

Biodiesel from *moringa stenopetala* seed oil

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Abstract

The aim of this paper is the production and characterization of biodiesel from *Moringa stenopetala* seed oil. Biodiesel is a renewable, biodegradable, and nontoxic biofuel. *Moringa stenopetala* seed oil is a potential feedstock that has not been discovered as an energy source. This study investigated the effects of process parameters: methanol to oil ratio, reaction temperature, and catalyst loading on the yield and quality of biodiesel. The experimental results showed that the maximum biodiesel yield was 94% and it was obtained at optimum process conditions: temperature 55°C, methanol to oil ratio 6:1, and catalyst loading 1.0. Biodiesel product was characterized based on international standards for testing fuels. According to the findings of the study, *moringa stenopetala* seed oil biodiesel has a specific gravity of 0.874 g/mole, a kinematic viscosity of 4.8 mm²/s, an acid value of 0.4 mg KOH/g, a saponification value of 196 mg KOH/g, FFA 0.2%, iodine value 104.5 high heating value 10,287 cal/g, flash Point 184°C, pour point 1°C, cloud point 10°C and cetane number 53. These results satisfy international standards for biodiesel.

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Introduction

Energy plays a crucial role in the socio-economic development of any country. The demand for energy sources has increased rapidly with the growing population in the world. Nowadays, the world is highly dependent on non-renewable fuels as an energy source. The current concerns such as high global warming, depletion of fossil fuels, and fluctuating price of conventional diesel fuels have pushed researchers toward finding new alternative, clean, renewable, and sustainable energy resources such as solar, hydroelectric, wind, and biomass-derived fuels (Kafuku, G. and Mbarawa, 2010).

Currently, fossil fuel is the dominant source of energy in the world though it is not renewable and has a high impact on global climate. The rapid increase in global energy demand, the depleting petroleum fuels, global climate change, the increasing price of fossil fuels, urbanization, and the

increasing population has driven the search for renewable energy sources which are clean, sustainable, and environmentally friendly (Atabani *et al.* 2013).

Biodiesel is a chain of mono-alkyl esters produced by the transesterification of vegetable oil and animal fats (Demshemino *et al.* 2013). Biodiesel is a promising biofuel that is renewable, biodegradable, and non-toxic. Biodiesel is gaining importance as one of the most important substitutes for depleting fossil fuels. There are many advantages of using biodiesel as an alternative form of energy. It can be used as such in the diesel engine without any engine modification indicating that it has similar physical and chemical properties as conventional diesel fuel. The combustion properties of biodiesel are also very close to those of petroleum diesel (Sreepada and Vijayalaxmi, 2013).

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The most common production technology of biodiesel is the transesterification process. Transesterification is a chemical reaction between one mole of triglyceride with three moles of alcohol to produce fatty acid methyl ester and glycerol in the presence of a catalyst or without a catalyst. Most transesterification reactions use ethanol or methanol alcohol. Many studies show that methanol is more practically used as alcohol than ethanol. Methanol is preferable due to its low cost and its physical and chemical advantages. Another advantage of using methanol is the ease of separation of glycerin which can be achieved by simple decantation (Marchetti and Yadessa, 2012).

Various catalysts can be used in the process. However, it was confirmed that the transesterification reaction was completed very fast when alkali catalysts are used. After the transesterification reaction is completed, glycerol, catalyst, alcohol, and soap are separated from biodiesel. The factors affecting biodiesel yield and fuel quality are alcohol-to-oil ratios, temperature, catalyst loading, and reaction time (Raj and Bhandari, 2017). Biodiesel is formed when triglyceride reacts with alcohol leaving glycerol as a byproduct as shown in Figure 1.

its advantages is it is considered a great natural cosmetic emollient and has high oxidative stability (Ayerza, 2012).

Moringa plant is a high-value tree of the Moringaceae family. It consists of 13 species highly distributed in Africa and Asia (Hamza and Azmach, 2017). Moringa is particularly suitable for dry regions since it can be grown using rainwater without expensive irrigation techniques. Moringa has been given less attention and ignored tree by communities to utilize it in different ways (Gemed and Desta, 2020). Flowering begins within the first six months. *Moringa stenopetala* seed is an available and underutilized resource. Ethiopia has a comfortable environment for mooring stenopetala plantations. Many parts of the country particularly southern parts such as Gamo Goffa, Sidama Wolayitta, Konso, and Darashe have planted moringa in their garden and farmland (Sreepada and Vijayalaxmi, 2013). Thus, this study investigated that *moringa stenopetala* seed oil is a potential industrial feedstock for biodiesel production as an alternative energy source.

Materials and methods

The sample of Moringa seed was collected from the Gamo Goffa Zone, Arba Minch area of Ethiopia. The seed sample

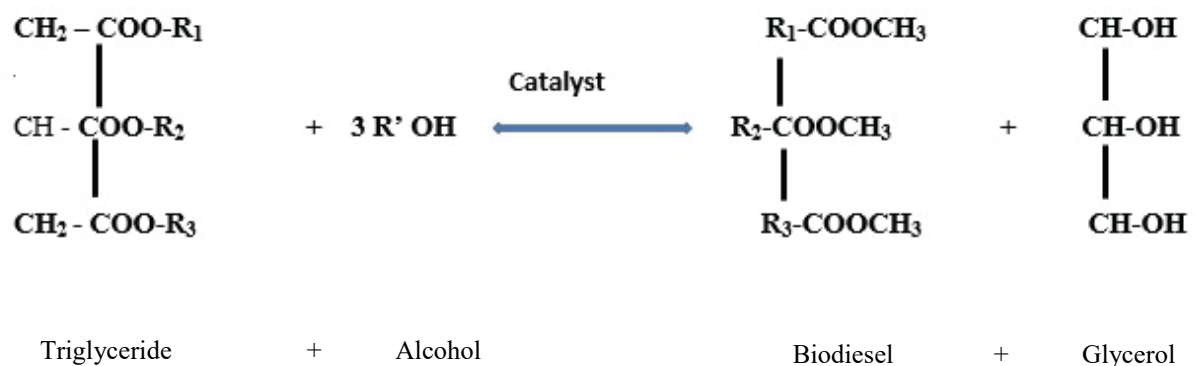


Fig. 1. Transesterification reaction

There are more than 300 vegetable oils identified as potential feedstocks for biodiesel production globally. The availability, characteristics, and variety of feedstocks make them promising ingredients for the sustainable production of biodiesel (Piyush *et al.* 2013). Researchers have proposed Moringa seed oil as an alternative potential feedstock to produce biodiesel. However, there is a research gap that promotes this feedstock for industrial processing. Moringa seed has an average of 35%-45% oil content with high oleic content of greater than 73% based on the type of the tree. Moringa oil has many various industrial and homemade benefits. One of

was dried and milled to reduce the particle size. Then, 80 g of sample was measured and put into the soxhlet apparatus thimble. Then, it was placed in a 500 mL capacity soxhlet chamber. 400 mL solvent (Hexane) was used for the extraction at different temperatures for 3 hrs. to 5 hrs. The extraction was conducted at 70°C, 75°C, and 80°C by varying extraction time to get the maximum possible oil yield. The soxhlet extraction method was selected since it results in high oil yield, is quick, the solvent can be reused, and is low cost (Sreepada and Vijayalaxmi, 2013). The unwanted substances were removed by evaporation and degumming. Evaporation was used to separate hexane from

oil by rotary evaporation at 80°C. Gum and wax were removed by a degumming method. Then, biodiesel was synthesized by transesterification reaction using a homogeneous catalyst (methanol) at different process parameters.

Experimental design

The experimental design used for this study was a Box Behnken design standard using design expert 11.0 software. The experimental design was done using three variables and three levels for a response surface method. 17 experimental runs were selected by design expert 11.0 software using the Box Behnken design standard method, with five central points per block. The main factors that affect biodiesel production by transesterification reaction are reaction time, reaction temperature, and alcohol-to-oil ratio (Seema and Meena, 2017). As indicated in Table I, the overall production of biodiesel was carried out at optimum parameters of catalyst load of 0.5 to 1.5 wt.%, reaction temperature of 45 to 65°C, and methanol to oil ratio of 3 to 9.

Table I. Independent variables and level of main factors that affect transesterification reaction

Variable	Unit	Level	
		-1	+1
Methanol to oil ratio	-	3	9
Catalyst loading	Wt.%	0.5	1.5
Reaction temperature	°C	45	65

The other parameters, reaction time and mixing intensity were kept constant at 1 hour and 500 rpm for all experimental runs. A combination of three factors and three levels of experimental runs was conducted. There are 17 experimental runs determined by Box Behnken's design arrangement as shown in Table II.

Production and purification of the product

The method used to produce biodiesel was a one-step transesterification reaction. Methanol was the alcohol selected for the transesterification reaction due to its availability, low cost, popularity, and ease of separation (Demirbas, 2005). The catalyst selected for this study was the base catalyst (KOH) since biodiesel can be produced at low temperatures and pressure reactions. Moreover, it is a homogeneous base catalyst that results in high conversion, minimum side reactions, short reaction time, and high yield (Marchetti and

Yadessa, 2012). Though it has many advantages, the disadvantage of the catalyst used is it cannot be reused.

When the reaction is completed, biodiesel and glycerol were separated by a separatory funnel after it settles for 24 hours. Then, biodiesel was washed with hot distilled water to remove soap, remaining methanol, and wax.

$$\text{Yield (\%)} = \frac{\text{Volume of pure biodiesel produced}}{\text{a volume of oil used}} * 100$$

Optimization of the process parameters using the Response Surface Method (RSM).

Optimization aims to determine the optimum value of the factors that affect the transesterification process to produce the maximum possible biodiesel yield at a minimum production cost. This is done by minimizing the methanol to oil ratio, reaction temperature, and amount of catalyst used as indicated in Table III.

Characterization of biodiesel

Characterization of fuel is determining the physical and chemical properties (Atabani *et al.* 2013). The physical and chemical properties of biodiesel were determined according to the American Society for Testing Materials (ASTM D 6751) and European standard (EN 14214) methods (Demirbas, 2005). Table IV showed the physical and chemical properties of biodiesel and the standard methods used to determine them.

Statistical analysis of experimental results

Data analysis of the experiment was done by design expert 11.0 software using Box Behnken design standard. The significance of experimental variables was obtained from analysis of variance (ANOVA).

Results and discussions

Yield

The maximum oil yield obtained at optimum conditions of temperature 80°C and extraction time 5 hours was 39.86 %. This result agreed with the average oil content of *Moringa stenopetala* seed oil which is 35% - 45% (Ayerza, 2012), and this result also agreed with the previous report of 38% which was reported by Azad *et al.* (2015) and by Gemechu (2021).

The maximum yield of biodiesel was 94% and it was obtained at optimum process parameters of methanol to oil ratio 6:1, temperature 55°C, and catalyst loading 1.0%. The yield of biodiesel obtained agrees with the previous report of

Table II. Box Behnken design arrangement (experimental design matrix)

Run	Factor 1 A: Methanol to oil ratio	Factor 2 B: Temperature	Factor 3 C: Catalyst loading	Yield response (%)
1	6	55	1.0	
2	6	65	0.5	
3	6	45	0.5	
4	3	55	1.5	
5	9	45	1.0	
6	9	65	1.0	
7	3	65	1.0	
8	6	55	1.0	
9	6	55	1.0	
10	9	55	0.5	
11	3	55	0.5	
12	6	55	1.0	
13	3	45	1.0	
14	9	55	1.5	
15	6	45	1.5	
16	6	65	1.5	
17	6	55	1.0	

Table III. The goal of optimization and limitation of process parameters

Name	Goal	Lower limit	Upper limit	Lower weight	Upper weight	Importance
A: Methanol to oil ratio	minimize	3	9	1	3	3
B: Temperature	minimize	45	65	1	3	3
C: Catalyst loading	minimize	0.5	1.5	1	3	3
Yield	maximize	65.67	94.0	1	3	3

Table IV. Standard methods (ASTM D 6751 and EN 14214) for physical and chemical properties of biodiesel

Property	Standard Methods
Specific gravity	EN 14214
Kinematic viscosity	ASTM D445
Saponification value	ASTM D5558
Acid value	EN 14104
Calorific value	ASTM D240
Flash and fire point	ASTM D93
Moisture content	EN 14774 -1
Iodine value	EN 14111
Pour point	ASTM D5771
Cloud point	ASTM D6950

Azad *et al.* (2015) which was 91%. The yield increased as transesterification parameters increased until the optimum value and decreased beyond the optimum value. At the lower value of parameters, the yield decreased due to the incomplete conversion of reactants to the product. Table V showed the yield response of all experimental runs.

Characterization of biodiesel

Physical and chemical properties of biodiesel were evaluated using standard methods and the results were compared with ASTM and EN standard methods and previous reports.

Density

The value of the density of biodiesel was 874 kg/m³. This result is within the standard range of density (860 – 900 kg/m³) (Demirbas, 2005) and agrees with the previous report by Kafuku and Mbarawa (2010) which was 900 kg/m³. Higher density increases the size of fuel droplets and causes higher emission of particulate matter and NO_x emission in a diesel engine while the lower density of fuel expands the efficiency of atomization (Choi and Reitz, 1999).

Table V. Biodiesel Yield response of experimental runs

Std	Run	Factor 1	Factor 2	Factor 3	Response 1
		A: Methanol to oil ratio	B: Temperature °C	C: Catalyst loading wt.%	Yield %
10	1	6	65	0.5	86.67
3	2	3	65	1	78.18
13	3	6	55	1	93.81
2	4	9	45	1	78.18
9	5	6	45	0.5	76.36
4	6	9	65	1	81.82
12	7	6	65	1.5	88.34
8	8	9	55	1.5	85.45
15	9	6	55	1	93.84
7	10	3	55	1.5	80
5	11	3	55	0.5	72.71
11	12	6	45	1.5	76.67
14	13	6	55	1	94
17	14	6	55	1	93.27
16	15	6	55	1	93.92
6	16	9	55	0.5	83.63
1	17	3	45	1	76.67

Kinematic viscosity

The experimental result showed that the value of kinematic viscosity was 4.8 mm²/s. This result is within the range of the ASTM standard value of biodiesel which is 1.6-6 (Eman and Cadence, 2013; Demirbas, 2005). Highly viscous fuel is difficult to pump and operate in internal combustion engines while lower kinematic viscosity of fuels increases wear leakage due to insufficient lubrication (Rao, 2011).

Acid value

The value of an acid value obtained was 0.4 mm KOH/g. This result agrees with the previous report (Kafuku and Mbarawa, 2010), with a value of 1.2 mmKOH/g. Higher acid-value fuels need further acid treatment costs and it also lowers the quality of the fuels. In this study, the lower acid value of biodiesel indicates that it has higher fuel quality.

Calorific value

The experimental result showed that the value of biodiesel was 10287 Cal/ g or 43 MJ/kJ. This result agrees with the previous report of Mahamudul *et al.* (2017) which was 39

MJ/kg, and the previous report of Agarwal and Das (2001) which was 43.2 kJ/kg. The result obtained is above the standard ASTM value which is 42-42.5 MJ/kg. This result indicates the higher fuel quality of biodiesel. The calorific value obtained is similar to that of conventional diesel fuels. This is the novel result of this study.

Flash point

The flash point obtained from the experimental result was 184°C. This result agrees with the standard ASTM value of biodiesel flash point which is greater than 130°C. From this study, the higher flash point of biodiesel indicates that it is safer to handle, store and transport the fuel (Jayed *et al.* 2011).

Cetane number

The value of the cetane number observed was 53. This result agrees with the standard ASTM value of the cetane number of biodiesel which is greater than 47 (Demirbas, 2005). Fuels with higher cetane number benefit the engine performance since the engine starts rapidly and run smoothly without causing much noise.

Table VI. Summary of physicochemical properties of *moringa stenopetala* seed biodiesel

Property	Result	Unit	Standard method used
Moisture	1.4	%	ASTM D4442-07
Ph	7.9	-	
Density at 15°C	874	Kg/m ³	EN 3675
Kinematic viscosity at 22°C	4.8	mm ² /s	ASTM D445
Acid value	0.4	mg KOH/g	ASTM D664
Saponification value	196	mg KOH/g	
Free fatty acid	0.2	%	
Iodine value	104.5	g I ₂ /100 g	EN 14111
Calorific value	10,287	Cal/g	ASTM D240
Flash point	184	°C	ASTM D93
Cetane number	53	-	ASTM D 6751
Fire point	190	°C	ASTM D93
Specific gravity	0.874	g/mole	EN 14214
Cloud point	10	°c	ASTM D6950
Pour point	1	°c	ASTM D5771

Cloud and Pour point

The experimental result showed that the cloud and pour points of biodiesel were 10°C and 1°C respectively. This result agrees with the previous report of Kafuku and Mbarawa (2010) which was 10°C and 3°C respectively. Cloud and pour point of fuel indicates the fuel performance at low ambient temperature (Agarwal and Das, 2001). Table VI showed the result of all main physicochemical properties of biodiesel synthesized.

The Lack of Fit F-value of 5.69 implies there is a 6.31% chance that a Lack of Fit F-value this large could occur due to noise. Lack of fit is bad -- we want the model to fit. This relatively low probability (<10%) is troubling.

The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around that average based on the

Table VII. Analysis of variance (ANOVA)

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	1316.65	9	146.29	380.98	< 0.0001	significant
A-Methanol to oil ratio	35.15	1	35.15	91.55	< 0.0001	
B-Temperature	332.56	1	332.56	866.06	< 0.0001	
C-Catalyst loading	7.55	1	7.55	19.65	0.0030	
AB	0.6972	1	0.6972	1.82	0.2198	
AC	0.3481	1	0.3481	0.9065	0.3727	
BC	0.0380	1	0.0380	0.0990	0.7622	
A ²	393.68	1	393.68	1025.23	< 0.0001	
B ²	458.66	1	458.66	1194.44	< 0.0001	
C ²	16.58	1	16.58	43.18	0.0003	
Residual	2.69	7	0.3840			
Lack of Fit	2.18	3	0.7259	5.69	0.0631	not significant
Pure Error	0.5101	4	0.1275			
Cor Total	1319.33	16				

Statistical analysis of experimental results

Analysis of variance (ANOVA) for the quadratic model

Analysis of variance determines the significance of the experimental results using coded variables as indicated in Table VII.

The Model F-value of 380.98 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise.

P-values less than 0.0500 indicate model terms are significant. In this case, A, B, C, A², B², C² are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

factor settings. When the factors are orthogonal the VIFs are 1; VIFs greater than 1 indicate multi-collinearity, and the higher the VIF the more severe the correlation of factors. As a rough rule, VIFs less than 10 are tolerable.

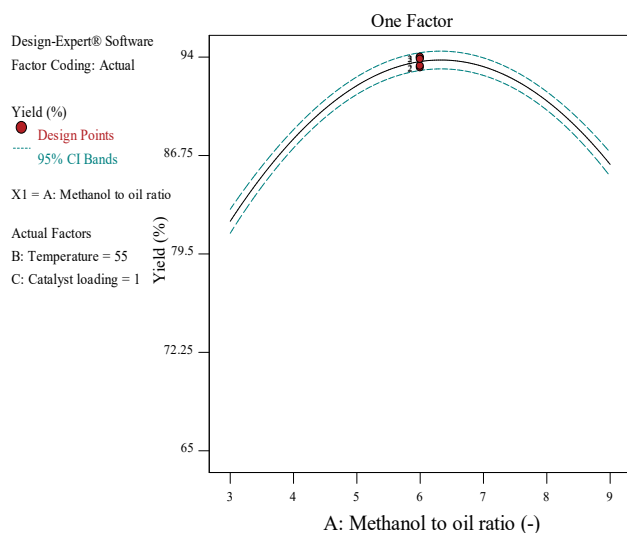
The effect of process parameters on the yield of biodiesel

Effect of methanol to oil ratio

Figure. (2) Shows the effect of methanol to oil ratio on biodiesel yield at constant temperature and catalyst loading. It showed that the yield of biodiesel increases as the methanol-to-oil ratio increases. Increasing the methanol-to-oil ratio beyond the optimum value results in excess methanol which decreases the yield of biodiesel. The maximum yield of biodiesel (94%) was obtained at a methanol-to-oil ratio of 6: 1. Therefore, this ratio was taken as an optimum value of the methanol-to-oil ratio. Decreasing the methanol-to-oil ratio

Table VIII. Optimum numerical solution by RSM

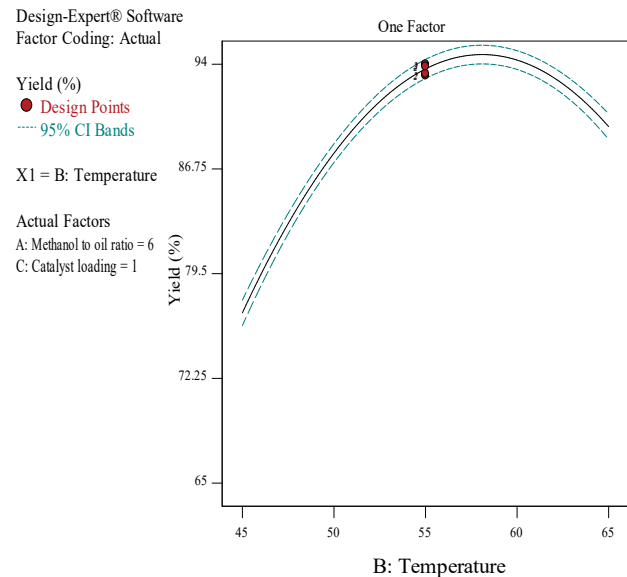
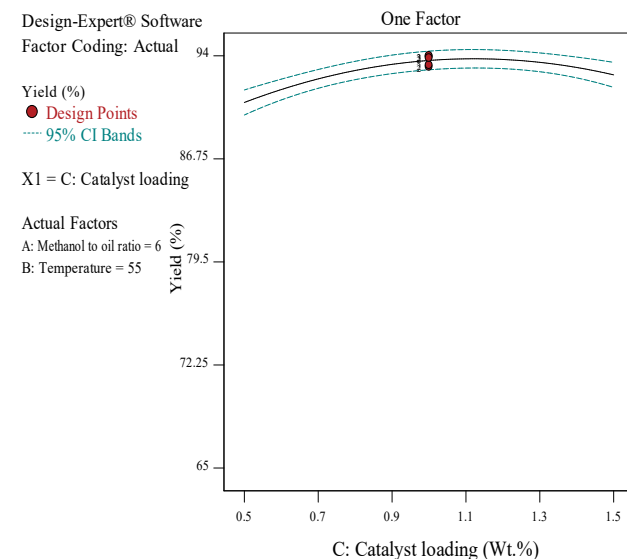
Number	Methanol to oil ratio	Temperature	Catalyst loading	Yield	Desirability	
1	4.489	51.537	0.500	83.683	0.753	Selected
2	4.511	51.501	0.500	83.722	0.753	
3	4.513	51.518	0.500	83.753	0.753	
4	4.487	51.430	0.500	83.529	0.753	
5	4.500	51.630	0.500	83.850	0.753	
6	4.444	51.617	0.500	83.610	0.753	
7	4.521	51.382	0.500	83.599	0.753	
8	4.450	51.419	0.500	83.369	0.753	
9	4.419	51.558	0.500	83.428	0.753	
10	4.512	51.729	0.500	84.028	0.753	
11	4.422	51.412	0.500	83.243	0.753	
12	4.472	51.818	0.500	83.981	0.753	
13	4.421	51.813	0.500	83.769	0.753	
14	4.773	50.864	0.500	83.781	0.751	
15	5.068	53.527	0.500	87.898	0.737	

**Fig. 2. Effect of methanol to oil ratio on yield of biodiesel**

below the optimum value (3:1), decreased the conversion efficiency of the transesterification reaction and resulted in lower biodiesel yield.

Effect of reaction temperature

Figure (3) shows the effect of reaction temperature on the yield of biodiesel at a constant methanol-to-oil ratio and catalyst loading. The yield of biodiesel highly increased as

**Fig. 3. Effect of temperature on yield of biodiesel****Fig. 4. Effect of catalyst loading on yield of biodiesel**

temperature increased from 45°C to 55°C. Maximum yield was obtained at 58°C and the yield started to decrease slightly from 58°C to 65°C due to the vaporization of methanol resulting in soap formation. The boiling point of methanol is 64.7°C. Increasing reaction temperature above this point leads to methanol loss by evaporation and decreases the conversion efficiency of the reaction. At 45°C, the yield is about 75%.

Effect of catalyst loading

Figure (4) shows the effect of catalyst loading on the yield of biodiesel at a constant methanol-to-oil ratio and temperature. As the amount of catalyst increased from 0.5% to 1.0%, the yield of biodiesel increased from 89% to 94% and the yield starts to decrease slightly as the amount of catalyst increased from 1.0% to 1.5% due to the formation of soap and emulsion. At a lower amount of catalyst (0.5%), the yield of biodiesel decreased due to the incomplete conversion of triglycerides of oil to fatty acid methyl ester.

Optimization of transesterification process parameters using the Response Surface Method (RSM)

The Response Surface Method determined the optimum value of process variables that maximize the yield of the product and minimize production cost

Table VIII showed that Response Surface Method found 15 solutions and selected the optimum values to minimize the economic cost of Temperature, Methanol, and Catalyst required for the reaction and to maximize the yield of biodiesel. The optimum yield of biodiesel (83.772%) was observed at a temperature of 51.537 and catalyst load of 0.500 wt.%.

Conclusions

Biodiesel is a promising biofuel that is synthesized by the transesterification of vegetable oils and animal fats. In this study, biodiesel was synthesized from *moringa stenopetala* seed oil using an alkali catalyst. The effect of methanol to oil ratio, reaction temperature, and catalyst weight on the yield and quality of biodiesel have been studied. Biodiesel produced was characterized according to international fuel testing standards methods. The optimization of process parameters was carried out using Response Surface Method (RSM).

The experimental result showed that the highest yield of biodiesel was 94% and this result was obtained at an optimum value of temperature 55°C, methanol to oil ratio of 6:1, and catalyst load of 1.0%. Increasing methanol to oil ratio, temperature, and catalyst load up to the optimum point increased the yield of biodiesel. However, further increase of the parameters above the optimum point decreased the yield of biodiesel. The temperature has the highest significant effect while the catalyst load has a lower effect.

Besides to characterization of different basic fuel properties of it, biodiesel can be blended with petrol-diesel at different percentages and applied in compression ignition engine to evaluate the effect of using a blend of biodiesel using differ-

ent parameters such as engine power, brake thermal efficiency, specific fuel consumption, exhaust gas temperature, and greenhouse gas temperature and gas emission. However, the performance analysis of the blend of biodiesel with petrol diesel in a compression ignition engine was not done due to a lack of laboratory setup and equipment required to measure these parameters. This study did not compare biodiesel with diesel fuels used in diesel engines. Moreover, it did not characterize the blend of biodiesel with petroleum fuels and did not compare biodiesel produced from other feedstocks other than *moringa stenopetala*. Thus, further research should be done on these areas.

Generally, it can be concluded that *moringa stenopetala* is a promising alternative feedstock for biodiesel production for the reasons: it does not affect food security, has a good oil content, and can be harvested throughout the year. The biodiesel produced meets the standard fuel quality and international standard specifications.

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