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PHYSICOCHEMICAL AND SPECTROSCOPIC ANALYSIS OF OILS FROM *TERMINALIA IVORIENSIS* A CHEV COMBRETACEAE

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ABSTRACT

Terminalia ivoriensis A. Chev Combretaceae is a West African medicinal plant widely used by the Ijaws in the Niger Delta region of Nigeria for the treatment of malaria and management of erectile dysfunction in men. The Seeds were extracted using successive maceration at room temperature using n-hexane, dichloromethane and 70% methanol exhaustively and concentrated *in vacuo,* yielded 11.29, 10.03, and 0.954 percent of oily extracts respectively. The samples were subjected to physicochemical analysis and the following parameters were obtained; 0.9104-0.9303, 1.466-1.474, 38.15-58.34mg/KOH, 84.2-115mgKOH/g, 15.23-24g I_2/100g and 26-80meq/Kg, for relative density, refractive index, acid value, saponification value, iodine value, peroxide value respectively. The GC-FID analysis of the oils showed that the oils contained 11 fatty acids and FT-IR result of the samples reveals that the oils contained fatty acid with a prominent characteristics peak at 1740 and 3004 cm^{-1} due to carbonyl and methoxyl group of esters respectively. The physicochemical analysis revealed that the oils could be used for skincare, emollient, vehicle for topical agents and as an insect repellant in agriculture because of the pungent and irritating odour produced by the oils.

KEYWORDS

Terminalia ivoriensis, Physicochemical parameters, GC-FID, FT-IR Analysis and Skincare.

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INTRODUCTION

Terminalia ivoriensis A. Chev Combretaceae is a West African medicinal plant widely distributed in the tropics. It is a nut producing plant used medicinally for the management of infectious diseases and erectile dysfunction in men by the Ijaws in the Niger Delta region of Nigeria[1,2]. Seeds,

fruits and nuts are good source of natural antioxidant which protects cells against harmful effect of free radicals implicated in diabetes, arthritis, cancer, and cardiovascular disorder[3]. Evaluation of *Terminalia* species has shown that they possess antioxidant, anticancer, trypanocidal, aphrodisiac, anti-inflammatory activity, hepatoprotective effect, wound healing activity and are used in the treatment of schistosomiasis and management of sickle cell disease[4-9].

Terminalia ivoriensis is a nut producing plant and all over the world nuts and seed producing plants are major source of edible oils consumed globally. Edible oils are vital constituent of our daily diet which provide energy, essential fatty acids and serve as a carrier of fat soluble vitamins[10]. About 90% of the World production of oils and fats come from animal, marine and vegetables which are used as food or as an ingredient in food, cosmetics and vehicle in certain Pharmaceutical formulations such as suspension, injections, and emulsion. Oils and fats are sources of vitamins, amino acid, minerals and fatty acids which are essential to the biological function of the body[10,11].

Plants producing seeds have been exploited commercially for the production of oils. Plants that produce oil bearing seeds include; soya beans, cotton seed, palm seed, groundnut, corn and sunflower oil[3]. Most plant produced seeds contained essential fatty acids such as Omega-3 and omega-6 which play a crucial role in brain development, cardiac functions and lipid lowering ability.

MATERIAL AND METHODS

Materials and Chemicals

All chemicals and reagents used were of analytical grade: sigma and JHD products. The Infrared analysis was carried out using Agilent Cary 630 FTIR using ATR accessory and GC-FID Agilent Auto sampler 7890 A model.

Collection and Identification of Plant Materials

The fresh seeds were collected from the wild at Otabi community in Oloibiri district of Ogbia Local Government Area of Bayelsa State-Nigeria. The plant was authenticated by Prof. Ajibesin Kola of Department of Pharmacognosy and Herbal Medicine, Niger Delta University Wilberforce Island, Bayelsa State, herbarium voucher specimen UUH66 was deposited in the Department of Pharmacognosy and Herbal Medicine, Faculty of Pharmacy, University of Uyo, Uyo.

Extraction

The dried powdered seed weighing 1505g was extracted via successive maceration at room temperature using 2x2.5L each of n-Hexane, dichloromethane and 70% methanol for 7-days respectively. Each of the extract was concentrated at 40^{o}C using rotary evaporator. The concentrated oils were subjected to physicochemical analysis[12].

Density Measurement

A 25ml clean density bottle was washed and rinsed with acetone, allowed to dry and weighed. The weight of the empty bottle determined (W_1). The density bottle was filled with test sample to the fluidicial mark and the weight determined (W2). The density bottle was cleaned, dried and filled with Carbon free water weighed on analytical balance and the weight recorded as W_3[13-15].

Refractive index Measurement

The refractive indices of the oils were determined at 20^{o}C using the Abbe refractometer. The sample was introduced into the prism surface and the required field was adjusted using the knob and readings recorded[14].

Viscosity Measurement

The viscosities of the samples were determined at 60, 30, 12 and 6 rpm at 20^{o}C using NDJ-5S Viscometer with a n^{o} 3 spindle. The sample was stirred for 1 minute waiting the reading on the meters monitor to be stable[16, 17].

Acid Value Measurement

The test sample weighing 0.5g was dissolved in 20ml of a mixture of methanol: ether (1:1) and 3 drops of Phenolphthalein solution was added as an indicator and titrated using 0.1M solution of Potassium Hydroxide solution. A blank determination was carried out without the test sample and the acid value determined[15,16].

Saponification Value Measurement

The test sample weighing 0.5g was dissolved in 25ml of 1 M KOH and refluxed on a boiling water

bath for 1hour cool and back titrate the excess KOH with 1M Hydrochloric acid (a ml) using phenolphthalein as the indicator and blank determination was carried out by omitting the test sample (b ml). The titration was carried out in duplicate and the saponification values determined[15,19].

Peroxide Value Measurement

The test sample weighing 0.5g was dissolved in 10ml chloroform and 15ml glacial acetic acid and 1ml 10% saturated KI Solution were added to the content of iodine flask, shaken, allowed to stand for 1 minute in the dark and 30ml of distilled water was added and titrated to a faint yellow colour with 0.01 M Sodium Thiosulphate solution and 1ml of Starch indicator added towards end point (a ml). A blank determination was carried out (titration b ml) and the peroxide value determined[10-13].

Iodine Value Measurement

The sample weighing 0.5g was transferred into a dry 250ml iodine flask and 10ml of chloroform added to dissolve the sample and 20ml of 0.1 M iodine monochloride was added and the mixture was kept in the dark for 30 minutes, 25ml of 10% KI and 100ml of distilled water were added. The liberated iodine was titrated using 0.1M Sodium Thiosulphate and starch mucilage was added towards end point as indicator. A blank determination was carried without the test sample[13,15].

Ester Value Measurement

The ester value will be determined by subtracting the acid value from saponification value.

RESULTS AND DISCUSSION

The seeds of *Terminalia ivoriensis* extracted with n-hexane, dichloromethane and methanol yielded 11.29, 10.03 and 0.954 percent of oily extracts which were subjected to physicochemical, FT-IR and GC-FID analysis.

The relative density of the experimental oils ranges 0.9104 - 0.9303, this implies that water is denser than the oils; oily extracts fall within the relative density range for fixed oils comparing with arachis oil with a relative density of 0.918[20,21].

The refractive index is unique characteristics that show how oils respond and bend light. Refractive indices of the oily samples ranges from 1.466 - 1.474 as shown in Table No.1. This implies that the values obtained are in line with standard values for fixed oils. When compared with olive oil with a refractive index of 1.4600 and arachis oil, 1.468-1.472, it means that light travels slowly in the experimental oils than olive and arachis oils; this could be due to different composition of the oils and interaction with light[10].

Viscosity is a measure of the internal frictions of the oil molecules and depends on the nature of the triglyceride and chemical constituent present in the oils. The viscosities of the oils at 6, 12, 30 and 60 rpm exhibited same pattern due to similar chemical characteristics of the oils but differ in their response when shear stress is applied as shown in Figure No.1 above. Viscosity is related to the chemical properties of the oils the more viscous oil is better its use as lubricant. Oils with low viscosity indicate they are light and probably highly unsaturated[10,22].

The acid value of the oils extracted ranges; 38.5-58.4, which revealed that the oils have undergone hydrolysis when compared with acid values of reference standard of olive oil 0.3-1.0 and arachis oil 0.5-0.8, however, the oils showed high level of acid value similar to wool fat with an acid value of 59.8 especially by n-hexane and methanol oily extracts. Low acid value indicates that the oils will be stable for a long period of time. High acid value showed that the oils may not be suitable in cooking (edibility), however, could be used for the production of paints, liquid soap and Shampoos[22].

Saponification value is an index of the average molecular weight of the fats and oils. The higher the molecular weight the smaller the saponification value and the higher the saponification value the greater is the percentage of short chain fatty acids present in the glycerides of the oils or fats. The saponification values of n-hexane, dichloromethane and methanol oily extracts ranges; 84.2-115.0mg/KOH which is below the standard stated by International Codex for edible oils compared with a reference standard 196-205mg/KOH. This implies that oils with high saponification value will

be suitable for industrial purposes such as soap making, the oils obtained cannot be used for soap making[23-25].

Iodine value is an indication of degree of unsaturation in a molecule. The high iodine values mean the oils contained fatty acids that are highly unsaturated and it varies for oils. The experimental values of the oils obtained ranges from 15.23-24.75g I_2/100g. The oily extracts obtained contain high level of saturated fatty acids which reflect on low iodine value obtained from the oily extracts, however, the oily extracts could be classified as nondrying oils with an iodine number less than 115gI_2/100g. The physicochemical analysis revealed that the oils could be used for skincare, emollient, vehicle for topical agents and as an insect repellant in agriculture because of the pungent and irritating odour produced by the oils. The experimental iodine values of the oily samples when compared to standard reference values of fixed oils such as; arachis oil, 88-98, olive oil 79-88, and wool fat 17-29gI_2/100g which showed that arachis and olive oils have high iodine value than the oily samples. The iodine value of the n-hexane and methanol oily samples are within the standard reference range for wool fat, 17-29g I_2/100g, however, dichloromethane oily sample has a lower iodine value compared to wool fat. High iodine value is an indication of decline in stability and susceptibility to oxidative rancidity[17,19,25,26].

Peroxide value is a useful indicator of spoilage of samples containing fats and oils. The peroxides values of the oily samples ranges from 26 - 80meq/Kg. The samples showed values above recommended standard of 10meq/kg for oils; however, the experimental values of the oils are above the standard recommended by Standard Organization of Nigeria and Nigerian Industrial Standards for edible oils which is an indication of rancidity and it showed the level or extent of lipid oxidation. The oxidation of fats and oils are undesirable due to off flavors, toxins and loss of fat soluble vitamins. Fixed oils are used in food, cosmetics and medicine because increase in peroxide value is a threat to human health and suggest absence or low level of antioxidant activity[22, 27-29].

The IR spectral showed that the oils contained fatty acids with a characteristics peak at 1740cm^{-1} due to carbonyl functional group of esterified carbonyl and absorption at 1237.5cm^{-1} due to C-O stretch in esters and appearance of 3004cm^{-1} -OCH_3 group of the methyl ester proved by the GC-FID analysis to contain fatty acid of methyl ester derivatives. The peaks at 2918 and 2851cm^{-1} is due to CH_2 and CH_3 scissoring of the oils and prominent peak at 1461.1cm^{-1} is due to CH_2 and CH_3 bending vibration of the aliphatic groups were observed in all the samples. There is a characteristic peak at 3480cm^{-1}which is due to hydroxyl functional group from residual solvent or water as contaminant present in oil extracted with 70% methanol[10,17,30-32].

The GC-FID analysis of the oily samples as shown in Table No.2 proved that the oils obtained from *Terminalia ivoriensis* seed contained saturated, monounsaturated and polyunsaturated fatty acids. The oily samples showed different degree of chemical composition having more of saturated, followed by polyunsaturated fatty acid, and n-hexane oily extract does not contain monounsaturated fatty acids however, dichloromethane and methanol oily extracts showed low level of monounsaturated fatty acid composition. This is in line with the result of the iodine value which indicated that the oily samples contained high level of saturated fatty acid.

Table No.1: Physicochemical properties of oils

S.No	Properties	N-Hexane Oil	Dichloromethane Oil	Methanol Oil
1	%yield	11.29	10.03	0.954
2	Relative density	0.9104	0.9303	0.9145
3	Refractive index	1.473	1.466	1.4740
4	Acid value	58.34	38.15	52.37
5	Saponification value	115.01	84.15	88.83
6	Iodine value	18.53	15.23	24.75
7	Peroxide value	36.0	26.0	80.0
8	Ester value	56.67	46.0	36.46

Table No.2: Viscosity of the oils

S.No	Rpm (viscosity)	N-Hexane Oil	Dichloromethane Oil	Dichloromethane Oil
1	6	500	760	60
2	12	360	470	42.5
3	30	100	136	12
4	60	24	0.00	18.5

Table No.3: GC- FID Result of Fatty Acids

S.No	Compound	Mol. Formula	N-Hexane	Dichloromethane	Methanol
1	Caproic acid	$C_6H_{12}O_2$	54.35	47.73	27.97
2	1-Heptadecanoic acid	$C_{18}H_{37}O_2$	3.9	21.99	-
3	Linoleic acid methylester	$C_{19}H_{35}O_2$	2.22	7.5	4.22
4	Linolenic acid methylester	$C_{19}H_{33}O_2$	39.47	10.09	40.81
5	Myristic acid methylester	$C_{15}H_{31}O_2$	-	3.19	-
6	Pentadecanoic acid methylester	$C_{16}H_{32}O_2$	-	2.28	3.59
7	Eladic acid methylester	$C_{19}H_{37}O_2$	-	1.97	-
8	Behenic acid methylester	$C_{23}H_{46}O_2$	-	5.16	-
9	Caprylic acid methylester	$C_9H_{19}O_2$	-	-	12.93
10	Caprylic acid methylester	$C_{17}H_{35}O_2$	-	-	4.76
11	Stearic acid methylester	$C_{19}H_{39}O_2$	-	-	5.71

Table No.4: FTIR Analysis Result

S.No	IR	N-Hexane	Dichloromethane	Methanol	Remark
1	1740cm^{-1} carbonyl group	Carbonyl	Carbonyl	Carbonyl	Presence o
2	Carbonyl in esters	C-O stretch	C-O stretch	C-O stretch	C-O stretch
3	96cm^{-1}	C=C	C=C	C=C	unsaturation
4	3004cm^{-1} methoxyl methyl group	OCH_3	OCH_3	OCH_3	Due to
5	2918 and 285cm^{-1} stretch	CH_3, CH_2	CH_3, CH_2	CH_3, CH_2	Due to C-H
6	1461.1cm^{-1} deformation	C-H	C-H	C-H	C-H
7	723.1cm^{-1} to methylene group	CH_2	CH_2	CH_2	Rocking due
8	3480cm^{-1} hydroxyl group.	-	-	-OH	Due to

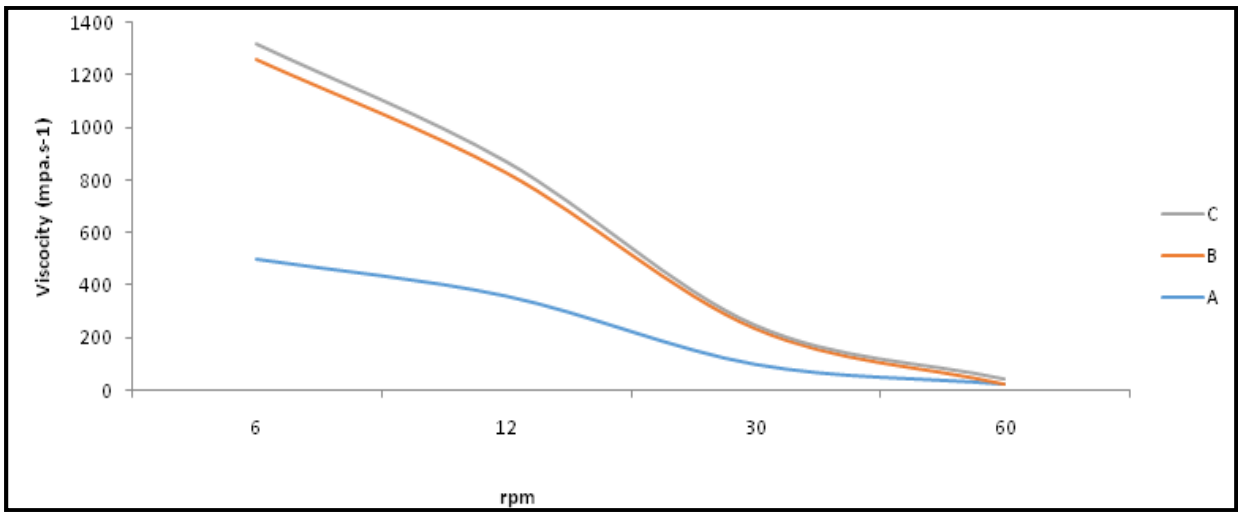

Figure No.1: Graph of viscosity versus Rpm

A = n-hexane extract; B = dichloromethane extract; C = methanol

CONCLUSION

The oils obtained from the seeds of *Terminalia ivoriensis* exhibited different physicochemical properties due to chemical composition of the oils however, the values obtained are below the minimal standard for edible oils, therefore the oils could be used for skincare, emollient, lubricant, vehicle for topical agents, as an insect repellant in agriculture because of the pungent and irritating odour produced by the oils and as a substitute for wool fat because of similar physicochemical parameters.

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CONFLICT OF INTEREST

We declare that we have no conflict of interest.

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