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Characterization of Non-Edible Plantsfor Biodiesel Production

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ABSTRACT: *Oil obtained from orange peels and velvet tamarind nut by solvent extraction (n-hexane and acetone) using the soxhlet extractor were investigated as feedstock's to determine some of the suitable parameters such as iodine value, saponification value, specific gravity, density, acid value and free fatty acid value, and their percentage yield that are suitable for biodiesel production. The density of orange and tamarind oil were found to be 0.9326g/cm^3 and 0.8541g/cm^3 respectively, their acid values were 19.81gNaOH for orange oil and 18.93gNaOH for velvet tamarind oil. The degree of unsaturation of both oils as determined by their iodine values were; 10.47gI_2/100g for orange oil and 5.39gI_2/100g for velvet tamarind oil. The viscosities of the oil were 11.29mPa.s and 26.55mPa.s for orange and velvet tamarind oil respectively. The various values obtained were compared with specifications of ASTM D6751 and it is established that the oil obtained from orange peels and velvet tamarind nuts could be used as alternative to/or blended with petro diesel.*
KEYWORDS*- Orange peel, Velvet Tamarind nut, Characterization, Solvent extraction, Biodiesel.*

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I. INTRODUCTION

The concern for fast depletion of petroleum oil and its environmental impact has shifted interest to alternative sources of fuels, particularly biofuels, which are renewable and environmentally friendly (Demirbas, 2008). Thus, varieties of virgin, non-edible and waste vegetable oils have been sourced for the production of biofuels (Maniak *et al*., 2009). The choice of biofuel over diesel fuel includes its portability, availability, renewability, higher combustion efficiency, higher octane number, higher biodegradability, high flash point, inherent lubricity, lower sulphur and aromatic contents (Knothe *et al*., 2005). The results have shown that some plants are reasonable sources of ascorbic acid and oils (edible and non-edible) which are potential sources of biodiesel fuels for diesel engines. Thus, in view of the need to find alternate sources of energy to power diesel engines, the present work is a study of some seeds which are known to contain oils and are also non-edible, such as orange oil, mango oil, tangerine oil, watermelon oil, avocado oil, velvet tamarind oil etc. The results of these studies are discussed in this work.

There are two types of oils; edible and non-edible oils. Edible oils are the major sources to produce biodiesel fuel like sunflower, soybean, and palm oils. Due to higher prices of edible vegetable oils compared to diesel fuel, waste vegetable oils and non-edible crude vegetable oils (mango oil, orange oil, pumpkin oil, avocado oil, beniseed oil etc.) are now being used as biodiesel sources. However, there could be disadvantages of using edible oils such as higher viscosity, lower volatility, the reactivity of unsaturated hydrocarbon chains etc. It is known that the density of biodiesel fuel mainly depends on its esters content and the remained quantity of alcohol; hence this property is influenced primarily by the choice of vegetable oil (Encinar et al., 2010). Due to these disadvantages, vegetable oils are not used directly as biodiesel, so there are methods to enhance the vegetable oil's characteristics for biodiesel production by blending the biodiesel from these oils with ozonated vegetable oil(vegetable oils containing a significant amount of ozone) which increases the acid value and decreases the viscosity of the oil and also prevents the agglomeration of biodiesel into a solidified material, giving crystals. Biodiesel is basically more preferable to the conventional diesel fuel because it is environmentally friendly in the aspect of pollution, as it is known to reduce the levels of emission of heavy pollutants such as carbon monoxide, nitrous oxide, and sooth. Several properties such as cetane number, density, viscosity, iodine number, pour point, cloud point are tested to check the properties of the different seed oils to be used as biodiesel fuel. Both pour and cloud points must be sufficiently low, because if the biodiesel is frozen, it will affect the equipment (Akpan, 2012; Alnuami et al., 2014). Neat biodiesel has a flash point over 300

Fahrenheit, well above the flash point of petroleum based diesel fuel (Sanjay, 2013; Vuppaladadiyam et al., 2013).

Orange oil usually appears as a yellow-orange liquid that smells strongly of the orange fruit. It is almost completely composed of a substance called *d-limonene* and is classified as an essential oil. It is produced from the orange fruit (*Citrus sinensis*) by the cells within the rind. Unlike most essential oils, orange oil is produced as a by-product of orange juice production by centrifugation, producing cold-pressed oil. It is non-edible and will cause digestive problems and extreme stomach aches if it is directly consumed due to its high concentration of the slightly toxic *d-limonene.* Consuming small amounts of *limonene* from substances like orange juice has been observed to produce no effect on human health. Due to the high percentage of *d-limonene* content in the orange oil, the oil is a mild irritant, as it dissolves protective skin oils. Limonene and its oxidation products are skin irritants, and limonene 1, 2-oxide (formed by aerial oxidation) is a known skin sensitizer. Limonene causes cancer in male rats, by reacting with α2u-globulin, which is not produced by female rats. However, limonene is not carcinogenic or genotoxic in humans (Bauer et al., 2001).

On the other hand, velvet tamarind *Dialium indicum* (known as Awin and Icheku in Yoruba and Igbo native Nigerian languages respectively) is a tall, tropical, fruit-bearing tree. It belongs to the *Leguminosae* family, and has small, typically grape sized edible fruits with brown hard inedible shells. It has been described as one of the common and most important trees of India. India is the world's top producer, exporting several thousands of tonnes of seed, seed powder and fruit pulp each year. The fruit is also very popular in Ghana and Nigeria and can grow 20 meters height and stays evergreen, and can be used as an ornamental tree and to provide shade. Each fruit typically has one hard, flat, round, brown seed, typically 7-8 millimetres across and 3 millimetres thick. The seed somewhat looks like a watermelon seed. Some have two seeds. The seeds are shiny, coated with a thin layer of starch. The oil is usually extracted from the dried and crushed velvet tamarinds, physic-nut and nicker-nut seeds by solvent extraction method using the soxhlet extractor (LPWG, 2017).

II. EXPERIMENTAL

2.1 Materials

Orange fruits and the velvet tamarind fruits were obtained from a local market in Owerri, Nigeria. A blender was used for size reduction after sun drying. soxhlet extractor set up was used for oil extraction. All the as received reagents such as n-hexane, acetone, petroleum ether, ethanol, phenolphthalein, sodium hydroxide, carbon tetrachloride, sodium thiosulphate, potassium hydroxide, wiji's reagent, and potassium iodide were of analytical grade.

2.2Characterisation procedure for orange oil

Orange fruits were obtained from a local market in Owerri, Nigeria. The orange fruits were washed with well water as soon as they were collected. After washing, the rinds of the oranges were removed from the orange fruit and they were sun dried to reduce the moisture content in order to obtain more oil. The dried orange rinds (peels) were blended with a blender for size reduction and sieved to remove unwanted particulate substances and the final product was an averagely coarse aggregate. The coarse aggregate was then transferred to the soxhlet extraction set up. The liquid n-hexane which acted as a solvent for extraction was poured in a round bottom flask (600ml) and heated with a heating mantle until the solvent was evaporated and condensed in the Soxhlet extractor that was connected to the water condenser. The oil which has been drained passes through the thimble of the extractor and drops into the round bottom flask and a mixture of hexane and the oil was formed. After the oil had been drained out from the sample completely, it was removed and the extraction chamber was filled with fresh weighed sample. This was continued until significant amount of oil was extracted. The resulting solution of the oil and the hexane was then heated to evaporate n-hexane and obtain the pure orange oil. The same procedure wererepeated for the extraction of the velvet tamarind seed using acetone as the extraction solvent. The viscosity of the oil samples was determined using a digital rotational viscometer. It is expressed in units of mPa.s.

2.2.1Specific gravity and density

25ml of water was poured into already weighedpycnometer bottle and subsequently weighed. The bottle was emptied, dried and filled with the extracted orange seed oil and tamarind oil which were subsequently weighed respectively. The specific gravity and density were determined according to Equation 1a and 1b.

$$\mathbf{SG}=\frac{\mathbf{Weightof25mlofoil}}{\mathbf{Weightof25mlofwater}} \quad (1a)$$

$$\mathbf{Density}=\frac{\mathbf{Weightof25mlofoil}}{\mathbf{Volumeofoil}} \quad (1b)$$

2.2.2Acid value or free fatty acid (FFA)

25ml of petroleum ether were mixed with 25ml of ethanol and 4 drops of phenolphthalein indicator was added into a conical flask containing 1.03g of the orange oil sample and was titrated with aqueous 0.1MNaOH. The solution was shake vigorously until a dark pink colour which persisted for 25 seconds was obtained. The acid value and FFA were calculated respectively using Equation 2a and 2b. The same procedure were repeated for the tamarind oil.

$$\boldsymbol{acidvalue} = \frac{\boldsymbol{Titrevalue \times M \times W}}{\boldsymbol{Weightofsampleused(g)}} \quad (2a)$$

$$\boldsymbol{FFA} = \frac{\boldsymbol{Acidvalue}}{\boldsymbol{2}} \quad (2b)$$

Where M is the molarity of the base and W is the molecular weight of the base.

2.2.3Iodine value

0.4g wt. of the respective sample were weighed into a conical flask and 20ml of carbon tetrachloride was added to dissolve the oil. Then 25ml of wiji's reagent was added to the flask using a safety pipette in fume chamber. A stopper was then inserted and the content of the flask was vigorously swirled. The flask was then kept in the dark cupboard for 2hours 30minutes. At the end of this period, 20ml of 10% aqueous potassium iodide and 125ml of water were added using a measuring cylinder. The content was titrated with 0.1M sodium thiosulphate solution until the yellow colour almost disappeared. Few drops of 1% starch indicator was added and the titration continued by adding sodium thiosulphate drop wise until blue coloration disappeared after vigorous shaking. The same procedure was used for blank test which was carried out without the oil sample. Equation 3 was used to calculate the iodine value.

$$\boldsymbol{Iodinevalue} = \frac{\boldsymbol{(V_1 - V_2) \times C \times 12.69}}{\boldsymbol{Weightofsampleused\ (g)}} \quad (3)$$

Where V_1 is volume of sodium thiosulphate used for blank, V_2 is volume of sodium thiosulphate used for the sample and C is concentration of sodium thiosulphate.

2.2.4Saponification value

2g of the orange oil sample was added into a conical flask containing 25ml of 0.1N ethanolic potassium hydroxide solution together with bumping granules in very little amounts to prevent explosion. The flask was sealed and allowed to boil gently in a water bath for 60minutes (1hour) at 100^0C. Few drops of phenolphthalein indicator were added and a pink colouration was observed. The solution was then titrated with 0.5M HCl when hot until the pink colouration disappeared. A blank was carried out at the same time (without oil). This same procedure were repeated for tamarind oil. The saponification value is determine using Equation 4.

$$\boldsymbol{SaponificationValue} = \frac{\boldsymbol{56.1 \times (V_1 - V_2) \times C}}{\boldsymbol{Weightofsampleused\ (g)}} \quad (4)$$

Where C is concentration of HCl used, V_1 is volume of hydrochloric acid used for blank and V_2 is volume of hydrochloric acid used for sample.

III. RESULTS AND DISCUSSION

The extracted orange oil was yellow in colour and the velvet tamarind oil was dark brown in colour with yields of 16.92% and 36.04% respectively per 25g of pulverized seed using n-hexane and acetone as extraction solvents respectively. The results of the physical and chemical properties of orange oil and velvet tamarind oil is as shown in Table 1.

From the experimental results obtained, the density of orange oil (0.9326g/cm^3) is greater than the density of velvet tamarind oil (0.8541g/cm^3), but slightly less dense than water and hence suitable as an alternative fuel as it tallies with the ASTM D6751 standard. The viscosities of the orange oil (11.29mPa.s) and tamarind oil (26.55mPa.s) tallies with the ASTM D6751 standard (35max) and hence suitable for biodiesel production.

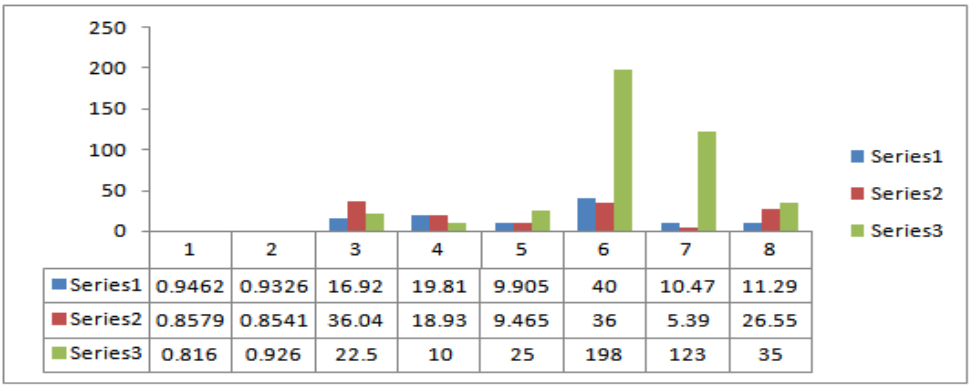
The free fatty acid value of both samples falls within the standard which is an indication that they will not be corrosive when used in making biodiesel which can affect the fuel pumps and filters. The degree of unsaturation for both oil samples is very low since their iodine numbers falls below the standard. Also, saponification value of both oil samples were found to be below ASTM D6751 standard which indicates the absence of unsaturated fatty acid characteristics of foaming ability.

The yield of velvet tamarind oil is highest using acetone as the solvent for extraction and this corresponds to the recommended standard ASTM D6751.

Table 1. Results for characterization of orange oil and velvet tamarind oil

Property/unit	Orange oil (citrus sinensis)	Velvet tamarind oil (Leguminosae)	ASTMD Standard
Specific gravity	0.9462	0.8579	0.816

Density (g/cm^3)	0.9326	0.8541	0.818-0.926
Oil yield (%)	16.92	36.04	22.5
Acid value (gNaOH/g)	19.81	18.93	10
Free fatty acid (FFA)	9.905	9.465	25 max
Saponification value (mgNaOH/g)	40	36	189-198
Iodine value (gI_2/100g)	10.47	5.39	123
Viscosity (mPa.s.)	11.29	26.55	35



	1	2	3	4	5	6	7	8
Series1	0.9462	0.9326	16.92	19.81	9.905	40	10.47	11.29
Series2	0.8579	0.8541	36.04	18.93	9.465	36	5.39	26.55
Series3	0.816	0.926	22.5	10	25	198	123	35

Figure 1: Graphical result of characterization

Series 1(blue) =orange oil, Series 2(red) =velvet tamarind oil & Series 3(green) = ASTMD standard

Key:

Column 1	Specific gravity
Column 2	Density
Column 3	Oil yield
Column 4	Acid value
Column 5	Free Fatty Acid value
Column 6	Saponification value
Column 7	Iodine value
Column 8	Viscosity

IV. CONCLUSION

This work has clearly demonstrated that the properties of both orange oil and velvet tamarind oil have proven to be useful feed to produce high quality methyl ester (biodiesel).Similarly, the properties of the oil such as their saponification value, acid value, density and specific gravity, iodine value and kinematic viscosity falls within the standard as recommended by ASTM D6751.It was equally established that oil obtained from velvet tamarind using acetone as the solvent of extraction produced the highest yield and hence more economical than oil obtained from orange oil using n-hexane as the extraction solvent. In the test for the iodine value of oil, during titration with sodium thiosulphate, the mixture must be vigorously stirred before 1% starch indicator (prepared by dissolving 1g of starch in 100ml of water) is added in order not to use up the thiosulphate solution in the burette.When testing for the saponification value of oil, it is advised to add bumper granules in minute quantities before heating in order to avoid explosion.

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