

# Enhanced biodiesel production from waste cooking oils catalysed by sodium hydroxide supported on heterogeneous co-catalyst of bentonite clay

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**Abstract:** Various proportions of bentonite clay performing as co-catalysts were evaluated for the production of biodiesel from waste cooking oil (WCO). The results showed that the use of bentonite as a heterogeneous co-catalyst could significantly increase the biodiesel yield by approximately 50% of the control. The heterogeneous co-catalyst of bentonite clay improved the properties of the produced biodiesel including acid number, free fatty acids (FFA), relative density, kinematic viscosity and flash point fulfilling with the standard ASTM limits and the European Biodiesel Standard (EN 14214). The use of bentonite clay in the transesterification of WCO could also enhance the conductivity of the produced biodiesel from 11 to 100  $\mu\text{S}\cdot\text{m}^{-1}$ .

**Keywords:** biofuel; heterogeneous catalyst; biodiesel, used frying oil

Fossil fuels have been the primary energy sources in the production of the worldwide energy that is derived primarily from petroleum. Due to the limited and non-renewable resource of the fuel and the high cost of the petroleum diesel, current research has mainly concentrated on the development of alternative fuels to oil production (Mahmudul et al. 2017; Oliveira et al. 2019). Biodiesel would be potential to be the main alternative fuel used to cut the excessive use of petroleum diesel. This is because biodiesel can be sustainably produced from renewable resources, such as animal fats and vegetable oils (Ramos et al. 2019).

Biodiesel is formed by alkyl esters of fatty acids and generated from the transesterification processes

of triglycerides and the esterification of free fatty acids (FFA) using alcohols, such as solvents, in the use of a catalyst to produce alkyl esters (biodiesel) as the main product and glycerine as the by-product. Some benefits derived from using biodiesel as a fuel may include its biodegradability, non-toxic nature and a sulfur-free composition (Alhassan et al. 2014). The low cost of the feedstock would be the main economic reason in the sustainability of biodiesel production (El-Gharbawy et al. 2021). A low-cost feedstock, such as waste cooking oils (WCOs) would be precious as the use of this feedstock would remarkably cut the costs of biodiesel production.

Even if using WCO as an alternative low-price feedstock would be valuable for biodiesel produc-

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tion, some obstacles may arise when conducting the transesterification of the feedstock (Esmaeili 2022). This is because the feedstock typically contains a significant amount of FFA and water that may hinder the transesterification processes (Jacobson et al. 2008; Darwin et al. 2020). A feedstock with a high FFA content would produce low methyl ester, and generate the unexpected reaction of saponification (Bokhari et al. 2016). This condition would easily occur when the catalyst used in the process is a homogeneous alkali catalyst, such as NaOH and KOH (Darwin et al. 2020). Hence, the use of heterogeneous catalysts would potentially help to optimise the methyl ester production and avoid the excessive formation of soap during the transesterification processes (Borges and Díaz 2012; Jamil et al. 2020). Furthermore, the use of heterogeneous catalysts in biodiesel production would have a valuable impact on some concerns including economic as well as environmental ones. This is because it could significantly lower biodiesel production costs, could eliminate the chemical residue in the effluent, could simplify the separation processes between the reactive products and catalyst residue, and is reusable and high stable in various media, such as acids, bases and/or alkalis (Oliveira et al. 2019).

Bentonite clay is a porous solid material that could be used for highly reactive catalysts in some organic reactions. Due to its high surface area, it could potentially be used as a solid catalyst for supporting in any kind of reaction (Adamis et al. 2005; Amalia et al. 2010). Chidi and Peter (2016) revealed that bentonite clay is considered as an eco-friendly solid catalyst material that may enhance the production of ester. Hence, it would be potentially used as a heterogeneous catalyst to enhance transesterification reactions for the synthesis of biodiesel. The current study aimed to evaluate the use of bentonite clay as a heterogeneous co-catalyst to optimise biodiesel production. The assessments of some different proportions of bentonite clay applied on the WCO transesterification processes to the biodiesel yield and its characteristics are also included.

## MATERIAL AND METHODS

**Materials preparation.** In this study, the oil used for each experiment sample was about a half a kilogram. Hence, the collection of waste cooking oil from chicken street vendors was sufficient. The WCOs used for the experiment were collected from

fried chicken street vendors situated in Kopelma, Darussalam, Banda Aceh, Indonesia. The oils were immediately cleaned and screened to remove any substance impurities that could influence the transesterification process during the biodiesel synthesis. The cleaned WCO was then placed in a glass container and stored at room temperature. Before starting the experiments, the oil sample was placed in a flask and heated to a temperature of  $108 \pm 2$  °C to vapourise the water contained within it. The oil sample was then screened using a 200 mesh-screen to remove any particle impurities (Darwin et al. 2020).

**Catalyst preparation.** The main catalyst used for the experiment was sodium hydroxide. Bentonite clay was used as a co-catalyst to enhance the biodiesel production from WCO. A gram of catalyst consisting of NaOH and bentonite was used for the transesterification processes. A series of NaOH/bentonite clay catalysts used for the synthesis of biodiesel was based on the following procedure: The ratios of NaOH and bentonite clay applied in the experiments were 9 : 1 as treatment 1 (T1), 8 : 2 as treatment 2 (T2), 7 : 3 as treatment 3 (T3), and 6 : 4 as treatment 4 (T4). Moreover, the control solution used sodium hydroxide as the only catalyst without using bentonite clay or any other catalysts (Darwin et al. 2021a). To ensure reliability and reproducibility, each experiment was performed in duplicate.

**Transesterification reaction procedure.** The transesterification reaction was conducted using 96% ethanol (technical grade) as the solvent. The catalyst (1 g) was dissolved in 100 mL of the solvent to have a highly concentrated hydroxide alcoholic solution. The prepared solution was mixed with WCO in a ratio between catalyst and WCO of 1 : 5 in which the total volume of the mixture was 500 mL. The mixture was continuously mixed at 50 rpm for approximately 60 minutes. The transesterification process was carried out under the temperature of 55 °C. The process was conducted for approximately 12 h to enable the natural separation between the crude glycerine and the biodiesel. The biodiesel synthesis occurred via a transesterification reaction in which a mole of triglyceride reacting with three moles of alcohol could generate three moles of methyl ester and one mole of glycerine. Hence, to generate a litre of the required biodiesel, at least 900 g of triglyceride and/or roughly about a kilogram of raw oil is required.

The crude biodiesel was washed three times with 1 litre of distilled hot water (80 °C) to clean it from the transesterification by-products (i.e., soap

components). During the washing, a mini air pump was used to circulate the crude biodiesel in the chamber. The crude biodiesel was removed from the mixture using the syringe and gravity method. The clean biodiesel was purified by heating it at  $107 \pm 3.0^\circ\text{C}$  to remove any impurities (Darwin et al. 2020).

**Analytical methods.** The generated biodiesel was analysed to measure its viscosity, density, FFA content, water content, acid number, conductivity, flash point, fire point, cloud point and pour point. The relative density was measured by a pycnometer under the temperature of  $15^\circ\text{C}$ . The viscosity analysis was conducted by using a vertical falling sphere viscometer equipped with digital timer at a temperature of  $40^\circ\text{C}$  (Yuan and Lin 2008). The FFA content and acid number and pH were analysed using a pH meter multifunction model benchtop complete probe and burette tube filled with using a titre of 100 mmol/L KOH (Rice et al. 2017; Darwin et al. 2019a). The conductivity was analysed by using a lab benchtop conductivity meter. To ensure its accuracy and reproducible results, the conductivity meter was calibrated with a potassium chloride (KCl) standard solution.

The water content was measured by a laboratory heating and drying oven, in which the sample was heated for approx. 24 h at  $105^\circ\text{C}$  (Rice et al. 2017; Darwin et al. 2019b; Darwin et al. 2021b). The flash, fire cloud and pour points were measured using a laboratory hot plate equipped with a digital thermocouple. The sample was placed inside the flash point apparatus with a cotton thread, and heated with the hot plate (Karmakar et al. 2018). To analyse the conversion effectiveness of the WCO during the transesterification reaction, the products were divided on the basis of the raw material to assess the biodiesel yield as the primary product and glycerine as the by-product. To have an accurate data analysis, all the sample measurements were carried out in replicates. All the biodiesel parameters were analysed according to the ASTM D6751 Standards and Testing Methods (Burton 2008).

**Statistical analysis.** The experimental data taken during the synthesis of the biodiesel were statistically analysed with single factorial ANOVA or a one-way ANOVA. The sample data collected were performed in duplicate. Furthermore, the data analysed by ANOVA were tested with a 5% ( $\alpha = 0.05$ ) level of significance to evaluate the effects of the various proportions of bentonite used as a heterogeneous co-catalyst towards the characteristics as well as to determine the generated biodiesel yield.

## RESULTS AND DISCUSSION

The physicochemical characteristics of the WCO used as feedstock for the production of biodiesel are presented in Table 1. As shown in Table 1, the FFA content of the WCO was about 1.8%, which was feasible to be processed directly in the transesterification reactions to synthesise the biodiesel without any pre-treatment of the esterification processes. The low water content of the WCO (0.1%) was quite valuable in avoiding unexpected products generated during the transesterification process. The viscosity of the WCO was somewhat high, which was around  $13\text{ mm}^2\text{s}^{-1}$ . The high viscosity of the WCO was expected to be significantly lower when it was processed in the transesterification reactions.

The results showed that with the higher proportion of bentonite clay used in the transesterification of the WCO, a lower amount of glycerine was generated. As depicted in Figure 1, the formation of glycerine tended to decrease from 31 to 18% when the proportion of bentonite used as the co-catalyst was increased from 30 to 40%. This indicated that the use of bentonite clay as a co-catalyst in the transesterification of WCO was quite valuable in cutting the excessive use of chemical catalysts (Alade et al. 2021). The results showed that even if bentonite clay was used in high proportions, it still has potential to reduce the glycerine formation, and could subsequently enhance the biodiesel production during the transesterification.

The glycerine generated in the study was much lower than that of the other studies which revealed that, during the transesterification reactions for biodiesel production, the glycerine produced would range from 30 to 60% (Hájek and Skopal 2010; Darwin et al. 2020). The low production of glycerine

Table 1. Physical chemical properties of the waste cooking oils used for the biodiesel production

Parameters	Unit	WCO
pH	–	5.95
Water content	%	0.10
Relative density ( $15^\circ\text{C}$ )	$\text{kg}\cdot\text{m}^3$	914.00
Kinematic viscosity ( $40^\circ\text{C}$ )	$\text{mm}^2\cdot\text{s}^{-1}$	13.20
FFA	%	1.79
Acid number	$\text{mg KOH}\cdot\text{g}^{-1}$	3.60
Electrical conductivity	$\mu\text{S}\cdot\text{m}^{-1}$	4.00

FFA – free fatty acid; WCO – waste cooking oils

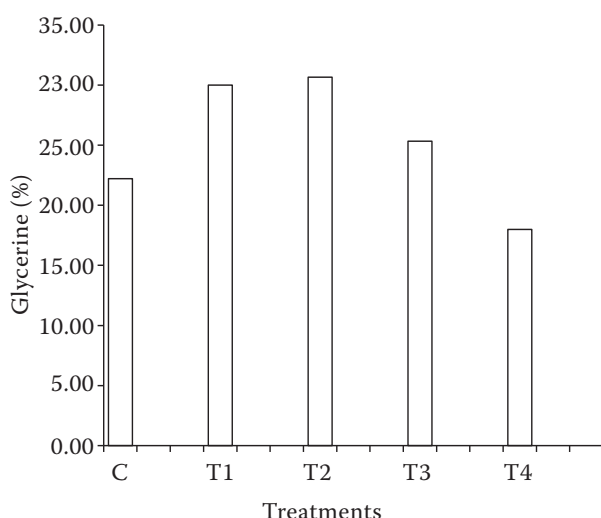


Figure 1. Glycerine yield from the various applied treatments in the transesterification processes

C – control; T – treatment

in this current study could be attributed to the use of bentonite clay as a heterogeneous co-catalyst. This is because bentonite may perform as a water adsorbent that could enhance the efficiency of transesterification reaction, and could thereby reduce the formation of soap in the produced crude biodiesel (Wu et al. 2016).

The results revealed that the bentonite clay used as a heterogeneous co-catalyst could significantly enhance the biodiesel yield (Figure 2). In this study, all the treatments with the bentonite addition in the transesterification of WCO successfully increased the biodiesel yield by more than 75%. The result was

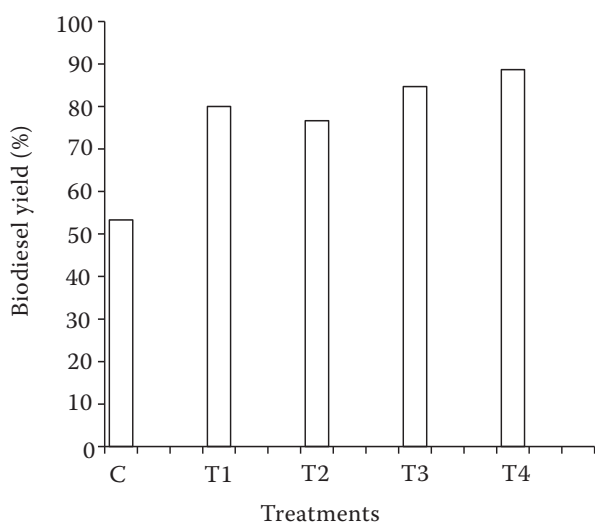


Figure 2. Biodiesel yield of the various applied treatments in the transesterification processes

C – control; T – treatment

in agreement with the study by Okechukwu et al. (2022), who revealed that the use of a heterogeneous catalyst would significantly improve the biodiesel yield. In this study, a 40% addition of bentonite as co-catalyst in the transesterification of WCO could improve the biodiesel yield to approximately 90%. This is quite significant since the control test or the sample of the biodiesel production without using bentonite as the co-catalyst only had a 50% of biodiesel yield. This suggested that high proportion of bentonite (40%) would still be feasible to be applied as co-catalyst in the transesterification processes of WCO, and thereby would reduce the excessive use of any chemical catalyst. The statistical analysis using the ANOVA test with the 5% level of significance showed that there is significant difference between the composition of added bentonite as the co-catalyst and the biodiesel yield ( $P$ -value =  $1.72 \times 10^{-6}$ ;  $F_{\text{test}} = 152.6$ ;  $F_{\text{crit}} = 5.32$ ;  $df = 1$ ). This finding is highly significant in that could it be applied in the bio-fuel industry to minimise the production costs and sustainably enhance the production of biodiesel as an alternative fuel.

A study by Ulakpa et al. (2022) on the statistical optimisation of biodiesel synthesis from waste cooking oil using a NaOH/bentonite impregnated catalyst revealed that the highest biodiesel yield of 93.22% was achieved with a bentonite to sodium hydroxide (NaOH) ratio of 1 : 10. This is quite different from the current study finding that the best biodiesel yield of 90% was obtained with a bentonite to NaOH ratio of 2 : 3 suggesting that the higher proportion of bentonite could be feasible to produce biodiesel from waste cooking oil. The different results could be attributed to the solvent used and the impregnated bentonite. In this current study, the solvent used was ethanol while methanol was used in their study and the used bentonite was not impregnated.

The results revealed that the use of bentonite clay as a heterogeneous co-catalyst could effectively reduce the FFA content of the biodiesel generated from the transesterification processes of WCO (Figure 3). All the samples of the produced biodiesel using the co-catalyst of bentonite had an FFA content of 0.28% that was extremely lower than that of the control (0.94%), which did not use the co-catalyst of bentonite (Figure 3A). Furthermore, the addition of bentonite clay as the co-catalyst in the transesterification process of WCO successfully decreased the acid number of the produced biodiesel (Figure 3B). Okechukwu et al. (2022) revealed that the use of



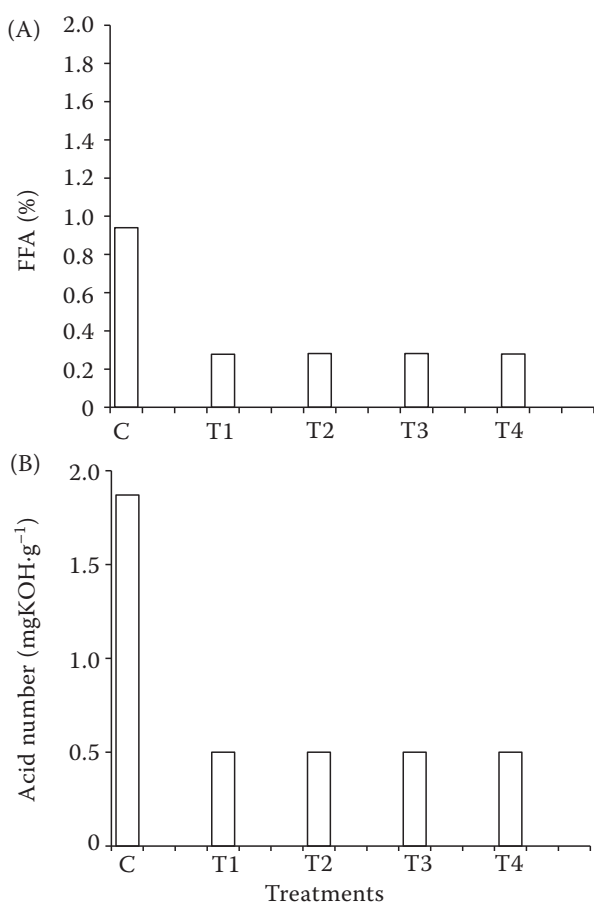


Figure 3. (A) FFA and (B) acid number of the biodiesel in the case of using various proportions of bentonite  
FFA – free fatty acid; C – control; T – treatment

a heterogeneous catalyst would potentially enhance the transesterification processes involving low-quality feedstock containing a high FFA content due to its stability and insensitivity to the FFAs.

The results showed that the acid number of the produced biodiesel co-catalysed with bentonite clay was about three times lower ( $0.5 \text{ mg KOH}\cdot\text{g}^{-1}$ ) than that of the biodiesel solely catalysed with sodium hydroxide ( $1.8 \text{ mg KOH}\cdot\text{g}^{-1}$ ). This suggested that a low acid number of the biodiesel could be effectively obtained by adding bentonite as a co-catalyst during the transesterification processes. Even though the acid number of the produced biodiesel ( $0.5 \text{ mg KOH}\cdot\text{g}^{-1}$ ) was somewhat higher than that of petro-diesel ( $0.35 \text{ mg KOH}\cdot\text{g}^{-1}$ ), it might not ruin the diesel engine parts (Singh and Padhi 2009; Karmakar et al. 2018). This is because the acid number of the produced biodiesel using bentonite clay as the co-catalyst successfully fulfilled the ASTM and European biodiesel standards, which were about  $0.5 \text{ mg KOH}\cdot\text{g}^{-1}$  (Sakthivel et al. 2018).

The results of the current study found that the more bentonite clay that was added as a co-catalyst in the transesterification of WCO, the higher the density of the produced biodiesel would be. The density of the biodiesel increased from  $872$  to  $886 \text{ kg}\cdot\text{m}^{-3}$  when the bentonite was increased from 10 to 40% (Figure 4). The density of the biodiesel is an essential property that could affect the characteristics of the engine performance. The study revealed that the fuel density could estimate the amount of fuel injected via the injection system to give feasible ignition (Sakthivel et al. 2018). The authors added that it has a vital role in the design of the injector nozzle as it directly affects the operation of the engine. The fuel density that was out of the standard bounds tends to lower the performance of the engine (Guo et al. 2016). In this study, the density of all the tested samples was still in the range of the ASTM standards of biodiesel that were around  $860$  and  $900 \text{ kg}\cdot\text{m}^{-3}$  (Karmakar et al. 2018; Darwin et al. 2020). Hence, the produced biodiesel would be feasible to be used as an alternative diesel fuel that may enhance the engine performance.

The results showed that the addition of bentonite as the co-catalyst in the transesterification of WCO may increase the viscosity of the produced biodiesel (Figure 5). The lowest viscosity of the tested samples was the T1 treatment containing 10% bentonite clay in which its viscosity was  $2.1 \text{ mm}^2\cdot\text{s}^{-1}$ . However, the highest viscosity of the produced biodiesel was obtained with the T4 treatment having 40% bentonite clay in which its viscosity was about  $6.0 \text{ mm}^2\cdot\text{s}^{-1}$ .

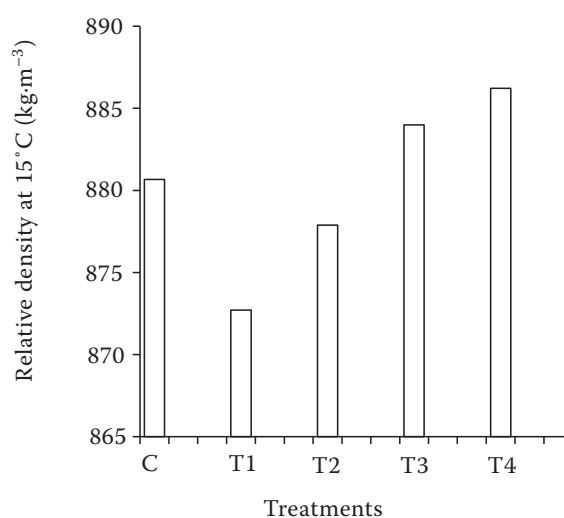


Figure 4. Relative density of the biodiesel using a heterogeneous catalyst of bentonite

C – control; T – treatment

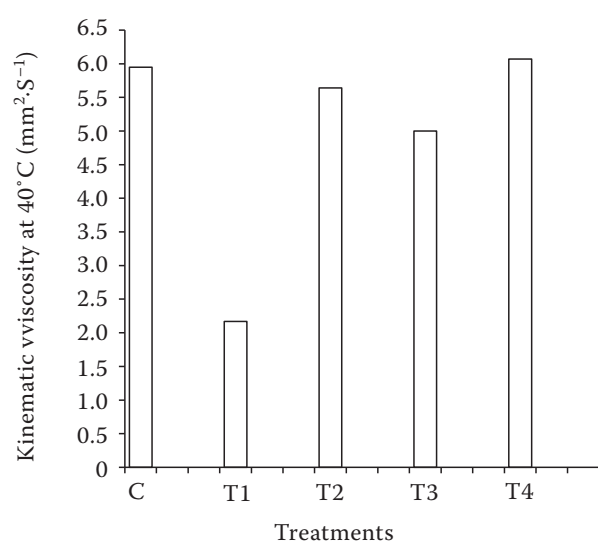


Figure 5. Viscosity of the biodiesel using a heterogeneous catalyst of bentonite

C – control; T – treatment

These results were still within the standard limits of kinematic viscosity of biodiesel established by ASTM, which range from 1.9 to 6.0 mm<sup>2</sup>·s<sup>-1</sup>. This suggested that all the treatments applied in this performed study of the transesterification of WCO using bentonite clay as a co-catalyst was feasible to generate the optimal viscosity of the produced biodiesel. This is highly significant since a higher viscosity of the fuel may induce deficient fuel atomisation which lowers the thermal efficiency and generates soot deposits. Furthermore, a lower fuel viscosity may cause finer fuel droplets that enable the injector to inflate the fuel into the combustion space (Sakthivel et al. 2018).

The use of bentonite clay as a co-catalyst in the transesterification of WCO could affect the flash and fire points of the produced biodiesel (Figure 6). In this study, the bentonite clay applied in the transesterification of WCO as a co-catalyst could lower the flash points of the produced biodiesel. The results showed that all the samples with the added bentonite clay as the co-catalyst generated lower flash points (103–105 °C) in comparison to that of the sample without using a co-catalyst or the control, which had a flash point of 111 °C. The use of bentonite clay as the co-catalyst in the transesterification of WCO not only could lower the flash point of the produced biodiesel but may also decrease its firing points. The firing points of the samples with the added bentonite clay ranged from 110 to 115 °C, which were lower than that of the control (126 °C).

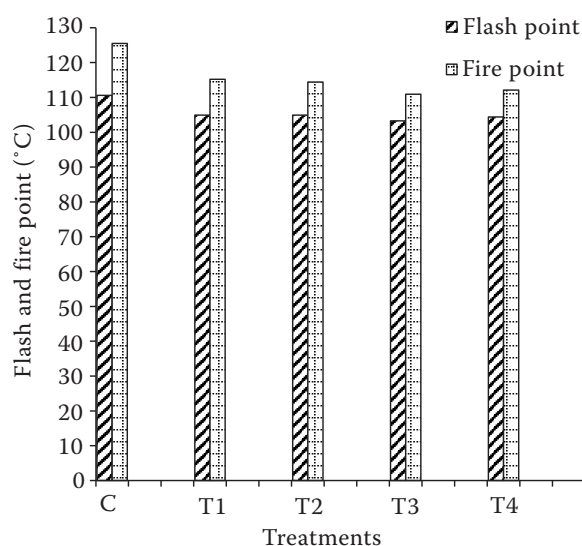


Figure 6. Flash and firing points of the produced biodiesel from the different proportions of bentonite as the co-catalyst

C – control; T – treatment

The flash points of all the tested samples were still within the standard ASTM and European Standard limits for biodiesel, which were between 101 and 130 °C (Buasri et al. 2012; Sakthivel et al. 2018). These were somewhat feasible since higher flash and firing points of biodiesel would minimise the possibility of any unanticipated fire risk (Karmakar et al., 2018). Hence, the produced biodiesel would be useful in terms of safety use, storage and transportation (Guo et al. 2016).

The results of the current study revealed the use of bentonite clay as a co-catalyst may lower the cloud point of the produced biodiesel. However, the addition of bentonite may not significantly affect the pour point of the biodiesel. As presented in Table 2, all the tested samples had cloud points (11–17.8 °C) and pour points (3.1–5.3 °C) higher than that of the standard limits of biodiesel established by ASTM, which are about –3 to –12 °C (cloud point) and –15 to –16 °C (pour point). These results indicated that the produced biodiesel still could be used in cold atmospheric conditions. However, the produced biodiesel was not feasible to be used in freezing temperatures and may need some biodiesel additives if the temperature was to be lower than 0 °C (Karmakar et al. 2018; Sakthivel et al. 2018).

The results of the current study revealed that the use of bentonite as a co-catalyst in the transesterification of WCO could significantly enhance the conductivity of the produced biodiesel. The results

Table 2. Physicochemical properties of the biodiesel made of the waste cooking oil catalysed with sodium hydroxide supported with bentonite

Parameters	Unit	Biodiesel				
		C	T1	T2	T3	T4
Cloud point	°C	17.80	15.00	11.00	10.60	12.30
Pour point	°C	4.00	3.40	4.60	3.10	5.30
Electrical conductivity	$\mu\text{S}\cdot\text{m}^{-1}$	11.00	100.00	100.00	110.00	70.00
Phosphorous	$\text{mg}\cdot\text{kg}^{-1}$	0.10	0.33	0.31	0.32	0.33
Water content	%	0.55	0.30	0.16	0.17	0.19
pH	–	7.86	7.33	7.30	7.52	7.22

T – treatment; 1–4 – No. of treatments

showed that all the biodiesel generated from the transesterification of WCO with the added bentonite clay as the co-catalyst had higher conductivity ( $70\text{--}110\ \mu\text{S}\cdot\text{m}^{-1}$ ) compared to that of the control ( $11\ \mu\text{S}\cdot\text{m}^{-1}$ ) or to the biodiesel production without using the bentonite co-catalyst. This result is highly significant as electrical charges of the biodiesel would not simply be accumulated at a high conductivity. Hence, the dissipation heat in the form of a spark would be minimised (Darwin et al. 2021a).

## CONCLUSION

Bentonite clay used as co-catalyst in the transesterification process of WCO could significantly enhance the biodiesel yield to nearly 90%. The addition of bentonite clay as the co-catalyst in the transesterification of WCO significantly reduced the FFA content of the produced biodiesel from 0.94 to 0.28%. The properties including the acid number ( $0.5\ \text{mg KOH}\cdot\text{g}^{-1}$ ), relative density ( $870\text{--}886\ \text{kg}\cdot\text{m}^{-3}$ ), kinematic viscosity ( $2.1\text{--}6.0\ \text{mm}^2\cdot\text{s}^{-1}$ ), flash point ( $103\text{--}104\ ^\circ\text{C}$ ) of the produced biodiesel using bentonite clay as the co-catalyst in the transesterification of WCO successfully fulfilled the ASTM and European Standard biodiesel limits.

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