Extraction and Physicochemical Characterization of Seed Oil from *Terminalia Mantaly* Seed

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Abstract: **- The increase in demand and different applications of oils foster the search for vegetable and seed oils that are of high quality to meet up with the increasing rate of its demand worldwide. In this research, the extraction and physicochemical characterization of the oil from *Terminalia Mantaly* was carried out. The seed oil of the plant was extracted using solvent (N-Hexane). Standard method was adopted to extract the seed oil of the plant. The parameters of seed oil determined include Physico-chemical properties such as; acid value 0.56 mg KOH/g, peroxide value 4 mEq/Kg, ester value 195.79 mgKOH/g, oil content 37 %, iodine value 53.30 gI_2/100g, saponification value 196.35 mgKOH/g, moisture content 0.4, density 0.92 g/ml, kinematic viscosity 4.80, specific gravity 1.08. These make the oil suitable for use in the industry except in the ink and paint industries because of its non drying property which is due to its low iodine value content.**

Key-Words: **Terminalia Mantaly, Extraction, Seed Oil, Nutritional Value**

I. INTRODUCTION

The use of fats and oils by man dates back to antiquity (Emmanuel *et al*, 2009). Vegetable oils are widely consumed domestically in Nigeria (Kayode, 2015, Nkafamiya *et al*, 2010). The interest in vegetable oils with bioactive compounds, such as the ones extracted from fruit seeds, is growing. (Jorge *et al*, 2014). Almost every part of the tree; roots, trunk, bark, leaves, flowers, fruits and seeds, is known to have some uses. They could also contribute to the supply of nutrients to the soil via nitrogen fixation as leguminous does (Awadia *et al*, 2016, Bello and Abdu, 2011).

Plants were used as a source of medicine in from the centuries ago and today the scientists and the general public recognize the value of plants as a source of new or complimentary medicinal products (Martin and Nazeema, 2015, Priya, 2011).

Nuts oils, seed oil and oils of fruit and vegetables are receiving growing interest due to their high concentration of bioactive lipid components, such as polyunsaturated fatty acids and phytosterols, which have shown various health benefits. Fats and oils, and their several lipid components are extensively used in the food and also in cosmetics, pharmaceuticals, oleochemicals and other industries (Maria *et al.*, 2012, Duman *et al*, 2011)Their chemical composition and specific properties have allowed them to find use as foods, fuels and lubricants. Their sources are numerous, encompassing vegetable, Animal, and marine sources (Emmanuel *et al.*, 2009, Nwobi *et al.*, 2006).

Biodiesel is produced from vegetable oils and fats by a transesterification reaction with mono- or dialcohol (Jose *et al*, 2014,Openshaw,2000) The vegetable oils are considered sources of these compounds, especially carotenoids, phenolic compounds, tocopherols, and phytosterols (Jorge *et al*,2014, Malacrida *et al.*, 2012). As it is with all matter, their usefulness to man is determined by their chemical nature; and all fats and oils have certain characteristics in common. Fats and oils are naturally occurring substances which consist predominantly of mixtures of fatty acid esters of the trihydroxy alcohol or glycerol (Emmanuel *et al*, 2009, Nwobi *et al*, 2006).

Different fats and oils come about due to the fact that there are numerous fatty acids of various kinds and these can be combined in an infinite number of ways on the hydroxyl centers of glycerol. Moreover, the physical properties of fats and oils are dependent on the nature of fatty acids involved in the ester. Hence the traditional distinction of fats as solids and oils as liquids arises from the fact that due to the different chemical structures of the different fatty acids combined in the esters, the bonding forces in existence vary in strength resulting in different melting points. These differences are manifested in different chain lengths, the presence or otherwise of unsaturation as well as geometric conformations (Emmanuel *et al*, 2009).

The present emphasis on conservation and environmental friendliness has brought about renewed interest in the use of these "natural oils" for non edible purposes. Their established superiority in terms of biodegradability(Emmanuel *et al*, 2009), when compared with mineral oils, as well as the fact that they are renewable and generally non toxic has focused attention on technologies that would enhance their usefulness as bio fuels and industrial lubricants (Emmanuel *et al.*, 2009). There are also concernsas to what to expect in case of vegetable oil spills. (Emmanuel *et al. 2009*, Li *et al*, 2005) including the process of remediating such spills (Emmanuel *et al*, 2009,Wincele *et al*, 2004).

II. MATERIALS AND METHOD

2.1 Materials / Equipment

Smooth stones together with stainless steel and container were used , mortar & pestle were be provided then soxhlet extractor set-up, rotary shaker(Bio Techno Lab Mumbai India)and retort stand were used, water bath HHW420(B-scientific England), heating mantle, beaker, *Terminalia mentaly* seed and Reflux condenser were also provided.

2.1.1 Chemical and Reagent

Distilled water, Dam's reagent together with some important reagent such as carbon tetra chloride,(May and Baker limited DangenHam England) diethyl ether (Sigma Aldrich Germany) and ethanol(JHD China) were used , then sodium hydroxide(JHD China), potassium hydroxide(JHD China), starch solution, phenolphthalein indicator (Sigma Aldrich Germany) and glacial acetic acid, Ammonia, Hydrochloric acid, N-Hexane, sulphuric acid, (E.merck, Darmstadt Germany), Boric acid(BDH Chemicals limited poole England), Iron(iii)chloride, potassium iodide, ammonium thiocyanate, orthophosphoric acid , Silver nitrate (HEZEDATONG CHEMICAL CO., LTD Sandong-China) were also be provided, sodium thiosulphate, ethanoic potassium hydroxide were also used in this research.

2.2 Sample Collection and Preparation

The fresh seeds of *Terminalia mantaly* were collected from Fadaman Madda, Bauchi Local Government Area of Bauchi state, and identified at the department of Biological Sciences, Abubakar Tafawa Balewa University (ATBU) Bauchi. Sample was then washed with distilled water 3 times, dried under shade and stored for further use.

2.2.1 Solvent extraction techniques

Terminalia Mantaly seeds were crack and the shells were carefully removed. The kernels thus obtained were used for oil extraction.

The kernels were grounded using mechanical method (mortar and pestle). The oil was extracted using organic solvents N-hexane. 352.0 g of grounded seeds was placed in a cellulose paper cone and extracted using N-Hexane in a soxhlet extractor for 6 hours. The extracted lipid was obtained by different techniques such as filtration, centrifugation and separating funnel; in order to get rid of the solid from solvent before the solvents was removed. Extracted seed oil was stored in freezer at -20°C for subsequent physicochemical analysis (Petal *et al.*, 2011)

2.3 Soxhlet extraction

The method described by Petal *et al.*, 2011 and Akbar *et al*. (2009) with slight modification was used. The seed kernels (352 g) were grounded using a mechanical method and defatted in a soxhlet apparatus. The extraction was carried out by using organic solvents such as N-hexane. The process was continued for 6 h. Solvent will be removed by vacuum evaporation and exposure to heat in a drying oven at 50°C. The amount of oil recovered will be calculated as percentage of total oil present in *Terminalia Mantaly* seed kernels. Each extraction was run in triplicate and the final value is the average of all.

2.4 Acid value

2.4.1 Principle: The acid value is determined by directly titrating the oil/fat in an alcoholic medium against standard potassium hydroxide/sodium hydroxide solution (F.S.S.A.I, 2015).

2.4.2 Procedure: A 5 ml' of diethyl ether and 5 ml of ethanol was mixed in a 250 ml beaker. The resulting mixture was added to 2.0 g of oil in a 250 ml conical flask and few drops of phenolphthalein were added to the mixture. The mixture was titrated with 0.1 M NaOH to the end point with consistent shaking for which a dark pink colour was observed and the volume of 0.1 M NaOH (V_0) was noted. The expression for acid value (AV) is given by:

$$AV=\frac{(Vb-Va)cm3\ X5.61}{Wt\ sample\ \ used}\ (\text{mg KOH/g})\ \text{----------i}$$

Va = sample titre value, Vb = blank titre value(Saeed and Shola, 2015)

2.5 Determination of saponification value

*2.5.1 Principle***:** The oil sample is saponified by refluxing with a known excess of alcoholic potassium hydroxide solution. The alkali required for saponification is determined by titration of the excess potassium hydroxide with standard hydrochloric acid (FSSAI, 2015).

*2.5.2 Procedure***:** The indicator method was used as specified by kyari, 2008, 2 g of the sample was weighed into a conical flask; 10 ml of 0.1 N ethanoic potassium hydroxide was then be added. The content was constantly stirred and allowed to boil gently for 30 min. 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 colour of the inculcator just disappeared. The same procedure was used for other samples and a blank. The expression for saponification value (SV) is given by:

$$SV=\frac{56.1\ N\ (Vo-Vi)}{m}\text{ - - - ii}$$

Where: *Vo* – the volume of the solution used for the blank test, *Vi* – the volume of the solution used for determination,

N– Actual normality of HCl used m– mass of the sample.

2.6 Determination of iodine value

2.6.1 Principle: The oil/fat sample taken in carbon-tetrachloride is treated with a known excess of iodine monochloride solution in glacial acetic (Wij's solution). The excess of iodine monochloride is treated with potassium

iodide and the liberated iodine estimated by titration with sodium thiosulfate solution (FSSAI,2015).

*2.6.2 Procedure***:** The method specified by kyari (2008) was used. A 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 25 ml of Wij's reagent was added to the flask using a safety pipette influenced chamber. A stopper was then be inserted and the content of the flask was vigorously swirled. The flask was then placed in the dark for 2 h and 30 min. 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 0.1 M sodium-thiosulphate solution until the yellow colour almost disappeared. A few drops of 1% starch indicator were added and the titration was continued by adding thiosulphate drop-wise until blue coloration disappeared after vigorous shaking. The iodine value (IV) is given by the expression:

$$IV = \frac{12.69C(V1-V2)}{m} \quad \text{iii}$$

2.7 Determination of Moisture Content of the Seeds

A 40 g of the sample was weighed and dried in an oven at 80°C for 3 hrs and the weight was taken after every 30 min. The procedure was repeated until a constant weight was obtained. After each 30 min., the sample was removed from the oven and placed in the desiccator for 30 minutes to cool. It was then removed and re-weighed. The percentage moisture in the seed was calculated from the formula:

$$\text{Moisture} = \frac{W1-W2}{W2} X\ 100\% \quad \text{-iv}$$

where W_1 = Original weight of the sample before drying; W_2 = Weight of the sample after drying.

2.8 Determination of the Percentage Oil Extracted

A 50 g of the sample was placed in the thimble and about 250 ml of normal hexane was poured into the round bottom flask. The apparatus will be heated at 60°C and allowed for 3hrs continuous extraction using Soxhlet apparatus. The experiment was repeated for different weights of the sample. At the end, the solvent was distilled and the percentage of oil extracted was determine

2.9 Determination of peroxide value

To 1 g of the oil sample, 1 g of potassium iodide and 20 ml of solvent mixture (glacial acetic acid/chloroform, 2/1 by volume) was 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 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:

$$PV = \frac{SN\ 1000}{W} \text{----------v}$$

where: S – ml of $Na_2S_2O_3$, N – normality of $Na_2S_2O_3$,

W – weight of oil sample (g).

2.10 Determination of Ester Value

The ester value was determined by using the procedure described by (Ebenezer, 2015).

Ester value = saponification value – Acid value ---------vi

III. RESULTS AND DISCUSSION

Table 1: Summary of physical properties of the oil extract

Parameters	Value
Density (kg/L)	0.92
Colour	Amber
Moisture (%)	0.4
Viscosity (mm^2/s)	4.8
Specific gravity L/Kg	1.08

Table 2: Summary of chemical properties of oil

Properties	Values
Oil yield (%)	37.00
Acid Value (mg KOH/g)	0.56
Peroxide Value (meq/Kg)	4.00
Iodine Value (gI2/100g)	53.30
Ester Value (mg KOH/g)	195.79
Saponification value (mgKOH/g)	196.35
FFA (%)	0.28

Table 3: Combined result of physicochemical propertied of some edible oil

Sample	Acid value	FFA	Peroxide value	Iodine value	Saponification value
Terminalia mantaly	0.56	0.282	4.00	53.30	196.35
Moringa seed	0.51±0.03	0.23±0.01	5.82±0.87	68.00±0.1	182±0.76
Cashew seed	0.84±009	1.34±0.34	3.45±0.41	48.45±0.43	162±0.05
Sesame seed	0.62±0.02	0.28±0.02	8.33±0.18	116.05±0.54	192.05±0.56
Bitter cola seed	0.06±0.01	0.096±0.01	10.22±0.24	53.99±034	229.45±0.04
Melon seed	0.43±0.11	0.24±0.05	7.19±0.03	124.40±0.67	247.50 ±0.34
WaterMelon seed	0.51±0.04	0.19±0.02	13.41±0.43	114.4±0.87	192.09±0.09
A.O.A.C. 1990 Standard	<= 4	,= 1.30	2-10	8-100	.= 180

Discussion

The results for the physical, chemical and combined result of physicochemical propertied of some edible oil of the extracted seed oil were given in tables 1, 2, and 3 . The oil is Amber in colour and less viscous, 4.8 mm^2/s at room temperature when compared to Bebra seed oil($40.59mm^2/s$) and cashew nut oil($56mm^2/s$) (Andualem and Gassesse, 2014). It has density and specific gravity of 0.92 Kg/L and 1.08L/Kg at 20 ^oC, respectively. The density of oil in this study(0.92Kg/L) is lower than the density of Bebra seed oil (0.94Kg/L) and the specific gravity of this study(1.08L/Kg) is higher than that of Bebra seed oil (0.926L/Kg), Cashew nut (0.964) (Aremu et al.,2006) and caster seed oil(0.958) (Akpan et al., 2007,Andualem and Gassesse, 2014).

The percentage oil yield of the seed was 37%. This value is similar to that of the percentage oil yield of Terminalia Catappa 38%, melon seed 38.30% moringa seed 40.60%, less than that of cashew seed 49.34% sesame seed 47.80% and higher than Bitter kola seed 11.92% as reported by (Saeed and shoal, 2015) Ebenezer, 2015, Abdulhamid *et al.,* (2014), and also higher than that of some conventional oil seed crops like cotton (15.0-24.0%) and soybean (17.0-21.0%) (Ebenezer, 2015). And it is within the A.O.A.C 1990. This high percentage oil yield indicates its potential use in detergent/ soap making industries and edible purposes.

The Saponification value of the oil was found to be(196 mgKOH/g) which is higher than that of Bebra seed oil (174.95 mgKOH/g), and some of common oil like castor seed oil(185.83mgKOH/g) (Andualem and Gassesse, 2014), Moringa Seed 182.89, cashew seed 169.42, sesame seed 192.70, water melon seed 192.09 (Saeed and Shola, 2015) and also *Terminalia Catappa*(140.275mgKOH/g), beeswax (93.0mgKOH/g) which is commonly used in soap making (Ebenezer,2015, Mabrouk, 2005). The value is similar to that of palm oil (196-205mgKOH/g), ground nut oil (188-196mgKOH/g), corn oil (187-196mgKOH/g) and lower than coconut oil (253mgKOH/g) and palm kernel oil (247mgKOH/g) as reported by Andualem and Gassesse, 2014 bitter kola seed 229.45, melon seed 247 (Saeed and shoal,2015) this is within the A.O.A.C.Standard 1990. Oils with lower saponification values contains high amount of long chain fatty acids (Andualem and Gassesse, 2014).

The acid value in this study was (0.56 mgKOH/g) which is higher than that of Bebra seed oil (0.052mgKOH/g), as reported by Andualem and Gassesse, 2014, melon seed 0.43, water melon seed 0.51 moringa seed 0.51,bitter kola 0.06 and lower than that of cashew seed sesame seed 0.62 (Saeed and Shoal,2015).This value falls within the AOAC Standard of < 4.0.According to Andualem and Gassesse, (2014) Aremu *et al.* (2015), low acid value in oil indicates that the oil will be stable over a long period of time and protect against rancidity and peroxidation. This could be attributed to presence of natural antioxidants in the seeds such as vitamins C and A as well as other possible phytochemicals like flavanoids. Acid value is used as an indicator for edibility of an oil and suitability for use in the paint and soap industries. High acid value in oil (e.g. luffa gourd) showed that the oil may not be suitable for use in cooking (edibility), but however, be useful for production of paints, liquid soap and shampoos (Aremu *et al.*, 2006a).

The peroxide value in this study was (4 mEq/Kg) which is higher than that of Terminalia Catappa(2.60mEq/Kg) as reported by Ebenezer,2015, cashew seed 3.45 Saeed and Shola,2015, and lower than Bebra seed oil 6.88 mEq/Kg. and also was greater than the peroxide value of 3.1 mEq/Kg of cashew nut oil (Aremuet *et al.,* 2006), moringa seed 5.82, sesame seed 8.33, bitter kola 10.22,melon seed 7.19 and water melon seed 13.41and the value is fall within the AOAC 1990 standard Saeed and Shola, 2015.Peroxide value (PV) is the most common indicator of lipid oxidation. The unrefined vegetable oils are characterized by greater PV, compared to refined oils. High values of PV are indicative of high levels of oxidative rancidity of the oils and also suggest absence or low levels of antioxidant (Andualem and Gassesse, 2014, Aremu, *et al.*, 2015).

The iodine value in this study was (53.30 $gI_2/100g$), which is similar to that of bitter kola 53.99,Terminalia Catappa (54.567 $gI_2/100g$) as reported by Ebenezer (2015) Saeed and Shola, 2015, and higher than that of some common seed oils like 44.4 $gI_2/100g$ cashew nut oil (Andualem and Gassesse, 2014, Aremuet *et al.*, 2006), 38.1 $gI_2/100g$ citrullus vulgaris cashew seed 48.45,Saeed and Shola, 2015 (Andualem and Gassesse, 2014), *Citrus sinensis* seed oil (38.50 $gI_2/100gm$) (Andualem and Gassesse, 2014, Oladimeji *et al.*, 2001). On the other hand, the Iodine value of this study (53.30 $gI_2/100g$) was lower than the range of110–115, 125–135, 125–140, and 115–124 $gI_2/100g$value of rapeseed oil, sunflower oil, soybean oil and corn oil, respectively (Andualem and Gassesse, 2014), moriga seed 68.00, sesame116.05, melon seed 124.40and water melon seed 114.4 and it is within the AOAC 1990 Standard Saeed and Shoal, 2015. The oil in this study was considered nondrying oil since drying oils have an iodine value above $100gI_2/100g$. The iodine value could be used to quantify the amount of double bond present in the oil which reflects the susceptibility of the oil to oxidation. Oils with iodine value less than 100 $gI_2/100g$ of oil are non-drying oils, correspondingly, Arem *et al.*,(2006a) reported that the lower the iodine value the lesser the number of unsaturated bonds; thus, the lower the susceptibility of such oil to oxidative rancidity. Therefore, non-drying oils are not suitable for ink and paint production due to their non-drying characteristics but may be useful in the manufacture of soaps (Ebenezer, 2015, Kochhar, 1998) and can be regarded as liquid oil. A good drying oil should have iodine value of 130 and above (Ebenezer, 2015, Aremu, *et al.*, 2015).

IV. CONCLUSION

It can be shown that from the result of the analysis, the seed of Terminalia Mantaly has higher Percentage oil content of

37%. The oil obtained in this research was analyzed for specific gravity at 20°C, viscosity at room temperature, acid value, saponification value, iodine value, peroxide value and ester value. Their respective values are 0.56 mg KOH/g, 196.35 mg KOH/g, 53.30 gI_2/100g, 4 meq/Kg and 195.79 mg KOH/g respectively.

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