, making it an effective catalyst for biodiesel production from palm oil.

Calcination temperatures below 600 °C resulted in lower biodiesel yield, possibly due to the excess porous nature of the catalysts. Catalyst calcined at temperature beyond 600 °C might vitrify the surface of CaO catalyst, where the particles become larger and smoother, indicated by decrease of catalyst surface area at 800 °C , as shown in Table 1. This phenomenon decreases the catalyst activity. In contrast, catalysts calcined at 600 °C possess large surface area, which contributes to a higher catalytic activity during transesterification. Moreover, thermogravimetric analysis (TGA) result also indicates that the optimum calcination temperature for CaO and CaO-$TiO_2$ catalysts is 600 °C. As portrayed in Section 3.2.1, the recommended calcination temperature of the catalysts was between 550 and 730 °C.
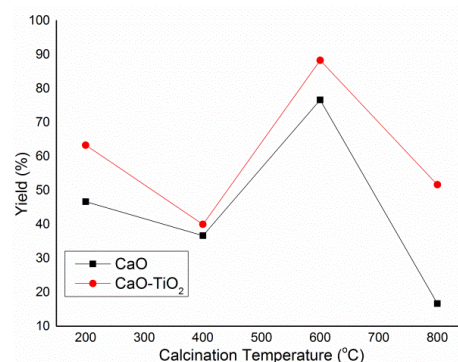


**Figure 2.** Effect of catalyst calcination temperature on percentage yield of biodiesel. (0.5 wt% catalyst dosage, 3:5 for ratio of VPO to methanol, 60 min, 65 °C, stirring speed ±500 rpm)

**TABLE 1.** Surface of calcined catalyst temperature

| Calcined temperature (°C) | BET area (m²/g) | |
| --- | --- | --- |
| | **CaO** | **CaO-TiO₂** |
| 200 | 1.87 | 14.37 |
| 400 | 1.60 | 10.56 |
| 600 | 7.00 | 22.96 |
| 800 | 6.02 | 15.43 |

Hence, the optimum calcination temperature for CaO and CaO-TiO$_2$ at calcination temperature of 600 °C was selected for further studies on effects of reaction parameters. This temperature is lower than the optimum calcination temperature in the previous study [23], as the water contained in the catalyst precursor was removed prior to the activation.

**3. 1. 2. Effect of Reaction Time**  As shown in Figure 3, biodiesel yield increases gradually with reaction time during transesterification using CaO and CaO-TiO$_2$ as catalysts. The highest yield was observed at 150 minutes, which were 90% (CaO) and 93.33% (CaO- TiO$_2$), respectively. At the initial time (30 – 60 min) the reaction was slow due to inadequate stirring time to enhance proper mixing and dispersion of catalyst and methanol in the vegetable oil [24]. The interaction between the phases increased starting from 90 until 150 min due to adequate stirring time, which also enhanced the reaction rate. Moreover, the transesterification reaction can remain for a longer time mainly due to the evaporation of methanol in the reaction mixture because it was carried out at maximum temperature (65 °C). This phenomenon maintained the temperature without increasing dramatically, by some cooling on the surface of the reaction mixture [24]. It was noted that the biodiesel yield increased further until 150 min. Besides that, the TiO$_2$ electrons migrate to the catalyst surfaces which induced the reduction of CaO catalyst, resulting in increase of catalytic activity [17]. Hence, transesterification of CaO-TiO$_2$ catalyst exhibited higher biodiesel yield. It was expected the excess reaction rate after 150 min would result in slight reduction in the biodiesel yields due to reversible reaction. Thus, 150 min was deemed  as the optimum reaction temperature for CaO and CaO-TiO$_2$ catalysts in this study. Based on the findings in Section 3.2.4, addition of TiO$_2$ to the surface of CaO improved the catalyst ability and increased the biodiesel yield at longer reaction time.

The X-Ray diffractogram shows the presence of anatase TiO$_2$ in the crystallite structure of the catalyst, which confirms the substitution behavior during the reaction. The Fourier transform infrared (FTIR) spectrum (Section 3.2.6) also shows the formation of TiO$_2$ band, which is in agreement with the XRD data. These findings show the basic site of the catalyst can perform well and provides a higher biodiesel yield at longer reaction time [25]. From these observations, the XRD and FTIR results support the stability and activity of CaO-TiO$_2$ catalyst during transesterification.

## 3. 2. Catalyst Characterizations

**3. 2. 1. Thermogravimetric Analysis (TGA)** Figure 4 illustrates the TGA as well as DTG curves of CaO and CaO-TiO$_2$. Four mass losses are observed at temperature ranges of 40-270 °C, 300-450 °C, 500-730 °C, and 750-950 °C. The first mass loss was almost constant due to the loss of absorbed water at the precursor surface by physisorption and chemisorption. Both catalysts exhibit single thermal decompositions at the second mass loss, 300-450 °C which are attributed to the loss of water of crystallization in CaO and Ca(OH)$_2$. The mass loss of constitutional water in the form of OH- at 500-730 °C resulted in the Ca(OH)$_2$ decomposition to CaO and CO$_2$ along with some losses of inorganic impurities. Finally, catalyst sintering took place at 750-950 °C and the mass loss remains constant afterwards.

The description of the TGA curves in this finding  is consistent with other findings [24, 26]. Hu, Wang and Han [15, 26] reported the crystal water loss at 350-451 °C and sintering of the preformed compound at 688-749 °C. The presence of Ca(OH)$_2$ and the formation of CaO in the catalyst at 600 °C are also confirmed by the XRD pattern obtained (Section 3.2.4). The results of thermal analysis suggested that the proper calcination temperature of the CaO-TiO$_2$ catalyst is in the range of 550-730 °C. A higher activation temperature is required to enhance the catalytic activity. This  was confirmed by the catalyst test study in the transesterification process.
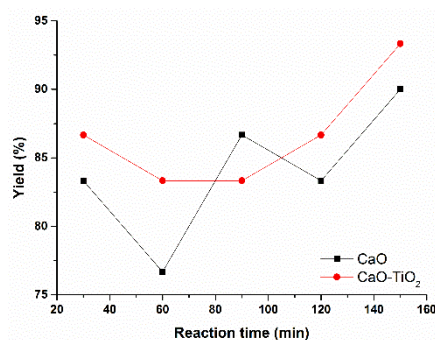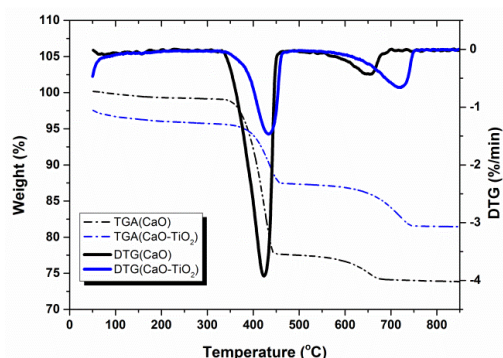


**Figure 3.** Effect of reaction time on percentage yield of biodiesel. (catalysts calcined at 600 °C, 0.5 wt % catalyst dosage, 3:5 for ratio of VPO to methanol, 65 °C, stirring speed at ±500 rpm)



**Figure 4.** TGA and DTG curves for CaO and CaO-TiO$_2$ catalysts

**3. 2. 2. Surface Area**    Table 1 illustrates the effect of calcination temperature on the surface area of CaO and CaO-TiO$_2$ catalyst. The surface area of CaO increased from 1.87 to 7.01 m$^2$/g when the calcination temperature increased from 200 to 600 °C, respectively. The same pattern was observed for CaO-TiO$_2$ catalyst. Increasing the calcination temperature from 200°C to 600 °C resulted in the increment of surface area from 14.37 to 22.96 m$^2$/g, respectively. However, the surface area of the catalysts decreased with calcination temperature due to the presence of Ca(OH)$_2$ which might reduce the biodiesel yield [24]. The calcination temperature beyond 600 °C can be attributed to the porosity development in the catalyst which was caused by the evolution of carbonization products (CO$_2$ in this case). This explanation is supported by the XRD chromatogram in Section 3.2.4. Moreover, the addition of TiO$_2$ increased the surface area of the catalyst. The strong interaction of TiO$_2$ with CaO gave an increment in the surface area of the catalysts, which reduced the surface diffusion of CaO. The interaction also gave a sintering and stabilized crystal structure of CaO-TiO$_2$ [27]. Boro, et al. [28] stated the further increase in surface area of the catalysts might have been caused by the crystal growth of CaO at higher calcination temperature. Based on the results obtained, therefore, only CaO and CaO-TiO$_2$ catalysts which are calcined at the temperature of 600 °C were subjected to the rest of the characterizations.

**3. 2. 3. Temperature-programmed Desorption of Carbon Dioxide (TPD-CO$_2$)**    Figure 5 indicates the presence of CO$_2$ desorption peaks at temperature ranges of 50-210 °C, 220-420 °C, and 430-850 °C. The peaks in the range of 50-420 °C are attributed to the interaction of CO$_2$ with weak and medium basic sites, and peaks at 430-850 °C indicate the presence of stronger basic site in the catalysts. It was found that the higher amount of the strong basic sites in catalyst led to increased biodiesel yield. Similar finding was reported in several studies [19]. The desorption peaks of both catalysts were observed between  50 - 210 °C and
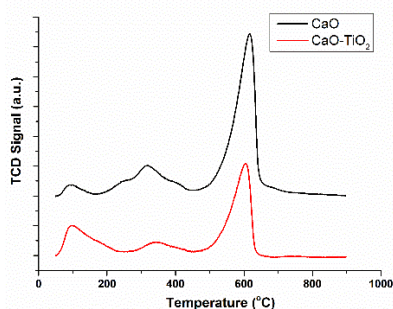


**Figure 5.** TPD-CO$_2$ profiles of (a) calcined CaO at 600 °C (b) calcined CaO-TiO$_2$ at 600 °C

220-420 °C, which are related to the basic sites with weak and medium strength concerned with Ca$^{2+}$-O$^{2-}$ and Ti$^{4+}$-O$^{2-}$ anions. In comparison, the strength of basic sites of CaO was higher than that of CaO-TiO$_2$, as evidenced by higher temperature as well as larger area for CO$_2$ desorption peaks associated with CaO. When Ti ions are substituted into the calcium lattice, some vacancies are created to maintain neutral charge in the ionic crystal. The Ti ion has a valence of 4, which is higher than that of Ca ion (2). These vacancies cause defects or weaknesses which are important for the heterogeneous catalyst [20]. Thus, the basicity of CaO-TiO$_2$ is less than that of CaO. The CO$_2$ desorption peaks was gradually shifted towards higher temperatures ranging from 450 to 650 °C, demonstrating the increase of catalyst basic strength with the present CaO crystallinity [21]. Based on the discussion of the TPD results, the mixed metal oxide system still provides a stronger basic strength on the catalytic site of CaO-TiO$_2$ catalyst even though the CO$_2$ desorption peak is less than the pure metal oxide because of higher basic sites of the catalysts (450-650 °C) which enabled high yield biodiesel to occur. Thus, both catalysts provided strong basic functions on the surface.

**3. 2. 4. X-ray Diffraction (XRD)**    Figure 6 depicts the XRD patterns of CaO and CaO-TiO$_2$ catalysts calcined at 600 °C. In such analysis, each inflection point is related to a pore in mesoscale, while the sharpness of the curves is related to the uniformity of the mesopore size distribution of the catalyst . The peaks obtained were compared with the Joint Committee on Powder Diffraction Standards (JCPDS) file. From the figure, the principle components of CaO catalyst are CaO, CaCO$_3$ (calcite and aragonite), and Ca(OH)$_2$, while CaO, TiO$_2$, CaCO$_3$, and Ca(OH)$_2$ occur in CaO-TiO$_2$

For parent CaO, intensified and narrow peaks at 2θ = 32.2°, 37.3°, 53.8°, 60.7°, 62.5°, 64.1°, 67.3° were observed. In contrast, these peaks were observed only at low intensity in CaO-TiO$_2$ catalyst. In addition, for both catalysts, peaks were observed at 2θ = 17.9°, 28.6°, 34.1°, 46.9°, and 50.7°. Such peaks are the characteristics peaks for Ca(OH)$_2$ when hydration of CaO occurs. CaCO$_3$ peaks were observed for both catalysts at 2θ = 29.2°, 36.37°, 38.9°, 39.48°, 43.55°, 47.87°, 49.05°, 57.89°. These CaCO$_3$ peaks confirm its reflection to the exposure of fresh CaO and CaO-TiO$_2$ catalysts in the atmosphere during calcination. TiO$_2$ peaks was observed in the CaO-TiO$_2$ catalyst at 2θ = 25.5°, 37.3°, 47.0°, 54.8°, 55.2°, 63.9°, 69.20°, 70.4°, and 75°.

In comparison, the areas and intensities for diffraction peaks of impregnated CaO-TiO$_2$ particles are slightly lower than those of parent CaO.
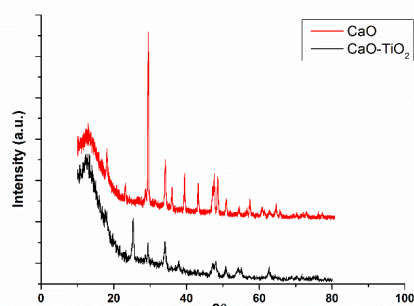
**Figure 6.** XRD pattern of calcined CaO and CaO-TiO$_2$ calcined at 600 °C

The TiO$_2$ impregnation onto CaO catalyst caused small decrease in CaO-TiO$_2$ crystallinity, due to structural collapse during calcination at high temperature and the presence of transition metal species support which tends to decrease the crystallinity of the parent CaO. Such observation is in agreement with the catalyst characteristics in study by Wong et al. [16]. The larger crystallite sizes led to lower surface area of the catalyst [14]. The intensity observed in the XRD pattern of both CaO and CaO-TiO$_2$ point to the large dimensions of the catalyst crystallite and confirm the formation of the CaO, CaCO$_3$, Ca(OH)$_2$, and TiO$_2$ moities at calcination temperature at 600 °C.

**3. 2. 5. Scanning Electronic Microscope (SEM) and Energy Dispersive X-Ray Spectroscopy (EDX)** The morphology of CaO and CaO-TiO$_2$ is shown in Figure 7. CaO calcined at 600 °C consists of particles with spherical shape on its surface, while CaO-TiO$_2$ calcined at 600 °C shows tiny aggregates particles on the surface. The presence of small spherical particles (similar to structure in pure CaO) is also observed in CaO-TiO$_2$. Homogeneous distribution of CaO particles in CaO-TiO$_2$ was in agreement with the XRD result, which the CaO content in the catalyst was low. The morphology of CaO-TiO$_2$ is similar to observations made by Taufiq- Yap et al. [29] and Tang et al. [14], where tiny aggregates of particles were observed on the surface of CaO modified with bromoocatane and CaO modified with ZnO, respectively. The presence of aggregates particles increased the catalyst surface area
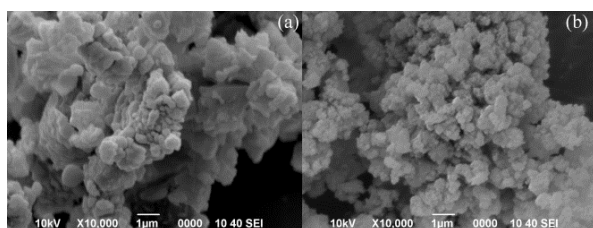
(shown in Table 1), hence the catalytic property in biodiesel production.

**3. 2. 6. Fourier Transform Infrared (FTIR) Spectroscopy** FTIR spectra of CaO and CaO-TiO$_2$ calcined at 600°C (Figure 8) showed similar absorption modes, including those at 866, 871, and 873 cm$^{-1}$, respectively. These peaks represent the out-of-plane bending and in-plane bending from CO$_3^{2-}$ group [24]. Peaks were also observed at ~1421 cm$^{-1}$ for both samples, which is assigned to asymmetric stretching from CO$_3^{2-}$ group. Another peak observed at 3639 cm$^{-1}$ is assigned to OH- stretching [30]. However, two additional peaks at ~1600 cm$^{-1}$ and ~3350 cm$^{-1}$ are exhibited by the CaO-TiO$_2$ sample, but not CaO. Such peaks are probably originated from the CaO lattice structure [28].

From Figure 7, it is suggested that the modified CaO with TiO$_2$ produce strong basic property at ~1600 cm$^{-1}$ and ~3350 cm$^{-1}$ which is caused by the absorption of gaseous CO$_2$ from atmosphere onto the catalysts, where the intensity still increased after modification [20]. This explanation is supported by TPD-CO$_2$ curves profile, as shown in the Figure 4. Therefore, the IR spectra result has proved that the CaO catalyst was successfully modified with TiO$_2$ and resulted in overall improvement of the product yield.

Similarly, the variation in elemental compositions was vividly noticeable from the EDX graphs.  The EDX analysis of the surface elemental composition revealed the presence of calcium (89.71 wt.%), oxygen (2.45 wt. %), carbon (7.50 wt. %) and water (0.34 wt. %) for CaO. Besides that, the surficial elemental composition by EDX analysis also revealed the presence of 85.18 wt. % calcium, 3.79 wt. % titanium, 5.02 wt. % oxygen, 5.95 wt. % carbon and 0.06 wt. % water in CaO-TiO$_2$.

# 4. CONCLUSION

This study focused on transesterification of vegetable palm oil (VPO) catalyzed by CaO impregnated with TiO$_2$.



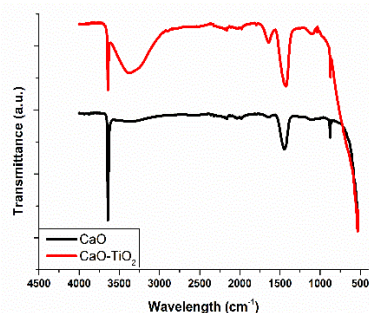**Figure 7.** Scanning electron micrographs of (a) CaO and b) CaO-TiO$_2$ catalysts



**Figure 8.** FTIR spectra of (a) CaO calcined at 600 °C and (b) CaO-TiO$_2$ calcined at 600 °C catalysts

The biodiesel yield increased with catalyst calcination temperature and reaction time. The optimum reaction conditions are 600 °C and 150 minutes for reaction time, and the biodiesel yields are 90% (CaO) and 93.33% (CaO-TiO$_2$), respectively. Impregnation of TiO$_2$ on CaO increased the biodiesel yield, due to the photocatalytic action of TiO$_2$. Comprehensive characterization of catalyst showed that the addition of TiO$_2$ to CaO resulted in the substitution of Ti and Ca ions in the calcium lattice, which improved the catalyst activity. Homogeneous distribution of TiO$_2$ particles on CaO surface also increased the surface area of CaO-TiO$_2$. TPD-CO$_2$ analysis result also showed that the synthesized CaO-TiO$_2$ had strong strength basic sites, although slightly lower than CaO catalyst. Moreover, calcination at 600 °C was in agreement with the TGA profiles. The characterization results showed that impregnation of TiO$_2$ on CaO improved the catalyst stability while maintaining catalytic activity. Overall, CaO-TiO$_2$ exhibits excellent catalytic property in transesterification of VPO, and its potential in transesterification of waste cooking oil should be investigated.

## 5. ACKNOWLEDGEMENT

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# Synthesis and Characterization of CaO-TiO$_2$ for Transesterification of Vegetable Palm Oil

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چکیده

این تحقیق، پتانسیل اکسید تیتانیوم آغشته به اکسید کلسیم (CaO-TiO$_2$) که به عنوان کاتالیزور در انتقال الیاف روغن های نباتی روغن نخل سبزیجات(VPO) برای تولید بایودیزل استفاده می شود را مورد بررسی قرار می دهد. عملکرد بایودیزل با دمای کالیبراسیون کاتالیزور و زمان واکنش افزایش یافته و استفاده از CaO-TiO$_2$ باعث افزایش تولید بایودیزل در مقایسه با واکنش کاتالیز شده توسط CaO می شود. هنگامی که CaO-TiO$_2$ در شرایط واکنش بهینه سازی شده استفاده شد, بایودیزل بازدهی به مقدار 93.33٪ را به دست آورد. تعاریف کاتالیستی نشان داد که افزودن TiO$_2$ به CaO, به دلایلی مثل افزایش سطح کاتالیزور و همچنین افزایش قدرت سایت های فعال کاتالیزور, خواص کاتالیزوری را افزایش داده که  باعث افزایش عملکرد کاتالیزوری CaO-TiO$_2$ می شود. این مطالعه پتانسیل CaO-TiO$_2$ به منظور تبدیل VPO به بایودیزل و همچنین توانایی کاتالیزور را به منظور تبدیل پسماند روغن مصرفی (نباتی) به سوخت تجدید پذیررا نشان می دهد.

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