International Journal of Agriculture and Environmental Research

ISSN: 2455-6939

Volume:03, Issue:01

EXTRACTION AND CHARACTERIZATION OF WATERMELON SEED OIL

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ABSTRACT

This work is about the production of biodiesel from watermelon (*Citrullus lanatus*) seed oil. The oil extraction was done using solvent extraction method. The basic test for suitability of the obtained watermelon oil was carried out. Using trans-esterification process, watermelon oil was heated to 60°C, solutions of methoxide ($NaOCH_3$) were also added and stirred continuously for an hour. It was left to settle for 24 hours. After shaking, the mixture was poured into a separating funnel and was allowed to stand for 24 hours. The biodiesel was obtained using the separating funnel, separating biodiesel and glycerine. The washed biodiesel was collected into a beaker and gently heated in an oven at 105°C to evaporate the excess water and methanol in the biodiesel. The results of the test classified the watermelon biodiesel within limits set for biodiesel's properties by ISO 14214 specifications on Biodiesel.

Keywords: Biodiesel, Watermelon seed oil; Methoxide; Glycerine; Trans-esterification; Solvent extraction; Methanol; Evaporate

INTRODUCTION

In simple words, biodiesel can be defined as a natural and renewable domestic fuel made from vegetable oil. Biodiesel can also be defined technically, as a fuel comprised of mono-alkyl esters of long chain fatty acids derived from vegetable oil or animal fats, and is typically made by reacting lipids with an alcohol. The "mono-alkyl ester" in the biodiesel is the product of the reaction of a straight chain alcohol, such as methanol or ethanol, with fat or oil (triglyceride) to form glycerol (glycerine) and the esters of long chain fatty acids. The world's major sources of energy today are petroleum, coal and natural gas which are fossil-derived and non-renewable which have been and is the major source of development in some countries. The world at large also depends on petroleum products as the main energy source. The way countries are over-

ISSN: 2455-6939

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dependence on petroleum products has caused increase in emission of combustion which generates pollution into the environment and the use of the energy sources over many years have also resulted in the rise of global temperature levels also known as global warming which is due to high levels of carbon that are released as by-products and exhaust gases. There are also serious cases of oil spillage which tends to destroy the immediate or remote marine life and environment where petroleum resources are found. Most of the political and socio-economic problems facing countries today are because of the instability of petroleum product.

Due to these problems it has become necessary to diversify and find alternative sources for fuel. Rudolph Diesel was the first to test peanut oil in his combustible ignition engine in the 1800's, during this period various oils where extracted from different plants used to run the engines such as melon seed oil, jatropha seed oil, soya bean, palm oil, sunflower, peanut oil, corn oil, sugar cane and olive oil which are produced in large quantities for use as fuel substitutes. The advantage of biodiesel fuel is that it is a renewable energy source unlike petroleum-based diesel. Another main biodiesel fuel advantage is that it is less polluting than petroleum diesel and the lack of sulphur in 100% biodiesel extends the life of catalytic converters. Its primary advantage is that it is one of the most renewable fuels currently available and it is also non-toxic and biodegradable. It can also be used directly in most diesel engines without requiring extensive engine modifications. Biodiesel fuel can also be used in existing oil heating systems and diesel engines without making any alterations. The lubricating property of the biodiesel may lengthen the lifetime of engines.

The aim of this research work was to produce biodiesel and to characterize the produced biodiesel from water melon seed.

MATERIAL AND METHODS

Procedure for watermelon oil extraction

The watermelon seeds were removed manually and carefully blended using an electric blender. The blended samples were sieved to remove unwanted particulate substances and the final product was an averagely coarse aggregate. The initial moisture content was calculated at first using the formula:

$$Mc = \frac{W_1 - W_2}{W_1} \times 100$$

Soxhlet Extraction Method

ISSN: 2455-6939

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The Soxhlet apparatus used for solvent extraction where 300ml of n- Hexane was poured into round bottom flask.10 grams of powdered *Watermelon seed* was placed in the thimble and inserted in the centre of the extractor. The Soxhlet was heated at 60^{o}c. When the solvent was boiling, the vapour rises through the vertical tube into the condenser at the top. The liquid condensate drips into the filter paper thimble in the centre, which contains the oil to be extracted. The extract seeps through the pores of the thimble and fills the siphon tube, where it flows back down into the round bottom flask. This was allowed to continue for 30 minutes. It was then removed from the tube, dried in the oven, cooled in the desiccators and weighed again to determine the amount of oil extracted. The experiment was repeated by placing 5g of the *Watermelon seed into* the thimble. The weight of oil extracted was determined at 30 minutes interval. At the end of the extraction, the resulting mixture containing the oil was distilled off using simple distillation to recover solvent from the oil. The oil extracted was stored in a plastic container for further use.

Transesterification

Transesterification process using magnetic stirrer, A 500 ml 3-necked round bottom flask equipped with mechanical stirrer, thermometer and condenser with guard tube to prevent moisture entering into the system, is heated to expel residual moisture. On cooling, 200 ml of oil was then added to the flask. The oil was stirred and heated in a silicon oil bath to 60 ^{0}C at which a prepared sodium methoxide (40 ml methanol and 1 g NaOH) was added rapidly under stirring condition and the reaction continued for at least two hours at the same temperature. Two layers were observed clearly after cooling. The top and lower layers observed were biodiesel (refined oil) and glycerin respectively. The suspected biodiesel layer was neutralized by diluted acetic acid and then washed with distilled water.

Determination of the Percentage of Watermelon Oil

The crude and the refined oil were weighed separately and their percentage yield was calculated on dry matter basis as shown in equation.

$$\% \text{ of yield} = \frac{\text{Weight of oil}}{\text{Weight of sample on dry matter basics}}$$

Determination Density and specific gravity

An empty washed and dried beaker was weighed on the top load weighing balance. The weight of the beaker was recorded. Exactly 50 cm3 of each of the oil sample were measured and pour into the beaker and weighed. The weights of the 50cm3 of the samples were recorded. The

ISSN: 2455-6939

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procedure was repeated with water and the weight of 50cm3 of water was obtained. The density and the specific gravity were calculated thus

$$\text{Density of oil} = \frac{\text{Weight of oil sample}}{\text{Volume of oil sample}}$$

$$\text{Specific gravity of oil sample} = \frac{\text{Weight of oil}}{\text{Weight of equal volume of water}}$$

Determination Acid Value

1g of refined oil was weighed separately in 250ml conical flasks. 5ml of isopropyl alcohol was added into the conical flasks containing the oil samples with thorough stirring. Three drop of phenolphthalein indicator was added and titrated against 0.1N of KOH solution while shaking constantly until a faint pink persist for 30s. The end point was recorded and the acid value was calculated as;

$$\text{A.V} = \frac{\text{Titre value} + \text{molar Conc. of KOH} + 56.1}{\text{Weight of oil sample}}$$

$$\%\text{ of FFA} = \frac{\text{Titre value} + \text{molar Conc. of KOH} + 56.1}{\text{Weight of oil sample}}$$

Determination of Saponification Value

2g of the samples were weighed separately in 250ml conical flasks. 50 ml of ethanoic potassium hydroxide was added into the conical flasks containing the oil samples with thorough stirring. The resulting mixtures were boiled until the oil dissolves. Three drops of phenolphthalein indicator was added and titrated against 0.1N of KOH solution while shaking constantly until a faint pink persist for 30s.

$$\text{S.V} = \frac{(B - R) + \text{Molar Conc. Of HCl} + 56.1}{\text{Weight of oil sample}}$$

Determination of Iodine Value

0.4g of the samples was weighed into a conical flasks and 20ml of carbon tetra chloride was added to dissolve the oil samples. Then 25ml of Dam's reagent was added to the flasks using a safety pipette in fume chamber. Stoppers were then inserted and the content of the flasks were

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vigorously swirled. The flasks were then placed in the dark for 2 hours 30 minutes. At the end of this period, 20ml of 10% aqueous potassium iodide and 125ml of water were added to each sample using a measuring cylinder. The contents were titrated with 0.1M sodium-thiosulphate solutions until the yellow colour almost disappeared. Few drops of 1% starch indicator was added and the titration continued by adding thiosulphate drop wise until blue coloration disappeared after vigorous shaking. The same procedure was used for blank test and other samples. The iodine value is given by the expression.

$$I.V = 12.69 \quad \frac{C\,(V_1 - V_2)}{M}$$

Where C = Concentration of sodium thiosulphate used; V_1 = Volume of sodium thiosulphate used for blank; V_2 = Volume of sodium thiosulphate used for determination, M = Mass of the sample

Determination of Refractive Index

Refractometer was used in this determination. Few drops of the samples were transferred into the glass slide of the refractometer. Water at 40°C was circulated round the glass slide to keep its temperature uniform. Through the eyepiece of the refractometer, the dark portion viewed was adjusted to be in line with the intersection of the cross. At no parallax error, the pointer on the scale pointed to the refractive index. This was repeated and the mean value noted and recorded as the refractive index.

Determination of Viscosity

A clean, dried viscometer with a flow time above 200 seconds for the fluid to be tested was selected. The samples were filtered through a sintered glass (fine mesh screen) to eliminate dust and other solid material in the liquid samples. The viscosity meter was charged with each of the samples by inverting the tube's thinner arm into the liquid samples and suction force was drawn up to the upper timing mark of the viscometer, after which the instrument was turned to its normal vertical position. The viscometer was placed into a holder and inserted to a constant temperature bath set at 40°C and allowed approximately 10 minutes for the sample to come to the bath temperature at 40°C. The suction force was then applied to the thinner arm to draw the samples slightly above the upper timing mark. The afflux time by timing the flow of the samples as it flow freely from the upper timing mark to the lower timing mark was recorded.

RESULTS AND DISCUSSION

The result obtained for various tests carried out on *Watermelon* seed oil the analysis of essential oil. Table1 show some of the properties of Watermelon seed oil (Crude Oil).

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Table 1 some of the properties of Watermelon seed oil (Crude Oil).

Percentage oil yield (%)	85
Free fatty acid (%)	6.7
Acid value (mg NaOHg-1 of oil)	0.7
Saponification value (mg K0Hg-1 of oil)	49
Bulk Density (gcm^{-3})	8.44
Specific gravity (40°C)	0.9
Iodine value (gI^2/100gof oil)	0.8
Refractive index at 40oC	5.21
Viscosity at 40°Cmm^2/s	4.4

CONCLUSIONS

The oil from *Watermelon* seed was extracted using n- hexane as a solvent by soxhlet apparatus and the transesterification of this oil into biodiesel was then carried out using methoxide and magnetic stirrer.

The oil from *watermelon* seed was extracted using n-hexane as a solvent by soxhlet apparatus and the transesterification of this oil into biodiesel was then carried out using methoxide and magnetic stirrer. Table above shows some of the properties of *Watermelon* seed oil. The percentage yield values for crude oil were (39%). The Free fatty acid value was found to be 6.7. The specific gravity for crude oil were respectively 0.9. The value of the viscosity of the crude oil ($4.4mm^2/s$) was found to be outside the recommended standard range 4.1 mm^2/s. This may be attributed to the fact that some impurities and other components were removed during refining. The refractive index of the value obtained for crude oil, 5.21. The chemical properties analysis shown in Table 1 indicates that the acid value of crude oil was 0.7mg NaOH/g of oil respectively. The results for the saponification value of the crude oil that were found to be 49mg KOH/g of oil respectively. The saponification value of crude oil, are the result specified for quality Watermelon oil. Also, the result obtained for the Iodine value of crude oil is 1.13.

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