The Potential of *Singgora* Roof Tiles/ZnO as a Heterogeneous Catalyst in Biodiesel Production

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Abstract Biodiesel from waste cooking oil (WCO) was produced using the transesterification method utilizing *Singgora* roof tiles as a heterogeneous catalyst. Due to their fragility and porosity, *Singgora* roof tiles are commonly regarded as non-recyclable waste. However, exploring their potential as solid catalysts for biodiesel production is a promising avenue for their utilization. To enhance the chemical reaction, the wet impregnation method incorporates zinc oxide (ZnO) as an additive during catalyst production. The study aims to decrease the quantity of free fatty acid (FFA) in WCO through a two-step transesterification process. The process involves an initial acid treatment using sulfuric acid, followed by the utilization of a *Singgora* heterogeneous catalyst to produce high-quality methyl ester. Furthermore, the effect of important variables such as methanol to oil molar ratio, catalyst concentration, reaction time, and reaction temperature was investigated in this study. The type of esters content in the biodiesel was determined by GCMS analysis. The fuel properties of the biodiesel produced were compared with the ASTM D6751, EN 14214, and AOCS methods in accordance with the standard. The results showed that the biodiesel production reached an optimum yield of 96.96% under the optimal condition of a 12:1 methanol to oil ratio, 1% catalyst concentration, reaction temperature at 65 °C, and a reaction time of 2 h. The results indicate that *Singgora* roof tiles possess promising potential as a heterogeneous catalyst in biodiesel production.

Keywords *Singgora* roof tiles · Biodiesel · Transesterification

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M. A. Salim et al. (eds.), *Proceedings of the 9th International Conference and Exhibition on Sustainable Energy and Advanced Materials*, Lecture Notes in Mechanical Engineering, https://doi.org/10.1007/978-981-97-0106-3_56

1 Introduction

Biodiesel, also known as fatty acid methyl ester (FAME), offers numerous advantages over diesel. It is derived from long chains of free fatty acids (FFA) or triacylglycerol (TAG) from edible or non-edible oil sources such as animal fats or waste cooking oil [1]. Heterogeneous catalyst has received extensive attention due to their ability to yield higher biodiesel yield and their potential for reusability compared to homogeneous catalyst. Heterogeneous catalysts are considered a greener technology as they can be easily separated from biodiesel and glycerol through filtration, eliminating the need for neutralization and reducing the environmental impact associated with wastewater generation [2].

In this study, *Singgora* roof tiles, a type of clay-based roofing material were employed as the heterogeneous catalyst for biodiesel production. *Singgora* roof tiles are commonly used to cover various buildings such as palaces, mosques, and private residences. However, they have certain limitations, such as being thin, lightweight, and prone to breakage [3]. Due to these characteristics, they often end up as construction waste with no possibility for reuse. Nonetheless, the potential of *Singgora* roof tiles as a heterogeneous catalyst for biodiesel production has been largely unexplored. To date, there has been a notable absence of research investigating the use of *Singgora* roof tiles as solid catalysts in biodiesel production.

In response to this issue, this study was done to explore the viability of utilizing waste *Singgora* as the main heterogeneous in boosting the performances of biodiesel production from WCO. *Singgora* roof will be fused with ZnO by the wet-impregnation method. The two-step transesterification method will be utilized in this biodiesel production, starting with oil pre-treatment and transesterification. All the important parameters such as the effects of methanol to oil molar ratio, catalyst loading, reaction time, and reaction temperature were also investigated.

2 Materials and Methodology

2.1 Materials

The raw WCO was obtained from Dietatic and Food Service Department, Hospital Melaka. All the 99.9% pure methanol, 98% sulphuric acid (H_2SO_4) and ZnO chemicals were purchased from Polyscientific Chemicals Sdn Bhd, Melaka. The waste *Singgora* roof tiles were acquired from one of the resident's houses at Ayer Keroh, Melaka.

2.2 Methods

Catalyst Preparation and Characterization

The *Singgora* roof tiles were cleaned and crushed and ground into powder which was passed through a sieve mesh to obtain particle sizes lesser than 1 mm using a pestle, mortar, and dry blender. The *Singgora* powder was then fused with ZnO by wet impregnation method where the powder and 1% of zinc nitrate hexahydrate, $Zn(NO_3)_2.6H_2O$ were mixed with some amount of distilled water and stirred for 4 h. The sample was dried for 24 h in the oven at 100 °C for 24 h to remove the excess water. Then, the dried waste *Singgora* was calcined in a furnace at 900 °C with a heating rate of 10 °C/min for 4 h to increase the catalytic reaction and convert $Zn(NO_3)_2.6H_2O$ to zinc oxide (ZnO). To prevent contamination from carbon dioxide, CO_2, and humidity, all catalyst samples were stored in tightly sealed glass jars. The chemical composition of the sample was observed using X-ray fluorescence (XRF) model Epsilon3-XL.

Pre-treatment and Transesterification

The detailed process of two-step transesterification was reported in previous paper [4]**.** The most optimum biodiesel is produced under the condition of a 12:1 molar ratio of oil to methanol, a 1% weight concentration of catalyst, a reaction time of 2 h, and 65 °C of reaction temperature. The composition of the biodiesel was analysed with gas chromatography-mass spectrometry (Agilent Technologies 7890A) and the fuel properties which are acid value, water content, and flash point, are tested according to ASTM D6751, EN 14214, and AOCS.

3 Results and Discussion

3.1 Catalyst Characterization-XRF

XRF was used to analyse the elemental chemical composition of the *Singgora*-ZnO catalyst. Table 1 shows that silica dioxide (SiO_2) is the highest component at 55.15% followed by aluminium oxide (Al_2O_3) at 19.18%.

Table 1 Chemical composition of *Singgora*/ZnO

Formula	SiO_2	Al_2O_3	Fe_2O_3	K_2O	ZnO
Concentration (%)	55.15	19.18	4.85	1.74	1.28

Table 2 FAME composition for B100 *Singgora*-ZnO

#Peak	Retention time	Compound name	Yields (%)
2	13.5372	11-Hexadecanoic acid	0.9761
3	13.7505	Hexadecanoic acid	38.1489
4	15.5418	9,12-Octadecadienoic acid (Z,Z)	10.7738
5	15.6092	9-Octadecenoic acid	40.9984
6	15.6654	11-Octadecenoic acid	1.1533
7	15.8844	Octadecanoic acid	4.1742
9	17.4903	5,8,11,14,17-Eicopentaenoic acid	0.7323
Total			96.96

3.2 Gas Chromatography-Mass Spectrometry (GC–MS)

All the FAME compounds of the produced biodiesel identified by the GCMS were tabulated in Table 2. 9-Octadecenoic acid was found to be the largest compound followed by hexadecenoic acid. The optimum conversion of the *Singgora*-ZnO biodiesel yield was achieved at 96.96%.

3.3 Biodiesel Oil Properties

The biodiesel properties results are tabulated in Table 3. For the data confirmation, several tests were assigned, such as acid number, kinematic flash point, and density. The assessments were determined using the ASTM D6751, EN 14214, and AOCS methods [5].

Table 3 Comparison of biodiesel properties with ASTM standard

Properties	Testing method	Result	Range	
			ASTM D6751	EN14214
Acid value	ASTM D664	0.224 mg	0.5 max	0.5 max
Density	ASTM D1298	872 kg/m^3	880	860–900
Flashpoint	ASTM D93	195 °C	130 min	101 min
Water content	EN ISO 12937	0.021% V	0.05% V max	0.05% V max
Iodine value	AOCS Cd 1c-85	66 g/100 g		120 max
Saponification value	ASTM D5558	195.4 mg/KOH	370 max	-

4 Conclusion

The results showed that the optimum condition for the biodiesel production that achieved maximum yield at 96.96% is a 12:1 methanol to oil ratio, 1% catalyst concentration, reaction temperature at 65 °C, and reaction time of 2 h. The results show that *Singgora* roof tiles have the potential to be a heterogeneous catalyst in biodiesel production.

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