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## Mango (*Magnifera indica*) seed oil grown in Dilla town as potential raw material for biodiesel production using NaOH- a homogeneous catalyst

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### ABSTRACT

Biodiesel produced by transesterification process from vegetable oils or animal fats is viewed as a promising renewable energy source. Now a day's diminishing of petroleum reserves in the ground and increasing environmental pollution prevention and regulations have made searching for renewable oxygenated energy sources from biomasses. Biodiesel is non-toxic, renewable, biodegradable, environmentally benign, energy efficient and diesel substituent fuel used in diesel engine which contributes minimal amount of global warming gases such as CO, CO<sub>2</sub>, SO<sub>2</sub>, NO<sub>x</sub>, unburned hydrocarbons, and particulate matters. The chemical composition of the biodiesel was examined by help of GC-MS and five fatty acid methyl esters such as methyl palmitate, methyl stearate, methyl oleate, methyl linoleate and methyl linolenate were identified. The variables that affect the amount of biodiesel such as methanol/oil molar ratio, mass weight of catalyst and temperature were studied. In addition to this the physicochemical properties of the biodiesel such as (density, kinematic viscosity, iodine value high heating value, flash point, acidic value, saponification value, carbon residue, peroxide value and ester content) were determined and its corresponding values were 87 Kg/m<sup>3</sup>, 5.63 Mm<sup>2</sup>/s, 39.56 g l/100g oil, 42.22 MJ/Kg, 132°C, 0.12 mgKOH/g, 209.72 mgKOH/g, 0.04%wt, 12.63 meq/kg, and 92.67 wt% respectively. The results of the present study showed that all physicochemical properties lie within the ASTM and EN biodiesel standards. Therefore, mango seed oil methyl ester could be used as an alternative to diesel engine.

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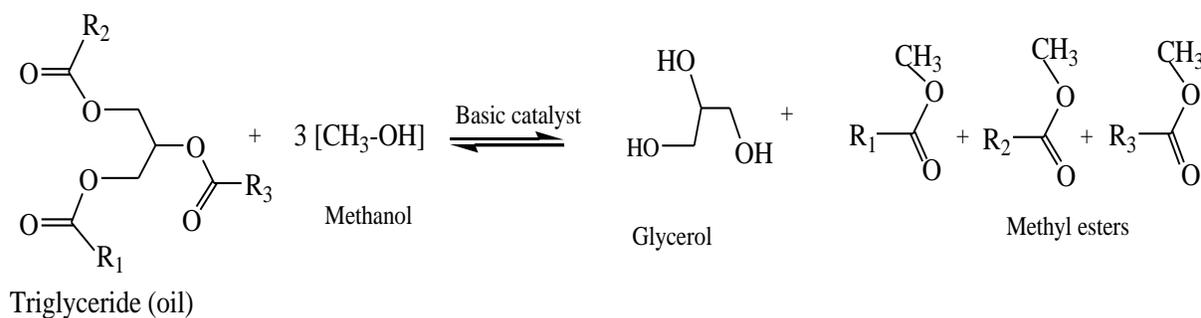
**Capsule Summary:** The biodiesel production efficiency from mango seed oil was evaluated. methylpalmitate, methyl stearate, methyl oleate, methyl linoleate and methyl linolenate were the main components of the biodiesel and mango seed oil methyl ester could be used as an alternative energy resource in diesel engine.

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### INTRODUCTION

Increasing energy demands, depletion of fossil fuels and environmental pollution make the world under crises now a day's. This is because of many countries worldwide are still

heavily dependent on petroleum as their main source of electricity and transportation fuel. The only possible solution to solve this crisis is to find a sustainable (renewable), economically feasible and environmentally friendly source of alternative energy is necessary. There are many alternative energy sources such as hydropower, wind, solar, geothermal



**Fig. 1:** base catalyzed transesterification processes

**Table 1:** Experimental treatments

| Methanol to oil molar ratio( $T_1$ ) | Temperature in °C ( $T_2$ ) | %w/w of NaOH to oil ( $T_3$ ) |
|--------------------------------------|-----------------------------|-------------------------------|
| 3:1                                  | 45, 55, 60 and 65           | 0.5                           |
| 6:1                                  |                             | 1                             |
| 9:1                                  |                             | 1.5                           |

Where: T = treatment

and biomass. Biodiesel fuel is a renewable energy resource which is made from vegetable oils available around us (Kondamudi et al., 2008; Lebedevas et al., 2006; Nebel and Mittelbach, 2006).

Mango seed (*Mangifera indica*) is a waste where so many people are throwing away after using the fruit flesh. It is one of the most popular fruit in gedeo zones as a result there is a significant rise in mango fruit consumption and consequently an increase in the mango seed waste generation. Therefore, alternative routes are needed for this waste management.

This waste can be used for various applications. The presence of nitrogen allows it to be directly used as fertilizer or as soil improver (or compost) (Lebedevas et al., 2008). On the other hand, waste mango seed have oil content in the order of 9-13wt% depending upon its varieties which very less to compare other oil seeds. The oil is high in unsaturated fatty acid such as oleic acid with 46.22 % (Nzikou et al., 2010).

Biodiesel is a clean, renewable, biodegradable, environmentally benign, energy efficient and diesel substituent fuel used in diesel engine. It is a carbon neutral fuel because there is no overall increase in CO<sub>2</sub> in the atmosphere due to recycling by the growing plants used to feed the biodiesel industry (Lebedevas et al., 2008; Ma and Hanna, 1999). Emissions of SO<sub>2</sub>, SO<sub>3</sub>, CO, unburnt hydrocarbons and particulate matter are lower than that of petroleum diesel (Coronado et al., 2009; Ma and Hanna, 1999). The most common process used to produce biodiesel is through transesterification, a reaction between triglycerides and an alcohol with a low molecular weight (ethanol or methanol) in the presence of a basic catalyst

(NaOH or KOH), to obtain esters and glycerol (Knothe et al., 2006; Mata et al., 2010 and 2011). Transesterification is a three-step reversible reaction of vegetable oils or animal fats with a methanol to form fatty acid methyl esters (FAMES) and glycerol as a final product (Morais et al., 2010).

The reaction mechanism for the formation of fatty acid methyl esters (FAME) is described as follows (Fig. 2).

## MATERIAL AND METHODS

### Chemicals and reagents used

Methanol (99%), sodium hydroxide, n-hexane, sodium thiosulfate, phenolphthalein, ethanol (96%), anhydrous sodium sulphate and mango seed oil.

### Instrumentations

Soxhlet apparatus, Rotary evaporator, GC-MS, bomb calorimeter, viscometer, hot plate, thermometer, round bottom flask, separatory funnel and grinder were used.

### Experimental design

A basic catalysts (NaOH) was used to study at which methanol/oil molar ratio, mass weight of catalyst and temperature an optimum biodiesel is produced from the oils of waste mango seed oil. Finally the physicochemical properties of the biodiesel were determined and compared with a set of parameter according to European Standard, EN 14214 and American standard test and material, ASTM6751.

### Seed material preparation

Undamaged thrown mango seeds were collected from Dilla town, cleaned and oven dried at 105°C for 1 hour to remove

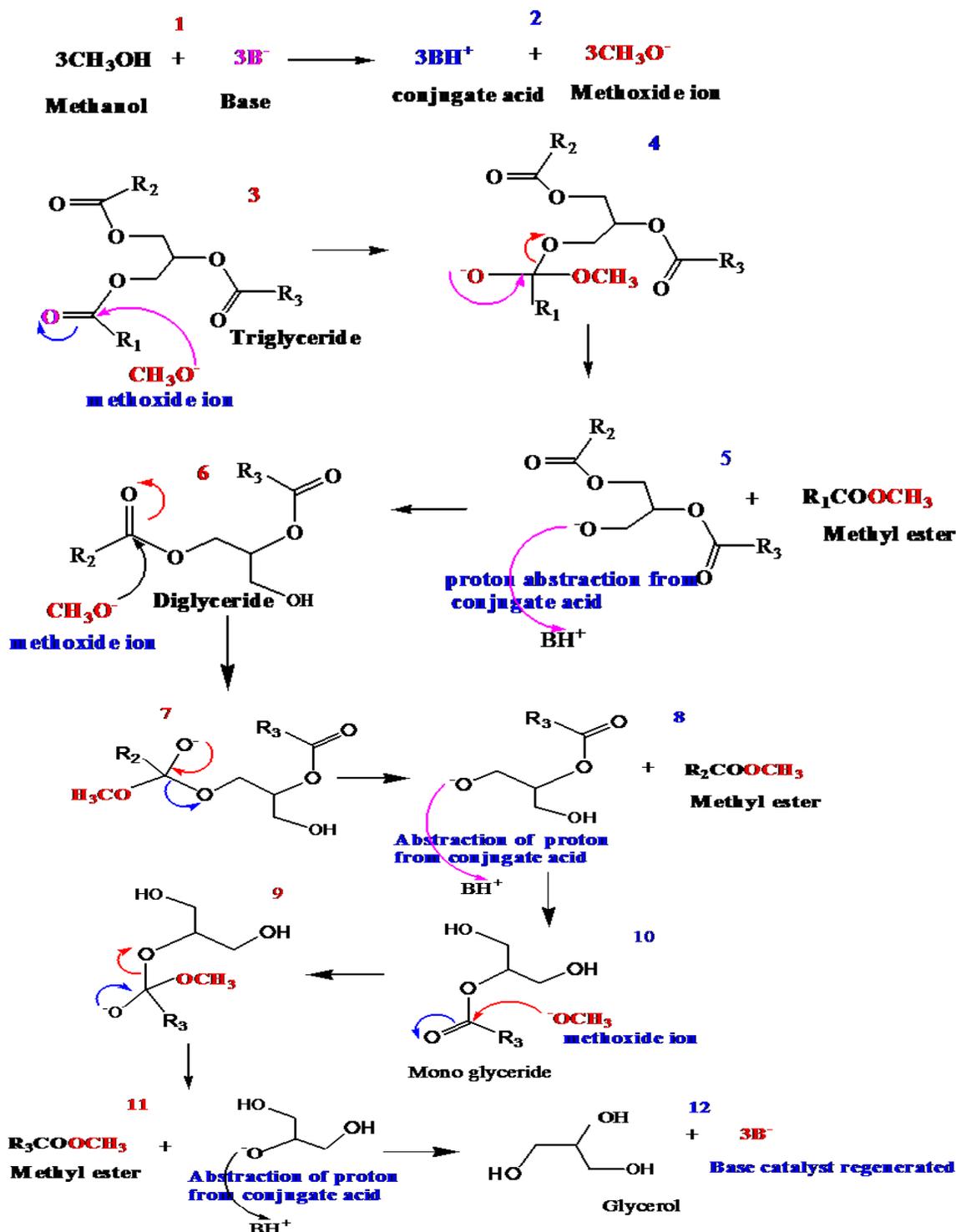


Fig. 1: Reaction mechanism for base catalyzed transesterification

the water content. After that the dried mango seeds were grounded using grinder and stored in sealed plastic bag for further use.

#### Oil extraction

Oven dried mango seeds were grounded in to powder by using grinder. After that 100g of the sample were loaded in to thimble in Soxhlet apparatus. Next to that extraction was carried out using 500 ml normal hexane at 68°C (boiling temperature of hexane) for 12 hours in an electrical heater. The mixture of the extracted oil and the hexane was

**Table 2:** Physiochemical properties of biodiesel prepared from mango seed oil, Petroleum diesel and biodiesel Standards (ASTM D6751& EN 14214)

| Fuel Properties             | Biodiesel | Standards(USA&EU) |           | Petroleum diesel. | unit                    |
|-----------------------------|-----------|-------------------|-----------|-------------------|-------------------------|
|                             |           | ASTM D6751        | EN 14214  |                   |                         |
| Specific gravity            | 0.87      | 0.88              |           | 0.845             | -----                   |
| Density at 15°C             | 87        | -----             | 860 – 900 | 845               | <i>Kg/m<sup>3</sup></i> |
| Kinematic viscosity at 40°C | 5.63      | 1.9 -6.0          | 3.5 - 5.0 | 2.86              | <i>Mm<sup>2</sup>/s</i> |
| Acid value                  | 0.12      | ≤ 0.8             | ≤ 0.5     | -----             | mgKOH/g                 |
| Saponification value        | 209.72    | -----             | -----     | -----             | mgKOH/g                 |
| Flash point                 | 132       | ≥ 120             | ≥ 130     | 67.5              | °C                      |
| Higher heating value        | 42.22     | -----             | -----     | 45.5              | MJ/Kg                   |
| Peroxide value              | 12.63     | -----             | -----     | -----             | meq/kg                  |
| Iodine value                | 39.56     | -----             | ≤ 120     | -----             | g I/100g oil            |
| Carbon residue              | 0.04      | 0.05 max          | -----     | 0.15Max           | %wt                     |
| Ester content               | 92.67     | -----             | ≥ 96.5    | -----             | -----                   |

**Table 3:** Methyl ester content, as function of mass weight of catalyst at methanol/oil molar ratio, 3:1, 6:1 and 9:1; reaction temperature, 60 °C; reaction time of 3 hours

| Methanol/oil molar ratio, | Mass weight of catalyst, wt. % | Temperature in °C | Methyl ester content, w/w% |
|---------------------------|--------------------------------|-------------------|----------------------------|
|                           |                                |                   | Dilla Town                 |
| 3:1                       | 1%wt                           | 60                | 84.65                      |
| 6:1                       | NaOH                           | 60                | 92.67                      |
| 9:1                       |                                | 60                | 89.57                      |

**Table 4:** Methyl ester content, as function of mass weight of catalyst at methanol/oil molar ratio,6:1; reaction temperature, 45 °C, 55 °C,60 °C and 65 °C; reaction time of 3 hours

| Methanol/oil molar ratio, | Mass weight of catalyst, wt. % | Temperature in °C | Methyl ester content, w/w% |
|---------------------------|--------------------------------|-------------------|----------------------------|
|                           |                                |                   | Dilla Town                 |
| 6:1                       | 1%wt                           | 45                | 88.78                      |
| 6:1                       | NaOH                           | 55                | 91.83                      |
| 6:1                       |                                | 60                | 92.67                      |
| 6:1                       |                                | 65                | 86.45                      |

separated by vacuum rotary evaporator (RE-52A, 220 V/50Hz) machine and the percentage of the oil was calculated by using the formula: % Oil = Mass of Oil x 100/ sample

## RESULTS AND DISCUSSION

The biodiesel characterization shows similarities to that of fossil diesel. The Physiochemical properties of the biodiesel were determined. Several tests of the properties of biodiesel such as specific gravity, density, kinematic viscosity, iodine value, high heating value, flash point, acidic value, saponification value, carbon residue, peroxide value and ester content profile were determined and all the result were lie within the ASTM and EN standards except the percentage

amount of ester produced (92.67%) which is less by 3.87% from its minimum value (96.5%) in EN standard as shown in Table 2.

Kinematic viscosity is a measure of resistance of fluid to flow under the influence of gravity (Gerpen et al., 2004). The result from this work shows a kinematic viscosity of 5.63 which is quite in agreement with the ASTM biodiesel standard but it is slightly higher than the European Standard and Petroleum diesel. Viscosity of a fuel is related to the fuel lubricity. Low viscosity fuels are unlikely to provide satisfactory lubrication in fuel injection pumps; these often lead to seepage and increase in wear (Raja et al., 2011). High viscosity in fuel are responsible for atomization of fuel, incomplete combustion and increased exhaust emissions, choking of the injections thereby forming larger droplets on

injector, ring carbonization and accumulation of the fuel in the engine (Wang et al., 2006).

Specific gravity of the fuel is very important in diesel engine because fuel injection system operates on a volume metering basis. The values of specific gravity obtained for mango seed oil methyl ester was 0.87 with a corresponding density value of 87kg/m<sup>3</sup>. This value lies within the ASTM standards and also in close proximity to the findings of other studies. The carbon residue is an indicator of residual carbon after combustion. The carbon residue for this study is measured to be 0.04 % wt which is less than the maximum value of ASTM standard (0.05% wt). (Gerpen *et al.*, 2004) reported that the major cause of surplus carbon residue in biodiesel is due excessive total glycerin present in it. The low value obtained in this study is due the complete separation and effective removal of glycerol after the transesterification process.

Flash point is the minimum temperature at which a fuel must be heated for it to ignite air -vapor mixture. The U.S.Department of Transportation specified 90 °C as the flash point for non-hazardous fuel (Kenneth and Bruce. 2010). The flash point for this work is 132°C. This result shows appreciable consistency with both ASTM, EN standard for biodiesel and works of other researchers. The high value obtained in this study clearly signifies that the biodiesel produced is basically free from methanol; this is because even small quantity of methanol can reduce the flash point reasonably and also negatively affects diesel engine parts such as fuel pumps, seals and elastomers. The acid value obtained in this work is very small (0.12) to compare with the ASTM and EN standard ( $\leq 0.8$  and  $\leq 0.5$ ) respectively and reported work of (Enweremadu and Alamu., 2010). Acid value measures directly the free fatty acids content of the methyl ester. It clearly helps to state the corrosive nature of the fuel, its filter clogging tendency and the amount of water that may be likely present in the biodiesel. This parameter can also be used to measure the freshness of the biodiesel. The higher the acid value the lower the quality of the fuel. Generally from the physiochemical properties of the biodiesel produced from mango seed oil table 2 above it can be used as diesel substituent fuel.

#### **Effect of different variables in the amount of biodiesel produced**

Several variables which affect the yield of FAMES were studied. These are mass weight of catalyst, methanol to oil molar ratio and temperature. Their effect of each variable was shown in Tables 3-5.

#### **Effect of variation of methanol/oil molar ratio in the amount of biodiesel produced**

The amount of methyl ester in the transesterification process activity depends on the molar concentrations of methanol to oil (Table 3). Large excess methanol is required to shift the equilibrium favorably during transesterification for better yields of biodiesel (Sree et al., 2009). When the methanol/oil molar ratio was increased from 3:1 to 6:1, an increase in

methyl ester content was observed but further increasing methanol/oil molar ratio from 6:1 to 9:1 a decrease in methyl esters content was observed. This is because the higher alcohol molar ratio interferes with the separation of glycerol from the biodiesel due to an increase solubility of glycerol in the alcohol (Table 3).

#### **Effect of variation temperature in the amount of biodiesel produced**

Temperature plays an important role during biodiesel production; this is because the rate of reaction is strongly influenced by the reaction temperature (Devanesan et al., 2007, Bajpai and Tyagi., 2006). Table 4 above shows the amount of biodiesel varies with temperature variation from 45°C to 65°C at a catalyst concentration of 1 % wt. As the temperature increase from 45 - 60 °C the conversion yields of biodiesel also increases considerably. Further increase in temperature results in decrease in the yield of biodiesel. Literatures have reported that alkaline transesterification are conducted close to the boiling point of the alcohol used and that temperature higher than this burns the alcohol resulting into lower yield. Patil and Deng (Patil and Deng, 2009), reported that alkaline transesterification at temperature above 60 °C cause excessive methanol loss due to evaporation and significantly reduce overall biodiesel yield. And that saponification of glycerides by alkali catalyst is much faster than the transesterification reaction above 60°C. This may be another plausible reason for the observed low conversion at 65°C.

#### **Effect of variation of mass weight of catalyst in the amount of biodiesel produced**

From the result obtained as shown in table 5 above, as the mass weight of catalyst increase from 0.5 to 1 %wt a progressive increase in percentage conversion in the reaction was achieved and thereafter experienced a decrease in the yield above this concentration (1 % wt of NaOH). It was obvious that increase in catalyst concentration beyond 1 % wt of NaOH results in a decrease in biodiesel yield. Hence the yield at 1.5 % wt (85.92%) was lower than the yield obtained at 0.5%wt (89.87%). This can be clearly explained by the reversible nature of transesterification reaction. The findings from this studies is very consistent with opinion of Darnoko and Munnir 2000), who reported that catalyst concentration greater than 1 % wt may have favored the backward reaction, thereby shifting the equilibrium from the right to the left, hence the formation of glycerol. In comparing to results of this findings with that of previous researchers such as (Tint and Mya, 2009) for *Jatropha Curcas* oil and (Oghome and Ibe, 2009) for Tiger nut oil as biodiesel resource showed efficient performance.

#### **GC-MS analysis of biodiesel prepared from waste mango seed oil**

Based on GC-MS analysis, the FAME of biodiesel prepared from waste mango seed oils grown in Dilla town, five compounds were identified as described in detail in Table 6.

**Table 5:** Methyl ester content, as function of mass weight of catalyst at methanol/oil molar ratio, 6:1; reaction temperature, 60 °C; reaction time of 3 hours

| Methanol/oil molar ratio, | Mass weight of catalyst, wt. % | Temperature in °C | Methyl ester content, w/w% Dilla Town |
|---------------------------|--------------------------------|-------------------|---------------------------------------|
| 6:1                       | 0.5                            | 60                | 89.87                                 |
| 6:1                       | 1                              | 60                | 92.67                                 |
| 6:1                       | 1.5                            | 60                | 85.92                                 |

**Table 6:** Chemical composition of FAMES of waste mango seed oil by GC-MS analysis

| Fatty acid methyl ester              | Position of double bond in FAMES | Molecular mass | Amount (%) |
|--------------------------------------|----------------------------------|----------------|------------|
| Methyl palmitate                     | ----                             | 270            | 9.72       |
| Methyl linolenate                    | 18:3, Δ 6,9,12                   | 292            | 37.45      |
| Methyl linoleate                     | 18:2, Δ 9,12                     | 294            | 44.10      |
| Methyl oleate                        | 18:1, Δ 9                        | 296            | 6.79       |
| Methyl stearate                      | -----                            | 298            | 1.94       |
| Saturated fatty acid methyl esters   | -----                            | ----           | 47.17      |
| Unsaturated fatty acid methyl esters | -----                            | -----          | 52.83      |

### Analysis of mass fragmentation of fatty acid methyl esters prepared from mango seed oil

Based on the GC-MS analysis on chemical composition of the biodiesel produced from mango seed oil grown in Dilla town, five fatty acid methyl esters such as methyl palmitate, methyl stearate, methyl oleate, methyl linoleate and methyl linolenate were identified. The molecular ion (parent) peaks of methyl palmitate, methyl linolenate, methyl linoleate, methyl oleate and methyl stearate were observed at 270, 292, 294, 296 and 298 respectively. The saturated FAMES detected in the biodiesel from mango seed oil (methyl palmitate and methyl stearate) show  $\text{CH}_3\text{OC}(=\text{OH}^+)\text{CH}_2$  fragment and appears at  $m/z = 74$  as the base peak (100%) which is the result of McLafferty rearrangement due to the formation of a six member ring structure of an intermediate. In the lower molecular weight region of methyl linolenate, hydrocarbon ions with a general formula of  $[\text{C}_n\text{H}_{2n-5}]^+$  were dominated with the ion at  $m/z = 79$  as the base peak.

Methyl linoleate shows  $[\text{CH}_2=\text{CHCH}=\text{CHCH}_2]^+$  fragment which appears at  $m/z = 67$  as the base peak (100%). Methyl oleate shows  $[\text{CH}_2=\text{CHCH}_2\text{CH}_2]^+$  fragment which appears at  $m/z = 55$  as the base peak (100%).

The methyl palmitate base peak ion at  $m/z = 74$  undergoes McLafferty rearrangement losing the methyl ester which is fragmented between the  $\alpha$  and  $\beta$  substituted carbons while the ion at  $m/z = 87$  is fragmented between C4 - C5 also losing methyl ester and a hydrogen atom. An ion with  $m/z = 57$  is fragmented between C3 and C4 losing a methylene diol and three hydrogen atoms via McLafferty rearrangement. methyl linolenate molecular ion occurs at  $m/z = 79$  and other dominant peaks in the molecule such as  $m/z = 93, 107, 121$  and  $135$  are due to fragmentation of the molecule hydrocarbon ions with a general formula of  $[\text{C}_n\text{H}_{2n-5}]^+$ . Methyl linoleate's molecular ion occurs at  $294 m/z$ . Both ions

at  $m/z = 67$  and  $81$  represent hydrocarbon fragments with general formula  $[\text{C}_n\text{H}_{2n-3}]$  losing dialkenes and a hydrogen atom. Methyl oleate's parent peak is observed at  $m/z = 296$ . The peak at  $m/z = 74$  represents the rearranged McLafferty methyl ester fragment while the peak at  $m/z = 87$  represents fragmented hydrocarbon ions with general formula  $[\text{CH}_3\text{OCO}(\text{CH}_2)_n]$ . Methyl stearate's molecular ion occurs at  $m/z = 298$ . The ion present at  $m/z = 74$  corresponds to the McLafferty rearranged methyl ester fragmented between the  $\alpha$  and  $\beta$  carbons while the ion at  $m/z = 87$  represents loss of methyl ester and a hydrogen atom fragmented between C3-C4 respectively. Molecular ion at  $m/z = 67$  and  $81$  represent hydrocarbon fragments with general formula  $[\text{C}_n\text{H}_{2n-3}]$  due to loss of alkenes and a hydrogen atom. Based on environmental pollution issues (Bhatti et al., 2016; Iqbal et al., 2013; Iqbal, 2016; Iqbal et al., 2015; Iqbal and Bhatti, 2015; Iqbal et al., 2016; Iqbal and Nisar, 2015; Iqbal et al., 2017; Manzoor et al., 2013; Mushtaq et al., 2016; Nadeem et al., 2016; Nisar et al., 2016; Nouren et al., 2017; Rashid et al., 2016; Shoukat et al., 2017; Tahir et al., 2016a; Tahir et al., 2016b; Ullah et al., 2013) and energy needs, there is need to use renewable energy sources and mango seed oil found to be efficient for the production of biodiesel.

### CONCLUSIONS

Biodiesel from mango seed oil is obtained by transesterification process using sodium hydroxide as catalyst. Optimum amount of methyl ester content (92.67%) was obtained at 60°C with methanol to oil molar ratio 6:1 and at 1%wt of NaOH. The physicochemical properties of the biodiesel were determined and compared ASTM and EN biodiesel standards and fulfills both properties. Therefore, mango seed oil methyl ester could be used as an alternative

energy resource to diesel engine. Engine test and further researches are needed to be conducted to determine the amount of biodiesel produced with heterogeneous catalysts and lipase enzymes. The future prospects of this study gives direction for those investors that wants to invest in biodiesel production industry and as a reference material for other researchers.

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