

The Effects of CaO-TiO₂ Calcination Temperatures on the Catalyst Characterization and Palm Biodiesel Psychochemical Properties

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Abstract

Biodiesel is the new innovative substitute for petroleum and there are many vegetable oils could be used as the feedstock and one of it is vegetable palm oil (VPO). This research is conducted to analyze CaO-TiO₂ catalysts under calcination process of temperature 200°C, 400°C, 600°C and 800°C then produce and test biodiesel oil. The catalysts then go through analysis using X-Ray diffraction analysis (XRD) analysis, Scanning electron microscopy (SEM) analysis and Fourier Transform Infrared Spectroscopy (FTIR) analysis. Through transesterification biodiesel is extracted with reaction time of 120 minutes and reaction temperature of 65°C and 10%wt of CaO-TiO₂ catalyst of 9:1 molar ratio of methanol and VPO. The biodiesel then will be tested for psychochemical properties that is the density and kinematic viscosity of the biodiesel. This analysis will be based on previous studies and also ASTM D6751 standards for biodiesel properties. The catalysts showed all the particles from different elements distributed accurately throughout the SEM test. Based on the result of density and kinematic viscosity it is proven that the biodiesel created from catalyst CaO-TiO₂ under calcination process of 800°C, shows accurate results accordance to ASTM D6751 standards.

1. Introduction

The petroleum fuels play a very important role in the development of industrial growth, transportation, agricultural sector and to meet many other basic human needs, however, these fuels are limited and depleting day by day as the consumption is increasing very rapidly [1]. By the 21st century an immense and intricate value chain was moving approximately 100 million barrels of oil per day from producers to consumers [2] While technically we could keep drilling for oil indefinitely, locating reserves will become harder and harder the deeper we dig into the ground - and the pricier, more complex and less efficient this enterprise will become [3]. Biodiesel produced from different triglyceride sources is an alternative fuel to Petro-diesel, the American Society for Testing and Materials (ASTM) defines biodiesel as mono-alkyl esters produced from various lipid feedstocks including vegetable oils, animal fats [4]. Furthermore, biodiesel is a nontoxic alternative fuel and biodegradable in freshwater and soil, making it unquestionably good for the environment [5]. The advantages of vegetable oil-based biodiesel over conventional diesel fuel include increased profitability, biodegradability and low aromatics

and sulphur content [6]. Edible vegetable oils such as soybean oil, palm oil and sunflower oil are first-generation biodiesel feedstocks because they were the first type of crop used to produce biodiesel [7]. Biodiesel production by transesterification reaction can be catalyzed with alkali, acidic or enzymatic catalyst [8]. Transesterification process is the most adaptive method for biodiesel production in which biodiesel and glycerol as a secondary product is obtained from the feedstock oil, by decreasing the activation energy, a catalyst speeds up the transesterification reaction [9]. 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 [10]. Among the transition metal oxides, titanium dioxide (TiO₂) has attracted researchers' attention for biodiesel production, 79% of biodiesel yield can be achieved using TiO₂ catalyst in grapeseed oil transesterification within temperature range of 200-270 °C [11]. It is proposed that the impregnation of TiO₂ onto CaO leads to the substitution of Ti ions on the Ca lattice, which results in defect creation and stable catalytic activity proves its suitable for catalytic activity [12].

2. Materials and Methods

The materials used for this research is mainly Calcium Oxide (CaO), Titanium Dioxide (TiO₂), Vegetable Palm Oil and methanol. For the first stage of the methodology is the catalyst preparation where the 1:1 in molar ratio of CaO-TiO₂ will go through process such as ball milling for grinding and blending, sieve to make particles smaller and finer. The next stage is calcination process, where the catalyst is divided into 4 samples where each sample are calcinated in different temperature of 200°C, 400°C, 600°C and 800°C for 5 hours. The catalysts then were analyzed using XRD, FTIR and SEM. Moving on to the next stage which is transesterification to produce biodiesel. In this process, 800°C calcinated catalyst of CaO-TiO₂ are used to produce biodiesel, methanol to palm oil molar ratio of 9:1 are also used in this process while heating under constant 65°C for 120 minutes. Then, the next phase is the separation, filtration and washing process of the biodiesel to remove any excess soap and esters also catalyst. After that, density and kinematic viscosity test are carried out on the biodiesel.

3. Result and Discussion

3.1 X-Ray diffraction analysis (XRD) Analysis

A wider peak indicates the possibility of a smaller crystal, a crystalline structure flaw, or an amorphous solid (one without perfect crystallinity) in the sample. Based on the XRD analysis in Figure 1, it showcases TiO₂ with the greatest anatase phase was calcined at 800 °C since its peak is the highest and thinnest. Follow alongside it, from 600°C to 200°C, the crystallinity along with amorphous structure decreases.

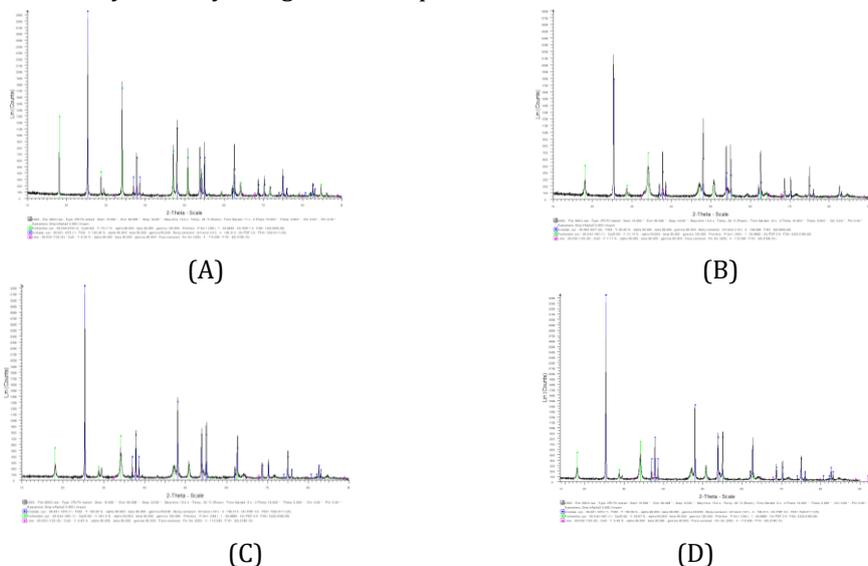


Figure 1 – XRD Analysis on Catalyst Calcinated at (A) 200°C, (B) 400°C, (C) 600°C and (D) 800°C.

3.2 Scanning electron microscopy (SEM) Analysis

In Figure 2. shows the SEM analysis of CaO-TiO₂ catalyst that has been calcinated in different temperatures of 200°C, 400°C, 600°C and 800°C. The morphology of the CaO-TiO₂ catalysts at 5.0 µm in particle size shows

agglomeration which means a lot of different element gathered together in tiny aggregate particles. The small particles that are is spherical shape is the structure of CaO means that CaO available in the catalyst, the homogenous distribution of CaO particles is accurate which in the XRD results shows that CaO is low in the catalyst.

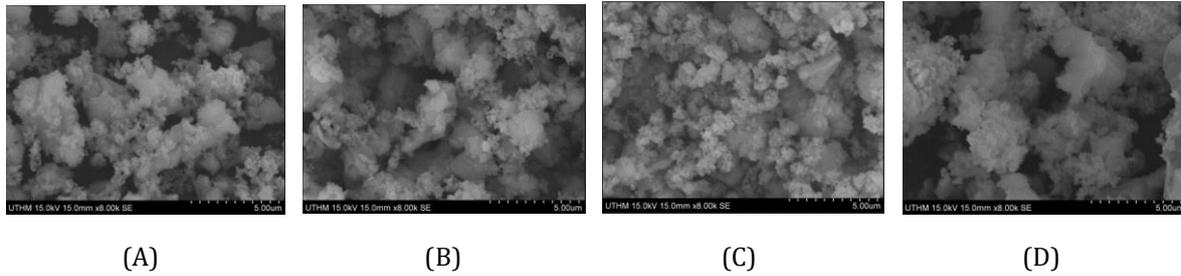


Figure 2 – SEM Analysis on Catalyst Calcinated at (A) 200°C, (B) 400°C, (C) 600°C and (D) 800°C

3.3 Fourier Transform Infrared Spectroscopy (FTIR) Analysis

All four samples for CaO-TiO₂ catalysts showed absorption mode in Figure 3 which are similar at 875.07cm⁻¹-875.16 cm⁻¹, 874.91 cm⁻¹ and 874.76 cm⁻¹. Also, peaks are observed at 1428.75cm⁻¹, 1418.03cm⁻¹, 1423.65cm⁻¹ and 1417.13cm⁻¹ for all four samples which is related to asymmetric stretching of CO group. Peaks at 3642.03cm⁻¹, 3641.16cm⁻¹, 3642.57cm⁻¹ and 3641.93cm⁻¹ are observed due to its relation OH-stretching.

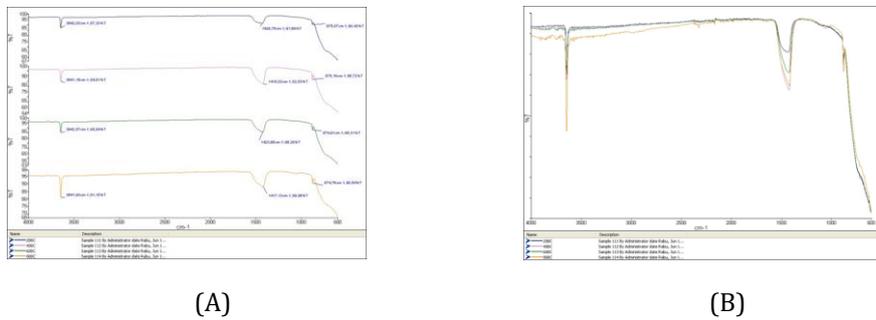


Figure 3 – FTIR Analysis of (A)Split Graph and (B) Mix Graph

3.4 Experimental Results and Analysis

Table 1. Results or Psychochemical Properties of Biodiesel

Sample/Standard	Density (15°C, g/cm ³)	Kinematic Viscosity (40°C, mm ² /s)	Biodiesel Yield (%)
CaO-TiO ₂ catalyst (800°C)	0.899	2.8	50%
ASTM D6751	0.86 - 0.90	1.9-6.0	95% - 100%

According to Table 1., density obtained is very much accordant with ASTM D6751 standards. The sample biodiesel with catalyst CaO-TiO₂ calcinated at 800°C, the kinematic viscosity is 2.8. This shows that the value is still comply with the standard and since the value of viscosity is low, this means that the oil is not thick and able to avoid issues like filter damage or poor combustion. However, the biodiesel yield is 50% which is lower than supposed standard, this might be because of long reaction time of catalyst that has thickening element (TiO₂) causes more soap and solidified catalyst and less biodiesel.

4. Conclusion

In conclusion, this research enlightens the knowledges in biodiesel production and how catalyst will affect the end product. Not only that, but also the reaction time, the mixing time, the calcination temperature, all have their impacts towards the end biodiesel product. This research mostly achieves all the results although the biodiesel yield does not reach the standards, which means there will always be a room for improvement to increase the quality of the biodiesel production which believe a great substitute to petroleum. The recommended improvement to be made are: -

- i. To increase biodiesel yield, reduce the reaction time during transesterification to avoid excessive soap or ester formation.
- ii. Carry out the transesterification process for all 4 samples of catalyst to observe if the lower temperature calcinated catalyst will results in lower yield, density and viscosity.
- iii. Repeat all test and analysis to gain average data for more accurate results.

These improvements could be conducted in future research as to increase the quality of biodiesel and also increase the understanding and knowledge in biodiesel production.

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