Extraction and Characterization of Oils from Some Local Seeds

Evwierhoma E. T. and Ekop I. E

Abstract: In this work, extraction and characterization were done for four local seed oils. The four seeds; sesame, soybean, avocado and jatropha considered have shown to contain certain percentages of oils, in the range of 2 to 50%. The oils were extracted using hexane as solvent in a soxhlet extractor. Characterization of the oils for the following parameters; oil content, pour point, viscosity, specific gravity, refractive index, acid value, peroxide value, free fatty acid and iodine value by standard techniques were carried out after extraction. Sesame seed yielded the highest oil content with an average of 51.34% while the lowest oil content was obtained from avocado seed of 2.51% average. Also the highest viscosity was obtained from avocado seed of 63.30mPa.s whereas the lowest value was obtained from jatropha seed of 52.60mPa.s.The values of the parameters suggest the potentials of the oils for domestic and industrial applications.

Keywords: Physiochemical characteristics, soxhlet extraction, solvent extraction, raffinate, oil content, supercritical extraction

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1.0 Introduction

Vegetable oils and fats are lipid materials derived from plants. Although many plant parts may yield oil, in commercial practice, oil is extracted primarily from seeds. Therefore seed oils can also be referred to as vegetable oils. Worldwide, these oils are increasingly becoming important and quite a number of analysis have been carried out primarily because of extensive demands for oils both for human consumption and for industrial applications; Seed oils are important sources of nutritional oils, and are of industrial and pharmaceutical importance. The characteristics of oils from different sources depend mainly on their compositions and no oil from a single source can be suitable for all purposes (Mohammed & Jorf-Thomas, 2003). Consequently with the increasing awareness of the importance of these seed oils there is need to search for oils from non-conventional sources to augment the available ones and also to meet specific applications.

Most seed oil extractions have been performed with organic solvents such as n-hexane but studies have shown that this solvent is subject to rapidly increasing costs and uncertain availability, highly flammable and explosive when mixed with air, leaves residues in the extract, and is not environmentally friendly. Hence supercritical fluid extraction (SFE) has become an important separation technique in the seed oil extraction process as it may be a viable alternative to current extraction methods. Extraction with the Soxhlet apparatus gives good yield,

due to the increased ability of the solvent to overcome forces that bind lipids within the sample matrix (Lumley et al. 1991).

The phenomenon of Supercritical fluid extraction was observed about a century ago (Hannay & Hogarth, 1879) but has been slow to find commercial applications mostly due to the sophisticated expensive high-pressure equipment and technology required. Supercritical fluids have gas-like diffusivities, but liquid-like densities, these properties vary as a function of pressure and temperature. With this technology, it is possible to extract heat-sensitive compounds such as unsaturated fatty acids and to avoid any toxic solvent residue in the product (King & List 1993; Rizvi 1994).

Oil can be extracted from various seeds; however this work focuses on four particular seeds, Sesame, Soybean, Avocado and Jatropha seeds.

The Sesame oil can be used as cooking oil due to its health benefits (blood pressure regulation, anti-depressant), for massage therapies, and cosmetics.

The Soybean oil is one of the most widely consumed cooking oils, can be used as a drying oil, be processed as a base for printing inks (soy ink) and oil paints, and as a fixative for insect repellents.

The Avocado oil can also be used as cooking oil for its health benefits (reducing cholesterol and fighting disease) and also in cosmetics (hand and body lotions, shampoos) as it is valued for its regenerative and moisturizing properties.

The Jatropha oil can be processed to produce jet fuels and high-quality biodiesel that can be used in a standard diesel combustion engine.

Extraction can be defined as a separation process consisting in the separation of a substance from a matrix; Oil is extracted from seeds, beans, and nuts for use as cooking or salad oil; as an ingredient in paint, cosmetics, and soap; and even as fuel. The first step in obtaining oil from the seed is to remove the seed-coat and husk in a process referred to as dehulling.

Historically, such oils have been extracted by wrapping seeds in cloth, and then using devices operated by stones and levers to exert pressure on them.

In Africa and some developing countries, manual processes, where hard objects are used to crack the shells are popular, but machines (dehullers) used in developed countries are being introduced. Once the nuts are cracked, the oil-bearing seeds are cleaned and dried. The seeds are cleaned by removing the seed coat and separating the chaff and dried by placing the seeds under the sun or by heating carefully on the fire for a short while. In the case of using crude means to extract the oil, drying can be followed by warming which ensures more oil yield. Once this is done, the next step is to begin the crucial extraction process.

Africans are known to derive body creams and cooking oil from nuts through traditional means. One of such means involves roasting palm kernel nuts in dried pans to "force out" the popular black palm kernel "pomade". Others prefer to extract their body cream from boiling coconut juice to separate the oil. Whatever the case or seed may be, this shows that seed oil extraction has come a long way in the history of our culture.

Seed oils are extracted in two primary ways, by mechanical pressing, or squeezing, and with petroleum solvents. Before the 1940's, mechanical pressing was the primary method used. Mechanical extraction, however, had its limits in terms of oil recovery. Because pressing generates heat and high temperatures which damage both the oil and meal, an oil content of the press cake below 5-6% was difficult to achieve. Solvent extraction was developed because it allows a more complete extraction at lower temperatures. It begins to be economically attractive where large quantities of seed can be processed (at least 200 tons per day for continuous-feed processes); where storage, transportation, power, water, and solvent supply are adequate; and where occupational safety and training standards can be enforced.

This work focuses on the extraction of oil from four local seeds using nhexane in a soxhlet apparatus, determine the physicochemical properties of the oils based on oil content, pour point, specific gravity, viscosity, refractive

index, iodine value, acid value and peroxide value. This study was designed to establish the suitability of the oils for domestic and industrial uses.

2.0 Methodology

Hexane, a hydrocarbon with chemical formula C_6H_{14} and boiling melting point 68.7 °C and   -95.3°C respectively has become the solvent of choice for solvent extraction(liquid-liquid extraction) because of its high stability, low evaporation loss, low corrosiveness, little greasy residue and better odour and flavour of the extracted products. Solvent extraction using hexane has several drawbacks including high capital equipment cost and operational expenditures, the perpetual hazard of fire and/or explosion as well as the residual solvent associated with both oil and meal.

The primary prerequisite for solvent extraction for oils is the rupturing of the seed or feed material to render the cell wall more porous, complete rupturing of the cell wall is necessary for rapid extraction.

In soxhlet extraction, normally a solid material containing the desired compound is placed inside a thimble made from thick filter paper, which is loaded into the main chamber of the Soxhlet extractor. The Soxhlet extractor is placed onto a flask containing the extraction solvent-hexane and is then equipped with a condenser.

The solvent is heated to reflux. The solvent vapour travels up a distillation arm and floods into the chamber housing the thimble of solid. The condenser ensures that any solvent vapour cools, and drips back down into the chamber housing the solid material.

The chamber containing the solid material slowly fills with warm solvent. Some of the desired compound will then dissolve in the warm solvent. When the Soxhlet chamber is almost full, the chamber is automatically emptied by a siphon side arm, with the solvent running back down to the distillation flask. This cycle may be allowed to repeat many times, over hours or days.

During each cycle, a portion of the non-volatile compound dissolves in the solvent. After many cycles the desired compound is concentrated in the distillation flask. The advantage of this system is that instead of many portions of warm solvent being passed through the sample, just one batch of solvent is recycled.

After extraction the solvent is removed, typically by means of a rotary evaporator, yielding the extracted compound. The non-soluble portion of the extracted solid remains in the thimble, and is usually discarded (Wikipedia, 2012).

2.1 Experimental Procedure

Sesame, soybean, avocado and jatropha seeds were gotten from local markets in Lagos state. The seeds were selected to remove those damaged and sundried for 24hrs to get rid of moisture as all apparatus were washed and oven dried, to get rid of moisture and the soxhlet apparatus set-up in readiness for the experiment. The seeds were ground mechanically to a particulate size of 1.0mm size, and in the case of soybean and jatropha, seeds were de-hulled and de-shelled respectively before grinding.

15g of the seed samples were weighed into a thimble (semi-permeable membrane) and placed into the soxhlet extractor with 200ml of hexane solvent for the first run, the solid was removed through filtration to get the extracted lipids. The apparatus was put on the heater and was left to run for 2hrs. Extracted oil was poured from the round bottom flask of the apparatus into a test-tube and kept aside to be degummed and characterized. Each sample was run in triplicate and the results tabulated.

3.0 Measurements.

Oil Content

Oil content was determined by drying the raffinate, measuring and calculating with reference to the initial amount of seed particle placed in the extractor.

$$\frac{15g - R(g)}{15g} * 100\% \qquad\qquad 1$$

Where R = amount of raffinate (Laboratory handbook,1997).

Pour Point

Complete pour point was determined using a pour point tester.

Specific Gravity

The specific gravity was determined by specific gravity bottles method using IS: 1460-2000. This was conducted at 25°C.

Specific gravity of sample=(W3 – W1)/(W2–W1) (Laboratory handbook,1997)   2

Where, W1 = Weight of empty specific gravity bottle, W2 = Weight of water + specific gravity bottle, W3 = Weight of test sample + specific gravity bottle.

Viscosity

A viscometer was used to measure the different oil viscosities. By this procedure, a concentric cylinder system was submerged in the oil and the force necessary to overcome the resistance of the viscosity to the rotation was measured. The viscosity value, in mPa.s, was automatically calculated on the basis of the speed and the geometry of the probe. The experiment was carried out by putting 2 ml of sample of oil in a concentric cylinder system using 60 sG1 as shear rate.

Refractive Index

A hand refractometer was used in this determination. The sample was transferred into the glass slide of the refractometer. In order to carry out determination of the refractive index of the oils, the measuring prism was opened and a drop of the oil is placed on it and closed with the small cover plate. The refractive index was read through the aperture in % Brix.

Iodine Value

The method specified by ISO 3961 (1989) was used. 0.4 g of the sample was weighed into a conical flask and 20 ml of carbon tetra chloride was added to dissolve the oil. Then 25ml of Dam reagent was added to the flask

using a safety pipette influenced chamber. A stopper was inserted and the content of the flask vigorously swirled. The flask was placed in the dark for 2 hrs and 30 min. At the end of this period, 20 ml of 10% aqueous potassium iodide and 125 ml of water was added using a measuring cylinder. The content was titrated with 0.1 M sodium-thiosulphate solution until the yellow colour almost disappears. A few drops of 1% starch indicator was added and the titration continued by adding thiosulphate drop-wise until blue coloration disappeared after the vigorous shaking. The same procedure was used for the blank test and for other samples.

The iodine value (*IV*) was given by the expression:

$$IV = 12.69c\ (V1\text{-}V2)\ m \quad \text{(Laboratory handbook, 1997)} \qquad\qquad 3$$

Where: *c* – concentration of sodium thiosulphate used,

*V*1 – volume of sodium thiosulphate used for the blank,

*V*2 – volume of sodium thiosoulphate used for determination,

m – Mass of the sample

Peroxide Value

1 g of potassium iodide and 20 ml of solvent mixture (glacial acetic acid/chloroform, 2/1by volume) was added to 1 g of the oil sample and the mixture was boiled for one minute. The hot solution was poured into a flask containing 20 ml of 5% potassium iodide. A few drops of starch solution was added to the mixture and the latter was titrated with 0.025 N sodium thiosulphate and the peroxide value was determined as follows:

$$PV = SN10^3 / W \quad \text{(Laboratory handbook, 1997)} \qquad\qquad 4$$

Where: *S* – ml of $Na_2S_2O_3$,

N – Normality of $Na_2S_2O_3$,

W – Weight of oil sample (g).

Acid Value

25 ml of diethyl ether and 25 ml of ethanol was mixed in a 250 ml beaker. The resulting mixture was added to 10 g of oil in a 25 ml conical flask and a few drops of phenolphthalein was added to the mixture. The mixture

was titrated with 0.1 m KOH to the end point with consistent shaking for which a dark pink colour was

observed and the volume of 0.1 m KOH noted.

Acid value = (V × N × 56.1) / W   (Laboratory handbook, 1997)   5

Where, V = volume of potassium hydroxide used, N = normality of

Potassium hydroxide, W = weight in g of the sample

Free fatty acid as oleic acid, per cent by weight = (28.2 × V × N) / W   6

% FFA × 1.99 = Acid value.

4.0 Results and Discussion

4.1 Results

Table 1: Oil extraction from 15g of Sesame seed

Run	Time (hrs)	Amt. of hexane used/ml	Raffinate (R)/ g	Extract (15g- R/g)	Oil content/%
A	2	150	7.3	7.2	48.00
B	2	200	6.9	8.1	54.01
C	2	250	7.23	7.77	51.33

Table 2: Oil extraction from 15g of Soybean seed

Run	Time (hrs)	Amt. of hexane used/ml	Raffinate (R)/ g	Extract (15g- R/g)	Oil content/%
A	2	200	12.34	2.66	17.73
B	6	200	12.27	2.73	18.20
C	6	250	13.01	1.99	13.27

Table 3: Oil extraction from 15g of Avocado seed

Run	Time (hrs)	Amt. of hexane used/ml	Raffinate (R)/ g	Extract (15g- R/g)	Oil content/%
A	2	200	14.55	0.45	3.00
B	6	200	14.49	0.51	3.40
C	6	250	14.83	0.17	1.13

Table 4: Oil extraction from 15g of Jatropha seed

Run	Time (hrs)	Amt. of hexane used/ml	Raffinate (R)/ g	Extract (15g- R/g)	Oil content/%

A	2	200	12.04	2.96	19.73
B	6	200	12.07	2.93	19.53
C	6	250	12.56	2.44	16.27

Table 5: Physicochemical Properties of the Oils

Properties/Seed	Sesame	Soybean	Avocado	Jatropha
Specific gravity (at 27^oC)	0.9145	0.9213	0.9122	0.9810
Viscosity (mPa.s)	54.37	60.35	64.30	52.60
Pour Point, ^oC	-11.8	-14.0	-15.0	-8.5
Refractive index (%Brix)	1.4760	1.4733	1.4691	1.4960
Iodine value ($gI_2/100g$)	117.45	126.25	80.00	100.50
Peroxide value (mequiv/kg)	0.60	0.95	3.30	3.50
Acid value (mgKOH/g)	1.560	0.995	1.650	11.720
Average Oil Content %	51.56	16.40	2.50	18.51
%Free Fatty Acid(%FFA)	0.78	0.51	1.68	5.89

4.2 Discussion

This work demonstrates the extraction of oil from some local seeds using n-hexane as the solvent in a soxhlet apparatus. This method is proficient in providing satisfactory results and the oil contents of the various seeds can be seen.

Oil Content

Table 1 shows oil content of the sesame seed to vary between 48 - 54%, and interestingly, it shows that as the amount of hexane in the apparatus was increased and the amount of seed (15g) in the thimble was held constant, the amount of oil extracted seemed to decrease. An optimum amount of 200ml of hexane was also noticed as this gives the highest amount of oil extracted with the Soxhlet apparatus. It is evident therefore that any increase in the quantity of the solvent, will give no increase in the amount of oil extracted.

Tables 2 to 4 also show this same trend of decrease in oil content with increasing solvent amount but a time variation was noted. As the time for the extraction run was increased from 2 to 6 hours, there was no significant change in the amount of oil extracted from the same quantity of seed. This means that the oil contained in the seeds can be recovered in 2 hours or less using the soxhlet apparatus. The oil extraction with the soxhlet has a very high yield due to the increased ability of the solvent to overcome forces that bind lipids within the sample matrix(Lumley et al, 1991).

Determination of oil content in seeds is important because it predicts the profitability of the given seeds as potential sources of oil. High oil content in seeds implies that processing it for oil would be economically viable (Ikhuoria et al, 2008). Thus the oil yield found in the seeds studied ranged from 2.5 to 51.34% as shown in table 5 and compares quite well with the oil yield reported for some commercial seeds such as cotton (36%), olive (17%), sunflower (44%) and corn (3.4%) (Rossel, 1987).

The sesame seed oil content obtained with an average of 51.34%.. Also the soybean and avocado seeds oil content obtained averaged 16.4% and 2.51% respectively. The jatropha seed oil content has an average of 18.51%.

Physiochemical Characteristics of the extracted oils

Acid Value
The acid value which is an index of free fatty acid content due to enzymatic activity was found to vary between 0.995 to 11.7% The sesame, soybean and avocado oils have relatively low acid values but require further reduction by refining for edible purpose. Oil that is low in acidity is suitable for consumption, acidity less than 0.1mgKOH/g is usually the best (Orthoefer, 2007). The Jatropha oil has a very high acid value (11.720 mgKOH/g)and therefore cannot be consumed as edible oil. It can serve as vegetable oil feedstock chemical industries such as biodiesel, soap, fatty acids, surfactants and detergents.

Peroxide value

The peroxide values of the oils studied were low; this may be due to the high stabililty of the seeds during the extraction operations (Besbes et al., 2004). Peroxide value and viscosity are monitored in order to study the effect of storage in accelerated conditions. The resistance against oxidation is evaluated by the peroxide value; we can say that sesame and soybean oil with very low peroxide values compared to avocado and jatropha oil would take a longer time to turn or to become rancid.

Pour point

The pour points of the oils are low. Pour point specifies the temperature at which the oil flows. Since the pour point of the oils is low, solidification point of the emulsion will be low and thus the freezing stability of the emulsion will be better. Viscosity also influences the pour point of oils. The viscosity of the oils is high. The higher the viscosity the lower the pour point as shown in table 5.

Specific gravity

Generally, oils are lighter than water. Some however are heavier than water, especially those which contain larger amounts of oxygenated constituents of the aromatic series (Stasosphere, 2007).

Most popular seed oils have specific gravity ranging from 0.9100 to 0.9400 and specific gravity of 0.92 is considered a good number for any edible oil (Elert, 2000). Some authors have stated that the specific gravity suitable for edible oils range from 0.8800 to 0.9400 and for oils used for fuel from 0.8200 to 1.0800 at 15°C. Using the equation $SG_{12} = -0.0006x + SG_{11}$ (Wan Nik et al, 2007), the above specific gravity intervals can be converted at room temperature to 0.87163 to 0.93136 for edible oils and from 0.8114 to 1.0714 for biofuels. These specific gravity ranges compared to those of the current study in table 5 indicate that both the edible oils; sesame, soybean, avocado and the biofuel; jatropha are in the range of common cooking oils and biofuels with respect to their specific gravity values

Viscosity

It is known that the chain length, saturation and unsaturation position of hydroxyl group of the fatty acids directly influence the viscosity of oils, hence oils having greater chain length have greater viscosity. As seen in Table 5, oils in the range of 58-66 mPa.s can be said to consist of long chain carbon atoms.

Refractive index

The refractive index for all the oils was found to be between 1.46 and 1,49 ; this falls within the range of values reported for some common seed oils, 1.48 for Teleferia occidental seed oil and 1.47 for corn oil. Refractive index is used to check purity and to follow and control hydrogenation and isomerization processes.

Iodine value

The Iodine values obtained were between 80 and 118g/100g; According to Ikhuoria and Maliki (2007), iodine value gives an indication of the degree of unsaturation of oils, higher iodine values can be attributed to high unsaturation. In fact, the iodine values of the oils were 117.45, 126.35, 80 and 100.50g of I_2 /100g oil, respectively. These values were higher than those of olive oil. This result indicates that these oils under study, are nondrying, highly unsaturated and it suggests that they contain high levels of oleic and linoleic acids (Elleuch et al., 2007). For biodiesel standards the iodine value should be less than 115 as per biodiesel as poor oxidation stability can cause fuel thickening, formation of gums and sediments, which in turn, can cause filter clogging and injector fouling, the jatropha oil studied gave an iodine value of 100.5 therefore it is a good biofuel which could be used for biodiesel synthesis. For fuels, oils that are denser contain more energy.

Conclusion

It can be seen that all the seeds analysed have appreciable oil contents, especially the sesame and soybean seeds. The high percentage makes these seeds distinct potential for the oil industry in Nigeria for economic purposes. Although the seed oils extracted and characterized had good structural values, refining is necessary to improve the utilization and consumption of these oils as edible oils. The high acid value and the % FFA of the jathropha oil make it non-edible vegetable but great potential for biodiesel production feedstock.

The physiochemical properties of the oils studied revealed that they are all in the range of both edible oils and

biofuel synthesis. The results therefore suggest the suitability of the oils for domestic and industrial uses.

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Evwierhoma E. T.

Department of Chemical Engineering,
University of Lagos.

*Ekop I. E

Department of Agricultural Engineering,
Akwa Ibom State University.
inemesitekop@aksu.edu.ng
* Correspondence

IJSER