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Research Paper

Compositional Analysis of Tea (*Camellia sinensis*) Seed Oil and Its Application

***Yahaya L.E [1], Adebowale K.O,[2] Olu-Owolabi B.I[2], Menon A.R.R.[3]**
[1]Cocoa Research Institute of Nigeria, Ibadan, NIGERIA.
[2]Chemistry Department, University of Ibadan, NIGERIA.
[3]National Institute for Interdisciplinary Science and Technology, Trivandrum, INDIA.

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Abstract-*Tea (camellia sinensis) seed oil, TSO was subjected to physical, chemical and instrumental analyses with a view for assessing its composition and technical applications. Physical and chemical properties such as color (lovibond), specific gravity, acid value, iodine index, hydroxyl value, saponification value etc., were studied. Gas chromatographic analyses of the oil indicated that TSO comprises 25% saturated fatty acids (palmitic and stearic) and 75% unsaturated fatty acids (C18:1, C18:2 and C18:3). The iodine value of TSO (74gI_2/100g) suggests that it is non-drying. ^{1}H and ^{13}C NMR spectrometry and Infra red spectroscopy (FTIR) analyses of the oil were also carried out and these further confirm the existence of these fatty acid chains. This study therefore indicates that TSO is a promising oleochemical and can therefore be harnessed for this purpose.*

Key words: Tea seed oil, Hydroxyl value, Gas chromatography, fatty acids, oleochemicals, acid value, etc.

Introduction

Vegetable oils represent important raw materials that are being used in the chemical industry. With the escalating prices of petroleum resources coupled with its finite and non renewable nature, researcher's interest has been rekindled in the development and utilization of alternative renewable feedstock. Vegetable oils are alternate energy source to petrochemicals because they are renewable, biodegradable, less toxic and eco-friendly. [1]

Tea (*camellia sinensis)* seed oil is one of such vegetable oil that has been underutilized and exploited in major part of the world, especially Nigeria. Tea plant originated in south-east Asia, probably in the region incorporating the sources and high valleys of the Brahmaputra, the Irrawaddy, the Salween and the Mekong rivers at the border separating India, China and Burma. In Nigeria, for example, tea cultivation is mostly practiced in the upland of the Mambilla plateau and production figure is enormous thus contributing to the world production figure of 3.6million tones per annum. [2] The implication of this is that the seed resulting from the cultivation of tea is often regarded as byproduct. Oil from tea seed could thus be harnessed to serve useful purpose such as oleochemical or chemical intermediate. Most industries in Nigeria that depends on vegetable oils have these raw materials such as olive oil, linseed oil, soybean oil etc sourced by importation and this is rather expensive and uneconomical.

Generally, vegetable oils are well known for both their edible and technical applications. [3, 4, 5]The need to reduce the competitive demand for these oils requires looking inward for other sources. To the best of our knowledge, a comprehensive characterization of tea seed oil using modern instrumental analyses has not been documented. It therefore becomes imperative that the would-be renewable resources be assayed to give insight about its potential use. The study therefore reports the physical, chemical and instrumental analyses of tea seed oil.

Material and methods

Tea seeds used for this study were obtained from the Mambilla station of the Cocoa Research Institute of Nigeria. Laboratory grade chemicals such as hydrochloric acid, potassium hydroxide, potassium iodate, sodium thiosulphate, starch, iodine monochloride, potassium iodide etc., employed for the characterization of the oil were obtained from sd fine chemicals, Mumbai, India. The oil content was determined according to the method of AACC 30-25 [6]. The seeds were thoroughly dried to low moisture content and the oil was expressed using soxhlet extractor and n- hexane as the extracting solvent. In this case, known weight of the finely ground tea seed was introduced into a thimble in a soxhlet apparatus and extraction was carried out for 8 hours under heat supply. The solvent was then removed using rotor vapor and extraction were performed in replicate the oil yield was calculated based on weight difference as follow:

Oil yield (%) = weight of oil/weight of sample × 100

Physicochemical characteristics

The physical and chemical characteristics of tea seed oil (TSO) like color, specific gravity, acid value, iodine value, hydroxyl value, saponification value were determined using IUPAC standard methods [7]. These were performed in replicate.

Nuclear Magnetic Resonance, NMR Spectroscopy

Proton and carbon NMR spectra of TSO were recorded using a 500MHz Bruckner NMR spectrophotometer in $CDCl_3$ containing small amount of trimethylsilane as the internal standard. In a typical experiment, 10-20mg of TSO was dissolved in 1ml $CDCl_3$ in an NMR tube and readings were taken between 0-14ppm for ^{1}H and 0-200ppm for ^{13}C NMR respectively. For ^{13}C NMR experiment, the carbon peaks were taken as 77ppm, while all other peaks were assigned with respect to it.

Fourier Transform Infrared, FTIR spectroscopy

Infra red spectra of TSO were recorded using an impact 400D Nicolet FTIR Spectrophotometer. Thin film of TSO was spread on NaCl plate and the FTIR spectrum was recorded in the range of 4000-400cm^{-1} wavelength.

Gas Chromatography

The fatty acid profile of TSO was determined as fatty acid methyl esters (FAME). The fatty acids composition of the oil was determined by using AOCS official methods Ce 1-62 [8], which constitute a base-catalyzed methylation reaction. The methyl esters were analyzed using a gas chromatograph (model GC-15A,Shimadzu corporation), an FID, a column (3mm×3.3mm i.d, packed with chromosorb WAW 60-80 mesh, percolated with 15% diethyl glycol succinate). The gas chromatograph was operated under the following conditions: nitrogen flow 40mL/min, hydrogen flow 40mL/min, air flow 30mL/min, column temperature 180^{o}C, injector temperature 200^{o}C and FID temperature 220^{o}C. The fatty acids were identified based on their retention times compared with standard fatty acids methyl esters (FAME).Samples were also performed in triplicate and mean values were obtained.

Results and discussion

Physicochemical characteristics of TSO

The physical and chemical characteristics of TSO are shown in Table 1.

TSO is a golden yellow colored liquid; the lovibond index (1.1R, 7.8Y) confirms this (table 1). The implication of this is that tea seed oil could be suitable in application where bright color is a requirement. The specific gravity obtained is in agreement with values for other known vegetable oils [9]. The saponification value obtained is an indication that tea seed oil is suitable for soap production by alkali hydrolysis and this is similar to coconut oil which is employed for similar purpose [10]. Tea seed oil has a low iodine value indicating that it is non-drying.

Gas Chromatography Analyses

The fatty acid profile of tea seed oil is shown in Table 2. It is obvious that TSO comprises essentially approximately 26% saturated fatty acids and 74% unsaturated fatty acids (table 2). The saturated fatty acids include palmitic and stearic acid while the unsaturated fatty acids include oleic, linoleic and linolenic.It is evident that oleic acid (C18:1), a monounsaturated fatty acid is the dominant fatty acid hence the low iodine value. Xu *et al* reported a similar value for hazelnut oil [11]. This therefore implies that tea seed oil may not be suitable for surface coating application; however, it could be useful upon modification.

The amount of various fatty acids in vegetable oils can serve as a guide in identifying the oil, hence the quality and purity of such oil could be guaranteed especially for industrial applications [12].The value obtained from this study could thus give insight to the identification of tea seed oil. Oils containing fully saturated acids tend to solidify in cold environment, thus limiting their low temperature functionalities [13].Tea seed oil with its unique high monoene and low percentage of saturated fatty acids could make it a promising candidate for oleochemical applications such as biodiesel fuel, biopolymers and biolubricants besides its use as edible oil.

Nuclear Magnetic Resonance of TSO

The proton NMR spectrum of TSO is shown in Figure 1 while the signal assignments are listed in table 3.Predominantly observable groups in the spectrum are the protons of the terminal methyl of the fatty acid chain (0.86-0.9 ppm), the allylic methyl group (1.59 ppm), the acyl methylene group (2.29 ppm), the methylene group of glyceryl in the alpha position (4.12-4.16ppm) etc. The carbon NMR spectrum of TSO is shown in figure 2 and the assigned signals in Table 4.

The signal at 173.2ppm is due to the presence of the carbon atom of the carbonyl group; 129.6-130ppm is due to the unsaturated carbon atoms; 62.08-68.85ppm is due to the glyceryl carbon atoms. The ^{13}C and ^{1}H NMR spectra of tea seed oil obtained from this study are analogous to known vegetable oils [14] and the results are useful for the purpose of identifying the quality of *Camellia sinensis* seed oil.

Fourier Transform Infrared Analyses of TSO

Figure 3 shows the FTIR spectrum of tea seed oil and table 5 shows the peaks and their assignments. The spectrum is characterized by five major peaks which of course is similar to other vegetable oil [15].

Potential Inedible Application

From this study, the physical and chemical characterization of tea seed oil has been evaluated. Instrumental analyses have also been carried out. Results indicate that TSO is a suitable feedstock for inedible applications. These potential applications may include surfactant production, biodiesel and lubricants, biopolymers etc. Tea seed oil may therefore serve as alternative to petrochemicals which are limited in supply.

Conclusion

The study was carried out to evaluate the physicochemical and instrumental analyses of tea seed oil. The results indicate that tea seed oil contains essentially monoene and low percentage of saturated fatty acids. GC, NMR and FTIR analyses of the oil also shows that it contains fatty acid chains which can be harnessed for technical applications.

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Table 1: Physicochemical characteristics of TSO

Parameters	Value
Appearance	Golden yellow
Yeild (%)	23
Color (Lovibond)	1.1R, 7.8Y
Specific gravity (29^0C)	0.891
Acid value (mgKOH/g)	23.69
Saponification value (mgKOH/g)	186.5
Hydroxyl value (mgKOH/g)	212.06
Iodine value ($gI_2/100g$)	74.23

***Results are mean standard deviation of triplicate determinations.**

Table 2: Fatty Acids Composition of Tea Seed Oil

Fatty Acid	%
Saturated	
Palmitic (C16.0)	21.88
Stearic (C18.0)	03.76
Total	25.64
Unsaturated	
Oleic (C18.1)	60.05
Linoleiec (C18.2)	13.01
Linolenic C18.3)	00.20
Total	73.26
Others	01.07

***Results are mean standard deviation of triplicate determinations.**

Table 3: ^{1}H NMR Spectrum Signals of Tea Seed Oil

Chemical Shift (ppm)	Assignment
0.86-.090	$-CH_3$ terminal methyl
1.25-1.35	$-CH_2$ saturated aliphatic chain
1.59-1.60	CH_3-C=C-,allylic methyl
1.99-2.05	$-CH_2$-C=C, allyllic methylene
2.29-2.33	$-CH_2$-COOR, acyl methylene
4.12-4.16	$-CH_2$-O-CO-, in alpha position in glyceryl
4.27-4-31	-CH=CH-, in olefinic fatty acid chain
5.33-5.54	-CH-O-CO-, in beta position n glyceryl

Table 4: ^{13}C NMR Spectrum signals of Tea Seed Oil

Chemical Shift (ppm)	Assignment
14.11	Saturated paraffin chain
22.56-22.	Saturated acyl carbon chain
27.1-27.2	allylic carbon
29.11-29.7	Methylene carbon
31.5-34.1	carbon alpha to carbonyl
62.08	C1 or C3 of glyceryl
68.85	C2 of glyceryl
129.6-130	olefinic carbon
173.2	carbonyl carbon

Table 5: FTIR Spectrum Peaks of Tea Seed Oil and Assignments

Peaks (cm^{-1})	Assignment	Remark
3000,2925,2854	C-H stretching	vibration (aliphatic)
1746.78	C=O stretching	vibration (ester)
1463.9	C=C bending	vibration (aliphatic)
1237,1163,1094	C-O-C stretching	vibration (ester)
722	C-H group	vibration (aliphatic)

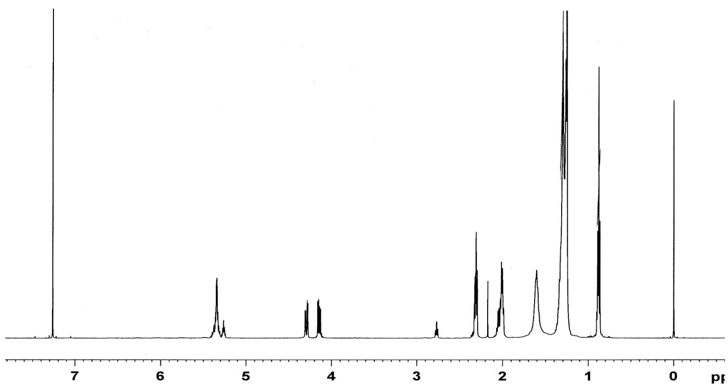

Fig. 1: ^{1}H NMR of Tea Seed Oil

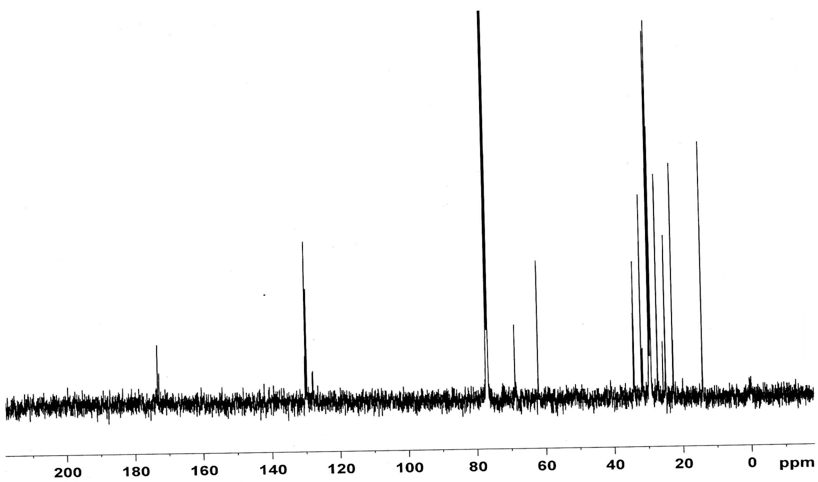

Fig. 2: ^{13}C NMR of Tea Seed Oil

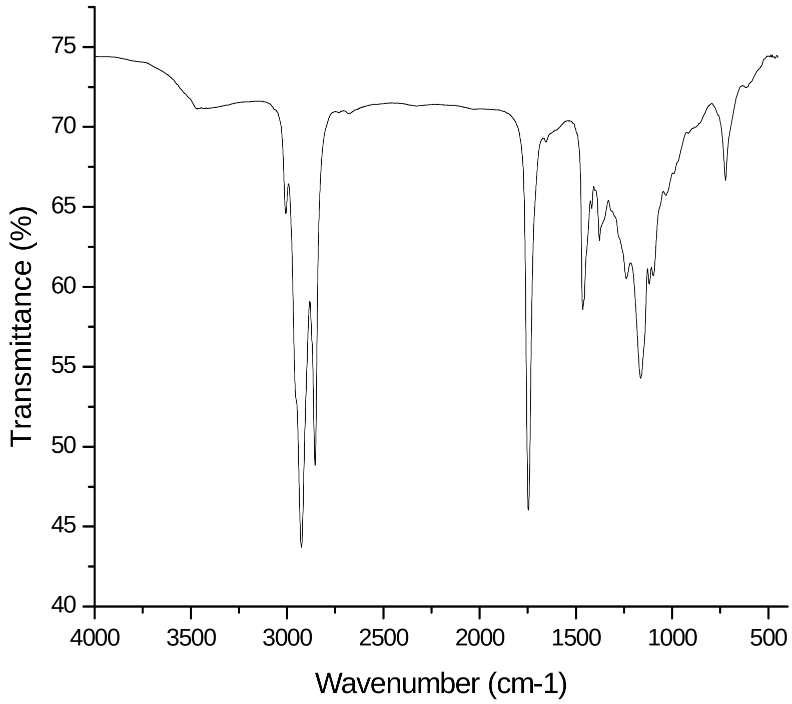

Fig. 3: FTIR Spectrum of Tea (*Camellia sinensis*) Seed Oil