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Characterization of Moringa Oleifera Seed Oil and its Extraction

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Abstract: *The extraction and characterization of both crude and refined of Moringa oleifera oil found in the North East Ecological Zone of Nigeria have been carried out. Normal hexane was used as a solvent for the extraction process. The oil produced was refined through a transesterification process using methoxide and a magnetic stirrer local. The characterization analysis revealed that tested parameters, which include specific gravity, refractive index, viscosity, density, acid value, saponification value, and iodine value for both crude and refined Moringa oil produced, were within the ASTM standard specifications. The oil was analyzed using the Gas Chromatography-Mass Spectrometer method showing the composition and components of the oil. The major component obtained is Octadecenoic acid having the highest abundance with respect to retention time as displayed by the highest peaks in the graphs. The oil is of good quality and could be recommended as suitable for industrial usage.*

Keywords: *Moringa oliefera,* biodiesel, solvent extraction, Transe, Gas Chromatography Mass Spectrometer

INTRODUCTION

Moringa oleifera plant which is the most widely cultivated species of the genus Moringa belongs to the family Moringa cease (Hsu *et al.,* 2006). It is considered very useful, as every part of it is used for food or other beneficial applications (Premi *et al.,* 2010). Moringa oil is the oil that can be extracted from Moringa seeds. The oil can be used for soap making and consumption. Besides the industrial uses such as fine lubricant and perfumery, the fatty acids profile of the oil with its very high content of oleic acid make its oil with high potential for further industrial application (Machell, 1994). The oil yield from the seeds depends on the nature of the solvent, the temperature of extraction, seed particle size, contact (residence) time between the solvent and the seed and pre-treatment conditions (Sayyar *et al.,* 2009).

Another potential use of Moringa oil is as biodiesel feedstock. Biodiesel is an alternative diesel fuel, made from renewable biological sources such as vegetable oils and animal fats. It is biodegradable and nontoxic, has low emission profiles and so is environmentally beneficial (Krawczyk, 1996). Biodiesel being a renewable diesel fuel substitute can be made by chemically combining any natural oil or fat with an alcohol such as methanol or ethanol. Methanol has been the most commonly used alcohol in the commercial production of biodiesel.

This study is aimed at extraction and characterization of *Moringa oleifera* Seed oil. This will be achieved through the realization of the following objectives:

- Extraction of Seed oil from Moringa seeds through solvent extraction Process;
- Refining the crude *Moringa Oleifera* seed oil;
- Characterization of crude and refined Moringa seeds oil for easy identification, and also to assess its quality.

MATERIALS AND METHODS

Moringa seeds from wild trees in Nigeria were purchased from custom market in Maiduguri, Borno state. The seeds were separated from the membranes and broken to remove the kernel from the hard shell. The kernels were sun-dried so as to reduce the moisture content. The dried kernels were crushed into fine particles with blender to make solvent extraction easier. The sample was stored in a safe place for solvent extraction.

Soxhlet Extraction Method

The Soxhlet apparatus used for solvent extraction (SE) where 300ml of normal Hexane was poured into round bottom flask.10 grams of powdered *Moringa Oleifera* 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 *Moringa oleifera* 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 (180 g) of oil (crude grade) 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 Moringa Oleifera Oil

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

$$\%\ oil\ yeild = \frac{weight\ of\ oil}{weight\ of\ sample\ on\ dry\ matterbasis}$$

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 cm^3 of each of the oil sample were measured and pour into the beaker and weighed. The weights of the 50cm^3 of the samples were recorded. The procedure was repeated with water and the weight of 50cm^3 of water was obtained. The density and the specific gravity were calculated thus;

$$Density\ of\ oil = \frac{weight\ of\ oil\ sample}{volume\ of\ the\ oil\ sample}$$

$$specific\ gravity\ of\ oil\ sample = \frac{weight\ of\ oil\ sample}{weight\ of\ equal\ volume\ of\ water}$$

Determination Acid Value

1g of the crude and refined oil were weighed separately in 250ml conical flasks. 5cm^3 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;

$$A.V = \frac{Titre\ value + Molar\ Conc\ of\ KOH + 56.1}{weight\ of\ sample}$$

$$\%FFA = \frac{Titre\ value + Molar\ Conc\ of\ KOH + 28.2}{weight\ of\ sample}$$

Determination of Saponification Value

2g of the samples were weighed separately in 250ml conical flasks. 50 cm^3 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

$$s.v = \frac{(B - R) + Molar\ Conc\ of\ HCL + 56.1}{weight\ of\ sample}$$

Determination of Iodine Value

The method specified by ISO 3961 (1989) was used. 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 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 (I.V) is given by the expression:

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

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.

Analysis of Essential Oils

Analysis of Essential Oil was done using Gas Chromatography with Mass Spectrometer (GCMS) to know the composition of oil and to the quantity of each composition. GCMS is a method that combines the features of gas-liquid chromatography and mass spectrometry to identify different substances within a test sample. The GCMS is composed of two major building blocks: the gas chromatograph and the mass spectrometer. The gas chromatograph utilizes a capillary column which depends on the column's dimensions (length, diameter, film thickness) as well as the phase properties. The difference in the chemical properties between different molecules in a mixture has separated the molecules as the sample travelled the length of the column. The molecules took different amounts of time (called the retention time) to come out of (elute from) the gas chromatograph, and the mass spectrometer downstream captured, ionized, accelerated, deflected, and detected the ionized molecules separately. The mass spectrometer did this by breaking each molecule into ionized fragments and detecting these fragments using their mass to charge ratio. These two components, used together, allow a much finer degree of substance identification than either unit used separately.

RESULTS

The result obtained for various tests carried out on *Moringa Oleifera* seed oil and biodiesel sample is presented below.

The analysis of essential oil obtained after Steam Distillation was done using Gas Chromatography Mass Spectrometer method for both *Moringa oleifera* Seed oil and biodiesel.

Table1 show Some of the properties of Moringa Oleifera seed oil (Crude Oil) and biodiesel (Refined Oil).

Property	ASTM	Oil	Bio diesel
Percentage oil yield (%)	-	39.0	98.0
Free fatty acid (%)	-	1.2	0.282
Acid value (mg $NaOHg^{-1}$ of oil)	0.4 - 4.0	0.7	0.562
Saponification value (mg $K0Hg^{-1}$ of oil)	-	49.0	50.0
Density (gcm^{-3})	0.878	1.17	0.9
Specific gravity ($40^{o}C$)	0.85	0.9	0.8
Iodine value (gI_2/100gof oil)	-	1.13	1.269
Refractive index at $40^{o}C$	1.476 – 1.479	1.4668	1.4662
Viscosity at $40^{o}Cmm^2/s$	1.9 – 6.0	4.4	3.2

Discussion

The oil from *Moringa oleifera* seed was extracted using normal 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 *Moringa oleifera* seed oil and biodiesel. The percentage yield values for both crude and refined oil were (39%) and (98%). The Free fatty acid value was found to be 1.2 and 0.282 KOH/g for crude and refined oil respectively. The specific gravity for crude and refined oil were respectively 0.9 and 0.8. Differences were observed between the value obtained for the viscosity of the crude and refined oil. The value of the viscosity of the crude oil ($4.4mm^2/s$) was found to be outside the recommended standard range of 1.3 – 4.1 mm^2/s (Joshi *et al.,* 2007), while the refined oil's viscosity of $3.2mm^2/s$ is quite within range. This may be attributed to the fact that some impurities and other components were removed during refining. The refractive index analysis shows that there is no significant difference between the value obtained for crude oil, 1.4668 and that of the refined oil, 1.4662. Comparing this result with the standard values that ranges from 1.476 – 1.479(ASTM specification for No. 2 Diesel fuel), a little difference was noticed. However, this little difference can be considered being within an acceptable experimental error range that can be attributed to the presence of some impurities and other component of the crude oil mixture. Thus, the refractive index of both crude and refined castor oil was in agreement with ASTM specification. The chemical properties analysis shown in Table 1 indicates that the acid value of crude and refined oil was 0.7mg NaOH/g of oil and 0.562mg NaOH/g of oil respectively. The value is a little bit higher in crude oil due to free fatty acid present; while it less for the refined oil as a result of the chosen strength 0.1M of NaOH used in the treatment of the crude oil, which must have

neutralized some of the free fatty acid present in it. In addition, both values fall within the ASTM specification (0.4 - 4.0 NaOH/g). The results for the saponification value of the crude and refined oil that were found to be 49mg KOH/g of oil and 50mg KOH/g of oil respectively. The saponification value of both crude and refined castor oil, are highly comparable with the result specified for quality Moringa oil. Also, the result obtained for the Iodine value of crude oil is 1.13 which a little bit lower compared to 1.269 after it was refined. As a result of their agreement with standard value, both the crude and refined oil could be classified as non-drying oils, since their iodine values are lower than 100.

Analysis of Essential Oil obtained was done using Gas Chromatography Mass Spectrometer method. The analysis shows the composition and components of the oil. These are shown in graphs produced by Gas Chromatography Mass Spectrometer apparatus. The GCMS spectrums of crude oil of March 25, 2015, which was extracted at 60 °C. Eleven spectrums were observed during the analysis with different peaks and retention times were observed. Figure 4.2 is the GCMS spectrums of the refined oil also extracted on the same day and at temperature. From the graphs it was observed that both the crude and refined Moringa oils are composed of the following compound: Glycerin, dl-Glyceraldehyde, 1,2,3,4-Butanetetrol, Hexadecanoic acid, Pentadecanoic acid, Tridecanoic acid, Ethyl tridecanoate, Octadecanoic acid, Docosanoic acid, Decanoic acid, 9,12-Octadecadienoic acid, 11,14-Eicosadienoic acid, 9,12-Octadecadienoic acid, 9,12-Hexadecadienoic acid, 9,12-Octadecadienoyl chloride, Heneicosanoic acid, Docosanoic acid, 7-Tetradecenal, Methyl ricinoleate Ricinoleic acid, E-11-Hexadecenoic acid, E-9-Tetradecenoic acid, Methyl ricinoleate, Oxiraneundecanoic acid, Kauran-18-al, Isoaromadendrene epoxide and Caryophyllene oxide, of all the components the major components is Octadecenoic acid, which is having highest abundance with respect to retention time as shown by the highest peaks in the graphs. Octadecenoic acid, has the following properties:

- Molecular formula: $C_{19}H_{36}O_2$
- Molar weight: 296
- RetIndex: 2085

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