

## INVESTIGATION ON THE CHARACTERISTICS OF BIODIESEL PREPARED FROM MEZE SEED OIL

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### Abstract

Production of biodiesel, a renewable fuel having environmental advantages, is a valuable process. This research is emphasized on the investigation of characteristics of biodiesel prepared from expelled Meze seed oil. Meze seed oil was firstly analyzed for its physicochemical properties and some of fuel properties. Biodiesel was prepared by two stage process; esterification followed by transesterification. Esterification step reduces the free fatty acid (FFA) value of the Meze seed oil to about 2% by using acid catalyst. Variation of different parameters such as volume of methanol, reaction time, reaction temperature, volume of acid catalyst (H<sub>2</sub>SO<sub>4</sub>) and amount of alkaline catalyst (KOH), were conducted to achieve the maximum yield of biodiesel. (400) mL (4:1% v/v) of methanol and (1.5) mL (1.5% v/v) of H<sub>2</sub>SO<sub>4</sub> for 90 min reaction time at 60°C were chosen as the most suitable conditions for esterification stage. In the transesterification stage, (50) mL (1:2% v/v) of methanol, (1) g (1% w/v) of KOH, 90 min reaction time and reaction temperature of 60°C were also selected as the most favourable conditions for the preparation of biodiesel from esterified Meze seed oil. Functional groups present in the prepared biodiesel was also investigated by FT-IR analysis. Determination of the fuel properties of prepared biodiesel such as flash point, pour point, kinematic viscosity, specific gravity and cetane number were conducted and compared with ASTM standard. Prepared biodiesel was found to have a moderate kinematic viscosity but relatively high flash point when compared with the standards outlined in ASTM Standards for Biodiesel.

**Keywords:** Meze seed oil, Biodiesel, Transesterification.

### Introduction

Biodiesel, a product of the transesterification of fats and oils by lower alcohols like methanol, is becoming increasingly popular as a fuel or as biodiesel blends through mixing with different levels of fossil diesel, in developing and developed countries. Vegetable oils, off-quality oils, used cooking oils and animal fats have been used as raw materials for biodiesel production using transesterification. Transesterification of these raw materials can be carried out in the presence of a catalyst, such as alkali, acid, or an enzyme (N. Saifuddin et al, 2009).

Mahua (*Madhuca longifolia*), belonging to the family Sapotaceae, is an important economic plant. It usually grows throughout the tropical and subtropical region. It is found abundantly in India, Sri Lanka and also Myanmar (tropical region). In Myanmar, it is called Meze or Myintzuthaka. It grows up to approximately 20 meters in height. Although having tremendous therapeutic and potential use, *Madhuca longifolia* is not fully utilized because of unawareness of people (Mishra & Padhan, 2013).

The flowers of *Madhuca longifolia* bloom with new leaves between February and April and the fruits ripen from May to August. Fruits are generally 2.5 to 5 cm long and ovoid or sub-globose shape. Their greenish colour changes to reddish-yellow or orange when they ripe and fleshy. (Mishra & Padhan, 2013).

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Seed oil can be produced by different methods such as, mechanical expression method, solvent extraction method, or both methods. The most common method for oil extraction is mechanical pressing using different kinds of press such as hydraulic press, screw press and rolling press. But, solvent extraction method is the one which is able to extract over 98% oil. However, specific process before conducting the next process is needed after extraction because of its disadvantages such as the necessary equipment for extraction is high in cost, the process is quite dangerous in correlation with fire and explosion and the solvent used (Deli, et al., 2011).

Meze (Mahua) seed oil can be used for skin care, for the manufacture of soaps, detergent, as vegetable butter and as fuel oil. Having emulsion property, it can also be used as an emulsifying agent in few pharmaceutical industries (Mishra & Padhan, 2013).

Thus, the objectives of this study are to extract the Meze (Mahua) seed oil and to apply for biodiesel preparation.

## Materials and Methods

### Materials

Mature meze seeds were purchased from Simehtun village, Amarapura Township, Mandalay Region. Best Oil Press machine was used to extract Meze seed oil. Analar grade of 98% methanol, sulphuric acid, potassium hydroxide and sodium hydroxide were also used in the preparation of biodiesel.



Meze Tree and Fruits



Meze Seeds



Meze Seed kernels

## Methods

### Pretreatment of Meze Seeds

Dehulling of Meze seeds were done manually to obtain the inner kernels. Then, the kernels were sun-dried to reduce the moisture content (4-6%) for 2-3 days. The dried Meze seed kernels were stored in storage bin at room temperature before extraction.

### Extraction of Meze Seed Oil

The dried Meze seed kernels were ground using expeller (Best Oil Press Machine with Heater ,Model No.02, Taiwan made) between 45-50°C. Meze seed oil and oil cake residues were obtained separately. The oil obtained was settled and filtered to remove the residual solids.

### Preparation of Biodiesel using Meze Seed Oil

In the preparation of biodiesel, a two-step transesterification process namely, acid esterification and alkali esterification was carried out. The esterification step firstly reduced the free fatty acid (FFA) content of Meze seed oil (MSO) to less than 2% using acid catalyst. Then, the alkaline catalyst transesterification process converted the low FFA of MSO to its ester.

About 100 mL of MSO was poured into 1L of flat-bottomed flask and heated to 60°C. Then 400 mL of 98% methanol and 1.5 mL of sulphuric acid was added to the preheated MSO and heating was continued at 60°C while stirring with magnetic stirrer at a speed of 500-600 rpm for 90 min. The product obtained was poured into a separating funnel and left for 2 hr. The excess methanol with sulphuric acid and impurities moved to the top surface and was removed. The lower layer was low free fatty acid esterified MSO. The oil sample was heated to 70 °C to remove the excess methanol and washed with distilled water in a separating funnel. Finally, the acid value of the product separated at the bottom was determined. The effects of amount of methanol, amount of catalyst, reaction time and reaction temperature on reduction of acid value of esterified MSO were investigated.

Then, 100 mL of esterified MSO was put into a 500 mL of flat-bottomed flask and heated to 60°C and stirred. The catalyst, 1g of potassium hydroxide which had already been mixed with 50 mL of 98% methanol, was gradually added into the preheated esterified MSO. Then, the mixture was stirred for 90 min at 60°C. When the reaction was complete, the reaction mixture was transferred into a separating funnel and allowed to settle overnight to enhance the separation at room temperature. When two layers separated sharply, the bottom glycerine layer was drawn off. The biodiesel was purified by washing gently with warm water to remove residual catalysts or soap. The washed solution was checked with phenolphthalein indicator to ensure that the biodiesel was free from residual catalysts or soap. The washed biodiesel was dried in an oven at 110 °C to remove residual methanol. The effects of amount of methanol, amount of catalyst, reaction time and reaction temperature on yield of prepared biodiesel were determined respectively.

### Methods of Identification and Analysis

Physico-chemical properties of Meze seed kernel were firstly investigated. To evaluate the quality of Meze seed oil, the characteristics such as specific gravity, refractive index, colour, moisture, acid value, saponification value, unsaponifiable matter, iodine value and peroxide value, kinematic viscosity, flash point and pour point were determined. Gas Chromatography analysis was also conducted to find out the fatty acid composition of Meze seed oil. The functional groups present in the prepared biodiesel were identified by FT-IR analysis. Fuel properties of biodiesel were also determined.

### Results and Discussion

The physico-chemical properties such as moisture, ash, protein, crude fibre, crude fat and carbohydrate content of Meze seed kernels are shown in Table(1). It is seen that the percentage of crude fat is the highest in Meze seed kernel and it is also higher than literature values. Its protein content is significantly different from the literature values (Mishra. & Padhan, 2013). It can be clearly seen that the kinematic viscosity of Meze seed oil is much greater than that of literature value (Padhi, 2010). Fatty acid composition of extracted oil investigated by Gas Chromatography is indicated in Figure (1) and Table (2). The results shown in Table (2) represent that extracted Meze seed oil mainly contains palmitic acid (25.9%), oleic acid (43.7%), stearic acid (19.3%) and linoleic acid (9.8%) as predominant compounds.. The results are in agreement with Mishra et al. (2013). The results shown in Table (3) represent the physico-chemical properties of extracted Meze seed oil. Peroxide value, acid value and iodine value of extracted Meze seed oil indicate the freshness and lower rancidity of extracted oil.

In this study, Meze seed oil was applied in the preparation of biodiesel by two stage process; esterification followed by transesterification. The first step reduced the free fatty acid (FFA) value of the Meze seed oil to about 2% using acid catalyst. The crude Meze seed oil had an initial acid value of 8.27 mg KOH/g of oil corresponding to FFA level of 4.16%. According to Kathirvelu (2014), the process of transesterification is complicated, if oil contains large amount of FFA content that will form soap with alkaline catalyst. The soap can prevent the separation of the biodiesel from the glycerine fraction. Therefore, pretreatment process using an acid catalyst (sulphuric acid) was conducted to reduce the acid value of Meze seed oil. The reduction of acid value of Meze seed oil on different volumes of methanol used is shown in Table (4). It is seen that the acid value reduced quickly at the initial stage and reduced slowly later. According to Kathirvelu (2014), this might be due to the effect of water formation during the esterification of FFAs, which prevented further reaction. So, 400 mL (4:1% v/v) of methanol was selected as the optimum amount to reduce the acid value of Meze seed oil.

Table (5) shows the effect of amount of acid catalyst on the reduction of acid value of Meze seed oil. The catalyst amount was varied in the range of 0.5-2.5 mL for five different volumes (0.5, 1.0, 1.5, 2.0, 2.5 mL) of sulphuric acid. At lower catalyst concentration, acid value could not be reduced sharply. In accordance with Kathirvelu (2014), the ester formation rate increased with increasing catalyst concentration. However, it was observed that addition of sulphuric acid in excess darkened the colour of the product and adding lower volume than a certain volume affected the yield of the subsequent step. Therefore, 1.5 mL (1.5% v/v) of  $H_2SO_4$  was selected as the suitable catalyst concentration that can provide the FFA content of less than 2%.

Effect of reaction time on reduction of acid value of esterified Meze seed oil is tabulated in Table (6). It is clearly seen that the acid value of the esterified oil dropped down to 2.37 (71.34% reduced) after 90 min. So, 90 min was chosen as the optimum reaction time for reducing the acid value of esterified Meze seed oil. Table (7) shows the effect of reaction temperature (40, 50, 60, 70, 80°C) on the reduction of acid value of Meze seed oil. The results show that the reaction temperature had an important role in the acid catalyzed esterification. The rate of reaction is increased by increasing the reaction temperature. This result was in accordance with Kathirvelu (2014). It was observed that the acid value of Meze seed oil could not be reduced to less than 2 mg KOH/g of oil at 40 °C. At 60 °C, the acid value was reduced to 2.37 (FFA content of 1.19%). Hence, the reaction temperature 60°C was considered to be the optimum temperature for this reaction.

The effect of methanol on yield of biodiesel in transesterification is described in Table (8). It can be seen that higher amount of methanol used (ie, 60, 70, 80 mL) gave high yield percent of biodiesel. But it is not too different of the yield percent that can be given by the use of 50 mL of methanol. In addition, high amount of methanol added to vegetable oil interfered with the separation of glycerine because there was an increase in solubility. When glycerine remains in the solution, it helps drive the equilibrium back to the left side of the equilibrium, resulting in lower yield of esters (Kathirvelu, 2014). So, 50 mL (1:2 v/v) of methanol was selected as the suitable condition for alkali catalyst esterification. The variation of the amount of alkali catalyst (KOH, 0.4-1.2 g/100 mL of oil) was carried out to prepare biodiesel and it is shown in Table (9). From this Table, the yield percentage of biodiesel at different amount of catalyst used can be seen. The results show that the reaction was highly dependent on the catalyst amount. It was

observed that the maximum yield of biodiesel was obtained at 1g of KOH. With further increase in amount of catalyst, there was a decrease in the yield of biodiesel. Addition of excess amount of catalyst give rise to the formation of soap and glycerol phase. Esterification does not take place effectively for insufficient amount of catalyst (Kathirvelu, 2014). Therefore, 1g (1% w/v) of KOH was chosen as the suitable amount for alkali catalyst esterification.

The different reaction times selected for this study was 45, 60, 75, 90 and 105 min and the effect of reaction time on yield of biodiesel is shown in Table (10). The results clearly show that the yield of biodiesel increased with increasing reaction time. In this study, 90 min was selected as the suitable reaction time for alkali catalyst esterification. Table (11) describes that the effect of different reaction temperature on the yield of biodiesel. It was observed that the yield of biodiesel decreased far beyond the boiling point of methanol. According to (Kathirvelu, 2014), the reaction temperature above boiling point of methanol is avoided since at high temperature it tends to accelerate the saponification of glycerides by the alkaline catalyst before completion of the alcoholysis. Therefore, 60°C was chosen as the optimum reaction temperature for alkali catalyst esterification.

The prepared biodiesel was identified by FT-IR Analysis. The spectra are shown in Figure (2). The functional groups in the compounds contained in prepared biodiesel are interpreted in Table (12). A strong signal was observed between wave number 1500  $\text{cm}^{-1}$  and 2000  $\text{cm}^{-1}$ . It was confirmed that the prepared biodiesel contained fatty acid methyl esters group. A group of carboxylic acid compounds was investigated between 2858.87  $\text{cm}^{-1}$  and 2926.11  $\text{cm}^{-1}$ . Aliphatic chloro and fluoro compounds were also found between 723  $\text{cm}^{-1}$  and 1170.83  $\text{cm}^{-1}$ . The comparison of fuel properties was conducted between prepared biodiesel with ASTM standard and it is described in Table (13). The results show that the properties of prepared biodiesel mostly agree with ASTM standard.

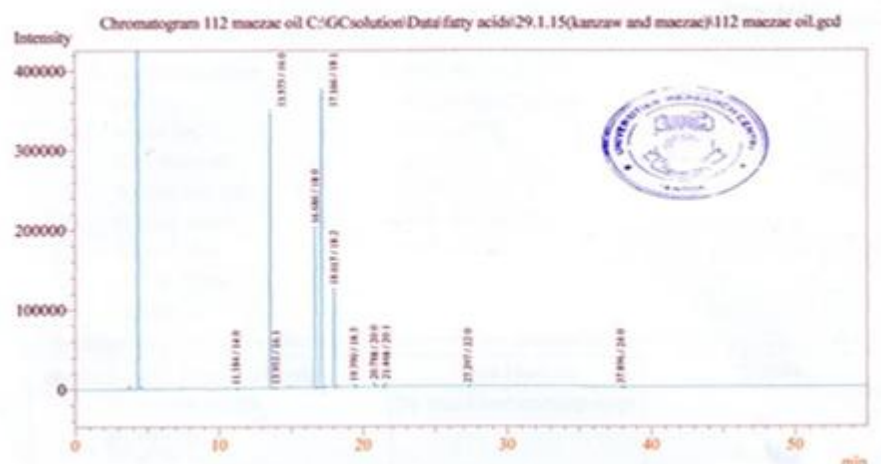


Meze Seed Oil by expeller    Biodiesel prepared from Meze Seed Oil

**Table 1 Physico-Chemical Properties of Meze Seed Kernels(Myanmar)**

Sr. no.	Characteristics	Meze seed kernel	Literature values *
1.	Moisture content (w/w%)	4.18	7.8
2.	Ash content (w/w%)	1.64	3.4
3.	Protein (w/w%)	5.90	16.9
4.	Crude fiber (%)	8.86	3.2
5.	Crude fat (%)	53.71	46.7
6.	Carbohydrate (%)	25.71	22

\* Mishra. & Padhan (2013)



**Figure 1** Fatty Acid Composition of Extracted Meze Seed Oil

**Table 2** Fatty Acid Composition of Expelled Meze Seed Oil by GC Analysis

Fatty acids composition analyzed by GC (%w/w)	Experimental value	Literature value *
C 12:0 (Lauric)	Not detected	-
C 14:0 (Myristic)	0.07	-
C 14:1 (Myristoleic)	Not detected	-
C 16:0 (Palmitic)	25.91	24.5
C 16:1 (Oleo-Palmitic)	0.06	-
C 18:0 (Stearic)	19.33	22.7
C 18:1 (Oleic)	43.72	37
C 18:2 (Linoleic)	9.82	14.3
C 18:3 (Linolenic)	0.28	-
C 20:0 (Arachidic)	0.53	-
C 20:1 (paullinic)	0.13	-
C 22:0 (Behenic)	0.05	-
C 22:1 (Erucic)	Not detected	-
C 24:0 (Lignoceric)	0.1	-

(\*) Mishra. & Padhan, 2013.

**Table 3 Physico-chemical Properties of Expelled Meze Seed Oil**

Sr.no.	Characteristics	Expelled Meze seed oil	Literature value *
1.	Refractive index	1.465	1.452-1.462
2.	Specific gravity	0.9089	0.862-0.875
3.	Colour	2.3R, 1.5B, 20Y	Pale Yellow
4.	Moisture (loss on drying,%)	0.046	-
5.	Acid value (mg KOH per g)	8.27	0.5-20
6.	Iodine value (mgI <sub>2</sub> /g)	53.170	58-70
7.	Saponification value (mg KOH per g)	195.499	187-196
8.	Unsaponifiable matter (%)	0.984	1-3
9.	Peroxide value (milliequivalents oxygen per kg)	9.369	-
10.	Kinematic viscosity (Cst) * *	51.8	25
11.	Flash point (°C) * *	204	226
12.	Pour point (°C) * *	15	15

(\*) Mishra and Padhan (2013), (\* \*) ( Padhi, 2010)

**Table 4: Effect of Volume of Methanol on Reduction of Acid Value (AV) of Esterified Meze Seed Oil Using Acid Catalyst**

Sr. no.	Volume of methanol (mL)	AV of Meze seed oil after reaction (mg KOH/g of oil)	(%) Reduction of acid value of esterified Meze oil
1	100	3.38±0.03	59.17±0.35
2	200	3.01±0.03	63.64±0.25
3	300	2.53±0.02	69.45±0.25
4	400*	2.37±0.03	71.34±0.10
5	500	2.35±0.04	71.54±0.45

(\*) Most suitable condition

**Table 5 Effect of Volume of Acid Catalyst on Reduction of Acid Value (AV) of Esterified Meze Seed Oil**

Sr. no.	Volume of catalyst (H <sub>2</sub> SO <sub>4</sub> ) (mL)	AV of Meze seed oil after reaction (mg KOH/g of oil)	(%) Reduction of acid value of esterified Meze seed oil
1	0.5	3.25±0.09	60.7±1.12
2	1.0	2.76±0.1	66.71±1.19
3	1.5 *	2.37±0.06	71.34±0.71
4	2.0	2.25±0.02	72.83±0.21
5	2.5	2.05±0.03	75.17±0.37

(\*) Most suitable condition

**Table 6 Effect of Reaction Time on Reduction of Acid Value (AV) of Esterified Meze Seed Oil Using Acid Catalyst**

Sr. no.	Reaction time (min)	A V of Meze seed oil after reaction (mg KOH/g of oil)	(%) Reduction of acid value of esterified Meze seed oil
1	45	3.9±0.06	52.84±0.78
2	60	3.3±0.07	60.10±0.84
3	75	2.97±0.07	64.09±0.89
4	90 *	2.37±0.08	71.34±0.99
5	105	2.36±0.04	71.46±0.49

(\*) Most suitable condition

**Table 7 Effect of Reaction Temperature on Reduction of Acid Value (A.V) of Esterified Meze Seed Oil Using Acid Catalyst**

Sr.no.	Reaction temperature (°C)	A V of Meze seed oil after reaction (mg KOH/g of oil)	(%) Reduction of acid value of esterified Meze seed oil
1	40	5.22±0.06	36.84±0.67
2	50	4.35±0.03	47.4±0.39
3	60 *	2.37±0.04	71.34±0.20
4	70	2.36±0.07	71.46±0.89
5	80	2.36±0.05	71.42±0.64

(\*) Most suitable condition

**Table 8 Effect of Volume of Methanol on Yield of Biodiesel Prepared from Esterified Meze Seed Oil in Transesterification**

Sr.no.	Volume of methanol (mL)	Volume of biodiesel (mL)	Yield (% v/v)
1	40	50.2±0.08	42.47±0.21
2	50*	65.5±0.17	55.55±0.16
3	60	65.8±0.12	55.8±0.11
4	70	66.3±0.22	56.23±0.11
5	80	66.8±0.12	56.66±0.19

(\*) Most suitable condition

**Table 9 Effect of Weight of Alkali Catalyst on Yield of Biodiesel Prepared from Esterified Meze Seed Oil in Transesterification**

Sr.no.	Weight of catalyst (KOH) (g)	Volume of biodiesel (mL)	Yield (% v/v)
1	0.4	62±0.45	52.58±0.09
2	0.6	62.3±0.12	52.84±0.08
3	0.8	63.5±0.22	53.86±0.07
4	1 *	65.5±0.25	55.55±0.05
5	1.2	64.4±0.02	54.62±0.07

(\*) Most suitable condition



**Table 10 Effect of Reaction Time on Yield of Biodiesel Prepared from Esterified Meze Seed Oil in Transesterification**

Sr.no.	Reaction time (min)	Volume of biodiesel (mL)	Yield (% v/v)
1	45	59.8±0.8	50.72±0.02
2	60	62.2±0.7	52.75±0.07
3	75	63±0.26	53.43±0.02
4	90 *	65.5±0.39	55.55±0.22
5	105	64.5±0.16	54.70±0.02

(\*) Most suitable condition

**Table 11 Effect of Reaction Temperature on Yield of Biodiesel Prepared from Esterified Meze Seed Oil in Transesterification**

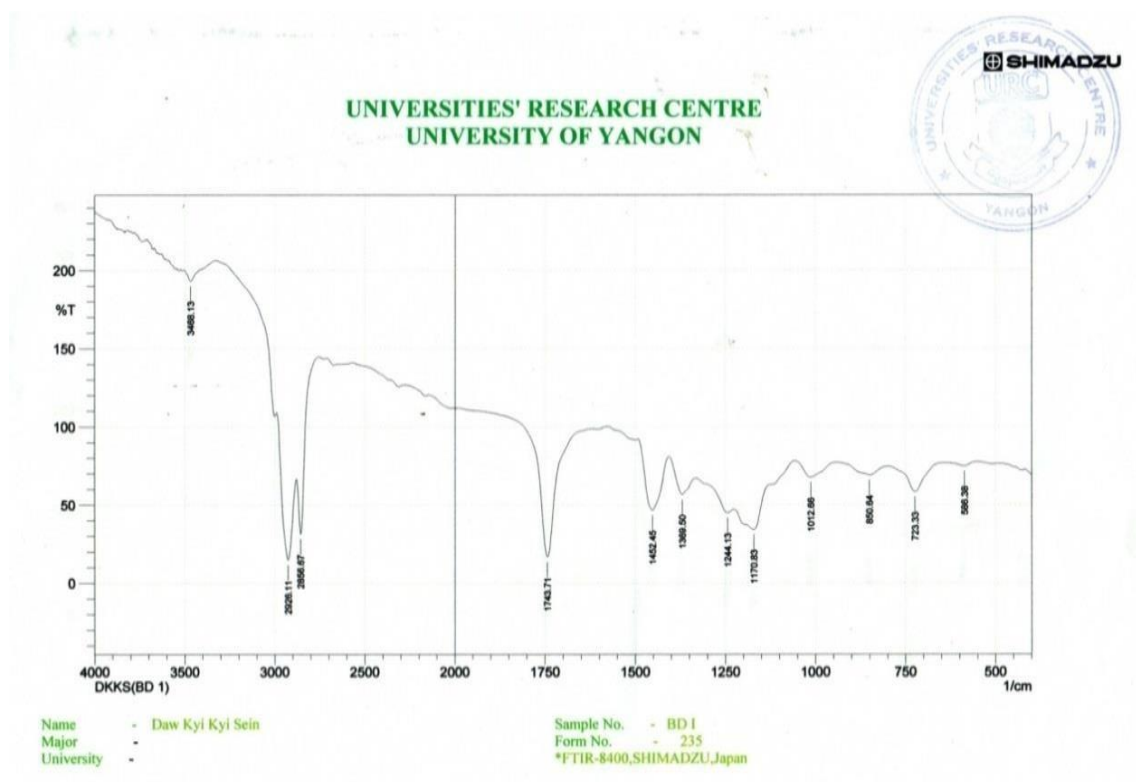
Sr.no.	Reaction temperature (°C)	Volume of biodiesel (mL)	Yield (% v/v)
1	40	55.7±0.16	47.24±0.02
2	50	62.3±0.12	52.84±0.08
3	60 *	65.5±0.16	55.55±0.05
4	70	65.8±0.33	55.81±0.12
5	80	60.2±0.14	51.06±0.09

(\*) Most suitable condition

**Table 12 FT-IR Spectra Data of Prepared Biodiesel**

Wave number, cm <sup>-1</sup>		Functional group	
Observed	Literature *		
3468.13	3500-3300	$\nu$ – NH	Stretching vibration of amines groups
2926.11 2856.67	< 3000	$\nu$ – CH	Stretching vibration of alkane group
1743.71	~1700	$\nu$ – C=O	Stretching vibration of carboxylic acid
1452.45	> 1400	$\delta_{as}$ - CH	CH asymmetric bending vibration
1369.50	< 1400	$\delta_s$ - CH	CH symmetric bending vibration
1170.83 1012.66	1260-1000	$\nu$ – C-O	Stretching vibration of alcohol and phenol group
850.64 723.33	900-700	$\nu$ – CH	Stretching vibration of alkene group

\* Mohan(2000)



**Figure 2** FT-IR Spectra of Prepared Biodiesel

**Table 13** Fuel Properties of Meze Seed Oil Biodiesel (MSOB)

Sr.no.	Property	MSOB*	ASTM (D 6751)
1	Flash point (°C)	226	130 minium
2	Pour point (°C)	12	Not available
3	Colour	< 2	-
4	Kinematic viscosity at 40°C (Cst)	5.4	1.9-6.0
5	Specific gravity at 30°C	0.8773	0.88
6	Cetane number	72.91	47 minium
7	Estimated heat of combustion (cal/g)	9442.228	-
8	Copper strip corrosion test	1a	Maximum-3
9	Saponification value (mg KOH/g)	205.08	-
10	Iodine value (mg I <sub>2</sub> /g)	61.032	-

1= slight tarnish ,      a= light orange, almost the same as fresh,      3 = dark tarnish

### Conclusion

In the preparation of biodiesel, a two stage transesterification process was selected to convert high free fatty acid (FFA) oil to its methyl ester. In the first stage of pretreatment process, the FFA content of Meze seed oil could be reduced to less than 2% using acid catalyst (1.5 % v/w H<sub>2</sub>SO<sub>4</sub>) reacting with methanol to oil ratio of 4:1 at 60°C and 90 min reaction time. Excess addition of sulphuric acid darkens the product. The first stage product having acid value less than 2% of FFA content was used for the second stage alkali catalyzed (1% w/w KOH) transesterification reaction. Methanol to oil ratio of (1:2 %v/w) favoured the completion of the reaction in 90 min at 60°C. The process gave a yield of 55.5% of biodiesel, which had comparable fuel properties with that of ASTM standard.

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