

# Methanolysis Transesterification of Beniseed Oil to Biodiesel Catalysed by Acid Activated Waste Bone

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**Abstract:-** The methanolysis transesterification of Beniseed oil to biodiesel using acid activated waste cow bone was carried out. Solvent extraction was used to extract oil from the seeds using n-hexane and characterized to determine its physiochemical properties. The catalyst was synthesized from waste cow bone by activating it with concentrated phosphoric acid (H<sub>3</sub>PO<sub>4</sub>) at a ratio of 1:4 (w/v) on weight basis. It was dried in a Memmert oven at 100 °C for 2 hours and characterized to determine its catalytic properties. The effects of process parameters on biodiesel yield were examined. The biodiesel was characterized to determine its physiochemical properties. The result showed that the seed yielded 54 % oil. The low acid value and free fatty acid value of the oil enable it to undergo direct transesterification without treatment with the acid activated waste bone. The transesterification of the oil gave a maximum yield between 75 – 80% and the process parameters significantly affected the biodiesel yield. The properties of the biodiesel were within acceptable standard.

**Keywords:-** Acid Activated Waste Bone, Biodiesel, Soxhlet Extraction, Beniseed Oil, Transesterification Reaction.

## I. INTRODUCTION

Biodiesel is a monoalkyl ester of long chain fatty acids originated from vegetable or animal fats. It has re-awaken the interest of researcher worldwide because it can be renewed, degrade biologically, non-toxicity and environmentally friendly benefits (Al Widyand Al-Shyouchk, 2002). Fossil fuels are non-renewable energy resources. Although, these fuels are contributing largely to the world energy supply, their production and uses have raised environmental concerns and political debates. It has been shown that 98% of carbon emissions are resulted from fossil fuel combustion (Yusuf *et al.*, 2010).

The need for energy is increasing continuously due to rapid increase in the number of industries and vehicles owing to population explosion. The source of this energy are petroleum, natural gas, coal, hydrocarbon and nuclear. The petroleum diesel combustion emits several greenhouse gases. Apart from these emissions, petroleum diesel is also major source of these air pollutants including NO<sub>x</sub>, SO<sub>x</sub>,

CO, particulate matter, and volatile organic compounds (Zhang *et al.*, 2012). Several alternative fuels have been studied to either substitute diesel fuel partially or completely. These studies give rise to invention of biodiesel.

Biodiesel is a close substitute to petrol-diesel, as they are produced from raw material in rural areas. The use of biofuels in place of conventional fuels would slow the progression of global warming by reducing sulfur and carbon oxides and hydrocarbon emissions. The more the amount of biodiesel in diesel, the lesser the carbon (IV) oxide emission. Blend of 20% biodiesel in diesel fuel, lowers carbon (IV) oxide emission by 16% while pure bio diesel drastically reduced carbon (IV) oxide emission to zero (Graboski *et al.*, 1998).

It is mostly produced via transesterification of refined vegetable oil, waste cooking oil, and used frying oil using alkaline catalysts (Chhetri *et al.*, 2008, Bai *et al.*, 2011). The type of catalyst used for conversion of triglyceride to fatty acid methyl ester (FFA) is very vital. Therefore, various types of catalysts have been developed to catalyze the transesterification reaction. The major catalysts used are categorized into homogeneous and heterogeneous catalysts. The conventional homogeneous base catalysts like sodium hydroxide, potassium hydroxide, sodium methanoate and potassium methanoate are frequently used in production of biodiesel (Sharma and Singh, 2008). The catalytic performance of these catalysts and their ability to perform under moderate conditions has led to their choice (Hideki *et al.*, 2001).

Conversely, high cost of refined feedstocks results in high price of biodiesel compared to diesel fuel (Bozbas, 2008). The cost of purifying feed stocks is almost 70% of the total cost of producing production (Zullaikah *et al.*, 2005). To that effect, various kinds of low grade vegetable oil and non-edible oils have been investigated (Lee *et al.*, 2007). The price of low-grade vegetable oil like: waste cooking oil is lower than pure oils. Nevertheless, the feedstocks contain higher amount of FFAs and water contents. These features make their processing challenging (Balat and Balat, 2010). Therefore, to overcome their processing difficulties, heterogeneous catalyzed transesterification is employed to circumvent the two-step transesterification technique thereby reducing the cost of

production. Hence, this study focused on methanolysis transesterification of beniseed oil to biodiesel catalyzed by alkaline activated waste bone.

## II. MATERIALS AND METHODS

### 2.1 Materials

Beniseeds were purchased from Ogbete Market Enugu, Enugu State, Nigeria while the waste bone was gathered from Gariki Abattoirs Market, Uguwoaba, Enugu State Nigeria. The methanol, and other chemicals were all purchased from De-Cliff Integrated Services Ltd, Enugu, Nigeria and they are of analytical grade.

### 2.2 Methods

#### 2.2.1 Extraction of oil from Beni seed.

200 g of the grounded Beniseed (particle size of 900  $\mu\text{m}$ ) was weighed and exposed to come in contact with n-hexane in a Soxhlet extractor. The mixture was then heated to 70 °C for 90 minutes. The percentage yield of the oil was calculated as thus:

$$\% \text{ yield} = \frac{\text{weight of the oil extracted}}{\text{weight of the sample used}} \times 100 \quad (1)$$

#### 2.2.2. Characterization of the Beni-seed Oil

The AOAC (1990) was used to determine the physicochemical properties of the beniseed oil while Fourier Transform infra- red spectrometer (FTIR, Shimadzu FTIR-8400S) and gas chromatography mass spectrometer (GCMS, Thermo Finnigan Trace GC/Trace DSQ/A1300) were used to determine its functional group and fatty acid profile respectively.

#### 2.2.3 Preparation and synthesis of the catalyst

The cow bones were immersed in a boiling to remove tissues and fats. It was rinsed with distill water and dried in an oven at 105 °C for 2 hours. It was then crushed with hammer mill to a particle size < 2 mm. The prepared waste bone was impregnated with concentrated phosphoric acid (1M  $\text{H}_3\text{PO}_4$ ) at a raw material to acid ratio of 1:4 w/v) on weight basis. The saturated samples were dried in a Memmert oven at 100 °C for 2 hours. After the activation, and slow cooling in air at room temperature, the slurry was washed with distilled water several times to reduce the pH value of the samples, filtered to be free from acid and then dried at a temperature of 110 °C for 4 hours and finally ground into a fine powder, sieved and stored in desiccators for further usage.

#### 2.2.4 Characterization of the catalyst

The synthesized catalyst was characterized to determine its morphology, elemental compositions, functional group, mineralogy and surface area using scanning electron microscope (SEM), X-ray fluorescence (XRF, ARL 9400XP), Fourier transform infra-red spectroscopy (FTIR BUCK model M530), X-ray diffractometer (XRD) and Brunauer-Emmett-Teller (BET, ASAP 2020) respectively.

### 2.2.5 Transesterification Reaction

50 g oil was weighed and placed in a flat bottom flask. Then 1.5 g of the catalyst (3 wt% of oil sample) and 13 mL of methanol was added to the oil. The flask was kept on a hot magnetic stirrer, heated to 60 °C for 2 hours and stirred with speed of 250 rpm. The mixture was then cooled, and allowed to stay for 24 hours. It was then separated into biodiesel and by-product. The yield was calculated using Equation (2).

$$Y = \frac{\text{weight of biodiesel}}{\text{weight of oil used}} \times 100 \quad (2)$$

The reaction was repeated with various methanol/oil molar ratio (6:1 – 14:1), amount of catalyst (1.0 – 5.0 wt%), reaction temperature (45 – 65°C), reaction time (1 – 5 h) and agitation speed (150 – 350 rpm).

### 2.2.6 Characterization biodiesel

The properties of the produced biodiesel were determined using ASTM D-6751. The properties of the biodiesel determined include: Acid value, FFA, saponification value, iodine value, density, viscosity, calorific value, flash point, cloud point, pour point and cetane number.

## III. RESULT AND DISCUSSION

### 3.1 Oil yield

The percentage of oil extracted from beniseed is 54%. The oil yield is relatively high and in reasonable agreement with the yield observed by Oje *et al.* (2021). The relatively high oil content of Beniseed will make it highly suitable and economical for industrial applications for FAME production, as any oil bearing seed that can produce up to 30% oil are regarded as suitable feedstock for FAME industrial production (Okolie *et al.*, 2012).

### 3.2 Characterization of oil

#### 3.2.1 Physicochemical Properties of the oil

The summary of the physicochemical properties of the oil is presented in the Table 1. The specific gravity obtained for Beniseed oil (BSO) lies within the standard range 0.87 – 0.98 for triglycerides (Oje *et al.*, 2021). The acid value of Beniseed oil extracted for this work is 6.0 mgKOH/g hence, the value signifies that it has good quality for biodiesel production. It was observed that the oil has moderate free fatty acid value of 3.3 %. This suggests that the pre-treatment of the oil is needed but this was avoided by use of heterogeneous catalyst. The oil has a saponification value of 185.1 mgKOH/g oil which is within the ranges of the standard 175 – 205 mgKOH/g oil (Okolie *et al.*, 2012; Oje *et al.*, 2021). This value shows that the Beniseed is a normal triglyceride and suitable for FAME production.

The density and high viscosity of the oil will cause atomization problem in internal combustion engine, therefore it cannot be used directly as bio-diesel. The low pour point shows that the oil will not solidify easily at room

temperature hence will have a longer storage life. The oxidation stability of the oil is within the acceptable limits and it is a good feedstock for biodiesel production.

**Table 1: The properties of the Beniseed oil (BSO).**

Properties	BSO
Density (g/cm <sup>3</sup> )	0.90
Kinematic viscosity (mm <sup>2</sup> S <sup>-1</sup> at 40°C)	45.94
Free fatty acid (mgKOH/g oil)	3.30
Iodine value (g/100g oil)	38.61
Saponification value (mgKOH/g oil)	185.10
Refractive index	1.42
Peroxide value (meq/kg)	0.54
Moisture content (%)	0.03
Acid value (mgKOH/g oil)	6.00
Calorific value (MJ/g)	30.31
Boiling point (°C)	200
Flash point (°C)	262.0
Fire point (°C)	274.2
Molecular weight (g/mol)	828.41
Oxidation stability 11°C (Hour)	5.50

### 3.2.2 Fatty acid profile of BSO (GC –MS)

The fatty acid compositions of BSO is presented in Table 2. From the table, it shows that the oil contained eight different main fatty acids: palmitic, stearic, oleic, Lignoceric, linoleic, Caproic, Lauric acid and Linolenic acid. Linoleic acid is the most abundant with 49.81% of the total fatty acid present. It consists of 25.57% of saturated acids (stearic acid, palmitic acid, Caproic acid, lauric acid and Lignoceric acid) and 74.43% unsaturated acids (oleic, linoleic, Linolenic acid). The dominant unsaturated fatty acid of the oil was Linoleic acid, which accounted for 49.81% of the total fatty acid content, hence, the oil belongs to oleic acid category (Sonntag, 2012). The oleic acid content of BSO as reported in Table 2 is comparatively higher than 7-40% reported for coconut oil, palm oil and cottonseed oil (Ampaitepin *et al.*, 2006; Rashid *et al.*, 2009). This shows that BSO oil is highly unsaturated triglycerides. However, the fatty acid components of the BSO is similar with the values obtained by Oje *et al.*, (2021).

**Table 2: Fatty acid composition of BSO oil.**

Fatty acid	Structure	Composition (%)	Molecular weight (g/mol)
Caproic acid	C6:0	7.90	116.20
Stearic acid	C18:0	7.99	284.48
Palmitic acid	C16:0	5.98	256.4
Oleic acid	C18:1	24.81	304.470
Linoleic acid	C18:2	49.81	294.48
	C24:0	0.34	200.32
Lignoceric acid	C12:0	3.36	200.32
Lauric acid	C18:3	0.38	368.63
Linolenic acid			

### 3.3 Characterization of waste bone Catalyst

#### 3.3.1 X-ray fluorescence (XRF) of waste bone catalyst

The elemental compositions of raw and acid activated waste cow bone catalyst revealed the presence of numerous elements as depicted in Table 3. The calcium and phosphorous are the most abundant elements. The calcium content is higher in raw and lower in acid activated waste bone. The large quantities of calcium and phosphorous confirmed that calcium phosphate and carbonate are abundant.

**Table 3: Elemental analysis of waste bone catalysts.**

Elements	Concentration (wt%)	
	Raw waste bone	Acid activated waste bone (AWB)
Na <sub>2</sub> O	0.720	0.003
MgO	0.500	0.246
Al <sub>2</sub> O <sub>3</sub>	1.180	0.859
SiO <sub>2</sub>	5.340	3.850
P <sub>2</sub> O <sub>5</sub>	26.45	33.32
SO <sub>3</sub>	1.780	0.001
Cl	0.390	0.511
K <sub>2</sub> O	0.340	0.091
CaO	59.95	79.16
TiO <sub>2</sub>	0.470	0.074
Cr <sub>2</sub> O <sub>3</sub>	0.000	0.004
Mn <sub>2</sub> O <sub>3</sub>	0.005	0.008
Fe <sub>2</sub> O <sub>3</sub>	1.470	0.107
ZnO	0.590	0.031
SrO	0.130	0.131

#### 3.3.2 Fourier transforms infrared spectrometer of waste bone catalyst

Fourier transform infra-red (FTIR) spectroscopy of the raw waste bone and acid modified waste bone catalysts were done to ascertain the functional groups present in them, the results are presented in Figures 1(a & b). The FTIR spectrum was scanned from 4000-400 cm<sup>-1</sup>. The presence of hydroxide and phosphate functional groups were identified by FTIR spectra. The characteristic PO<sub>4</sub><sup>3-</sup> absorbed bands of the acid activated waste bone was observed with the broad bands at 3281.4 to 2281.12 cm<sup>-1</sup>, attributable to adsorbed water. The sharp peaks at 1505.8 to 723.1 cm<sup>-1</sup> represent the vibration peaks of PO<sub>4</sub><sup>3-</sup>. The present of weak sharp transmittance band at 3261.4 to 3112.3, could be as a result of activation with high H<sub>3</sub>PO<sub>4</sub> concentration. This peak may be assigned to isolated O–H groups. Due to the activation process, the bands may be also ascribed to the stretching mode of hydrogen-bonded P=O, to O–C stretching vibrations in P–O–C and to P=OOH. The FTIR results were similar to those reported by Hengbo *et al.*, (2007), Oje *et al.*, (2021) and Ghobadian *et al.*, (2009).



surface area and pore volume when compared with the raw (Ajayi *et al.*, 2009).

Table 5: Surface area and porosity analysis of raw catalyst and acid activated waste bone catalyst.

Sample name	Raw waste bone	Acid activated waste bone
Surface area (m <sup>2</sup> /g)	11.234	114.913
Pore volume (cm <sup>3</sup> /g)	0.004	0.137
Pore size A <sup>o</sup>	5.901	2.647

### 3.4 Influence of process parameters on the yield of biodiesel

#### 3.4.1 Influence of catalyst concentration on biodiesel yield

The influence of catalyst weight was studied between 1 – 5 wt% (based on weight of the oil) while other variables were kept constant. Figure 3 showed that as catalyst weight increased, the yield of biodiesel also increased up to 4% weight of catalyst and decreased at higher catalyst weight. The more yield of biodiesel with increase in catalyst weight is due to presence of more catalyst in the reaction medium. Addition of the catalyst beyond 4% weight showed decrease in the amount of biodiesel yield. This may be due to excess catalyst causing dispersion and mixing bottlenecks, thereby inhibiting the formation of FAME (Ude and Onukwuli, 2019; Oje *et al.*, 2021).

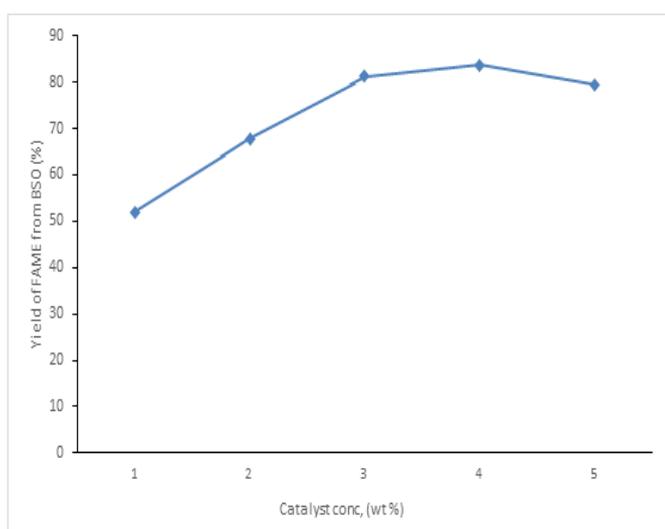


Figure 3: Influence of Catalyst Conc. on biodiesel yield: Reaction conditions: Time = 2 hr, Temperature= 60 °C, Speed= 250 rpm, mole ratio = 12:1

#### 3.4.2 Influence of methanol/oil molar ratio on FAME yield.

Figure 4 presents the influence of methanol/oil molar ratio on the yield of biodiesel produced. Methanol to oil molar ratio was studied between the range of 6:1 to 14:1 and other variables were constant. The result from the figure, indicates that the molar ratio positively influenced the yield. The more yield was gotten at a methanol/oil molar ratio of 12:1. The higher molar ratio resulted in higher yield of biodiesel. The yield decreased when the molar ratio was

above 12:1. This may be attributed to reduction in the activities of catalyst making it difficult to remove the glycerol (Leung and Guo, 2010; Oje *et al.*, 2021).

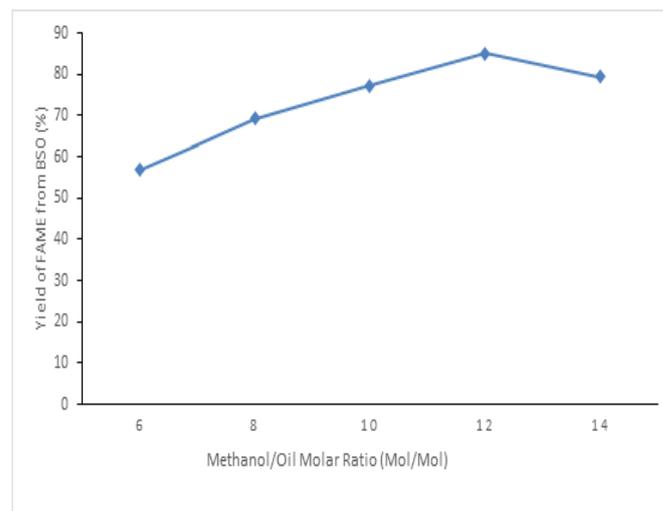


Figure 4: Influence of methanol /oil molar ratio on biodiesel yield. Reaction conditions: Time = 2hr, Temperature 60 °C, Speed= 250 rpm, catalyst conc. = 3.0 wt. %.

#### 3.4.3 Influence of temperature on biodiesel yield

The influence of temperature between 45 °C to 65 °C on biodiesel yield was studied keeping other parameters constant and depicted in Figure 5. The figure shows that the reaction rate was slow at low temperatures, but biodiesel yield first increased and then decreased with the increased of the reaction temperature beyond 60 °C. However, a more rapid reaction rate could be obtained at high temperatures, but at high temperatures, methanol was vaporized and formed a large number of bubbles, which inhibited the reaction on the two-phase interface. The result is in consonant with those of Ude and Onukwuli (2019) and Oje *et al.* (2021).

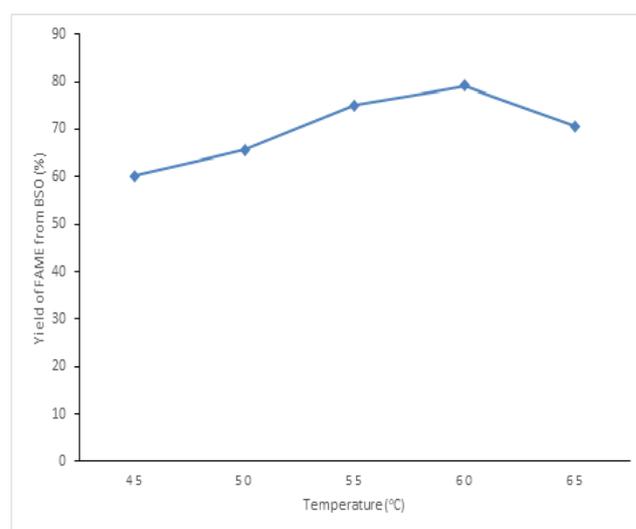


Figure 5: Influence of temperature on biodiesel yield. Reaction conditions: Time = 2 hr, molar ratio =12:1, Speed= 250rpm, catalyst= 3.0 wt. %

### 3.4.4 Influence of speed of agitation on biodiesel yield

The biodiesel yield at various rate of agitation is depicted in Figure 6. There was incomplete reaction at lower agitation rate resulting in low yield. The yield decreased as the rate of agitation was higher than 300 rpm. The backward reaction could be as a result of high entropy of the reacting system and reduction of contact time of the reactants thereby retarding the formation of biodiesel. These results are in conformity with observations made by Oje *et al.* (2021), who studied the effect of agitation speed on the transesterification of beniseed oil catalyzed by acid activated eggshell and concluded that higher agitation promoted the homogenization of the reactants and thus led to higher yields.

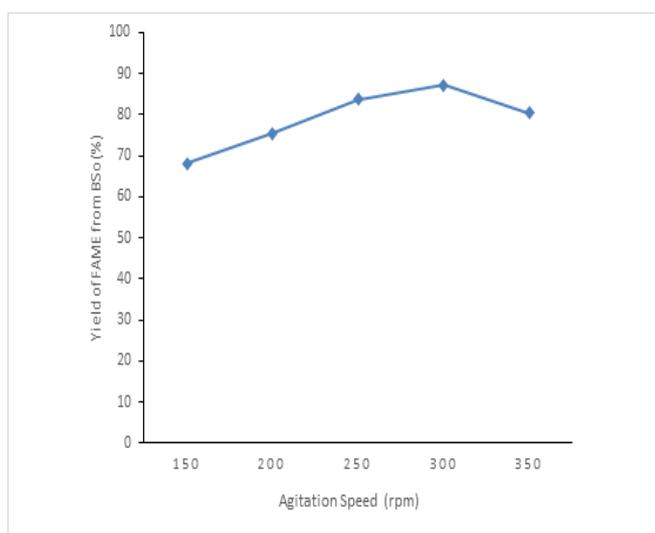


Figure 6: Influence of agitation speed on biodiesel yield. Reaction conditions: Time= 2 hr, Temperature = 60 °C, molar ratio=12:1, catalyst= 3.0 wt. %.

### 3.4.5 Influence of time on biodiesel yield

Reaction time was studied from 1 to 5 hours on biodiesel as other parameters were kept constant and presented in Figure 7. From the figure, it shows that there was a reaction delay at initial time due to dispersion and mixing of methanol and catalyst into BSO from 1 hour. But as the reaction time progresses, the yield rises and then declined as the time further goes up. This was due equilibrium attained and reverse reaction that occurred. From the figure, it shows that the maximum ester yield of 77% was obtained from transesterification of beniseed oil with acid activated waste bone. The figure also indicated that an increase of the reaction time from 4 hours to 5 hours would not yield further increase in biodiesel yield but leads to a reduction in the product yield. This is because longer reaction time enhanced the hydrolysis of esters (reverse reaction of transesterification), resulting in loss of esters (Eevera *et al.*, 2009). Similar result was also reported by Oje *et al.*, (2021).

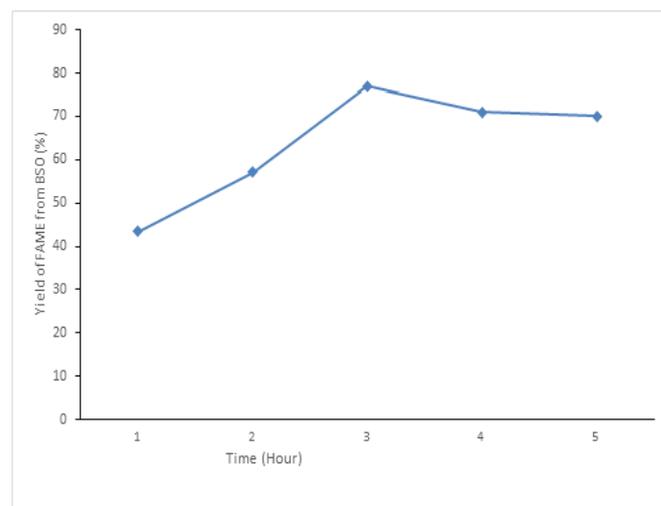


Figure 7: Influence of time on biodiesel yield. Reaction conditions: methanol/oil molar ratio= 12:1, Speed= 250 rpm, catalyst= 3.0 wt. %.

### 3.5 Characterization of biodiesel

The fuel properties of biodiesel produced are presented in Table 6, and the limits that they were compared with (ASTM D 6751 (2002) standards).

From the result it can be inferred that the viscosity of biodiesel is 3.90 Cst and within the acceptable limit. That is to infer that biodiesel from BSO via acid activated waste bone has a good injection and atomization performance. Furthermore, it will offer lubrication and protection for the moving parts of an engine superior to those of the diesel. Density is an important fuel property, because injection systems, pumps, and injectors must deliver an amount of fuel precisely adjusted to provide proper combustion. Biodiesel generally has a higher density than Petro-diesel. This has a significant impact on fuel consumption as fuel introduced into the combustion chamber is determined volumetrically. The default value of 40 °C density specified in ASTM D6751 is 0.82–0.90 g cm<sup>3</sup>. The result from Table 6 show that the density value of biodiesel obtained meet the density value specified by the ASTM standard for biodiesel. The flash point for the biodiesel produced from BSO falls within the ASTM standard, and makes it safe for handling and storage.

Cetane number is a measure of ignition quality of a fuel. This is the most pronounced change from vegetable oil to the trans-esterified product (biodiesel). Fuels with low cetane numbers show an increase in emission due to incomplete combustion. The lower limit for cetane index is 47 by ASTM standards. The cetane number of the BSO biodiesel is greater than 50 as presented in Table 6, which is above the lower limit for cetane number. The result obtained is acceptable.

**Table 6: Fuel properties of Biodiesel compared with ASTM limits.**

PROPERTY	UNITS	ASTM METHODS	BSO FAME by AWB	ASTM LIMITS
Density	kg/m <sup>3</sup>	ASTM D-1298	864.6	830-880
Kinematics Viscosity	Cst	ASTM D-445	3.91	1.6-6.0
Flash Point	°C	ASTM D-93	155	≥130
Pour Point	°C	ASTM D-97	4	+15 max
Cloud Point	°C	ASTM D-2500	2	-15 to 5
Acid Value	mgKOH/g	ASTM D-974	0.4	≤ 0.80
Low Heating Value	MJ/kg		49.8	≥ 35
Aniline Point	(°C)	ASTM D-4737	178	
Higher Heating Value	MJ/Kg		54	
Cetane number		ASTM D-130	58	47 min
Iodine Value	12/100g	ASTM D6751	45.01	120 Max

#### IV. CONCLUSION

The transesterification of beniseed oil catalyzed by acid activated waste bone was carried out. The oil from the seed was extracted by solvent extraction methods using n-hexane. The result revealed that the yield of oil was reasonably enough for biodiesel production. The low acid value, and FFA value of the oil enable it to undergo direct transesterification without treatment using the acid activated waste bone. Maximum yield of 75 – 80% was achieved and the process parameters have significant effects on the yield. The properties of the biodiesel such as density, viscosity, cetane index, oxidative stability and higher heating values of biodiesel produced meet the ASTM standard and were within the acceptable limits.

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