

## Production and Characterization of Biodiesel from *Aphanamixis polystachya* Seed Oil

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### Abstract

The present study focuses on the utilization of non-edible *Aphanamixis polystachya* seed oil as a potential feedstock for the production of biodiesel. The extracted oil from non-edible seed could not be directly exploited for biodiesel production owing to its high free fatty acid (FFA) content of 5.785%, so acid catalyzed esterification was applied to reduce FFA of oil to zero followed by base-catalyzed transesterification to convert esterified product to its mono-esters. Thermogravimetric analysis (TGA) revealed that the mass percentage corresponding to biodiesel under optimum conditions was 74.38%. The conversion of the triglycerides in the oil into biodiesel through transesterification was confirmed using FT-IR spectroscopy. The elemental analysis of the produced biodiesel was studied. The produced biodiesel had properties which were comparable with biodiesel standards and could be utilized as an alternative diesel fuel without any hardware modifications.

**Keywords:** *Aphanamixis polystachya* seed oil, Biodiesel, Acid-catalyzed esterification, Base-catalyzed transesterification.

### I. Introduction

The dwindling reserves of diesel have led researchers to find an alternative biodegradable, environmentally friendly diesel fuel 'Biodiesel'. Biodiesel is also known as the mono-alkyl ester of fatty acids can be derived by transesterification from oil seeds which grow abundantly in Bangladesh<sup>1</sup>. One such oil seed is the dark brown seeds of the plant, *Aphanamixis polystachya* (*A. polystachya*) belonging to the Meliaceae family. This plant locally known as Pithraj, Roina, Tiktaraj is found extensively in the forests and hilly areas of south Asian countries. The seed oil is non-edible and therefore does not possess any threat to food supplies compared to the conventional edible seed oils used in biodiesel production<sup>2</sup>. The seeds also contain about 44% oil and plantation of this tree can reduce petroleum imports by 21%<sup>3</sup>. The seed oil has a high degree of unsaturation making it a suitable candidate for cold weather operations of diesel engines<sup>3</sup>.

The present study also involves the extraction of oil from *A. polystachya* seeds using *n*-hexane as solvent and the optimized two-step esterification process for the production of biodiesel with improved fuel properties. This study also shows the detail structural, thermal, elemental and physico-chemical characterization of the produced biodiesel.

### II. Materials and Methods

#### Materials

*A. polystachya* seeds were collected from Mithila Seed Company, Tongi, Gazipur, Bangladesh. The seeds having an initial moisture content of 25.484 wt% were washed, cleaned and sun dried until they attained a moisture content of 6.71 wt%. The seeds were ground to fine powder using a mortar to utilize them for oil extraction. All the chemicals utilized in this research work were of analytical reagent grade. *n*-Hexane (96% pure) was purchased from Daejung Chemicals and Metals Company Limited, South Korea. Methanol (99.5% pure) and sulfuric acid (98% pure) were purchased

from Merck KGaA, Germany. Potassium hydroxide pellets were purchased from Riedel-de Haen, United States.

#### Extraction of Oil from Seed Powder

*A. polystachya* seed oil content in seed was determined by soxhlet extraction using *n*-hexane as solvent<sup>4</sup>. About 50 gm of dried seed powder was taken in a porous bag. The bag was placed inside a soxhlet apparatus that was suspended above a 500 mL round-bottom flask containing about 250 mL of *n*-hexane and below a reflux condenser. The flask was heated with a 500 mL heating mantle (Glassco Laboratory Instruments, India). The extraction process was optimized by carrying out the extraction of oil at five different extraction times (30, 60, 90, 120 and 150 min) in order to achieve the highest oil percentage. At the end of the extraction period, the flask containing *n*-hexane and the oil were removed and the residual solvent and moisture were separated by distilling the oil-solvent mixture at the boiling point of *n*-hexane (68.7 °C). After completion of distillation, the *n*-hexane recovered was further utilized for the extraction of oil. Dark brown viscous oil was obtained whose oil content is determined by the following formula:

$$\text{Oil (\%)} = \frac{\text{weight of oil obtained in gram}}{\text{weight of the seed powder in gram}} \times 100$$

The oil was then analyzed for its different physico-chemical properties to evaluate its use towards biodiesel production.

#### Production of Biodiesel

Two step esterification processes were carried out from oil conversion to biodiesel. In the first step is acid-catalyzed esterification is used, while in the second step, alkali-catalyzed transesterification process is personalized. The acid-catalyzed esterification is a pretreatment step that reduces the FFA content of the crude oil below 2% using an acid catalyst. 100 mL of *A. polystachya* seed oil was poured into the flask and heated to about 40 °C. The methanol was

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then charged into the flask and the mixture of methanol and preheated oil was stirred for a few minutes. Catalyst sulfuric acid was then added to the reaction mixture. Heating and stirring was continued until the reaction went to completion. The process was optimized by varying each experimental parameter while keeping other parameters constant. The following four parameters were varied:

1. Amount of methanol ( 30, 40, 50, 60, 70 and 80 vol% of oil)
2. Amount of sulfuric acid catalyst (1.0, 1.5, 2.0 and 2.5 vol% of oil)
3. Reaction temperature (40, 50, 60, 70 and 80 °C)
4. Reaction time (30, 45, 60, 75, 90 and 100 min)

The reaction mixture was poured into a separating funnel and allowed to settle under gravity for 1 h. The excess methanol, along with sulfuric acid and impurities moved to the upper phase and were removed. The lower layer which consisted of the esterified oil was separated and its acid value and FFA content was determined by standard titrimetric method. The optimum condition having the lowest FFA content was used for the main transesterification reaction.

In base-catalysed transesterification, KOH pellets were dissolved completely in required amount of methanol to prepare methanolic KOH solution. About 100 g of the pretreated oil was heated to 40 °C and then KOH methanolic solution was charged into the reaction flask and heating and stirring was continued at 400 rpm at the required temperature for the required time until the transesterification was completed. To achieve a higher yield of biodiesel, the process was optimized by varying the following four parameters.

1. Amount of methanol (20, 30, 40, 50, 60 and 70 wt% of oil)
2. Amount of KOH (0.5, 0.65, 0.8, 1.0, 1.2 and 1.4 wt% of oil)
3. Reaction temperature (40, 50, 60, 70 and 80 °C)
4. Reaction time (30, 60, 90, 120 and 150 min)

After the completion of the reaction, the reaction mixture was poured into a separating funnel and allowed to settle for 16 hrs. After 16 hours, the reaction mixture separated into two layers. The upper layer consisted of methyl ester and the lower layer consisted of glycerol and impurities. The lower layer was drawn off. Each experiment was conducted three times in order to reduce the experimental errors.

#### *Biodiesel Purification*

After the separation of glycerol, water washing of biodiesel was performed to remove the impurities present in the product. The methyl ester was mixed with hot (60 °C) distilled water (10 vol% of ester) and allowed to settle under gravity in the separating funnel. Two layers were formed, the upper layer was of biodiesel, and the lower layer comprised of water and impurities. This process was repeated until the lower layer had a pH similar to the pH of distilled water, indicating that the biodiesel was free of catalyst. This was achieved after washings and then the product was allowed to settle under gravity overnight. The

biodiesel collected from the upper layer was distilled to remove any traces of methanol in it and yield of biodiesel was calculated using the following equation<sup>5</sup>.

$$\text{Yield (\%)} = \frac{\text{Weight of produced biodiesel}}{\text{Weight of pretreated oil}} \times 100$$

### **III. Characterization of Produced Biodiesel**

The elemental analysis of the biodiesel sample was performed using CHNS elemental analyzer (varioMicro VI.6.1, GmbH, Germany). The samples were weighed into a silver capsule and placed in the auto sampler carousel and analyzed for 800 s. The combustion and reduction temperature were 1150 °C and 850 °C, respectively. The flow rate of helium and oxygen were 200 mL/min and 14 mL/min, respectively. One drop of the produced biodiesel and the extracted oil were separately placed in the sample holder. The FT-IR spectra was obtained by FT-IR Spectrophotometer (Model: IRPrestige-21, Shimadzu Corporation, JAPAN) equipped with an Attenuated Total Reflectance (ATR) device in the wave number range 700–4000 cm<sup>-1</sup> with 20 scanning rate with resolution of 4 cm<sup>-1</sup>. The FT-IR spectrum was taken in a transmittance mode. The thermogravimetric analysis (TGA) of biodiesel was conducted in a TGA-50H Thermogravimetric Analyzer, Shimadzu, Japan from room temperature (25.67 °C) to 599.29 °C with a heating rate of 10 °C /min under nitrogen atmosphere with a flow rate of 10 mL/min. The fuel properties of *A. polystachya* oil and the produced biodiesel were determined using standard test procedures.

### **IV. Results and Discussion**

#### *Effect of extraction time on oil content*

The extraction process was optimized by using six different extraction times (30, 60, 90, 120, 150 and 180 min) while keeping the amount of seed powder and volume of *n*-hexane constant at 50 gm and 250 mL, respectively. The variation of the oil percentage with the extraction time is shown in Fig. 1. With increase in the extraction time, the solubility of the oil in the solvent was enhanced so the extraction rate increased and the amount of oil extracted increased. When the extraction time was increased from 150 to 180 min, the oil content was almost constant. It was because nearly all the free oil on the surface of the seeds was solubilized in the fresh *n*-hexane over the course of 150 min. Therefore an extraction time of 150 min was used as the optimum time for extraction for which the oil content was found to be 39.33%. This oil content of was comparable to that of *jatropha* (55%)<sup>6</sup>, *castor* (55%)<sup>7</sup> and *rubber seed oil* (45%)<sup>8</sup> which are the conventional seed oils used for biodiesel production.

#### *Physico-chemical characterization of extracted seed oil*

The physico-chemical properties of the extracted seed oil were determined by the following standard test procedures and are listed in Table I. The free fatty acid (FFA) of the extracted oil was determined from its acid value. The FFA content of the extracted oil (5.785%) did not lie within the

acceptable limit (3-5%) for base-catalyzed trans-esterification and may lead to soap formation making it difficult to separate the glycerol layer from biodiesel layer and result in lower yield of the methyl ester<sup>9</sup>. The kinematic viscosity of the oil at 40 °C was very high (33.568 cSt) and would result in the formation of large droplets on direct injection which would cause poor combustion, increased exhaust smoke and emissions, wear of gear train, camshaft and tappets of the set of injection pumps due to the higher injection pressures, excessive pressure losses in the piping system and poor atomization. The high volatility of the oil due to its low flash point makes the oil unsuitable as a fuel for diesel engines. Therefore a dual step process was developed for the production of biodiesel from the extracted seed oil<sup>10</sup>.

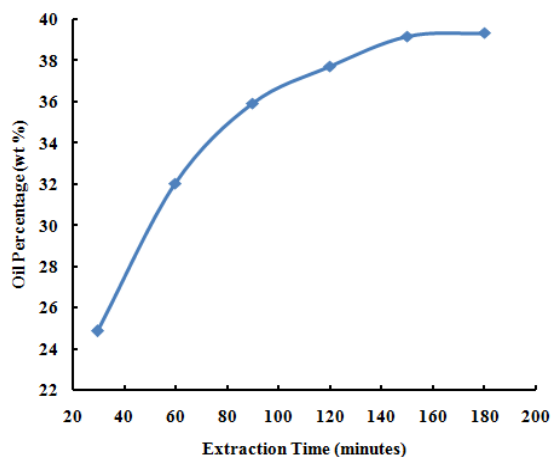


Fig. 1. Variation of oil content with extraction time.

### Acid-catalyzed esterification

#### Effect of amount of methanol

Methanol was used to carry out the esterification of the extracted oil. The acid value and FFA content was determined for different amount of methanol (30, 40, 50, 60, 70 and 80 vol% of oil) keeping the amount of H<sub>2</sub>SO<sub>4</sub> catalyst constant at 0.5 vol%, temperature constant at 60 °C and time constant at 90 min.

Table 1. Physico-chemical characterization of extracted *A. polystachya* seed oil

Parameters	Testing Procedure	<i>A. polystachya</i> oil
Colour Index	ASTM D 1500-64	0.5
Density at 15 °C, g/cm <sup>3</sup>	IP 160/57	0.924
Kinematic Viscosity at 40 °C, cSt	ASTM D 445-65	33.568
Kinematic Viscosity at 100 °C, cSt	ASTM D 445-65	9.79
Flash Point, °C	ASTM D 93-62	30
Pour Point, °C	ASTM D 97-57	-11
Acid value, mg KOH/gm	IP 1/58	11.57
FFA, %	IP 1/58	5.785
Water content, vol%	IP 74/57	0.36
Ash content, wt%	ASTM D 482-63	1.23
Carbon residue, wt%	ASTM D 189-65	1.17
Calorific value, KCal/kg	IP 12/58	8571.42

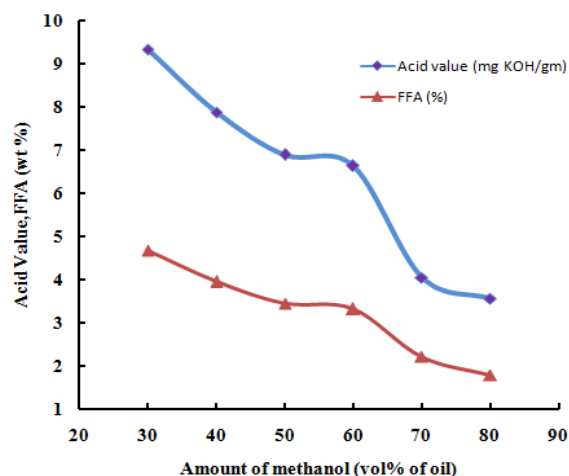


Fig. 2. Variation of acid value and FFA content with the volume of methanol.

The variation of the acid value and FFA content with the amount of methanol is shown in Fig. 2. With increase in the amount of methanol from 30 to 80 vol%, the FFA content was seen to decrease significantly from 4.670 to 1.780% due to the increase in solubility of methanol in the extracted oil which resulted in higher reaction rate with the FFA present in the oil. This is in agreement with other researchers who used excess quantities of methanol while using sulfuric acid as a catalyst<sup>11</sup>. Therefore the optimum amount of methanol to be used for acid-catalyzed esterification was 80 vol% of oil for which the FFA content was the lowest.

#### Effect of amount of sulfuric acid catalyst

Sulfuric acid was used as the catalyst for the acid pretreatment of the oil. The acid value and FFA content was determined for different amount of sulfuric acid catalyst (1.0, 1.5, 2.0 and 2.5 vol% of oil) keeping the amount of methanol constant at 80 vol%, temperature constant at 60 °C and time constant at 90 min.

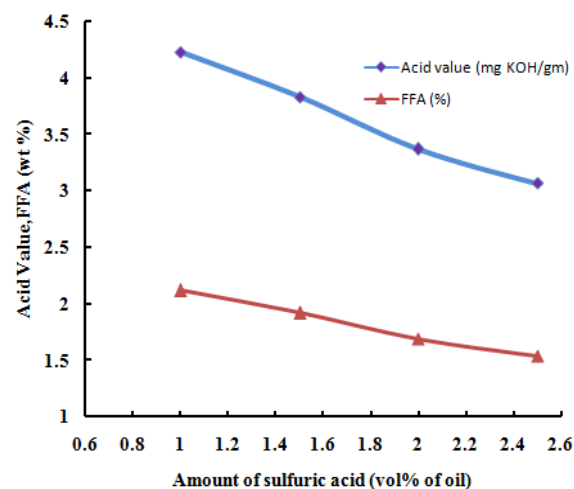


Fig. 3. Variation of acid value and FFA content with the amount of sulfuric acid.

The variation of the acid value and FFA content with the amount of sulfuric acid is shown in the Fig. 3. The lowest FFA content of 1.533 % was observed for 2.5 vol% sulfuric acid. It should be noted that when the experiment was carried out using 3.0 vol% sulfuric acid catalysts, a dark colored product was obtained and it was not possible to distinguish the methanol layer from the esterified layer, therefore excess amount of sulfuric acid should be avoided. A lower amount of sulfuric acid than 2.5 vol% would cause a reduction in biodiesel conversion.

#### Effect of reaction temperature

The acid value and FFA content of the oil was determined for different reaction temperatures (40, 50, 60, 70 and 80 °C) keeping the amount of methanol constant at 80 vol%, amount of sulfuric acid catalyst constant at 2.5 vol% and time constant at 90 min. The results are shown in Fig. 4. With increase in temperature from 40 to 70 °C, the FFA content decreased from 2.423 to 1.642% and then showed a rise in value of 1.710% on reaching 80 °C. This was because at low temperature, conversion of FFA to methyl ester was very low even after stirring the mixture for a long time but as the temperature increased, the conversion also increased simultaneously due to increase in diffusion rate owing to increase in solubility of the solvent in the oil. But as the temperature crossed 70 °C, the acid value and FFA content showed a slight rise which may be due to the loss of methanol from the reaction mixture and the product also exhibited a dark color at this temperature. Another reason for the increase in FFA content beyond 70 °C is the burning of the oil by sulfuric acid. Moreover, high temperature would also cause an increase in the production cost of biodiesel in industry. Therefore, the optimum temperature for acid pretreatment process was 70 °C.

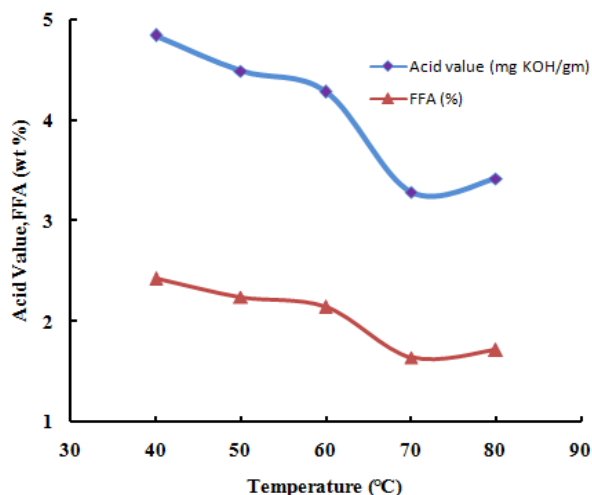


Fig. 4. Variation of acid value and FFA content with reaction temperature.

#### Effect of reaction time

The acid value and FFA content was determined for different reaction time (30, 45, 60, 75, 90 and 105 min) keeping the amount of methanol constant at 80 vol%,

amount of sulfuric acid constant at 2.5 vol% and reaction temperature constant at 70 °C. The results are shown in Fig. 5. With increase in reaction time from 30 to 60 min, there was a decrease in FFA content from 3.747 to 0.935% and then again from 60 to 105 min, there was increase in FFA content from 0.935 to 1.383%. The FFA content may have decreased up to 60 min because a time of 60 min was required owing to proper contact between the reactant and the oil resulting in higher conversion of the FFA into esters but beyond 60 min, the increase in FFA may be due to decomposition of methyl ester as a result of prolonged time of stirring. Therefore, a reaction time of 60 min was chosen as the optimum time for acid-esterification process.

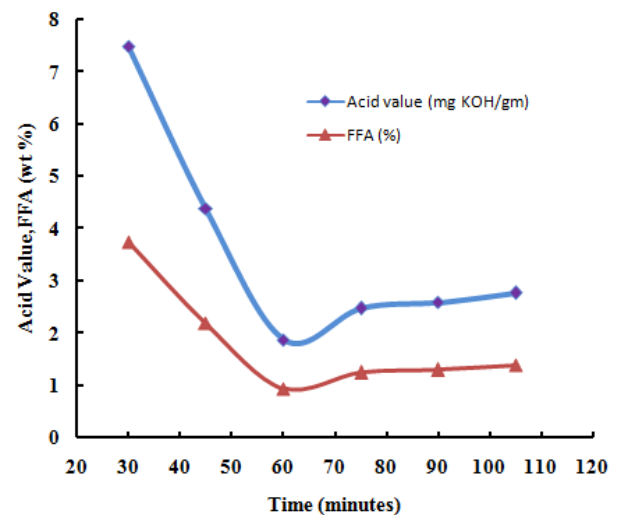
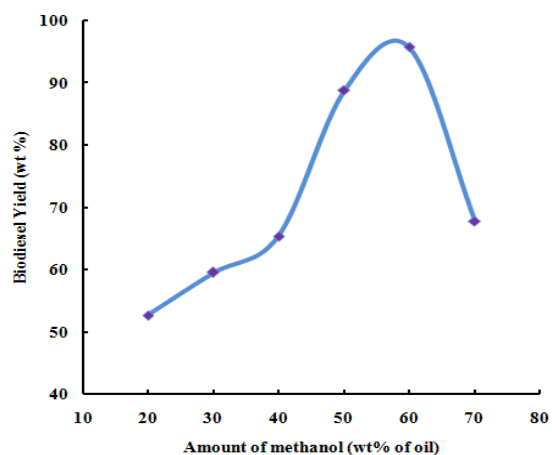


Fig. 5. Variation of acid value and FFA content with reaction time.

### Base-catalyzed transesterification

#### Effect of amount of methanol

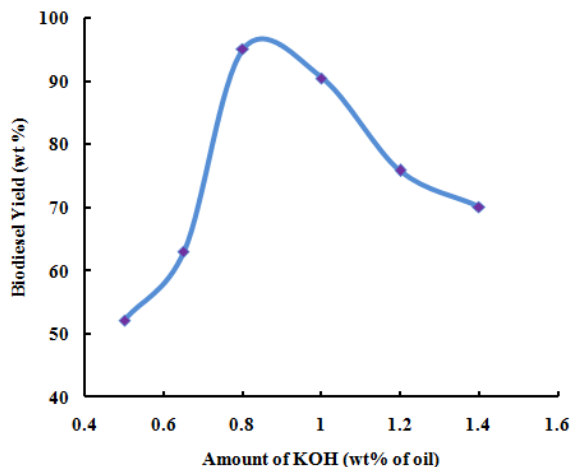
Methanol was used as the carrier of the catalyst, KOH in the transesterification of the pretreated oil. The yield of biodiesel was measured for different amount of methanol (20, 30, 40, 50, 60 and 70 wt% of oil) keeping the amount of KOH constant at 1.0 wt%, reaction temperature constant at 60 °C and time constant at 90 min. The result of the variation in the biodiesel yield with the amount of methanol is shown in Fig. 6. The biodiesel yield increased from 52.71 to 95.76 wt% with increase in the amount of methanol from 20 to 60 wt%. This is because higher amount of methanol results in higher diffusion rate of the catalyst in the oil phase and therefore higher reaction rate with the FFA or triglycerides. But on addition of 70 wt% of methanol to the reaction mixture, there was a decrease in the yield of biodiesel to 67.80 wt% because the excess methanol favored the conversion of the triglycerides into monoglycerides as well as enhanced the contact between the methyl ester and glycerol molecules and favored the backward reaction forming monoglycerides<sup>12</sup>. Therefore 60 wt% of methanol was considered as the optimum amount of alcohol required for transesterification.



**Fig. 6.** Variation of Biodiesel yield with the amount of methanol.

#### Effect of amount of KOH

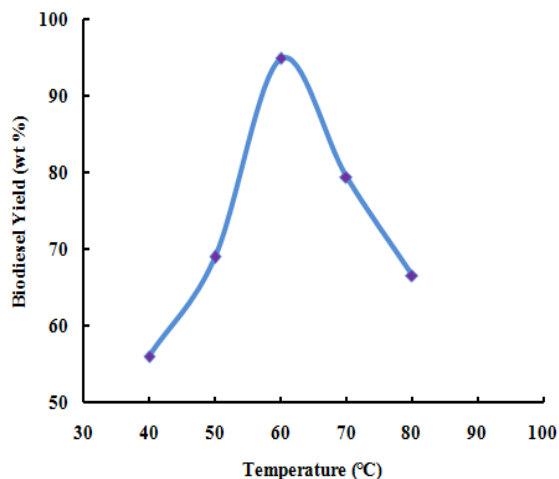
KOH was used as the catalyst to carry out the transesterification reaction. The yield of biodiesel was measured for different amount of KOH (0.5, 0.65, 0.8, 1.0, 1.2 and 1.4 wt% of oil) keeping the amount of methanol constant at 60 wt%, reaction temperature constant at 60 °C and time constant at 90 min. The result of the variation in the biodiesel yield with the amount of KOH catalyst is shown in Fig. 7. As the KOH concentration was increased from 0.5 to 0.8 wt%, the conversion of triglycerides as well as the ester content was increased from 52.15 to 95.06 wt%. Insufficient amount of KOH resulted in incomplete conversion of the triglycerides as indicated by lower ester content and no transesterification reaction was observed below 0.5 wt% of KOH. The ester content reached an optimum value at 0.8 wt% of KOH but beyond this concentration, the ester production decreased. This is because addition of excess alkaline catalyst caused the participation of more triglycerides in the saponification reaction and led to emulsion formation which resulted in increase in viscosity of the oil and gel formation. This gel hinders the separation of the glycerol from the biodiesel and thereby reduces the yield of biodiesel. These results were similar to biodiesel produced from *Brassica carinata* oil<sup>13</sup> and used frying oil.



**Fig. 7.** Variation of Biodiesel yield with the amount of KOH.

#### Effect of reaction temperature

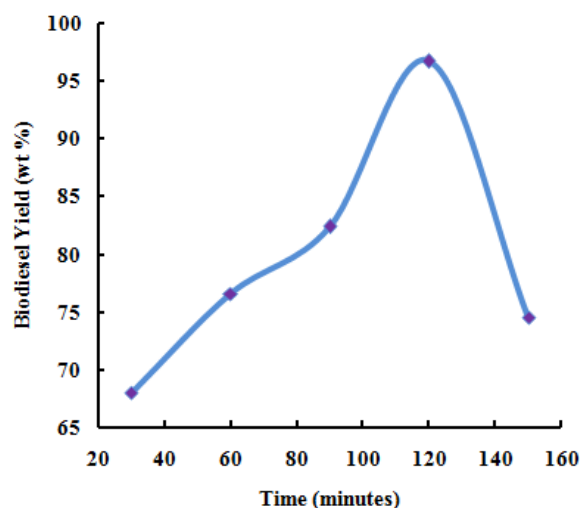
The yield of biodiesel was measured for different reaction temperatures (40, 50, 60, 70 and 80 °C) keeping the amount of methanol constant at 60 wt%, amount of KOH constant at 0.8 wt% and time constant at 90 min. The results of the variation in the biodiesel yield with the reaction temperature are shown in Fig. 8. With increase in temperature from 40 to 60 °C, there was an increase in biodiesel yield from 56.05 to 94.91 wt%. This was because with increase in temperature, the reaction rate increased and reduced the reaction time due to the reduction in viscosity of the oil. This increased the solubility of the oil in methanol and improved the contact between the oil and methanol molecules, thereby resulting in a better yield of methyl ester. Beyond 60 °C there was a decrease in biodiesel yield, the biodiesel yield was 79.47 and 66.58 wt% at 70 and 80 °C, respectively. This decrease in yield at higher temperature may be due to the evaporation of methanol (boiling point: 64.7 °C). High temperature also accelerates the saponification of the glycerides by KOH before completion of alcoholysis. Therefore, 60 °C was considered as the optimum reaction temperature for transesterification. Similar trend was observed by researchers for neat and used frying oil and tobacco seed oil<sup>14</sup>.



**Fig. 8.** Variation of Biodiesel Yield with reaction temperature.

#### Effect of reaction time

The yield of biodiesel was measured for different reaction time (30, 60, 90, 120 and 150 min) keeping the amount of methanol constant at 60 wt%, amount of KOH constant at 0.8 wt% and reaction temperature constant at 60 °C. The result of the variation in the biodiesel yield with reaction time is given in Fig. 9. With increase in reaction time from 30 to 120 minutes, the biodiesel yield showed a gradual increase from 68.03 to 96.67 wt%. This was because with increase in time, perfect contact between the reagents was achieved with constant stirring. At 150 min, the yield decreased to 74.47 wt% because prolonged time of stirring caused decomposition of the methyl esters into triglycerides. Therefore reaction time of 120 min was considered as the optimum time required for transesterification.



**Fig. 9.** Variation of Biodiesel Yield with reaction time.

#### Elemental composition of produced biodiesel

The elemental composition of the produced biodiesel from *A. polystachya* seed and biodiesel standard according to ASTM PS 121<sup>15</sup> are given in Table II. The elemental composition reveals that the produced biodiesel contains significant amount of oxygen which would lead to complete and cleaner combustion and cause reduction in combustion delay and probability of fuel-rich zone formation which are prone to hydrocarbon and carbon monoxide emissions<sup>16</sup>. The high nitrogen content of the produced biodiesel indicates that there will be increased NO<sub>x</sub> emissions on burning this fuel. NO<sub>x</sub> is a major contributor of acid rain and human disease. It is also a primary pollutant in the formation of tropospheric ozone which is an important greenhouse gas. Some researchers have concluded that increased NO<sub>x</sub> emissions are also associated with higher oxygen content of biodiesel<sup>17,18</sup>. The composition of sulfur remains within the permissible limit and therefore the SO<sub>x</sub> emission hazards from this fuel will be low.

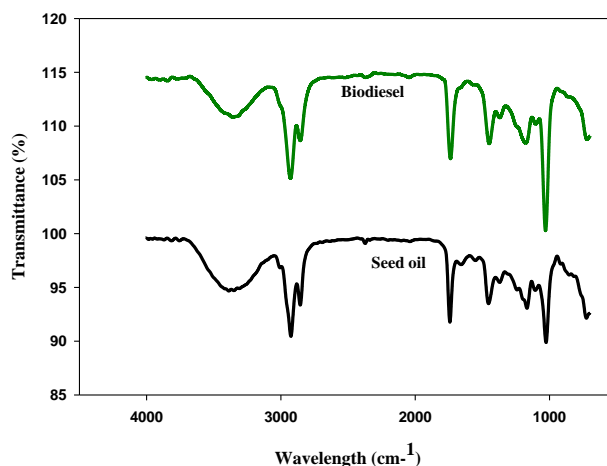
**Table 2. Elemental analysis of produced biodiesel and biodiesel standard.**

Sample	Produced Biodiesel	Standard Biodiesel
Weight (mg)	3.4030	-
C (%)	75.030	77
H (%)	12.619	12
N (%)	1.132	0.0
S (%)	0.00	0.0-0.0024
O (%)	11.219	11

#### Fourier Transform- Infrared Spectroscopic Analysis

The FT-IR spectra of the oil and biodiesel sample are given in Fig. 10 and are used to show that the triglyceride in the oil has been actually converted to biodiesel. On comparing the FT-IR spectra of *A. polystachya* oil and its biodiesel, subtle differences are observed because the product of transesterification (fatty acid methyl Ester) is chemically similar to its precursor (oil). The main functional group of

*A. polystachya* seed oil are carboxylic acid (C=O) appears as main peak in 1741.72 cm<sup>-1</sup> region and alkanes (C-H) that appears in 2856.58 and 2926.01 cm<sup>-1</sup>. The major peaks are in the region of 1735.93 cm<sup>-1</sup> and 2927.94 cm<sup>-1</sup>. So the main functional groups of *A. polystachya* seed oil biodiesel are ester (C=O) appeared in 1735.93 cm<sup>-1</sup> and sp<sup>3</sup> alkyl (C-H) that appears in 2854.65 and 2927.94 cm<sup>-1</sup>. The symmetrical stretching vibration of the ester carbonyl group (C=O) which is common in both samples<sup>19</sup>. Symmetric and asymmetric bending vibration of -CH<sub>2</sub> group are observed at 1371.39 cm<sup>-1</sup> and 1454.33 cm<sup>-1</sup>, respectively for oil sample and the same bending vibrations of the -CH<sub>2</sub> group of biodiesel was observed at 1369.46 cm<sup>-1</sup> and 1448.54 cm<sup>-1</sup> respectively<sup>20</sup>. The bands at 1026.13 and 1029.99 cm<sup>-1</sup> correspond to C-O stretching for oil sample and biodiesel, respectively. In addition, the peak at 1163.93 cm<sup>-1</sup> in the triglyceride spectrum corresponds to the stretching vibration of the C-O group attached to -CH<sub>2</sub>, which converts to the peak at 1176.58 cm<sup>-1</sup> in the methyl ester group (CO-O-CH<sub>3</sub>), attributed to the stretching vibration of C-O group attached to -CH<sub>3</sub><sup>21</sup>. The reduction of CH<sub>2</sub>-O-groups in oil and the appearance of CH<sub>3</sub>-O- vibrations in biodiesel were obvious<sup>22</sup>. After comparison of the two FT-IR spectra, it can be concluded that the transesterification reaction had progressed successfully.



**Fig. 10.** FT-IR spectra of oil and produced biodiesel.

#### Thermogravimetric Analysis

Thermogravimetric analysis (TGA) is a simple and convenient way of monitoring transesterification reaction and the boiling point of esters. A simple TGA curve for the biodiesel sample is shown in Fig. 11. A weight loss of 5.46% occurred in the temperature region of 25 to 76 °C due to the dehydration of moisture present in the sample. In the temperature region of 76 to 153 °C, a weight loss of 6.14% occurred due to the evaporation of trace amount of methanol that may have not been completely removed during the purification step. A significant weight loss of 74.38 % occurred in the region of 153 to 236 °C due to the evaporation of biodiesel. A weight loss of 9.24 % occurred in the temperature region of 236 to 347 °C due to the



vaporization of trace amount of esters of unsaturated fatty acids remaining in the sample. The next weight loss occurred from 347 to 599 °C due to the thermal decomposition of mono-, di- and triglyceride residues. The weight then started to show a steady decrease in mass and from around 460 °C, the weight of the sample became almost constant and at the maximum temperature of 600 °C, 2.28 % of the sample remained.

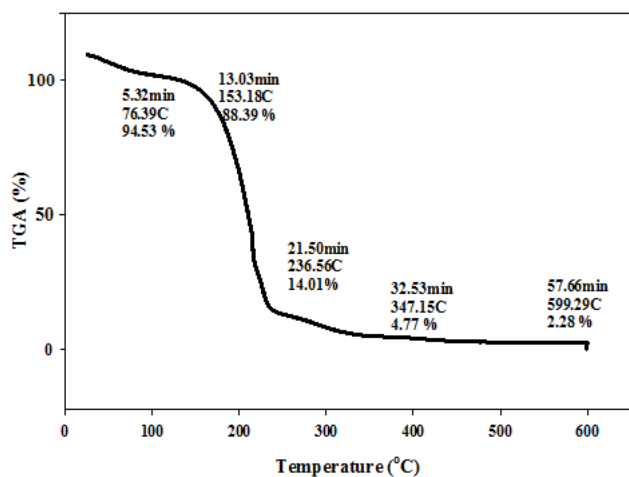


Fig. 11. TGA curve obtained for produced biodiesel.

#### Physico-chemical characterization of produced biodiesel

Various physico-chemical properties of the biodiesel were determined and compared with biodiesel standard. Table III lists some of the physico-chemical properties of the produced

biodiesel. On modification of the extracted oil into biodiesel by two-step process, the fuel properties had shown significant improvement. The density and viscosity of the produced biodiesel lied within the standard range and therefore it would lead to proper atomization resulting in proper combustion followed by lower emissions and exhaust smoke. The biodiesel would also be able to provide sufficient lubrication for the precision fit of fuel injection pumps. The biodiesel will be safe to handle and store owing to its high flash point and would not have any problems during cold weather operations because its pour point lies within the standard range. The acid value and FFA content indicate that there is less chance of this fuel to form acidic solutions which would cause corrosion and deterioration of fuel properties.

Almost no moisture was detected in the produced biodiesel which means that there is no chance of moisture to block the flow of biofuel, promote microbial growth and lead to increased corrosion of fuel systems. The ash content was lower than the biodiesel standard and it indicates that there is lower risk of smoke emission from this biodiesel. The high carbon residue can lead to clogging of fuel filters and this may be attributed to the presence of trace amount of glycerol and soap in the produced fuel. The calorific value of the biodiesel was almost 93 % of the standard and this lower calorific value is attributed to high oxygen content of the fuel which is also confirmed by elemental analysis. Since the cetane number of the fuel is high, its can easily combust in the internal combustion engines. Therefore this fuel can be implemented in internal combustion engines without any hardware modification.

Table 3. Physico-chemical characterization of produced biodiesel.<sup>23-25</sup>

Property Specification	Biodiesel Standard ASTM D6751		Produced Biodiesel	Commercial Biodiesel
	Test Method	Limits		
Density at 15 °C, g/cm <sup>3</sup>	ASTM D 1298	0.88	0.875	0.8445
Kinematic Viscosity 40 °C, cSt	ASTM D 445	1.9-6.0	4.278	2.71
Kinematic Viscosity 100 °C, cSt	ASTM D 445-65	-	2.152	-
Flash Point, °C	ASTM D 93	130 min	152	65
Pour Point, °C	ASTM D 97	-15 to -16	-5	-20
Acid value, mg KOH/g	ASTM D664	0.5 max	0.449	0.34
FFA, %	IP 1/58	0.40 max	0.325	
Water content, vol%	IP 74/57	0.05	-	Nil
Ash content, % (w/w)	ASTM D 482-63	0.02 max	0.0079	0.02
Carbon residue, % (w/w)	ASTM-D 189 -65	0.05 max	0.0166	0.05
Calorific value, MJ/kg	ASTM -D 240	38.586	39.0133	44.5
Sulfur content, %	ASTM -D 129-64	0.07	0.0	0.905
Cetane number	ASTM D613	47 min	47	51
Sulfated ash content, % mass	ASTM D874	0.002 max	0.0007	

## V. Conclusions

The present study was successful in investigating a low cost, high FFA feedstock for biodiesel production. The two-step process utilized for the production of biodiesel in the current research was successful in improving the properties of the extracted oil in terms of FFA content, viscosity, pour point, flash point, making the produced biofuel quite suitable as an alternative diesel fuel. The biodiesel was found to contain permissible amounts of sulfur and almost 0% moisture content. Although most of the properties were on par with international standards, the biodiesel was found to contain high carbon residue and to possess high nitrogen content and its calorific value was found to be 7% lower than the standard value. The high carbon residue is an indication of the presence of glycerol in the purified biodiesel. The presence of traces of methanol and glyceride residues was also detected by thermogravimetric analysis so further investigation on the purification process of the produced biodiesel should be carried out. Further research and development should be carried on additional fuel property measures, long-term run and wear analysis of biodiesel fueled engine.

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