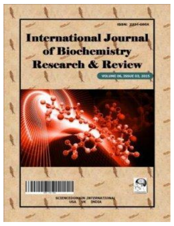

International Journal of Biochemistry Research & Review

26(3): 1-10, 2019; Article no.IJBCRR.47959
ISSN: 2231-086X, NLM ID: 101654445

Fourier Transform Infra-Red (FT-IR) Characterization of Plant Oils from Selected Cultivars Grown in Nigeria

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Authors' contributions

This first report research was carried out in collaboration among all authors. Author AAW initiated the concept of the research, conducted the literature search, collected the plant materials, performed the laboratory work, formulated recommendations and prepared the write-up for publication. Authors LJB, LGH, MNVP, A. A. Odutuga and A. A. Omodolapo identified some issues, formulated recommendations and reviewed the paper. All authors read and approved the final manuscript.

Article Information

DOI: 10.9734/IJBCRR/2019/v26i330098
Editor(s):
(1) Dr. Fidanka Trajkova, Assistant Professor, Faculty of Agriculture, Goce Delcev University of Stip, Macedonia.
(2) Dr. Jose Luis Ramirez Ascheri, Graduate Program in Food Science and Technology, Federal Rural University of Rio de Janeiro (UFRRJ), Brazil.
Reviewers:
(1) Paulo T. C. Freire, Universidade Federal do Ceará, Brazil.
(2) Jack D. Williams, Mercyhurst University, USA.
(3) Bidhan Mahajon, Central Council for Research in Ayurvedic Sciences, Ministry of AYUSH, Govt. of India, India.
Complete Peer review History: http://www.sdiarticle3.com/review-history/47959

Original Research Article

Received 02 February 2019
Accepted 11 April 2019
Published 19 July 2019

ABSTRACT

Background and Objectives: The developments of fourier transform infrared (FT-IR) spectroscopic instrumentation, and application, over the years has made it a powerful analytical tool in the study of oils and fats. This work has explored fourier transform Infra-Red for characterization of plant oils from selected cultivars grown in Nigeria. The selection of these plants oils that flourishes in Nigeria aside their used primarily for nutritional applications, is dependent on the fatty acid (FA) composition of triacylglycerol (TAG which make them potential for bioenergy and biofiuel production considering

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the explosion of energy demand through alternative energy sources. They are also needed for the preparations of chemical feedstocks, biopolymer and composites, skin care products. Oils from these plants can provide renewable sources of high-value fatty acids for both the chemical and health-related industries. We report here several areas where these plant oils can have a significant impact on the emerging bioeconomy.

Materials and Methods: The samples were each placed in contact with KBr disc and FT-IR spectra were collected in frequency 4500-400 cm-1 by coadding 32 scans and at resolution of 4 cm-1. All spectra were rationed against a background spectrum. In each scan, a new reference background spectrum was detected.

Results: The spectra of oils of the present investigation revealed the following bands 1522, 1449.55, 1364.68, 1444.73, 1364.68, 1445.09, 1369.50, 1447.62, 1362.75, 1449.55, 1371.43, 1447.62, 1366.54, 1447.62, 1450.5, 1360.82, 1370.47, 1446.66, 1246.06, 1364.47, 1448.59 for *Adansonia digitata, Ricinus communis*, *Sesamum indicum, Jatropha curcas, Allium cepa*, *Cucumis melo*, *Lannea microcarpa*, *Lagenaria vulgaris* and *Sesamun indicum* seed oil respectively. 1450 cm-1- 1444 is related to bending vibration of CH_2, and CH_3; (cis =C–H bending) and at 1360 - 1370 cm-1 is concerned with the bending vibrations CH_2 groups which showed total unsaturation. The results obtained shows that all the oils are unsaturated because there is no band around 3005- 3009 which is the band that normally determine the index of degree of unsaturation.

Conclusion: The FT- IR spectroscopy proved to be an important technique for identification, analysis, determination of degree of saturation of fatty acids in oils suitable for industrial applications.

Keywords: Fourier Transform Infra-Red; plant oils; fatty acids; industrial; bioeconomy.

1. INTRODUCTION

Plant wealth ranging from seeds powder, fats and oils, seed cakes etc. have played a significant role in improving the quality of human life for centuries and have served as valuable components of foods, cosmetics, pharmaceuticals and biofuel among others. The selected seed oils for analysis are those from indigenous plants [1]. In Nigeria, castor (*Ricinus communis*) is grown in the northern and middle belts where the weather is favourable [2]. It is a potential multi-purpose environmental crop [3]. Physic nut (*Jatropha curcas*) is a potential biofuel crop. The seed oil can be used as a feed stock for biodiesel. Alternatively, Jatropha oil is used in soap, glue or dye industry [4] Baobab (*Adansonia digitata*) is a multi-purpose tree species native to Africa [5] The seeds have a very high oil content useful for soap production [6] *Lagenaria siceraria* or *Lagenaria Vulgaris* and its fruits are widely cultivated in Nigeria from the savannah region of the North to the forest areas of the South. It belongs to the family Cucurbitaceae with the common name of bottle gourd, calabash gourd, etc [7]. The seed oil contain various phytochemical constituents like tannins, saponins, alkaloids, steroids and terpenoids [8]. Polyphenols and antioxidant activity of seed oils of bottle gourd cultivars was reported [9]. *Sesamum indicum* is an annual plant of pedaliaceae family [10]. Nigeria ranks second in the world for production and export of sesame seed [11]. The oil is also useful in the industrial preparation of perfumery, cosmetics (skin conditioning agents and moisturizers, hair preparations, bath oils, hand products and make-up), pharmaceuticals (vehicle for drug delivery), insecticides and paints and varnishes. Sesame seed has higher oil content (around 50%) than most of the known oil seeds [12]. Physico-Chemical, GC-MS analysis and cold saponification of onion (*Allium cepa*) seed oil was reported. The seed oil has potential in the production of soap, perfumery and Pharmaceuticals [13]. Physico-chemical, GC-MS analysis and cold saponification of canary melon (*Cucumis melo*) seed oil was first reported. The seed oil also has potential in the production of soap, perfumery and pharmaceuticals [14]. In this work industrial applications of the selected seed oils from FT-IR results were discussed.

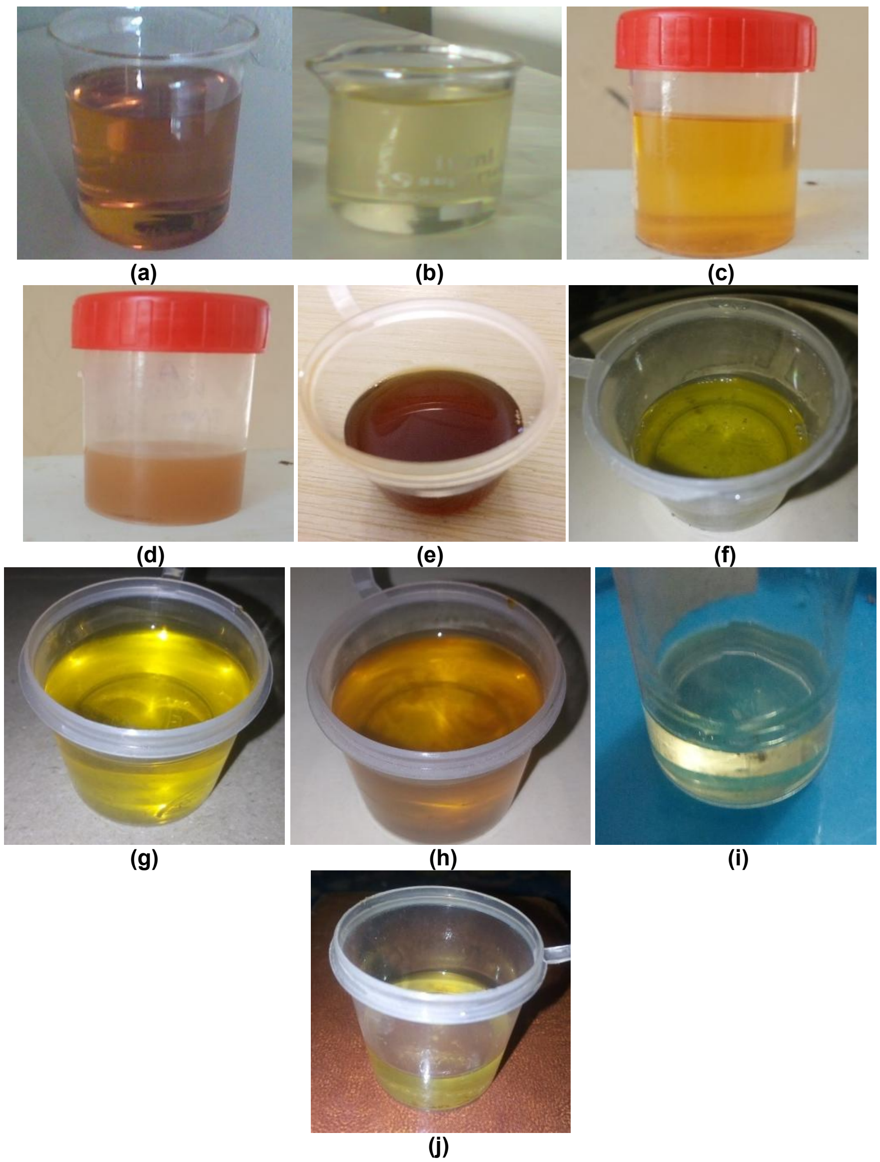
2. MATERIALS AND METHODS

2.1 Oil Extraction Procedure

The hexane extract was obtained by complete extraction using the Soxhlet extractor (GG-17, SHUNIU). The 50 g of each powdered sample was put into a porous thimble and placed in a Soxhlet extractor, using 150 cm3 of n-hexane (with boiling point of 40-60°C) as extracting solvent for 6 hours repeatedly until required quantity was obtained. The oils (Figs. a-i) were obtained after evaporation using Water bath at 70°C to remove the excess solvent from the extracted oil. The oil was then stored in refrigerator prior to FT-IR analysis.

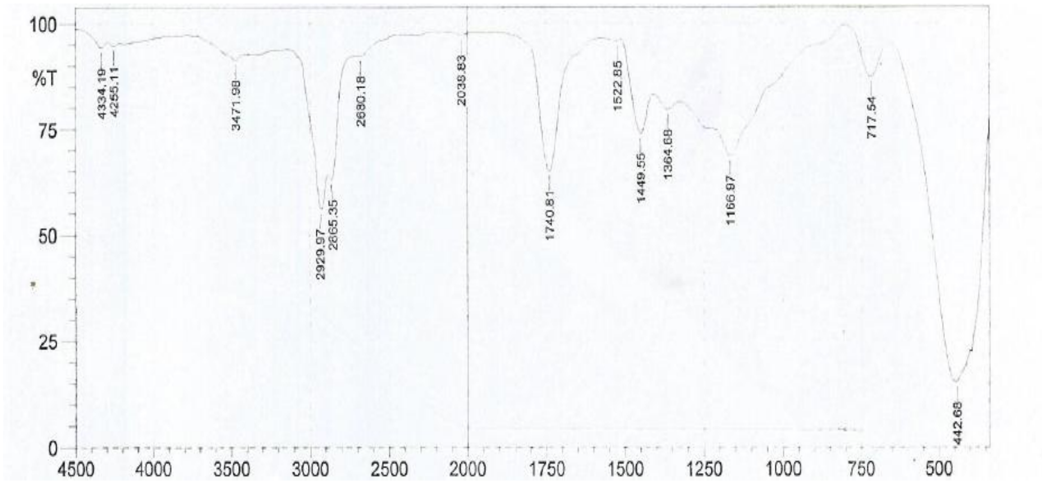
2.2 FTIR Spectrometric Analysis

Fourier Transform Infrared Spectrometer Nicolet 8400S equipped with a detector of deuterated triglycine sulphate (DTGS) and connected to software of OMNIC operating system (Version 7.0 Thermo Nicolet) was used to obtain FT-IR spectra of samples. The samples were each placed in contact with KBr disc and FT-IR spectra were collected in frequency 4500-400 cm-1 by coadding 32 scans and at resolution of 4 cm-1. All spectra were rationed against a background spectrum. In each scan, a new reference background spectrum was detected. These spectra were recorded as absorbance values at each data point in triplicate. The analysis was carried out at NARICT, Zaria, Nigeria.

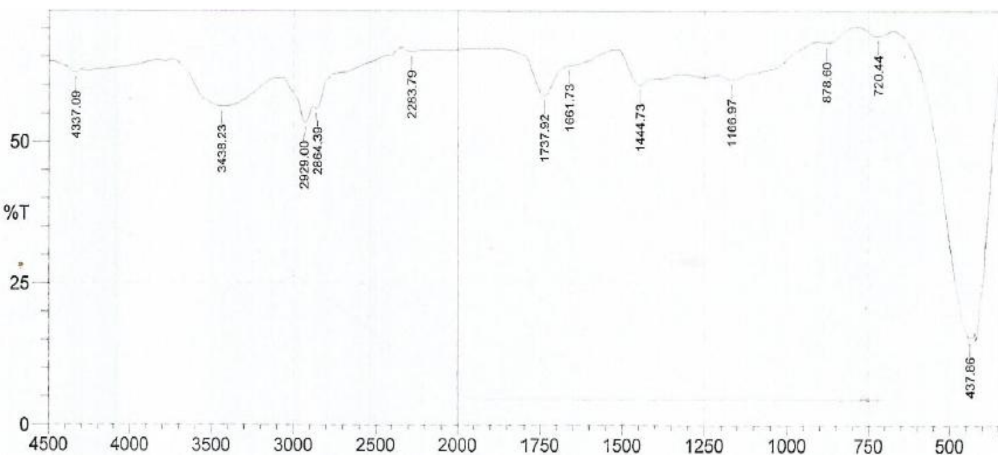


Figs. a-i. (a-b) *Ricinus communis* bean and wild *Ricinus communis* seed oil (c) *Adannsonia digitata* seed oil (d) *Lagenaria vulgaris* seed oil (e) *Lannea microcarpa* seed oil (f) *Allium cepa*, seed oil (g-h) Brown *Sesamum indicum* and white *Sesamum indicum* seeds oil (i) *Cucumis melo* seed oil (j) *Jatropha curcas* seed oil

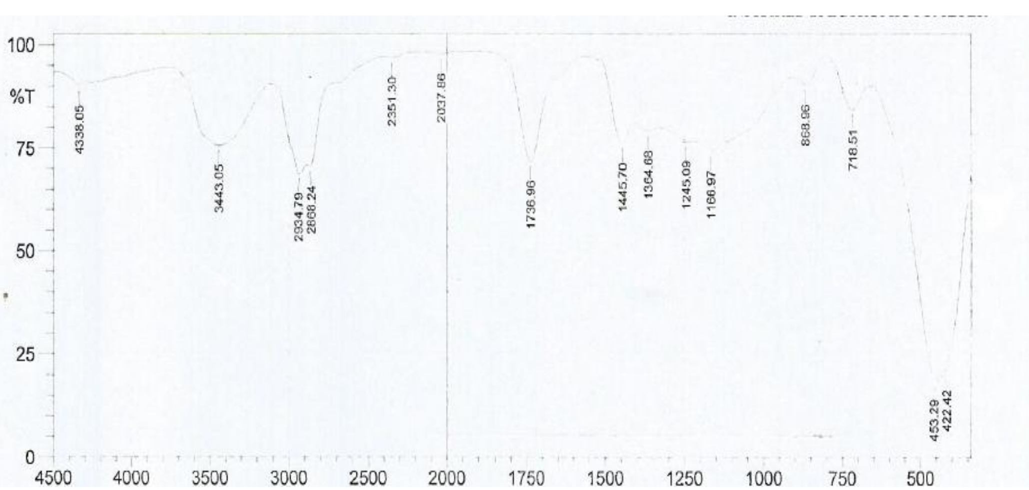
3. RESULTS



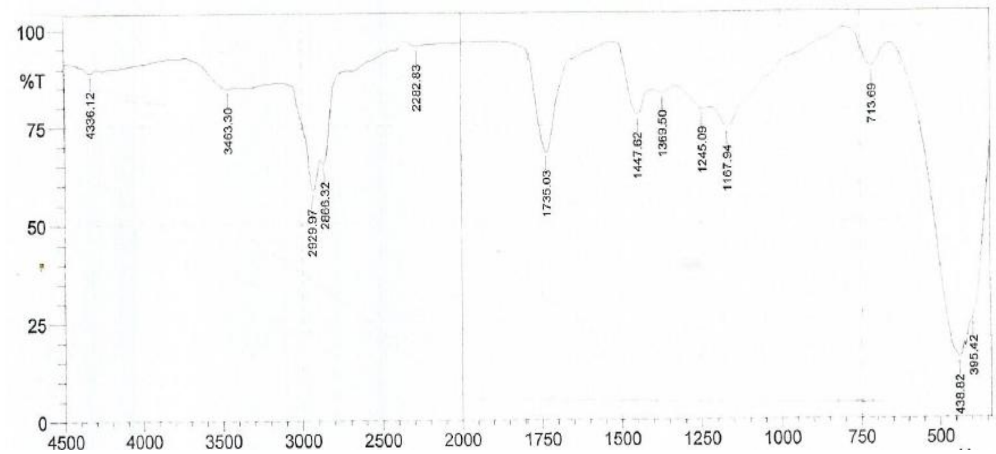
Adansonia digitata **seed oil**



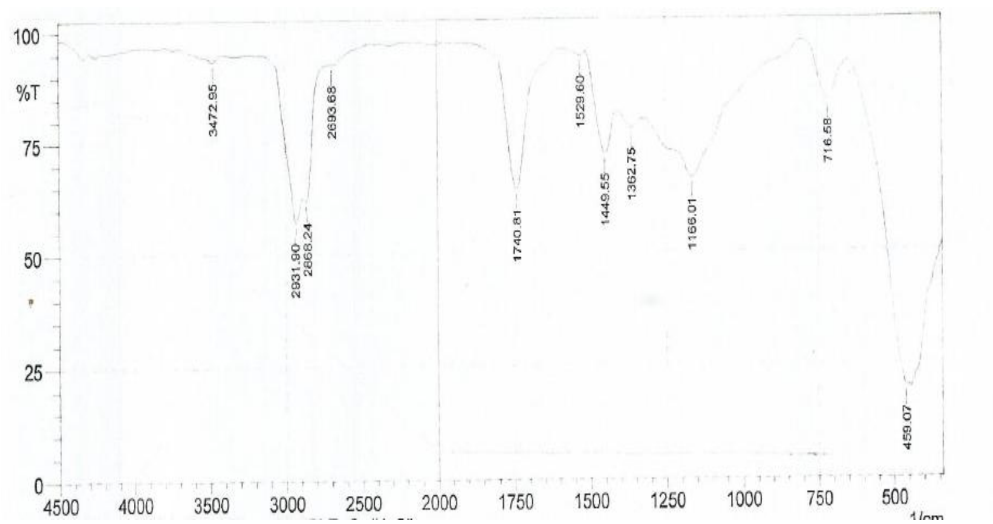
Wild castor (*Ricinus communis)* seed oil



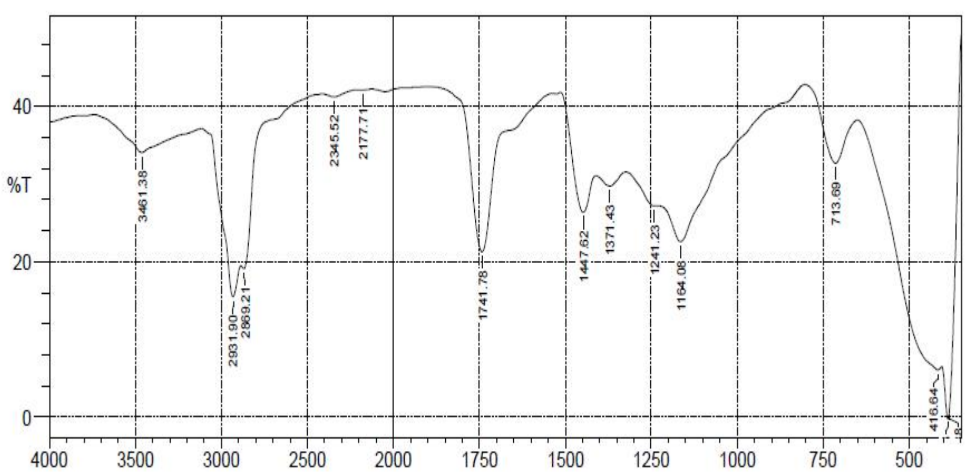
Castor (*Ricinus communis*) bean seed oil



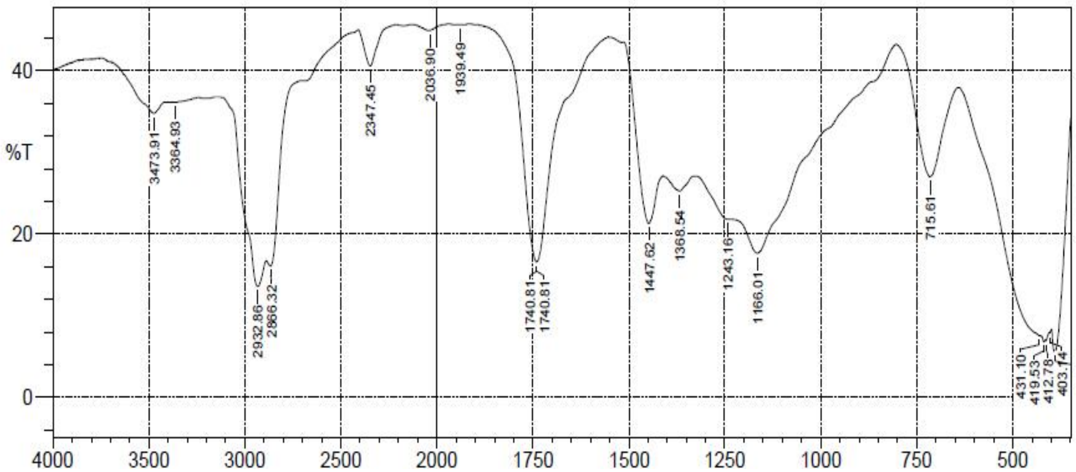
Brown sesame (*Sesamum indicum)* seed oil



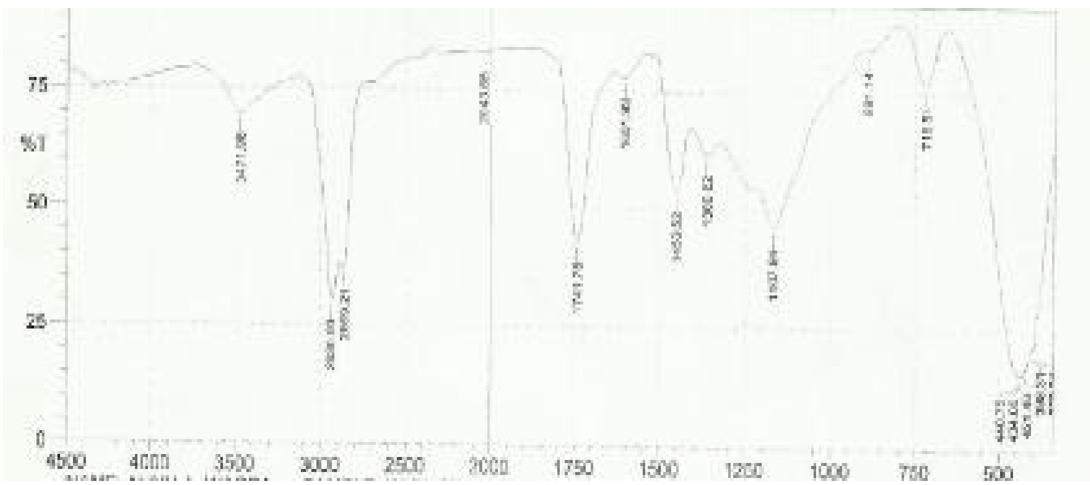
***Jatropha curcas* seed oil**



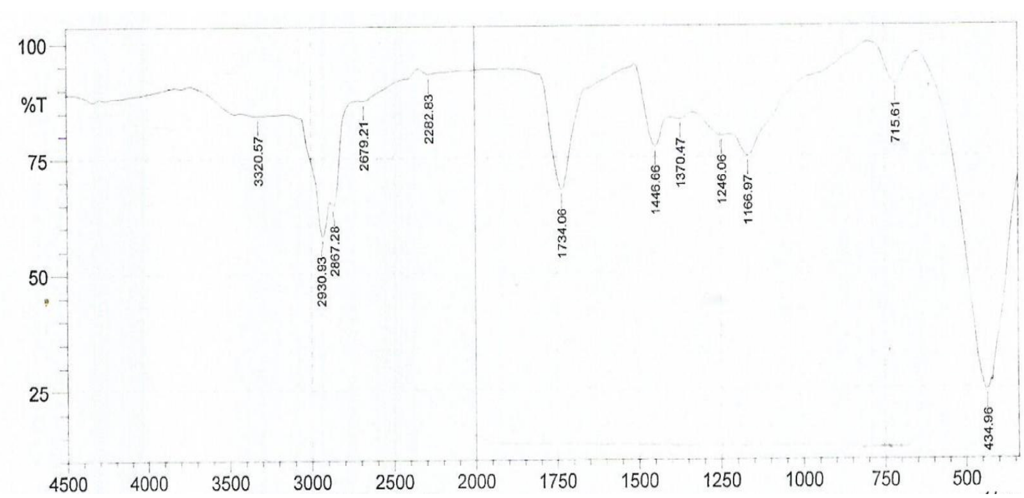
***Allium cepa* seed oil**



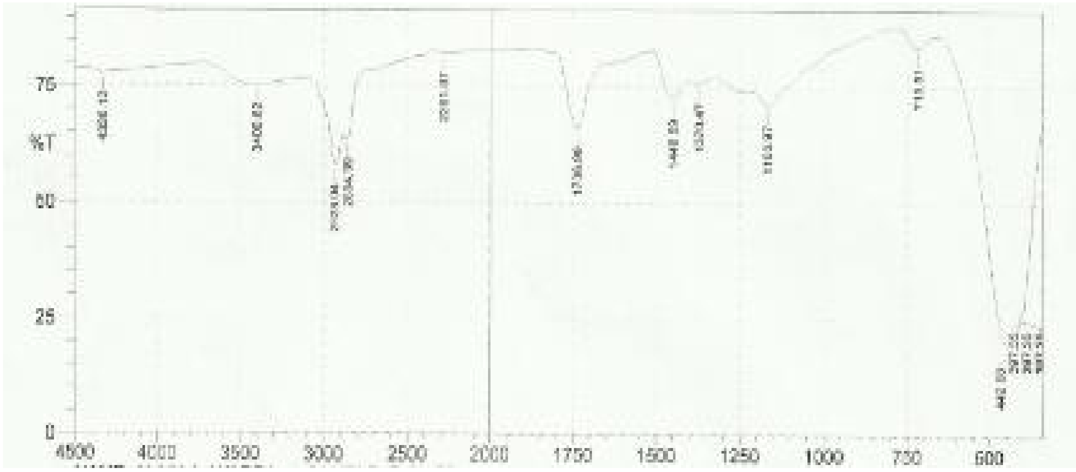
***Cucumis melo* seed oil**



***Lannea microcarpa* seed oil**



***Lagenaria vulgaris* seed oil**



White (S*esamum indicum*) sesame seed oil

FT-IR Spectra of the selected seed oils analysed

Table 1. Some common illegitimate absorption bands in infrared spectrum

Wave no (Cm-1)	Compound	Source
3700-3500	H_2O	Any Source
3450	H_2O	Hydrogen bonding in water,usually in KBr Disc
2000-1430		Atmosphere
2350	CO_2	Atmospheric absorption
1640	H_2	Water of crystallization
1270	$SiCH_3$	Silicone grease
1000	SiO	Glass
667	CO_2	Atmosphere

4. DISCUSSION

FT-IR spectroscopy is a rapid, on-destructive technique with less sample preparation. It allows the qualitative determination of functional group of organic compounds as the characteristic vibrational mode of each molecular group causes the appearance of bands in the infrared spectrum at a specific frequency [15] as the intensities of the bands in the spectrum are proportional to concentration (i.e. Beer's law is obeyed). Mid-infrared spectra have been used to characterize edible oils and fats, because they distinguish the intensity and the exact frequency at which the maximum absorbance of the bands appears, in accordance to the form and composition of the sample. The region used by the material scientists is the mid infra-red region extending from 4000 cm-1 to 200 cm-1.The region beyond 200 cm-1 is called the Far Infra-red region This region is concerned with low frequency vibrations and some molecular rotations. In infra-red spectrum, some spurious bands may be found due to various factors. A list of such bands is presented in Table 1.

As in Table 2, Region 1 (4500 cm^{-1} – 4259 cm^{-1}) shows band for Castor oil, *Adansonia digitata*,, Wild castor seed oil, Castor bean seed oil, Brown sesame seed oil and white sesame seed oil around this region but does not reveal any bands for Jatropha, Allium, Sesame cucumis, lannea and lagenaria This band is not really useful in identification of oil it show aliphatic C-H bond (RSC, 1992. 3471.98 cm^{-1}-3220.58 cm^{-1} was observed for all the oils which indicates the presence of stretching vibration of bonded and non-bonded –O–H groups (R.S.C, 2016), double bond's stretching and deformation/ bending of other bonds (Vlachos, et al. 2006) the band near 3008 cm^{-1} assigned to the C-H stretching vibration of the cis – double bond (=CH) is missing in all the oils. This band at 3008 cm^{-1}-3005 cm^{-1} can be the index of degree of unsaturation of edible oils and also used for their characterization.

Table 2. IR Region of the seed oils

Region Band	Characteristics wave number	Remark
I4500-3000	4334.19, 4265.11, 4337.09, 4336.12, 4259.93, 4335.16, 4336.12, 3471.98, 3438.23, 3443.05,	Aliphatic C-H bond
	3403.30, 3472.95, 3461.38, 3473.91, 3471.56, 3475.89, 3220.57, 3400.62, 3473.91, 3471.98, 3320.57, 3400.62	Stretching vibration of bonded and non-bonded –O–H groups
11 2900-2800	2929.97, 2865.35, 2929.00	
	2929.00, 2864.39, 2934.79, 2868.24, 2929.97, 2866.32, 2931.90, 2868.24, 2931.90, 2869.21, 2932.86, 2866.32, 2930.98, 2869.21, 2930.93, 2867.21, 2679.21, 2928.04, 2864.39.	CH Stretching Vibration of Cis double bond within unsaturated fatty acyl ester Asymmetric –CH2–, symmetric –CH3 and –CH2– stretching vibrations
III 1800-1600	1740.81, 1661.73, 1737.92, 1736.96, 1735.03, 1740.81, 1631.83, 1741.78, 1740.81, 1741.78, 1601.91, 1734.06, 1736.96	–C=O stretching vibrations (e.g. in the –COOR /–COOH groups of aminoacids),= C – Hstretching vibrations, C – C skeletal vibrations
IV 1500-1300	1522, 1449.55, 1364.68, 1444.73, 1364.68, 1445.09, 1369.50, 1447.62, 1362.75, 1449.55, 1371.43, 1447.62, 1366.54, 1447.62, 1450.5 1360.82, 1370.47, 1446.66, 1246.06, 1370.47, 1448.59.	C–O vibrations (e.g. in the –COOR /aromatic –C=C stretching vibrations), –OH bending vibrations, –C–O–H in-plane bending vibrations,–CH3 out-of-plane bending vibrations, –CH2–wagging and twisting vibrations
V 1300-1230	1166.97, 1166.97, 1166.97, 1167.94, 1166.01, 1164.08, 1166.01, 1243.16, 1167.94, 1166.97, 1166.9	C(O)–O stretching vibrations and –OH in plane vibrations (e.g. in aromatic ethers)
VI 1230-400	717.54, 442.68, 720.44, 720.44, 718.51, 453.29, 713.69, 416.64, 716.58, 459.07, 713.69, 416.64, 715.61, 431.10, 718.51, 440.75, 715.61, 434.96, 715.61, 442.68	C – O stretching vibrations (e.g. in in triacylglycerols), trans = C – Hout - of - plane bending, trans = C – Hout - of - plane bending

Region II (2900 cm^{-1}-2800 cm^{-1}) in this region IR spectrum of oils under study presents bands around 29297 & 2865, 2929 & 2864, 2934.79 & 2868.24, 2929.97 & 2866.32, 2931.90 & 2868.24 and 2931.90 & 2869.21 cm-1, for Castor oil, *Adansonia digitata,* Wild castor oil, Castor bean seed oil, *Sesamum indicum, Jatropha curcas, Allium cepa, Cucumis melo, Lannea microcarpa, Lagenaria Vulgaries* and White sesame seed oil respectively, which are characteristics to symmetrical and asymmetrical stretching vibration of aliphatic CH2 group of triglycerides, those around 2900 (–CH3 asymmetrical stretch). These bands are more significant in vegetable oils.

Region III (1800-1600 cm-1) The bands at 1735 cm-1-1741cm cm-1 and 1631-1661 cm^{-1}present in the spectra are concerned with double bond stretching. The band at 1735 cm-1-1741cm is C=O stretching vibrations e.g. in the –COOR /–COOH, spectral band at around 1631-1661 cm^{-1} was found in all the oils and these corresponds to the double C=C link, C – H stretching vibrations, C – C skeletal vibrations and may be related to the polyunsaturated fatty acids. It is found in spectra of edible and medicinal oil.

Region IV (1500-1300) corresponds to deformation and bending vibrations e.g –C–O–H in-plane bending vibrations,–CH_3 out-of-plane bending vibrations, –CH_2–wagging and twisting vibrations. The spectra of oils of the present investigation reveal the following bands 1522, 1449.55, 1364.68, 1444.73, 1364.68, 1445.09, 1369.50, 1447.62, 1362.75, 1449.55, 1371.43, 1447.62, 1366.54, 1447.62, 1450.5, 1360.82, 1370.47, 1446.66, 1246.06, 1364.47, 1448.59 for *Adansonia digitata,* Wild castor oil, Castor bean seed oil, *Sesamum indicum, Jatropha curcas, Allium cepa*, *Cucumis melo*, *Lannea microcarpa*, *Lagenaria vulgaris* and White sesame seed oil respectively. 1450 cm-1-1444 is related to bending vibration of CH_2, and CH3; (cis =C–H bending) and at 1360 - 1370 cm-1 is concerned with the bending vibrations CH2 groups. These bandscan be used to determine total unsaturation. Region V (1300- 1230) corresponds to C (O)–O stretching vibrations and –OH in plane vibrations (e.g. in aromatic ethers) which is found in all the oils.

Region VI (1230-400) bands were found near 750-400 cm−1 for all the oils indicating C – O stretching vibration (e.g. in triacylglycerol), (cis –CH=CH– bending out of plane) and CH_2 rocking. The results obtained shows that all the oils are unsaturated because there is no band around 3005- 3009 which is the band that normally determine the index of degree of unsaturation and Also that IR spectroscopy is an important technique for identification, analysis, determination of degree of saturation of fatty acids in Oils [16].

Unsaturated fatty acids (UFAs), as the constituent of lipids, can be oxidized to produce mono- and dicarboxylic acids which are valuable materials in different industries [17] as such theses plant oils find various industrial and technology applications in coatings and polymers, printing inks, lubricants, cosmetics/pharmaceuticals, leather processing, surfactants, solvents, hydraulic fluids, pesticide/herbicide adjuvants, glycerin (glycerol), and as fuels. Many lipid-derived products are meant to replace existing petroleum-based products, such as plastics and fuels [18].

5. CONCLUSION

In the present work we have investigated the FT-IR spectra of seed oils from *Adansonia digitata, Ricinus communis*, *Sesamum indicum, Jatropha curcas, Allium cepa*, *Cucumis melo*, *Lannea microcarpa*, *Lagenaria vulgaris* and *Sesamun indicum*. This study allow us to characterize the vibrations of the main constituents of the oils, improving the knowledge about important natural substances.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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Peer-review history:
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