## **Original Research Article**

# Evaluation of *Momordica Balsamina* Seed Oil for its Potential as Feedstock for Biodiesel Production

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### 6 Abstract

This paper sought to evaluate the potential of oil obtained from balsam apple (*M. balsamina*) 7 for biodiesel production. Oil was extracted from the seed using n-hexane in a soxhlet 8 9 extractor, then transesterified to biodiesel using single step alkali hydrolysis. The biodiesel so 10 produced was analyzed for their physicochemical and fuel properties using American Standards for Testing and Materials procedures. GC-MS was used to quantify the Fatty acid 11 methyl esters yield. Percentage oil yield was  $33.21 \pm 0.18$  %. The physicochemical properties 12 of the oil showed high acid values (*M. balsamina*  $9.958 \pm 0.5 \text{ mg/KOH/g}$ ). The percentage oil 13 degumming was  $10.12 \pm 0.6$  %. Some critical fuel parameters for the biodiesel produced from 14 the oil showed compliance with American Standards for Testing and Materials and European 15 standard specifications. The result of biodiesel produced showed; Colour 2.9  $\pm$  0.0, cetane 16 number 57.699  $\pm$  0.23, flash point 137  $\pm$  0.00 °C, cloud point 1.0  $\pm$  0.00 °C, pour point -0.4  $\pm$ 17 0.0 °C, sulphur content 0.021  $\pm$  0.01 ppm, kinematic viscosity 3.82  $\pm$  0.0 mm<sup>2</sup>/s and specific 18 gravity  $0.8615 \pm 0.0$  g/cm<sup>3</sup>. The percentage yield of biodiesel was  $89.12 \pm 0.04$  %. Stearic 19 acid methyl ester, a saturated ester, was dominant with percentage of 14.03 % in the biodiesel 20 produced. The yield of the oil is high enough and comparable with other oils that have been 21 used for biodiesel production. It can be inferred that oil from M. balsamina is suitable for 22 23 biodiesel production.

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25 Keywords: *M. balsamina*, transesterification, biodiesel, cetane number.

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### 29 **1.0 Introduction**

There is a need for alternative energy sources to petroleum based fuels due to the depletion of the world's petroleum reserves, global warming and environmental concern which makes it necessary to develop renewable energy sources of limitless duration and smaller environmental impact than the traditional one. One possible alternative to fossil fuel is the use of oils of plant origin like vegetable oils and tree borne oil seeds. These oils, after being formulated, have been found suitable for utilization in diesel engines [1].

Recently, environmentalists have started to debate on the negative impact of biodiesel production from edible oil due to large-scale production of biodiesel from edible oils which may bring global in balance to the food supply and market demand [2]. Hence, in this paper non edible vegetable oil from *Momordica balsamina* seed oil was assessed as potential alternative fuel for economic development and to solve environment issues.

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### 42 **2.0 Materials and Methods**

43 **2.1** Sample collection and preparation

# 44 Sample was collected from ripped seeds of balsam apple

Sample was collected from ripped seeds of balsam apple; *M. balsamina* in Kawo Area within Kaduna metropolis. The leaves and seeds were identified in the Herbarium section of Biological Science Department, Ahmadu Bello University Zaria with a Voucher numbers 1139 and was kept for future reference. The seeds were peeled to obtain the kernels, which was air dried and pulverized to a fine powdered form and stored in an air tight plastic containers.

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52	2.2 Extraction procedure
53	Two hundred gram (200 g) of air dried and pulverized plant seeds of M. balsamina was
54	weighed out and packed into a thimble, which were in turn placed into soxhlet extractor.
55	Extracting solvent n-hexane (500 $\text{cm}^3$ ) and anti - bumping chips were put into a 1000 $\text{cm}^3$
56	round bottomed flask and heated on heating mantle at 60 <sup>0</sup> C. The extraction was allowed to
57	continue until the solvent was cleared. The solvent in the round bottomed flask were
58	collected and concentrated in vacuo using a rotatory evaporator at 40 $^{0}$ C. The process was
59	repeated to obtain the mean of the percentage extraction and enough oil for further analysis.
60 61	% Extraction = <u>Weight of oil extracted</u> $\times 100$
62	Weight of sample
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64	2.3 Pre-treatment of the Oil (Acid Degumming)
65	Two hundred (200 gram) of <i>Cucurbita maxima</i> oil was weighed and heated to 70 $^{0}$ C on a heating
66	mantle. 25 % citric acid was prepared by dissolving 250 g citric acid in 1000 cm <sup>3</sup> of distil water
67	and was added in the ratio of 3:1 citric acid to oil then mixed for 5 min. This was centrifuge for
68	30 min for separation of gums and other impurities which settled at the bottom. Oil at the top of
69	the centrifuge test tubes, was decanted and reheated [3]
70	
71	2.4 Physicochemical Properties of the Seed Oil
72	2.4.1 Determination of the saponification value
73	The American Standard for Testing and Material (ASTM) method- (D 5558-95) was used for the
74	determination of the saponification values of the vegetable oil. The oil (5 g) was weighed into
75	Erlenmeyer flask and 0.5 M ethanolic KOH was prepared by dissolving 7 g of KOH in 250 $\text{cm}^3$
76	ethanol and 25 cm <sup>3</sup> of the prepared 0.5M ethanolic KOH was added and the resulting mixture

was refluxed for 60 minutes. The resulting solution was subsequently titrated against 0.5 M HCl 77 prepared by diluting 10.7 cm<sup>3</sup> HCl in 250 cm<sup>3</sup> of distil water using phenolphthalein as indicator. 78 The resulting end point was obtained when the pink colour changed into colourless. The same 79 80 procedure was used for the blank. The Saponification value (SV) was then calculated using the expression; 81 Saponification value (S.V.) = 5.61 (B-S) x M of HCl 82 Weight of sample 83 84 (Source: ASTM-D 5558 (95)) 85 86 Where; B – Vol. of HCl required by blank 87 S – Vol. of HCl required by sample 88 M – Molarity of HCl 89 5.61-Molar mass of KOH 90 91

92 2.4.2 Determination of acid value

Acid value of the oil was determined by ASTM method (ASTM – D 974(00). The oil (0.5 g) of 93 the oil was weighed into 250 cm<sup>3</sup> conical flask and 50ml of neutralized ethyl alcohol was added, 94 prepared by neutralizing a solvent mixture of 25 cm<sup>3</sup> ethanol and 25 cm<sup>3</sup> diethyl ether with 0.1M 95 ethanolic KOH prepared by dissolving 1.4 g KOH in 250 cm<sup>3</sup> of ethanol using phenolphthalein 96 as indicator. The mixture was added to the oil and heated on a water bath to dissolve the oil. The 97 solution was then titrated against 0.1 M KOH prepared by dissolving 1.4 g of KOH in 250 cm<sup>3</sup> of 98 distill water using phenolphthalein as indicator. The acid value was determined after which the 99 100 free fatty acid was calculated respectively as follows;

101	Acid Value = $\underline{A \times M \times 56.10}$
102	W
103	(Source: ASTM-D 974 (00))
104	Where,
105	A = ml of 0.1M KOH consumed by sample
106	M = Molarity of KOH
107	W = weight in grams of the sample
108	Then,
109	Free fatty acid = <u>Acid Value</u>
110	2

The oil (0.5 g) was weighed into conical flask and 20  $\text{cm}^3$  of carbon tetrachloride was added to 112 dissolve the oil. 25 cm<sup>3</sup> of Wijs reagent was added into the flask using a measuring cylinder in a 113 fume chamber and a stopper was inserted, the content of the flask was vigorously swirled and 114 kept in the dark for 35 minutes. 20 cm<sup>3</sup> of 10 % aqueous potassium iodide prepared by diluting 115 10 cm<sup>3</sup> of potassium iodide in 90 cm<sup>3</sup> of distill water was added into the content of the flask 116 using a measuring cylinder. The content was titrated with 0.1M sodium thiosulphate solution 117 prepared by dissolving 3.95 g of anhydrous Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> in 250 cm<sup>3</sup> of distill water. Few drops of 1 118 % starch indicator were added and the titration continued by adding the sodium thiosulphate drop 119 wise until coloration disappeared after vigorously shaking. The same procedure was used for the 120 blank test. The Iodine Value (I.V) is given by the expression; 121

122 Iodine Value (I.V) = 
$$\underline{126.9C} (V_1 - V_2)$$

124	Where,
125	C = concentration of sodium thiosulphate
126	V1 = volume of sodium thiosulphate used for blank
127	V2 = volume of sodium thiosulphate used
128	M = mass of sample
129	12.69= Constant.
130	
131	2.4.4 Determination of refractive index
132	Abbey refractometer was used in this determination. A drop of the sample was transferred into a
133	glass slide of the refractometer. Water at 30 °C was circulated round the glass slide to keep its
134	temperature uniform. Through the eye piece of the refractometer, the dark portion viewed was
135	adjusted to be in line with the intersection of the cross. At no parallax error, the pointer on the
136	scale pointed to the refractive index. This was repeated and the mean value noted and recorded
137	as the refractive index.
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- 2.7.0 Determination of Fuel Properties of Biodiesel 139
- 2.7.1 Determination of pour point 140

The pour point was determined by ASTM method (ASTM D - 97). The oil sample was poured 141 into the test jar to the level mark. The test jar was closed with the cork carrying the high – pour 142 thermometer. The position of the cork and the thermometer were adjusted for the cork to fit 143 tightly, the thermometer and the jar were coaxial and the thermometer bulb was immersed 3mm 144 below the surface of the sample. After this, the test jar was placed into the cooling medium. The 145 sample was cooled at a specified rate and examined at interval of 3 °C for flow characteristics 146

until a point was reached at which the sample showed no movement when the test jar was held in
a horizontal position for 5 seconds. The observed reading of the thermometer was recorded. 3 °C
was added to the recorded temperature and the result was recorded as the pour point

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### 1 2.7.1 Determination of kinematic viscosity

The temperature of the viscometer bath was adjusted to 38.9 °C. A calibrated thermometer was 152 held in upright position and inserted into the bath by a holder. A clean dry calibrated viscometer 153 was selected and carefully flushed with a dry nitrogen gas to remove the moist room air. A 154 sample of the biodiesel was drawn up into the working capillary of the viscometer and the 155 timing bulb was then allowed to drain back as an additional safeguard against moisture 156 condensing or freezing on the walls. The charged viscometer was inserted into the bath at a 157 depth such that at no time during the measurement of the flow time was any portion of the 158 159 sample in the viscometer less than 20 mm below the surface of the bath. The viscometer together with its content was allowed to remain in the bath for 30minutes to reach the test 160 temperature (38.9 °C). A suction bulb was used to adjust the head level of the biodiesel to a 161 position in the capillary arm of the viscometer about 7 mm above the first timing mark. The 162 biodiesel was then allowed to freely flow and the time required for the meniscus to pass from 163 the first to the second timing marks was noted with a stop watch. The procedure was repeated to 164 make a second measurement of flow time and the average of these determinations was used to 165 calculate the kinematic viscosity. The viscometer was thoroughly cleaned with sample solvent 166 167 and dried by vacuum. The procedure was repeated for the other samples of the biodiesel (ASTM D 445-97) 168

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170	Calculation:	
171		v = C x t
172		Where,
173		v = kinematic viscosity, mm2/s
174		C = calibration constant of the viscosity, (mm2/s)/s
175		t = mean flow time
176		

### 177 2.7.3 Determination of Cloud point

The cloud point was determined using ASTM D2500. A cylindrical test tube was filled with the biodiesel to a specific level (5 cm<sup>3</sup>) and clamped with a wooden clamp bearing thermometer. The test tube was placed on the ice/salt bathe and the set up inspected at intervals for cloud formation. The temperature at which a distinct cloudiness appeared at the bottom of the test tube was observed and recorded as the cloud point of the biodiesel.

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### 184 2.7.4 Determination of Flash point

The flash point was determined using ASTM D93. Seta MultiflashPensky-Martens Flash 185 186 Point Module Part Number 34100-2 was used to determine the flash point. The automatic PMCC module conforms precisely to national and international Pensky-Martens Closed 187 Cup flash point test methods. It comprises a heated cup and lid, and a DIPS pod 188 189 containing the dipping mechanism, gas and electric ignitors, fire detection system and a stirrer. The Pensky- Martens module was used with the Multiflash Universal Base unit 190 (p/n 34000-0). The base unit recognises the Pensky-Martens module is connected and 191 192 instantaneously sets up standard test parameters and calibration data

### 193 2.7.5 Determination of Cetane number

194	The cetane number was determined based on the formula proposed by Demirbas 2009 as
195	there was no enough biodiesel to carry out the test. The formula is given below.
196 197 198	$CN = \frac{46.3 + 5458}{S.V - 0.225 \times I.V}$
199	Where,
200	S.V = Saponification of the biodiesel
201	I.V = Iodine Value of biodiesel
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203	2.7.6 Determination of Specific gravity
204	Specific gravity bottle was washed, rinsed with acetone and dried at room temperature in
205	a dessicator and the weight of the empty bottle determined using an electronic weighing
206	balance. The weight of the bottle filled with water was recorded. The same procedure was
207	repeated with the oil and the specific gravity computed as follows;
208	Specific gravity = $W2 - W1$
209	W3 – W1
210	Where,
211	W1 = weight of empty bottle
212	W2 = eight of bottle + oil
213	W3 = weight of bottle + water
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#### 3.0 RESULTS

#### Table 1: Physicochemical properties of the seed oils

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Oil property Unit		M. balsamina	
		( Balsam Apple)	
Oil Content	%	$33.21 \pm 0.18\%$	
Saponification Value	mgKOH/g	$207.57\pm0.02$	
Iodine Value	gI <sub>2</sub> /100g	$171.31 \pm 059$	
Peroxide value	Meq/kg	$1 \pm 0.2$	
		0.050 - 0.5	
Acid value	mgKOH/g	9.958 ± 0.5	
Relative density	Dimensionless	$0.874 \pm 0.0$	
y			
Refractive index	Dimensionless	$1.481\pm0.0$	
Free fatty acid	Percentage	$4.979\pm0.6$	
Unsaponifiable value	mgKOH/g	$2.98\pm0.0$	
Gums	%	$10.12 \pm 0.6\%$	

### Table 2: Result of GC-MS for Biodiesel Produced from Balsam Apple (BOME)

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FATTY ACID	CARBON NUMBER	MOLECULAR FORMULAR	% COMPOSITION
Azelaic Acid	C10:2	$C_{11}H_{20}O_2$	0.72
Linoleic Acid	C18:2	$C_{19}H_{34}O_2$	11.48
Palmitic Acid	C16:0	$C_{17}H_{34}O_2$	7.94
Stearic Acid	C18:0	$C_{19}H_{38}O_2$	14.03
Alaidic Acid	C18:1	$C_{19}H_{36}O_2$	8.43
Cerotic Acid	C26:1	$C_{27}H_{54}O_2$	10.18
Behenic Acid	C22:0	$C_{23}H_{46}O_2$	10.18
Eicosanoic Acid	C20:1	$C_{21}H_{42}O_2$	4.68
Eicosatrienoic Acid	C20:2	$C_{21}H_{54}O_2$	8.09
Linolenic Acid	C18:2	$C_{19}H_{32}O_2$	9.67
Non-methyl esters			14.6
Total			_

Fuel parameter	BOMEs	ASTM Limits	European Standard
(%)Yield of Biodiesel	89.12 ± 0.06	-	-
Colour	$2.9\pm0.0$	3.5	-
Specific gravity g/cm <sup>3</sup>	$0.8615\pm0.0$	-	-
Kinematic viscosity (mm <sup>2</sup> /s)	$3.82\pm0.0$	6.0 Max	3.5-5.0
Pour point (°C)	$-0.4 \pm 0.0$	NS	NS
Cloud Point (°C)	$1.0\pm0.0$	NS	NS
Flash point (°C)	$137\pm0.0$	130 min	<120
Sulphur content (ppm)	$0.021\pm0.01$	0.050	-
Cetane number	57.699 ± 0.23	Min47	-

### Table 3: Fuel Properties of Biodiesel Produced from Balsam Apple (Mean ± SD)

NS = Not specified BOME = Balsam Oil Methyl Ester

### **4.0 Discussions**

The results of the physical and chemical properties of *Momordica balsamina* oil seed studied is presented in Table 1. The percentage oil content of *Momordica balsamina* is  $33.21 \pm 0.18$  %. According to Food and Agricultural Organization (FAO) as reported by [4], any seed containing greater than 17 % of oil is considered to be an oil seed and can be utilsed as feedstock for biodiesel production, as such *M*omordica *balsamina* seeds are good feedstock for biodiesel.

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The relative density of the *Momordica balsamina* oil was found to be  $0.874 \pm 0.0$ , which 241 is higher than the relative density of Africa star apple seed oil 0.711 [5] and Horse eve 242 seed oil 0.847 [6]. The higher relative density observed in the Momordica balsamina seed 243 oil when compared with other literatures is due to the fatty acid composition, minor 244 245 components and temperature. Since the relative density of seed/vegetable oils are dependent on this factors as reported by [7]. This in turn will affect the viscosity of the 246 Momordica balsamina seed oil resulting in poor atomization of the biodiesel produced 247 from the seed oil in the engine performance. 248

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The refractive index of the oil is  $1.473 \pm 0.0$ . This value is lower than that of beniseed oil (1.474) [8]. The refractive index of an oil is the ratio of speed of light at a defined wavelength to its speed in the oil/fat itself [9]. The difference in the refractive index of the *M. balsamina* oil compared with similar seed oils reported in literature is due to the variation in wavelength and temperature, degree of unsaturation and substitutions of component fatty acids.

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The acid value of *Momordica balsamina* was found to be 9.958  $\pm$  0.5 mgKOH/g. The 257 acid value of oil was higher in comparison to the acid value of Egusi seed oil (4.0 258 mgKOH/g) [10] and rubber seed oil (8.17 mgKOH/g) [11]. These indicate that the oil 259 will be unstable over a long period of time and will not be protected against rancidity and 260 peroxidation. This could be attributed to lack of natural antioxidants in the seeds such as 261 vitamins C and A as well as other possible phytochemicals like flavonoids [9]. The lower 262 the acid value, the lower the free fatty acid, the more suitable, the oil for 263 264 transesterification process.

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The saponification value for was found to be  $210.38 \pm 0.02$  mgKOH/g, which is lower when compared with *cocos nucifera* or coconut oil 246 mgKOH/g [11]. However biodiesel derived from oil with high saponification value causes exhaust emissions during burning in the engine [12].

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The iodine value was found to be  $182.74 \pm 0.4$  gI<sub>2</sub>/100g, which is higher than the iodine value of refined 87.72 gI<sub>2</sub>/100g and unrefined castor oil 84.8 gI<sub>2</sub>/100g [8]. This indicates that the oil studied is suitable for biodiesel production since low iodine value of vegetable oil produces biodiesel with high cloud and pour points; higher cloud and pour points means poor engine performance in cold temperatures [10].

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The viscosity for the oil was found to be  $25.0 \pm 0.0 \text{ mm}^2$ /sec, which is lower compared to the viscosity of *Cucurbita pepo* oil 93.65 mm<sup>2</sup>/sec [13] and *black benised* seed oil 33.2

279 mm<sup>2</sup>/sec [14]. According to [15] viscosity increases with molecular weight, as such 280 *Momordica balsamina* is having high molecular weight, which in turn will affect 281 injection lubrication, increase engine deposits and fuel atomization [16]

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The peroxide value of *Momordica balsamina* was  $1 \pm 0.2$  Meq/kg and is considered lower compared with Melon seed oil 5.63 Meq/kg [17] but higher than groundnut seed oil 0.74 Meq/kg that have been used in biodiesel productions [18]. The peroxide value of *Momordica balsamina* indicates that *Momordica balsamina* will have less level of deterioration when attacked or exposed to oxygen, since peroxide value indicates the level at which deterioration will take place as a result of oxidation owing to the availability of oxygen during storage [19].

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The unsaponifiable value for *Momordica balsamina* was found to be  $3.20 \pm 0.4$ mgKOH/g, which is higher compared to African pear seed oil 2.62 mgKOH/g and Soy bean seed oil 0.837 mgKOH/g [20]. The high level of the unsaponifiable matter in the oils indicates the presence of good lubricating properties [21].

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The percentage degumming after centrifugation of *Momordica balsamina* was  $10.12 \pm 0.6 \%$ . This is above the percentage degumming of crude Canola oil  $4.50 \pm 0.0 \%$  and crude Sunflower oil  $3.20 \pm 0.0 \%$  [3]. The value indicates that there is high level removal of hydratable and non-hydratable phosphatides, and other impurities especially metals contamination (P and Ca specifically) and also improvement in the yield of biodiesel production [22].

### **302 4.3 Fatty acid methyl esters**

The result of fatty acids methyl esters in Balsam Oil Methyl Esters (BOMEs) biodiesel shown in Table 2 indicate that stearic acid methyl esters been the dominant ester will help in enhancing the stability of the biodiesel, it is a saturated compound and less susceptibility to peroxidation [23]. It is also observed that balsam oil methyl ester is having more long chain of acid methyl esters, as such the long chain of balsam oil methyl ester will have effect on the fuel properties of the biodiesel produced [24].

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### 4.4 Fuel properties

The fuel properties of Balsam Oil Methyl Esters (BOMEs) produced is shown in Table 3. The percentage yield was recorded to be  $89.12 \pm 0.06\%$ , which is higher than the percentage yield of biodiesel produced from the oil seeds of *Gul Mohr* 87% [16]. The kinematic viscosity of the fatty acid methyl esters corresponded to biodiesel specification for both ASTM and European Standard limits, which indicate the presence of short chain unsaturated methyl fatty acid esters and is likely to produce less deposit when burnt in combustion engines [23].

318

The flash point of Balsam Oil Methyl Esters (BOMEs) conforms to the ASTM and (EN 2003) for biodiesel limit. This shows that the biodiesel produced has fewer tendencies to ignite accidentally, since flashpoint is the lowest fuel temperature at which application of ignition source causes the vapor of the fuel sample to ignite under the prescribed test conditions and also a parameter used to assess the overall flammability hazard of fuel [25].

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The cetane number of the Balsam Oil Methyl Esters (BOMEs) was found to correspond to the biodiesel specification for ASTM Standard limits. The higher the cetane number the better it is in its ignition properties. Biodiesel has higher cetane number than conventional diesel fuel, which results in higher combustion efficiency [26].

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331 The pour point of the Balsam Oil Methyl Esters (BOMEs) was found to be -0.3°C. There 332 is no standard specification for pour point due to variation in atmospheric condition.

The cloud point of Balsam Oil Methyl Esters (BOMEs) was found to be 1.1°C. Cloud point test characterizes the low temperature operability of diesel fuel. There is no standard specification for cloud point due to variation in atmospheric condition.

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The specific gravity of Balsam Oil Methyl Esters (BOMEs) was found to be 0.8713 g/cm<sup>3</sup> this is lower than the specific gravity of biodiesel produced from *Lagenaria Vulgaris* with specific gravity of 0.8879g/cm<sup>3</sup> [23]. This indicates that the biodiesel produced can be handled and stored safely. It is an important physical property in handling and storing of fuels [19].

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The sulphur content of Balsam Oil Methyl Esters (BOMEs) was 0.032ppm, and was found to correspond to the biodiesel specification for ASTM Standard limit of 0.050ppm.This indicates that the biodiesel produced could emit less SOx upon combustion [16].

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348	5.0 Conclusion
349	The oil content of the plant seed shows economic viability of feedstock for biodiesel
350	production and the physicochemical properties of the oil is promising with the exception
351	of saponification and free fatty acid values which were above their limits for biodiesel
352	production. The percentage of biodiesel production from the oil was high with more long
353	chain methyl esters.
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