



Characterization of (Taura) *Detarium microcarpum* Seed Oil for Fatliquor Production

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ABSTRACT

The seed oil of *Detarium microcarpum* gave 7.50% oil yield, the Physico-chemical properties (saponification value, iodine value, peroxide value, pH value, refractive index and its functional group using FT-IR) of the extracted oil were analyzed using standard official methods and found to contain oil in low levels. This suggests that they contain mainly saturated fatty acids judging by their low iodine value ($61.93 \text{ I}_2 \text{ g}100\text{g}^{-1}$ of oil) which did not exceed $88 \text{ I}_2 \text{ g}100\text{g}^{-1}$ and a refractive index of 1.56 at 40°C , while the peroxide value 6.00 meq^{-1} was an indication of a low level of hydroperoxides that could initiate or propagate further oxidation of the oil, thereby improving the oxidative stability of the oil for the manufacture of a blended fatliquor which can have ageing properties. The iodine value of the oil ($61.93 \text{ I}_2 \text{ g}100\text{g}^{-1}$ of oil) was found to be less than $115 \text{ I}_2 \text{ g}100 \text{ g}^{-1}$ (value for drying oil); this implies that the *Detarium microcarpum* seeds oil is non-drying oil. This was supported by its saponification value of $213.18 \text{ mg KOH g}^{-1}$ and it is an indication that oils of higher saponification value (that is oils of smaller molecules) have high penetrating powers into the leather giving rise to high leather suppleness. The FT-IR spectrums of the oil at various peaks were also observed. The carbonyl functional group: $\text{C}=\text{O}$ was observed at 1744.4 cm^{-1} frequency and frequency 3008 cm^{-1} , $=\text{CH}_2$ in unsaturated hydrocarbon was observed. This oil can have useful properties in the manufacturing of soap, cosmetics and lubricants.

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INTRODUCTION

Seed oil is a vegetable oil that is obtained from the seed (endosperm) of some plant, rather than the fruit (pericarp). An oil is any neutral, nonpolar chemical substance that is a viscous liquid at ambient temperatures and is both hydrophobic (immiscible with water, literally "water fearing") and lipophilic (miscible with other oil, literally "fat loving"). Oils have a high carbon and hydrogen content and are usually flammable and surface active. The general definition of oil includes classes of chemical compounds that may be otherwise unrelated in structure,

properties and uses. Oil may have an animal, vegetable, or petrochemical origin, and may be volatile or non-volatile (Brian *et al.*, 2002).

Fatliquor is a dispersing or lubricating agent in the form of emulsion used to replace the natural oils/fats lost by the skins/hides during leather production. During the preliminary stages of the leather manufacture, unhairing, liming, bating and degreasing, most of the natural oils from the skins are removed, and at the time of completion of tanning, the leather does not contain sufficient amounts of lubricants to prevent it from drying into a hard material. The term fatliquoring is

used for the incorporation of oils and fats into the leather in the form of an emulsion. The main characteristics of fat-liquored leathers are softness, feel suppleness and a certain degree of waterproof. It can penetrate to the collagen fibres, making the leather lubricant and plastic. The fatliqor enables easy movement of the molecular chain segment, giving the leather softness, waterproof, moisture and flexibility (Bin, 2009).

LITERATURE REVIEW

Description of *Detarium microcarpum*

Detarium microcarpum commonly known as sweet dattock or tallow tree is an under-utilized tree legume that grows naturally in the West and Central Africa region. It is also known as Taura, Kwakuragwahu, Ofo, Gungorochi, Sedum Ogbogbo all in Hausa, Kilba, Igbo, Nupe, and Yoruba language of Nigeria

respectively (Guill & Perr, 2007). It is a multi-purpose species with a plethora of use due to its medicinal properties, edible fruit (eaten raw, cooked, or made into the flour with many users of its own) and hardwood used as fuel-wood (Schmelzer and Gurib-Fakin, 2008). *Detarium microcarpum* occurs naturally in the dryer regions of West and Central Africa (Benin, Cameron, Central Africa Republic, Chad, Gambia, Ghana, Guinea -Bissau, Cote d Ivoir, Mali, Niger, Nigeria, Senegal, Sudan and Togo). Unlike the other species of its family, it grows in dry savanna while the *Detarium senegalense* grows in dry forest and *Detarium macrocarpum* grows in humid forests. Different vernacular names exist for this species including the English sweet dattock or tallow tree and the French Dankh or defar as well as Abu-laili (Sudan), and Tamba Dala (Mali) (Guill and Perr, Van Damme, 2006).



The Inner seed of *Detarium microcarpum* obtained directly from the local market



Detarium microcarpum fruits obtained directly from local market



Mature tree of *Detarium microcarpum* in the Nazinon Forest (www.wikipedia.org)



Medicinal Uses

The use of herbal medicine may be as old as the history of mankind (Reuben *et al.*, 2010). As reported, the use of the plant as a medicine is an ancient practice present to all society especially African societies of which Kilba community of Hong Local Government of Adamawa State is not left out in the use of *Detarium microcarpum* (Guill and Perr, 2007) stem, root and bark as a remedy for infant ailment gedigedi or Tando (Hausa), stomach aches, wounds etc, and the fruit is eaten (Kouyarte and Van Damme, 2008). Medicinal application of *Detarium microcarpum* includes the use of the tree bark, leaves and roots with medicinal properties to treat ailments such as tuberculosis, and meningitis (Van Damme, 2008). They have wide distribution areas because of their diuretic astringency properties, which can be obtained by infusion or decoction to treat rheumatism, venereal diseases, urogenital infection, stomach ache, intestinal worms and diarrhea including dysentery (Van Damme, 2008). The fresh bark or leaves are applied to wounds to prevent and cure infection (Van Damme, 2008). Despite the numerous medicinal application of this herbal plant, phytochemical analysis on it are scarce and thus, deserve more attention (Reuben *et al.*, 2013).

MATERIALS AND METHOD

Sample Collection

Detarium microcarpum (Taura) seeds were sourced from new market, Dutsin-Ma Local Government Area of Katsina State, Nigeria, in October, 2017. The seeds were transported to the quality control laboratory Nigerian Institute of Leather & Science Technology, (NILEST) Samaru-Zaria. The specie was identified as *Detarium microcarpum*, belonging to FABACEAE family, with verification number of 480 at the Faculty of Science, Department of Biological Science, Ahmadu Bello University, Zaria, Kaduna State, Nigeria.

Oil Extraction

The specimen seed were crushed using the laboratory mortar and pestle, wrapped in a thimble and placed into the Soxhlet apparatus. About 250ml of n-hexane was measured using a measuring cylinder, this was poured into the round bottom flask and it was then connected with the extraction chamber containing the sample seed. A condenser was placed above the extraction chamber and a cotton wool was used to cover the outlet to prevent the evaporation of the solvent. Water was refluxed through the condenser (inlet and outlet), heat was applied to the extraction apparatus at the bottom of the flask through a heating mantle. This treatment/procedure was done exhaustively with each sample. After the extraction, the seed content in the flask was evaporated to remove the solvent left in the extracted oil.

Determination of Saponification Value

The indicator method was used as specified by ISO 3657 (1988). 2g of the sample was weighed into a conical flask; 25ml of 0.1N ethanoic potassium hydroxide was then added. The content which was constantly stirred was allowed to boil gently for 60 minutes. A reflux condenser was placed on the flask containing the mixture and a few drops of phenolphthalein indicator was added to the warm solution and then titrated with 0.5 M HCl to the end point until the pink color of the inculcator just disappeared (Laboratory Handbook, 1997). The same procedure was used for other samples and a blank. The expression for saponification value (SV) is given by:

$$SV = \frac{56.1N (V_0 - V_i)}{m} \dots\dots\dots 1$$

where:

- V_0 – the volume of the solution used for the blank test,
- V_i – the volume of the solution used for determination,
- N– Actual normality of HCl used,
- m– Mass of the sample.



Determination of Acid Value

25ml of diethyl ether and 25ml of ethanol were mixed in a 250ml beaker. The resulting mixture was added to 2g of oil in a 25 ml conical flask and a few drops of phenolphthalein were added to the mixture. The mixture was titrated with 0.1 m NaOH to the endpoint with consistent shaking for which a dark pink colour was observed and the volume of 0.1 m NaOH (V_o) was noted. Free fatty acid (FFA) was calculated using the equation:

$$V_o/W_o = 82-100, \dots\dots\dots 2$$

(100 ml of 0.1 NaOH=2.83g of oleic acid)

where:

W_o = sample weight

Then acid value = FFA - 2 $\dots\dots\dots 3$

(Laboratory Handbook, 1997)

Determination of Iodine Value

The method specified by ISO3961 (1989) was used. 0.4g of the sample was weighed into a conical flask and 20 ml of carbon tetra chloride was added to dissolve the oil. Then 25 ml of Dam reagent was added to the flask using a safety pipette influenced chamber. A stopper was then inserted and the content of the flask was vigorously swirled. The flask was then placed in the dark for 2 hours and 30 minutes. At the end of this period, 20 ml of 10% aqueous potassium iodide and 125 ml of water were added using a measuring cylinder. The content was titrated with 0.1M sodium thiosulphate solution until the yellow colour almost disappeared. A few drops of 1% starch indicator were added and the titration continued by adding thiosulphate drop-wise until blue colouration disappeared after vigorous shaking. The same procedure was used for the blank test and other samples. The iodine value (IV) is given by the expression:

$$IV = \frac{12.69 \times (V_1 - V_2)}{m} \dots\dots\dots 4$$

where:

V_1 –the volume of sodium thiosulphate used for the blank,

V_2 –the volume of sodium thiosulphate used for determination,

m – Mass of the sample.
(Akpan *et al.*, 2006).

Determination of Peroxide Value

To 1g of the oil sample, 1g of potassium iodide and 20 ml of solvent mixture (glacial acetic acid/chloroform, 2/1 by volume) were added 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 the starch solution were added to the mixture and the latter was titrated with 0.025 N sodium thiosulphate and the peroxide value was determined as follows:

where:

Peroxide value (mol peroxide/kg sample)

$$= \frac{\text{titre} \times \text{molarity of Na}_2\text{S}_2\text{O}_3 \times 1000}{\dots\dots 5}$$

Sample weight (g)

(Akpan *et al.*, 2006)

Determination of Refractive Index

A refractometer was used in this determination. The sample was transferred into the glass slide of the refractometer. To carry out the determination of the refractive index of oil, the prism box was opened and a few drops of the oil were placed on the ground surface of the lower prism. It was then closed and the box flattened again, making sure that the oil did not flow away. The cross wires of the telescope were focused by rotating the eyepiece and adjusting the mirror to get good illumination. Using the lower knob, the prism box was turned slowly backwards and forwards until the field of view became coloured fringe. Using the upper knob, the compensator was rotated until the coloured fringe disappeared and the lighted image showed a sharp edge. The prism box was rotated until the sharp edge was in coincidence with the intersection of the cross-wires in the telescope. The index of refraction was then read off on the scale through the eyepiece. The third decimal place in the refractive index could be read directly and the fourth was estimated with an accuracy of about ± 0.0002 (Agro-



physics, 2008).

Determination of pH Value

2g of the sample were poured into a clean dry beaker and 25ml of hot distilled water was also added to the sample in the beaker and stirred slowly. It

then cooled in a cold-water bath to 25°C. The pH electrode was standardized with a buffer solution and the electrode was immersed into the sample and the pH value was read and recorded (Akpan *et al.*, 2006).

RESULTS AND DISCUSSIONS

Table 1: Chosen properties of oil extracted from *Detarium microcarpum*

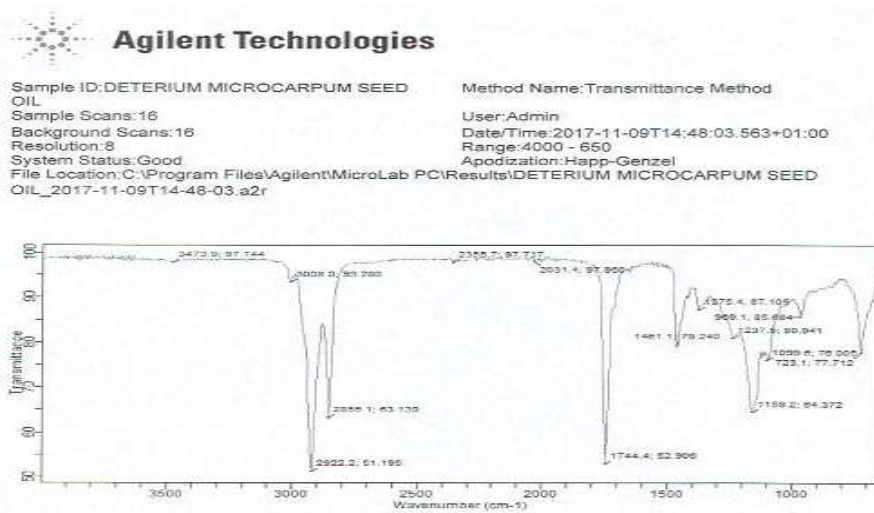
Parameters	<i>Detarium microcarpum</i>
Acid value (mg KOH g ⁻¹ of oil)	4.49
Saponification value (mg KOHg ⁻¹ of oil)	213.18
Iodine value (I ₂ g 100 g ⁻¹ of oil)	61.93
Peroxide (ml g ⁻¹ of oil)	6.00

Table 2: Physical Properties of *Detarium microcarpum* and the Oil

Parameter	<i>Detarium seeds and oil</i>
Moisture content (%)	0.850±0.26
Ash content (%)	0.765±0.03
Oil yield (%)	7.50±0.10
pH	6.20
Refractive index (27°C)	1.564
Solubility of oil in water	Insoluble

Table 3: Results for the FT-IR analysis

S/N	Frequency (-cm ⁻¹)	Frequency
1.	1744.4 cm ⁻¹	-C=O stretch for aliphatic ester
2.	3473.9 cm ⁻¹	-OH stretch for carboxylic acids
3.	2855 cm ⁻¹	-CH ₂ - In aliphatic compounds
4.	3008 cm ⁻¹	=CH ₂ stretch in unsaturated Hydrocarbons
5.	2922.2 cm ⁻¹	-CH ₃ stretch





The infrared spectrum of the n-hexane extracts functional group absorption bands are shown in table 3. This depicts the various wavelengths of the oil extract the peak observed at 1744.4 cm^{-1} is attributed to -C=O carbonyl group stretch for aliphatic ester, the absorption bands at 3473.9 cm^{-1} is due to -OH hydroxyl group which was stretch typically from carboxylic acids. Also, the absorption bands at 2855 cm^{-1} were due to $\text{-CH}_2\text{-}$ methylene groups stretch in aliphatic compounds, in 3008 cm^{-1} absorption bands is due to =CH_3 groups stretch in unsaturated hydrocarbons, and bend at 2922.2 cm^{-1} is as a result of -CH_3 groups stretch typically from the spectra. It can be observed that the peaks increase in the extract is due to the presence of -OH groups and =CH_2 which were stretched from carboxylic acids and unsaturated hydrocarbons respectively. Therefore, unsaturated fatty acids are more fluid, darken with sunlight, become sticky or gummy on oxidation by air, and readily combine with sulphur or iodine (that is easily sulphated). Thus highly unsaturated oils may cause trouble on the aging of the leather. In the painting trade, they are classified as semi-drying (that is castor oil) because they become gummy on exposure to air and drying oils (linseed oil) which on exposure "dry" to a hard, non-oily or non-tacky varnish.

Percentage Yield

The comparative analysis with oil seeds showed that the seeds can be classified as oil seeds or oil crop since it can serve as a commercial source of vegetable oils. This value for the oil content of the seed (7.50%) was higher compared with results of oil contents of *Brachystegia eurycoma* (5.87%), and *Mucuna flagellipes* (3.77%) (Ibironke et al., 2005).

Iodine Value

From Table 1, *Detarium microcarpum* seed oil is shown to contain a low percentage of fatty matter, with the

quality of iodine value of $61.0 (\text{I}_2 \text{ g } 100 \text{ g}^{-1} \text{ of oil})$ compared to 86.5, 81.5 and 81.0 of Karanja oil, sulphated Karanja oil and sulphated castor oil respectively (IOSR., 2015). The iodine value is used to determine the degree of unsaturation of fatty acids. Oil of iodine value above 140 is thus very reactive and are called as drying oil. Similarly when the iodine value is between 95-140 are known as a semi-drying oil. And non-drying oils should have iodine value below 95.

Saponification Value

The saponification value in mg KOH g^{-1} was 213. The saponification value of oil, fat or an ester is defined as the number of milligrams of potassium hydroxide (KOH) required to completely neutralize the free acids to saponify the esters present in 1gm of the substance. It also indicates the length of the carbon chain of the acid present in that particular oil or fat. An increase of saponification value of oil increases the volatility of the oil. It enhances the quality of the oil because it shows the presence of a lower molecular weight compound in 1g of the oil. The principle in the method of Jacobs (1958) reveals that the number of milligrams of KOH required to saponify the oil will be greater in the former than in the latter case. Therefore, the higher the saponification value, the lower the molecular weight of the fatty acids and the better the quality of the oil.

Acid Value (AV) And Peroxide Value (PV)

The acid and peroxide values were found to be (4.49 mgKOH/g and 6.00 milliEqu./kg , respectively) of *Detarium microcarpum* seed oil. These suggest that the oil has a low deteriorating rate and therefore can be stored for

a relatively long period. Both values are the indices of biogenic and oxidative rancidity, respectively, in neutral lipids; and higher values are associated with a higher rate of rancidity. Higher AV materials allow faster appearing but less stable suds creation. Lower AV materials



allow slower to appear but more stable suds formation. Lower acid value means more cleansing (detergency).

Refractive Index

The seeds oil had a refractive index of 1.56 at 40°C. Refractive index of the oil is the ratio of the speed of light at a defined wavelength to its speed in the fat itself. This value varies with wavelength and temperature and also varies with the degree and type of unsaturation as well as the type of substitutions of component fatty acids and with accompanying substances. Refractive index is widely used in quality control to check for the purity and adulteration of fatty materials and to follow hydrogenation and isomerization (Hoffman, 1986)

CONCLUSION

The crude extract of *Detarium microcarpum* seed oil examined in this work by its pH value, acid value, iodine value, saponification value, peroxide value, refractive index especially, the infrared spectroscopic analysis that shows that Unsaturated fatty acids are more fluid, darken with sunlight, become sticky or gummy on oxidation by air, readily combine with sulphur or iodine, that is easily sulphated. Thus highly unsaturated oils may cause trouble on the aging of the leather. In the painting trade, they are classified as semi-drying (for example castor oil) because they become gummy on exposure to air and drying oils (for example linseed oil) which on exposure "dry" to a hard, non-oily or non-tacky varnish. They may, however, be useful for other purposes such as soap, judging by their high saponification values in the range of 123-261 mg KOH⁻¹.

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