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Investigating the Potential of "White-Seed Melon" (*Cucumeropsis Mannii*) oil as a Biodiesel Feedstock.

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ABSTRACT

The controversial food-fuel debate over the conventional vegetable oil has inspired the continued quest among researchers for a more feasible crop oil. The present work is an approach to study white melon seed oil (WSMO) as a potential feedstock for biodiesel production. In this paper, crude WSMO was extracted from the seed via soxhlet extraction, characterized for its physicochemical properties and then transesterified with methanol using sodium hydroxide as catalyst at 60^0C for 1hr. Oil:methanol ratio of 1:6 was used to produce a yield of 82% of the corresponding methyl ester (WSMOME), thus confirming its feasibility for mass production. The fuel properties of WSMOME are in agreement with the American Society for Testing and Materials (ASTM) standard, suggesting that WSMO can be used for biodiesel production. The fuel properties of WSMOME were determined and the values are as follows: flash point (146^oC), cloud point (4.6^oC), carbon residue (64%), kinematic viscosity ($3.24mm^2/s$), ash content (0.03%), acid value (1.24mg KOH/g), moisture content (0.03%), specific gravity (0.87kg/l) and cetane number (54). These values suggest a good fuel performance when compared to the ASTM standard for conventional biodiesel and fossil diesel. Also, the FAME profile of WSMOME indicates a 59.82% and 40.18% unsaturation and saturation level respectively. The potential of white seed melon oil as a biodiesel feedstock is clearly presented in this study.

KEYWORDS: Biodiesel, Cucumeropsis mannii, Transesterification, Renewable energy, White melon, Seed oil, FAME.

1. Introduction

The widespread use of diesel fuel has raised concerns due to its contribution to air pollution and the gross dependence on finite fossil fuel resources. Biodiesel offers a cleaner alternative to traditional fossil diesel by emitting fewer harmful pollutants when burnt, thus emerging as a promising solution. The conversion of food to fuel has long been a controversial debate and this has inspired the continued quest among researchers for a more feasible feedstock. The widespread use of diesel fuel in various sectors has raised concerns due to its contribution to air pollution and dependence on finite fossil fuel resources. To address these challenges, biodiesel emerges as a promising solution. Biodiesel, derived from renewable sources such as vegetable oils or animal fats, offers a cleaner alternative to traditional diesel, emitting fewer harmful pollutants when burnt.

Fossil fuels (petroleum, coal and natural gases) are the world's major sources of energy. Considering the gross dependence on fossil fuel by nation's leading to its scarcity and consequent increase in the emission of combustion-generated pollutants, there is a need to explore the opportunities in renewable energy. With the increasing demand for sustainable fuel alternatives, the search for novel feedstock sources becomes crucial.

The use of vegetable oils such as olive, sunflower, soybean and peanut oil has been explored as an alternative fuel substitute to the conventional fossil-based diesel. Biodiesel is defined as the mono alkyl esters of long chain fatty acids obtained from renewable feedstock, such as vegetable oil or animal fats, for use in compression ignition engines [1]. In recent times, biodiesel has attracted more attention due to its environmental friendliness, derived from renewable sources, biodegradable and non-toxic nature. It can also be produced from any material that contains fatty acids, either linked to other molecules or present as free fatty acids [2]. Much studies have been made on the use of various vegetable fats and oils, animal fats, waste greases, and edible oil as a viable feedstock for biodiesel production [2]. Biodiesel is produced by the catalytic transesterification of triglycerides with methanol/ethanol.

Considering their availability, various oils have been used as feedstock for the production of biodiesel in different countries. Soybean oil in the USA, palm oil in Malaysia and Indonesia, coconut oil in the Philippines are being used for biodiesel production. Also, the jatropha tree (*Jatropha curcas*), karanja (*Pongamia pinnata*) and mahua (*M. indica*) are used as major biodiesel fuel sources in India [3]. The use of vegetable oils (palm, soybean, sunflower, rapeseed, etc.) as a feedstock for biodiesel production has continued to compete with its use for food thereby adding more stress on the price, production and availability of these oils [4]. Consequently, this has piqued more interest in the search of other more feasible feedstocks. Over 350 oil bearing crops have been identified, and recent studies made on the production of biodiesel from less common or conventional oils including *Moringa oleifera* [4], pumpkin [5], desert date [6], *Michelia champaca* and *Garcinia indica* [7], sea mango [8], and Rubber [9] seed oils.

Cucumeropsis mannii commonly known as white melon seed, “egusi” in Yoruba, “'egwusi” in Igbo and “agushi” in Hausa is a member of the Cucurbitaceae family. This plant family is known for its great genetic diversity and widespread adaptation which includes tropical and subtropical regions, arid deserts and temperate locations [10]. Cucurbits are known for their high protein and oil content. Seeds of cucurbits are sources of oils and protein with about 50% oil and up to 35 % protein [11]. This justifies the reason for its wide cultivation and consumption over the world. In Nigeria, it is used both as condiment and thickener in local soup, and the industrial scale production of the oil is yet to be utilized despite the huge potential [12]. Various studies have reported predominantly high linoleic fatty acid content in white seed melon oil.

Fig. 1. Image of white seed melon

For the purpose of this study, white seed melon (*Cucumeropsis mannii*) has been referred to as white seed melon (WSM) for proper identification and consistency with the literature. The main objective of the present study was to investigate the white seed melon oil (WSMO) as a potential feedstock for biodiesel production. The fuel properties of the white seed melon oil methyl ester (WSMOME) were determined and compared with fossil diesel and biodiesel from conventional vegetable oils.

Transesterification

Transesterification, also called alcoholysis, is the displacement of alcohol from an ester by another alcohol [13]. It is the most used method of conversion and refers to the reaction of a vegetable oil or animal fat with an alcohol in the presence of a catalyst to produce alkyl esters and glycerol. The alkyl esters are what are called biodiesel.

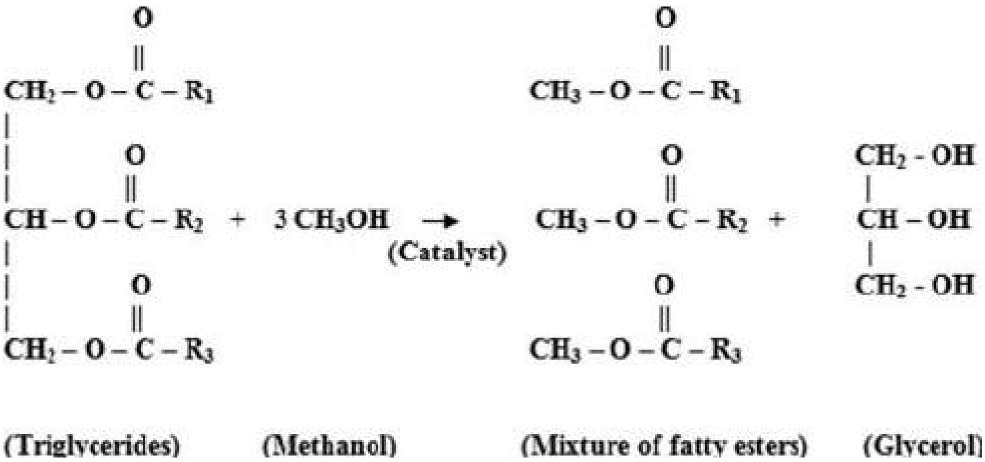

Fig. 2. An equation for a general transesterification reaction.

According to [14], the biodiesel yield is dependent on the oil/methanol/catalyst ratio. Three different ratios of oil/methanol/catalyst were used in the production of the biodiesel to study the effect of menthol that gives the best yield while keeping the temperature, reaction time and catalyst constant as given in the table below. The result shows that the ratio 1:9:0.3 gave the highest biodiesel yield.

	Oil	NaOH (g)	Methanol	% Yield	Temp. (^oC)	Time (mins)
Ratio 1	1	0.3	10	68	45	40
Ratio 2	1	0.3	9	77	45	40
Ratio 3	1	0.3	7	72	45	40

Table 1. Effect of oil/methanol/catalyst ratio on the yield of biodiesel (WSMOME)

2. Experimental section

2.1 Materials and methods

N-Hexane solvent, Methanol (99.8% purity) and sodium methoxide (95%) were purchased from a Chemicals store in Onitsha main market, Anambra state, Nigeria. All chemicals used for this work were analytical reagent grade. Experiments were done in triplicates to determine the physical and chemical properties of the WSMO and WSMOME and then expressed as mean values.

2.2 Sample collection and preparation

The white seed melon was purchased and dehusked from a local market in Gboko, Benue State, Nigeria and transported to FUTO for this study. This seed was sun-dried for seven days and ground using a manual hand grinder.

2.2. Extraction of oil from the seed using the soxhlet apparatus.

To extract the WMSO, soxhlet extraction was carried out using n-hexane. 30g of the grounded sample weighed into the extraction thimble. The thimble and the sample were inserted into the soxhlet apparatus before adding about 250 ml of n-hexane in a 500 ml flat bottom flask. The set-up was mounted on a heating mantle and heated at 68 °C for 4hrs. After the extraction process, the extract was exposed to the atmosphere and later dried in an oven at 40°C for 6hrs. The percentage oil yield was calculated using Equation (1). The average yield was reported after two batches of extraction.

$$\% \text{ oil Yield} = \frac{Weight\ of\ oil\ extracted,\ W2}{Weight\ of\ ground\ seed\ used\ W1} \text{ X } 100 \qquad (1)$$

2.3. Transesterification of WSMO

Transesterification of the oil was carried out in a round bottom flask reactor equipped with a thermometer, a heating mantle with magnetic stirrer, and a reflux condenser. 100g of the extracted WSMO which was preheated was measured into the round bottom flask. The reaction was carried out at 60^0C for 1 hour with 1wt% of sodium methanoate (catalyst) and oil: methanol ratio of 1:6 [4]. At the end of the reaction, the resulting mixture was cooled and transferred into a separating funnel and allowed overnight for the separation of glycerol and methyl ester by gravity. The upper part is composed of glycerol while the lower part is composed of methyl ester. The two phases were carefully collected separately. The resulting methyl ester obtained was purified by successive washing with warm deionized water to remove residual impurities (catalyst, glycerol methanol and soap). To neutralize the remaining soaps and catalyst, a small quantity of sulphuric acid was used to do the second washing. Finally, the resultant WSMOME was dried using anhydrous sodium sulphate to remove moisture. The % yield was calculated as follows:

$$\% \text{ Biodiesel Yield} = \frac{Weight\ of\ methyl\ ester\ produced, W1}{Weight\ of\ oil\ used\ in\ reaction, W2} \text{ x } 100 \qquad (2)$$

2.4 Characterization of WSMO and WSMOME

The physicochemical properties of the extracted WSMO and the biodiesel produced was carried out in the Chemistry laboratory of Federal University of Technology Owerri, Imo state, Nigeria using standard methods.

The Physical properties determined are density, kinematic viscosity, flash point, cloud point and pour point using ASTM: D1298. D445, D93, D2500 and D97 standard test procedure respectively.

Determination of the Flash point

Flash point indicates the least temperature at which a liquid ignites when exposed to an ignition source. For this study, the flash point of WSMOME was determined using the ASTM D93 Pensky- Martens closed cup tester. 50 ml of WSMOME was placed in the test cup and stirred at a constant rate of 250r/min ± 10r/min. The stirring was only stopped to apply the ignition source. This was heated at a slow constant rate and at every 10^oC temperature rise, a flame introduced using a shutter. The flash point was recorded as the least temperature at which a flash appears in the form of sound and light.

Determination of the Flash point

Flash point is the lowest temperature (corrected to a standard pressure of 101.3 kPa) at which the application of an ignition source causes the vapors of a liquid to ignite under specified test conditions. This was determined using the ASTM D93 Pensky-Martens Closed Cup Tester. 50mL of the sample was placed in the test cup. The sample was stirred at a constant rate of 250r/min ± 10r/min and the stirring was stopped only to apply the ignition source. The sample was heated at a slow constant rate. At every 10°C temperature rise, a flame was introduced with the help of a shutter. The temperature at which a flash appeared in the form of sound and light was recorded as the flashpoint.

Determination of the kinematic viscosity

The viscosity of the test sample was taken using the old oil glass viscometer, using the mouth the biodiesel in the lower bulb was sucked to a point above the top white ring mark of the second bulb of the old glass viscometer. The sample meniscus was adjusted by releasing the thumb till it is at the same level with white ring mark on top of viscometer second bulb. The sample was allowed to flow and a stop watch was used to take the time interval of the flow. The time for the biodiesel to pass the second ring mark was recorded. Similar procedure was repeated for water and the time recorded. The viscometer is calculated from the results obtained.

Determination of the Cloud Point

A portion of the biodiesel was poured into a test tube and the mercury point of the thermometer with calibration below 10ºC was inserted in the test tube. The set up was inserted in a beaker containing ice. The test sample was observed closely. After some time, the sample was observed to form a cloud of gel. The temperature was taken as the cloud point was recorded.

Determination of the Pour Point

The same set up as in the cloud point test was immersed in the ice and left to solidify. When the solidification was confirmed, the test tube was removed and tilted and closely observed till it started to flow. The instant temperature taken on observing the flow of solidified sample was recorded as the pour point temperature.

Specific gravity

The specific gravity of WSMO was determined using a specific gravity bottle. A clean empty specific gravity bottle was weighed on a digital weighing balance (Virgo V600 – W, Haryana, India) and the weight (w_1) was noted. It was then filled with distilled water and the weight (w_2) was noted. The water was removed and the specific gravity bottle was dried and cooled. It was then filled with WSMO and weighed (w_3). These were all carried out at 25°C. The specific gravity was calculated using Equation (3) below.

Specific gravity (cm^3) = $\frac{W3-W1}{W2-W1}$ (3)

Where W_1 is the weight of the empty specific bottle, W_2 is the weight of the specific gravity bottle and water, W_3 is the weight of the specific gravity bottle and the WSMO.

The properties determined include viscosity, iodine value, refractive index, specific gravity, acid value peroxide value and saponification value were analyzed. The fuel properties determined include flash point, cloud point, pour point, heat of combustion, moisture content and ash content. The free fatty acid content of the oil was determined using GC-MS analysis. The color of the oil at room temperature was determined through visual inspection.

Determination of viscosity of the WSMO

The kinematic viscosity of the oil was determined using a digital viscometer. The required spindle was selected and fixed on the viscometer. The spindle was then inserted into the WSMO until the level mark on the spindle reached the surface of the WSMO. The Enter button on the instrument was pressed and the viscosity of the MSMO was displayed on the screen.

Chemical Properties

The chemical properties were determined using the standard test procedure as given below.

Saponification Value determination

The saponification value was expressed as the number of milligrams of potassium hydroxide (KOH) required to saponify 1g of oil. Determination of saponification value was according to AOCS method Cd 3-25 (AOCS, 1990). The 2g of oil were dissolved with ethanol in the Erlenmeyer flask. Then connect it with an air condenser and boil gently for 1 hr in order that sample is completely saponified. After it cooled and added 1ml phenolphthalein indicator. The mixture was titrated with 0.5N HCl until the pink color had just disappeared.

S.V. = $\frac{(B-R)\ X\ 28.05}{Weight\ of\ sample}$ (4)

Where,

B = Blank titre

R= Real titre value

Peroxide value determination

Peroxide value is a measure of peroxides contained in the oil and is determined by measuring iodine released from potassium iodide. Determination of peroxide value was performed according to AOCS official method Cd 8b-90 (AOCS, 2005). The oil sample (5±0.01g) was dissolved in 30 mL acetic acid-chloroform (3:2) solution. Then saturated KI solution and distilled water were added and the flask was shaken vigorously to liberate iodine from the chloroform layer. The mixture was titrated with 0.01N sodium thiosulphate using starch solution as an indicator.

$$\text{Iodine Value} = \frac{(S-B)\ x\ Normality\ of\ Na2S2O3\ x\ 1000}{Weight\ of\ sample} \quad (5)$$

Where,

S= Sample titration (ml)

B= Blank titration (ml)

Cetane Number (CN)

The calculated saponification value (S.V) and iodine value (I.V) were used to calculate the cetane number (CN) which is the ability of fatty acid methyl esters as a fuel to ignite quickly after being injected. Empirical formula was proposed by [15] and was used in the work. The higher its value, the better is its ignition quality. This is one of the most important parameters which is considered during the selection of fatty acid methyl esters for use as a biodiesel. The cetane number was determined using the relation provided by [16].

$$\text{Cetane Number (CN)} = 46.3 + \frac{5458}{S.V - 0.225\ X\ I.V} \quad (6)$$

3.0 Results and Discussion

Physicochemical properties of white melon seed oil (WMSO)

Crude WSMO was freshly extracted from white seed melon and used for the purpose of this study. The oil was extracted by soxhlet extraction using hexane as solvent. The physicochemical properties of the oil were determined afterwards as shown in table 1. The saponification value and acid value were determined to be 207.2 mg KOH/g and 0.92 respectively. With an acid value of 0.92 mg KOH/g corresponding to 0.46% free fatty acid (FFA) content, transesterification of the oil was carried out (via a single-stage reaction process) [5]. Additionally, it has been reported that <0.5% FFA content is required for effective transesterification to avoid soap formation resulting from high fatty acid [3,5]. The kinematic viscosity of the crude WSMO was 32.2 mm^2/s which was similar to that reported for soybean oil [17]. The density of the oil was determined to be 908.4kg/m3. These results are within the limits reported for conventional vegetable oils [17]. All the properties of WSMO were in agreement with previous studies [18-20].

Physicochemical parameters	**values**
Peroxide value (mg/g oil)	1.32
Density @ 15°C (kg/m^3)	908.4
Kinematic viscosity @ 40°C (mm^2/s)	32.4
Saponification value (mg KOH/g)	206.1
Acid value (mg KOH/g)	0.89
Free fatty acid (%)	0.51
Calorific value (MJ/kg)	40.1
Yield (%)	68

Color	5Y + 0.4R
Ave. Molecular weight (g)	874

Table 2. Physicochemical properties of WSMO

Fatty Acid Methyl Ester (FAME) composition of WSMOME

The fatty ester profile of WSMOME, as determined by gas chromatography, is provided in Table 2. The result shows that WSMOME is composed of six FAMEs namely: palmitate, palmitoleate, stearate, oleate, linoleate and linolenate. From the Fatty acid profile, the total unsaturated fatty ester is 59.82% while the total saturated fatty ester is 40.18%. The FAME composition of WSMOME determined by the fatty acid profile influences the engine performance, fuel stability and emissions significantly thus impacting on the overall efficiency and environmental impact of the diesel engine. A higher unsaturation level will result to higher NOx emissions and peak pressure of the engine. However, hydrogenation can reduce the number of unsaturated bonds.

Fatty acid	**Structure**	**Weight (%)**
Methyl palmitate	16:0	14.65
Methyl palmitoleate	16:1	2.67
Methyl stearate	18:0	25.53
Methyl oleate	18:1	2.51
Methyl linoleate	18:2	43.40
Methyl linolenate	18:3	11.24

Table 3. A profile of the Fatty Acid Methyl Ester composition of WSMOME

Fuel properties of WSMOME compared to that of conventional biodiesel and fossil diesel.

The physicochemical properties of the WSMOME were determined by standard methods. From the result in the table, it shows that the WSMOME properties are in agreement with the ASTM standard for quality biodiesel.

Fuel properties	**WSMOME value (This study)**	**ASTM Limits**	
		Biodiesel D6751	**Fossil Diesel D975**
Specific Gravity @ 60^{o}C (kg/l)	0.87	0.88	0.85
Kinematic Viscosity (mm^2/s) @40^{o}C	3.24	1.9-6.0	1.3-4.1
Carbon residue (% wt)	64	77	87
Flash point (^{o}C)	146	130-170	60-80
Moisture content (% vol)	0.03	0.05 max	0.05 max
Ash content (%wt)	0.03	0.04	0.07
Acid value (mg KOH/g)	1.24	1.740	0.25
Cetane Number	54	47-65	40-55
Pour point (^{o}C)	5.2	-15 to 10	-35 to -15

Cloud point (°C)	4.6	-3 to 12	-15 to 5
Density (g/cm^3)	0.91	0.88	7.079

Table 4. Fuel properties of WSMOME

Specific gravity of WSMOME is a measure of lightness or heaviness of the methyl ester in relation to water. The agreement in the relative density value for the WSMOME (0.87) with fossil diesel (0.85) makes it possible to blend the two together.

The kinematic viscosity of the biodiesel is 29.8% higher than that of fossil diesel, which signifies a more resistance to flow of the biodiesel under gravity than the fossil diesel.

The carbon residue from the WSMOME shows 16.8% and 26.4% decrease in carbon residue when compared to a conventional biodiesel and fossil diesel respectively. This suggests that the WSMOME will leave a lower carbon residue during thermal combustion. This lower carbon residue suggests an improved engine cleanliness and thus reducing the maintenance cost over a long time.

Flash point suggests the fuel ignition temperature when exposed to flame. The flash point for WSMOME was determined to be 146°C which is within the accepted limit for conventional biodiesel by ASTM. The flash point of WSMOME being higher than that of fossil diesel (60°C-80°C) is an indication that it is less flammable than the conventional fossil diesel.

A 40% decrease in moisture was identified for WSMOME compared to fossil diesel. A low moisture content indicates less vulnerability to microbial growth and corrosion in storage tanks.

Ash content is a measure of the mineral content of the fuel. 25% and 57.1% decrease in ash content was identified for WSMOME compared to conventional biodiesel and fossil diesel respectively. This result indicates that fossil diesel contains almost double of noncombustible inorganic matter than biodiesel.

Acid value of WSMOME is 28.7% lower than that of conventional biodiesel, suggesting a lower free fatty acid content and thus lower moisture content. With 79.8% acid value higher than the fossil diesel, WSMOME has more free fatty acid content as expected.

4. Conclusion

WSMO was reacted with methanol through a transesterification reaction using sodium hydroxide as a catalyst to produce WSMOME. The high oil and biodiesel yield of 68% and 82% respectively is an indication of its potential for commercial production. Results from this study has shown that the fuel properties of WSMOME is in conformity with ASTM standard and also agrees with the values for biodiesel from sunflower and soybeans as established in literature. Thus, considering the good fuel performance of WSMOME and the under-utilization of white melon seed oil, it could be concluded that WSMO is a viable feedstock for the production of biodiesel.

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