



OPTIMIZATION OF BASED CATALYSED  
TRANSESTERIFICATION OF YELLOW OLEANDER  
(*Thevetiaperuviana*) OIL



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**Abstract:** Optimized production and direct effects of process variables on the production and quality of methyl ester biodiesel fuels from the non-edible seed oils of yellow oleander seed was carried out. Catalyst nature and concentration, oil to methanol volume ratio, reaction time and temperature were taken into consideration as variables to their individual response on the yields of the methyl esters produced. Optimized concentrations were 0.225 to 1.125% wt catalysts, and oil to methanol volume ratios from 5:1 to 5:2. Reaction time was varied (30 to 150 min) with temperatures range (30 to 70°C). It was found that, the highest Fatty Acid Methyl Ester (FAME) yield of 70 % was achieved at 120 min reaction time, with oil to methanol mass ratio of 5:1, catalyst (KOH) amount of 0.9 g and reaction temperature of 60°C.

**Keywords:** B iodiesel, FAME, Free fatty acid, Oleander seeds, transesterification.

### Introduction

Today, the world is confronted with two major problems, energy crisis and degrading environment (Betiku *et al.*, 2014). The major reason is excessive use of fossil fuels. So it has become a necessity to search for alternative fuels, which promises to solve both of these problem and offer sustainability to these problem as automobiles are one of the major factor toward the consumption of petroleum based fuels and also major source of greenhouse emissions. Wastes, and combustible renewable material have the highest potentials to supply the global energy requirement and they accounted for more than three quarters of the global energy supply in 2007 and 2008 (Betiku *et al.*, 2014). The current trend focuses on search for alternate fuel for replacement of fossil fuels due to the increase in the price and environmental concerns about air pollution.

With the advancement in technology and increasing industrialization, there has been an increase in energy

demand. This energy requirement is primarily met by petroleum based fuels, which being non-renewable in nature necessitates finding alternative fuels which are renewable. Biodiesel is one of the alternatives which are renewable and environmentally friendly. Biodiesel is a biofuel, produced from vegetable oils/fats through a process known as transesterification (Narwal and Sharma, 2014). Transesterification is the process of interchanging the organic alkyl group of an ester with alkyl group of an alcohol (Jain and Sharma, 2010) and this is done by adding methanol to the triglyceride, this removes the glycerol backbone and replaces it with a smaller methyl group, and thus, splitting part of the large molecules, methyl esters and glycerol are left as separate products as shown in the equation below, where R<sub>1</sub>, R<sub>2</sub> and R<sub>3</sub> are long hydrocarbon chains called fatty acid.

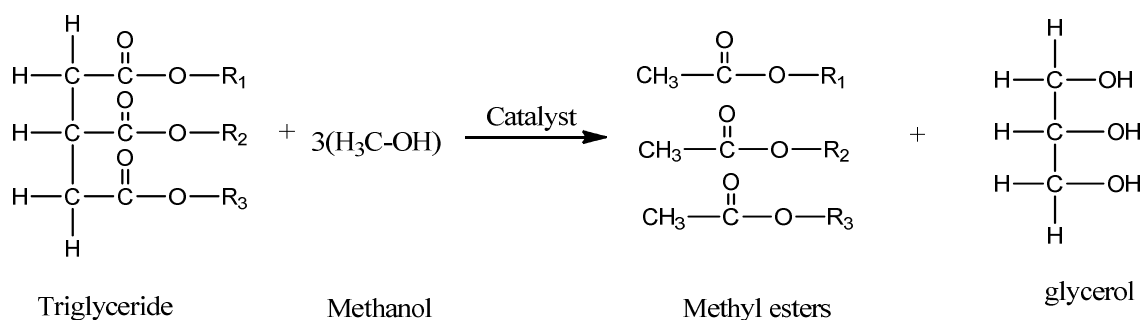


Fig. 1: Transesterification equation (Jain and Sharma, 2010)

Biodiesel possess many environmental benefits over petroleum diesel fuel. It also reduces petroleum dependency and enhances energy security (Ramadhas, 2005). The risk associated with handling, storing and transporting are lower due to its higher flash point and higher biodegradability (Ramadhas, 2005). It degrades about four times faster than diesel (Demirbas, 2010). Furthermore, it has a greater lubricity than petro-diesel which reduces corrosion in engines and increases durability. Finally it contains 10 to 11 wt% oxygen by weight which ensures more complete combustion of

hydrocarbons (Boocock *et al.*, 1998). Extensive research has been carried out to optimize the overall process (Shafiee and Topal, 2009) for biodiesel production, but transesterification reaction has been a priority in many studies.

Basumatary and Dekacarried out the transesterification of yellow oleander (*Thevetiaperuviana*) seed oil to its fatty acid methyl esters (FAME) in methanol by batch reaction using a heterogeneous catalyst derived from the rhizome of *Musa balbisiana* Colla (one variety of banana plant) and they obtained a yield of 95 wt% at room temperature

(32°C) in 3 h (Basumatary and Deka, 2014). The catalyst was thermally stable and better catalytic activity was observed when it was activated. Chindo *et al.* (2013) extracted oil from Yellow oleander (*Thevetiaperuviana*) seed with n-hexane solvent in a soxhlet extractor and transesterified with ethanol and methanol using KOH as a catalyst where 84.2% and 91.6%, respectively of FAME were attained.

Yellow oleander (*Thevetiaperuviana*) is an evergreen ornamental plant which grows to about 2-6 m in height. The plant belongs to the *Apocynaceae* family and is widespread in American, Asian and African continents. The plant starts flowering after one and a half year from plantation and there after blooms thrice a year. The part of the plant that produces oil is the seed, the seed has oil yield of 1575 kilogramme per hectare (kg/ha). The fruits are usually green in colour and become black on ripening. This fruits contains between one to four seeds in its kernel and the plant bears milky juice in all organs. Mature fruits contain about 22-23% Kernel and seeds have 60-65% oil content (Basumatary and Deka, 2014) closer to that obtained by Bashir *et al.* (2014) when using rubber seed. The aims and objectives of this study are to optimize the production of biodiesel from yellow oleander oil and also to investigate the influence of reaction parameters viz- viz reaction time, oil to methanol volume ratio, catalyst (KOH) amount and reaction temperature.

**Materials and Methods**

**Sample collection**

Samples were handpicked from the garden of Biological Science Department, Gombe State University, Nigeria and Staff Quarters of Federal Teaching Hospital, Gombe, Gombe State and dried under the sun for some hours to remove the remaining moisture, and then the kernels were separated by breaking the capsules. The black pericarps of the fruits were separated from their stony kernel. The stony kernels were cracked with a stone to remove the nut inside the hard kernels. Subsequently, the seed nuts were ground using manual grinding machine to increase surface area of sample, and in order to weaken and rupture of the cell.

**Oil extraction**

600 – 700 g of ground seed nut was placed into a bowl and 50 to 60 ml of water were added, stirred and pressed continually using bear hands, until oil was obtained. The process was repeated until reasonable amount of oil was obtained.

**Characterization of the oil**

The recommended methods of the Association of Official Analytical Chemists (AOAC, 1984) were adopted to determine the levels of moisture content, and acid value of the oil. Moisture content was determined by heating 5.0 g of the sample to a constant weight in a watch glass placed in an oven maintained at 105°C, while the acid value was determined using titrimetric method based on AOAC standard.

**Transesterification process**

The transesterification process was carried out in a 250 ml conical flask placed on a temperature and variable rotational speed controlled by heating mantle. As can be seen in Appendices 1–4, various amounts of oil, methanol and catalyst were appropriately measured for the optimisation studies, varying the amount of one of the

intervening variables one at a time while keeping the other parameters constant. All the transesterification process was carried out for two hours except when the effect of time on the biodiesel production was monitored. When transesterification was completed the reaction was terminated by the addition of 0.5M HCl. The mixture was transferred into a separating funnel and allowed to stand overnight. The product separated into two layers, with the lower layer consisting of glycerine, which was drained off. The upper layer which consists of FAME and small amount of catalyst was washed with warm distilled water (60 – 80°C) to remove the remaining impurities like soap, and methanol. Washing was completed when the water became clear. The transesterification procedure was repeated where the effects of reaction time, oil to methanol volume ratio, catalyst (KOH) amount and reaction temperature were investigated.

**Result and Discussion**

**Sample characterization**

As shown in Table 1, the moisture content obtained was 0.4% which will not affect the production of biodiesel. High moisture content reduces the conversion of triglycerides to biodiesel fuel (Atadashi *et al.*, 2012). The Acid Value obtained was 2.154 mg of KOH/g of oil. Production of biodiesel is best when the acid value is less than 1% (Mathiyazhagan and Ganapathi, 2011).

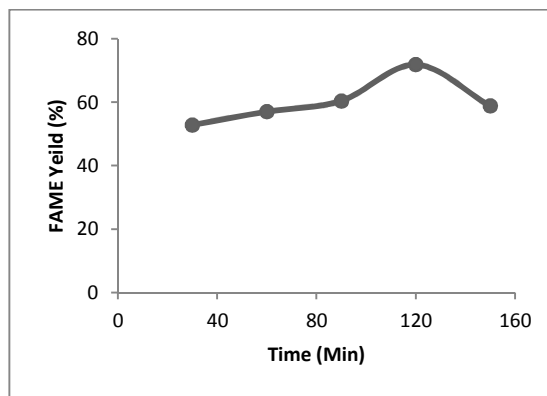
**Table 1: Characteristics of Oleander oil**

S/N	Parameters	Value
1	Free fatty acid (%)	1.077
2	Acid value (mg of KOH/g of oil)	2.154
3	Moisture content of oil (%)	0.4



(a) Yellow Oleander tree (b) Oleander fruits (c) Oleander seeds

**Fig. 2: Pictures of Oleander fruit**



**Conditions:** KOH (Catalyst) = 0.45g, Temperature = 50°C and Oil to methanol ratio = 5:1

**Fig. 3: Effect of reaction time on FAME yield**

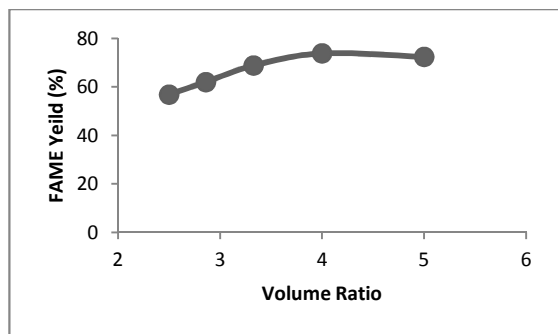
**Effects of reaction time**

Fig. 3 presents the result of effect of reaction time on the FAME yield where the reaction time was varied from 30 min until 150 min. As can be seen in the figure, there was an increase in the yield at the initial stage of the reaction

up to 120 min. This showed that 120 min is maximum time for the conversion of the oil to the methyl ester. The yield obtain at this condition is 71.8%. After 120 mins a decrease in the yield was observed due to the reversible reaction (Alptekin and Canakci, 2011) as equilibrium will shift to the left due formation of excess glycerol. Also previous study confirmed that, excess reaction time did not promote the FAME yield but may cause major soap formation (saponification) (Ramadhas *et al.*, 2005). Reaction time of 120 min used in this work is higher than 90 min when using chicken fat based biodiesel (Jagadale and Jugulkar, 2012).

**Effects of oil to methanol molar ratio**

One of the important variables affecting the ester yield during the transesterification process is the molar ratio of alcohol to vegetable oil. Molar ratio is the ratio of number of moles of alcohol to number of moles of glycerides in the oil. Theoretically, transesterification reaction requires three moles of alcohol for each mole of oil. However, in practice, the molar ratio should be higher than that of stoichiometric ratio in order to drive the reaction towards completion (Ramadhas *et al.*, 2005). In this research, various volume ratios of the oil to methanol starting with 5:1 until 5:5 were tested. The result for the effect of volume ratio of oil to methanol on the FAME yield result is shown in Fig. 4. The result of the FAME yield shows that, as the molar ratio increases the FAME yield increases. Thus it was found that, the highest FAME yield was achieved at the molar ratio of 5:1 (72.4 %) as shown in the Figure. However, a further increase in molar ratio did not show any improvement in the FAME yield instead a decrease was observed. This is because excess methanol may move over the ester layer and it can cause decrease the flash point of the biodiesel (Monteiro *et al.*, 2008). Hence 5:1 molar ratio is considered to be optimum ratio found in this study.



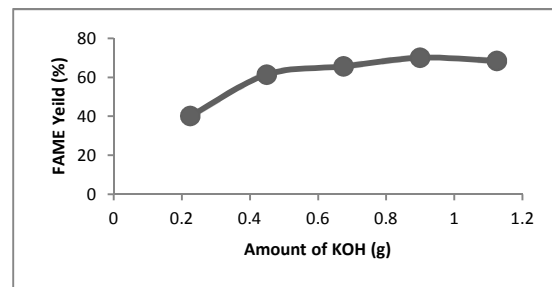
Conditions: KOH (Catalyst) = 0.45 g, Temperature = 50°C and reaction time = 2 h

Fig. 4: Effect of oil to methanol volume ratio on FAME yield

**Effects of catalyst concentration**

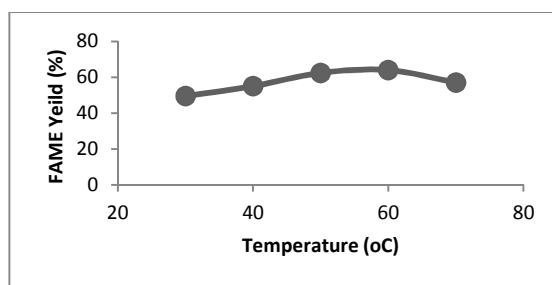
The alkaline catalyst, potassium hydroxide concentration in the range of 0.225–1.125 g was used in the present experimental analysis. The effect of catalyst amount on conversion efficiency is shown in Fig. 5. The maximum conversion of 70% was achieved at 0.9 g of KOH. Addition of excess amount of catalyst, gave rise to the formation of an emulsion, which increased the viscosity and led to the formation of gels and consequently decreased the yield (Ramadhas *et al.*, 2005).

As mentioned by (Monteiro *et al.*, 2008) from physical appearance, the higher catalyst concentration, the darker the biodiesel produced. Therefore the addition of a suitable catalyst concentration is important to the physical appearance of biodiesel. Amount of catalyst (0.45 g) used in this study is slightly lower than that reported by (Jagadale and Jugulkar, 2012).



Conditions: Volume ratio= 5:1, Temperature= 50°C and reaction time = 2 h

Fig. 5: Effect of catalyst concentration on FAME yield



Conditions: Volume ratio= 5:1, KOH = 0.45 g and reaction time = 2 h

Fig. 6: Effect of reaction temperature on FAME yield

**Effect of reaction temperature**

Temperature is one of the important aspects that affect the biodiesel production because it can facilitate the rate of reaction (Boocock *et al.*, 1998). The maximum yield of 64% was obtained at the temperatures of 60°C. The decrease in yield was observed when the reaction temperature went above 60°C. The reaction temperatures greater than 60°C should be avoided, because they tend to accelerate saponification reaction of the triglycerides by the alkaline catalyst before completion of the alcoholysis (Ramadhas *et al.*, 2005). Also above this temperature the methanol vaporizes as the boiling of the methanol is 65°C. From this study, it has confirmed that, 60°C was the optimum condition for transesterification of yellow oleander oil similar to (Okullo *et al.*, 2010).

**Conclusion**

The production of fuel-quality biodiesel from low-cost low FFA feed stocks (Oleander seed oil) was investigated in this research. The transesterification of yellow oleander seed oil using KOH as catalyst was successfully optimized. It was found that, the highest biodiesel yield obtained was 71.8% at 120 min reaction time, with oil to methanol mass ratio of 5:1, catalyst (KOH) amount of 0.9 g and reaction temperature of 60°C. This study reveals that biodiesel from unrefined oleander seed oil is quite suitable as an alternative feedstock for biodiesel. However, further research and development on additional fuel property

measures, long-term run and wear analysis of biodiesel-fueled engine is also necessary.

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**Appendices Optimization**

**Appendix 1: Oil to methanol volume variation**

Volume of oil (cm <sup>3</sup> )	Volume of methanol (cm <sup>3</sup> )	Mass of KOH (g)	Temp (°C)	Reaction time (h)
50	<b>10.0</b>	0.45	50	2
50	<b>12.5</b>	0.45	50	2
50	<b>15.0</b>	0.45	50	2
50	<b>17.5</b>	0.45	50	2
50	<b>20.0</b>	0.45	50	2

**Appendix 2: Catalyst variation**

Volume of oil (cm <sup>3</sup> )	Volume of methanol (cm <sup>3</sup> )	Mass of KOH (g)	Temp (°C)	Reaction time (h)
50	10	<b>0.225</b>	50	2
50	10	<b>0.450</b>	50	2
50	10	<b>0.675</b>	50	2
50	10	<b>0.900</b>	50	2
50	10	<b>1.125</b>	50	2

**Appendix 3: Temperature variation**

Volume of oil (cm <sup>3</sup> )	Volume of methanol (cm <sup>3</sup> )	Mass of KOH (g)	Temp (°C)	Reaction time (h)
50	10	0.45	<b>30</b>	2
50	10	0.45	<b>40</b>	2
50	10	0.45	<b>50</b>	2
50	10	0.45	<b>60</b>	2
50	10	0.45	<b>70</b>	2

**Appendix 4: Time variation**

Volume of oil (cm <sup>3</sup> )	Volume of methanol (cm <sup>3</sup> )	Mass of KOH (g)	Temp (°C)	Reaction time (min)
50	10	0.45	50	<b>30</b>
50	10	0.45	50	<b>60</b>
50	10	0.45	50	<b>90</b>
50	10	0.45	50	<b>120</b>
50	10	0.45	50	<b>150</b>

