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3 **Fourier Transform Infra-Red (FT-IR) Characterization of Plant oils from**
4 **Selected Cultivars Grown in Nigeria**
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6

7 **Abstract**

8 **Background and Objectives:** The developments of Fourier transform infrared (FT-IR)
9 spectroscopic instrumentation, and application, over the years has made it a powerful
10 analytical tool in the study of oils and fats. This work has explored Fourier Transform Infra-
11 Red for Characterization of Plant oils from Selected Cultivars Grown in Nigeria. The
12 selection of these plants oils that flourishes in Nigeria aside their used primarily for
13 nutritional applications, is dependent on the fatty acid (FA) composition of triacylglycerol
14 (TAG which make them potential for bioenergy and biofuel production considering the
15 explosion of energy demand through alternative energy sources . They are also needed for
16 the preparations of chemical feedstocks, biopolymer and composites , skin care products .
17 Oils from these plants can provide renewable sources of high-value fatty acids for both the
18 chemical and health-related industries. We report here several areas where these plant oils
19 can have a significant impact on the emerging bioeconomy. **Materials and Methods:** The
20 samples were each placed in contact with KBr disc and FT-IR spectra were collected in
21 frequency 4500-400cm⁻¹ by coadding 32 scans and at resolution of 4cm⁻¹. All spectra were
22 rationed against a background spectrum. In each scan, a new reference background spectrum
23 was detected. **Results:** The spectra of oils of the present investigation revealed the following
24 bands 1522,
25 1449.55,1364.68,1444.73,1364.68,1445.09,1369.50,1447.62,1362.75,1449.55,1371.43,1447.
26 62,1366.54,1447.62,1450.5, 1360.821370.47, 1446.66,1246.06, 1364.47, 1448.59 for
27 *Adansonia digitata*, *Ricinus communis* , *Sesamum indicum* , *Jatropha curcas* , , *Allium cepa*,
28 *Cucumis melo*, *Lannea microcarpa*, *Lagenaria Vulgaris* and *Sesamun indicum* seed oil
29 respectively.1450 cm⁻¹- 1444 is related to bending vibration of CH₂, and CH₃; (cis =C–H
30 bending) and at 1360 - 1370 cm⁻¹ is concerned with the bending vibrations CH₂ groups
31 which showed total unsaturation. The results obtained shows that all the oils are unsaturated
32 because there is no band around 3005- 3009 which is the band that normally determine the
33 index of degree of unsaturation.

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

37 **Keywords:** Fourier Transform Infra-Red, plant oils, fatty acids, , industrial, bioeconomy
38

39 **INTRODUCTION**
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41 Plant wealth ranging from seeds powder, fats and oils, seed cakes etc. have played a
42 significant role in improving the quality of human life for centuries and have served as

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

68 **Materials and Methods**

69 **Oil Extraction Procedure**

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

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77 **FTIR Spectrometric Analysis**

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

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89 **(a)**



(b)



(c)



(d)



(e)



(f)



(g)



(h)



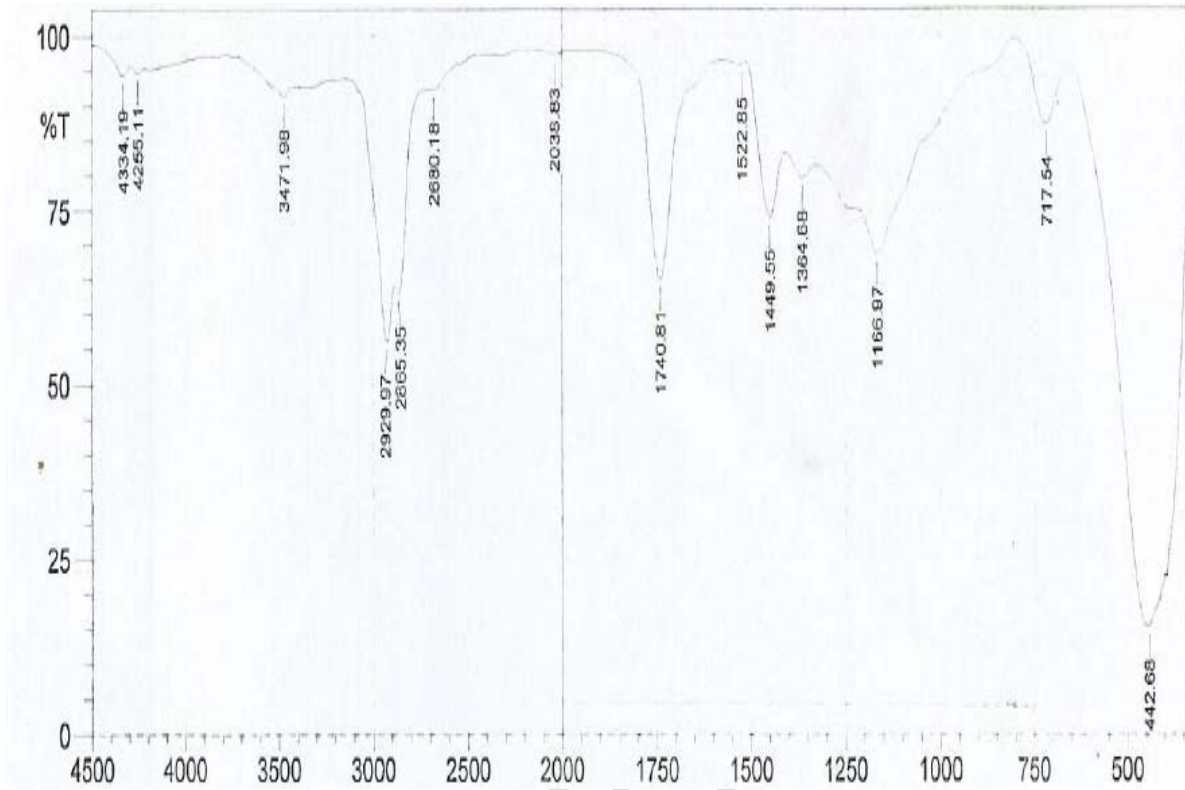
(i)

Figures a-i

(a-b) *Ricinus communis L.* bean and wild castor seeds oil (c) *Adannsonia digitata* seed oil (d) *Lagenaria Vulgaris* seed oil (e) *Lannea microcarpa* seed oil (f) *Allium cepa*, seed oil (g-h) *Sesamum indicum* brown and white seeds oil (h) (i) *Cucumis melo* seed oil

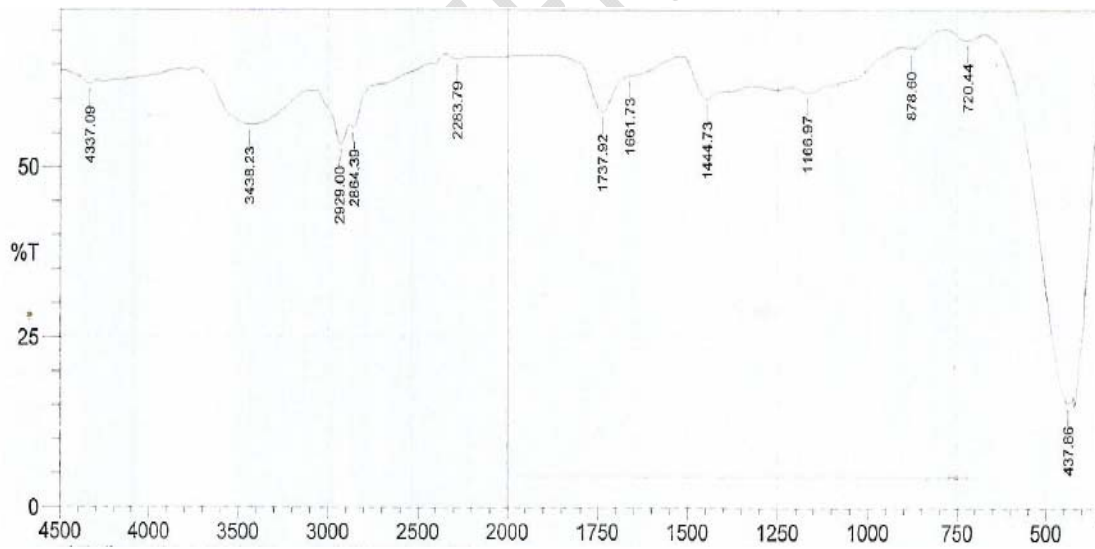
Results and Discussions

RESULTS



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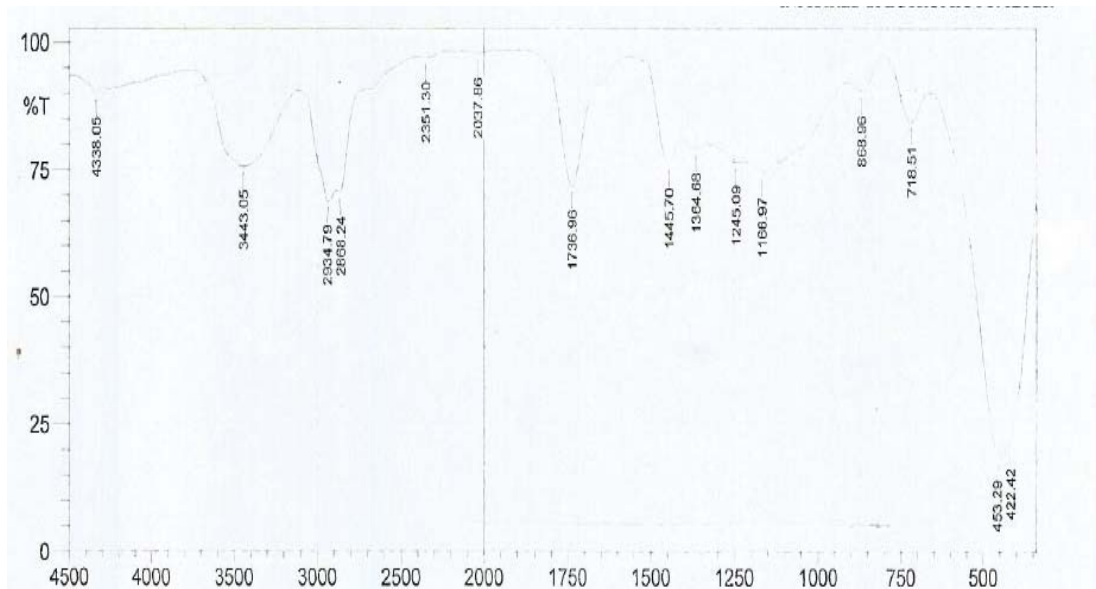
111 *Adansonia digitata* seed oil



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113 Wild castor seed oil

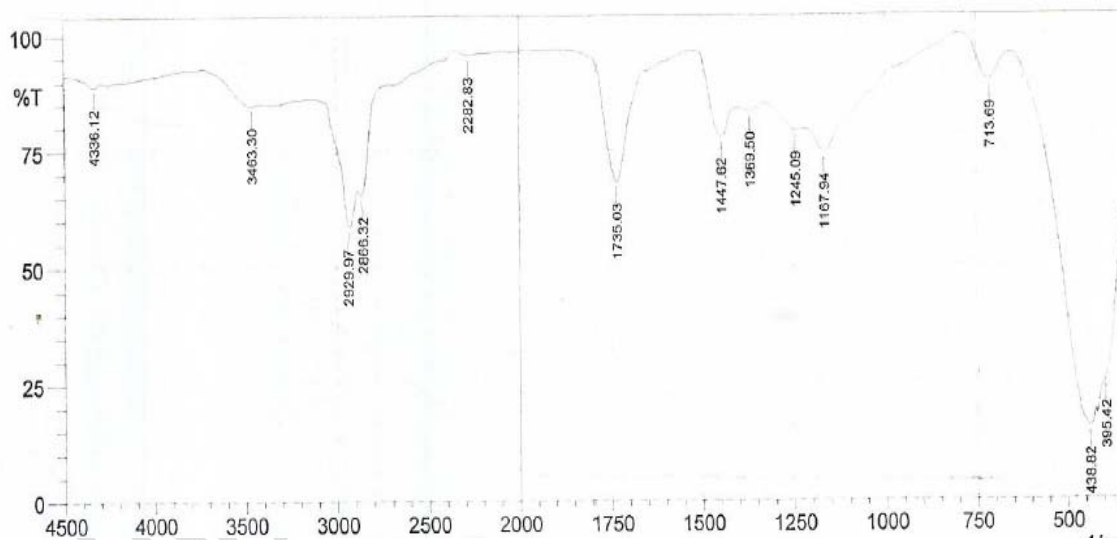
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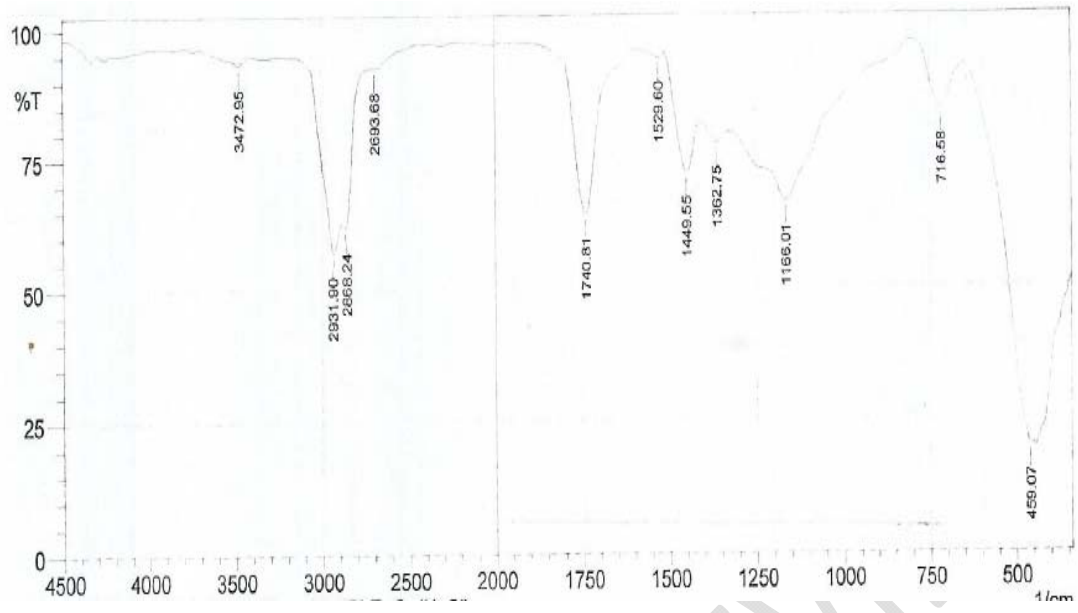
116 **Castor bean seed oil**

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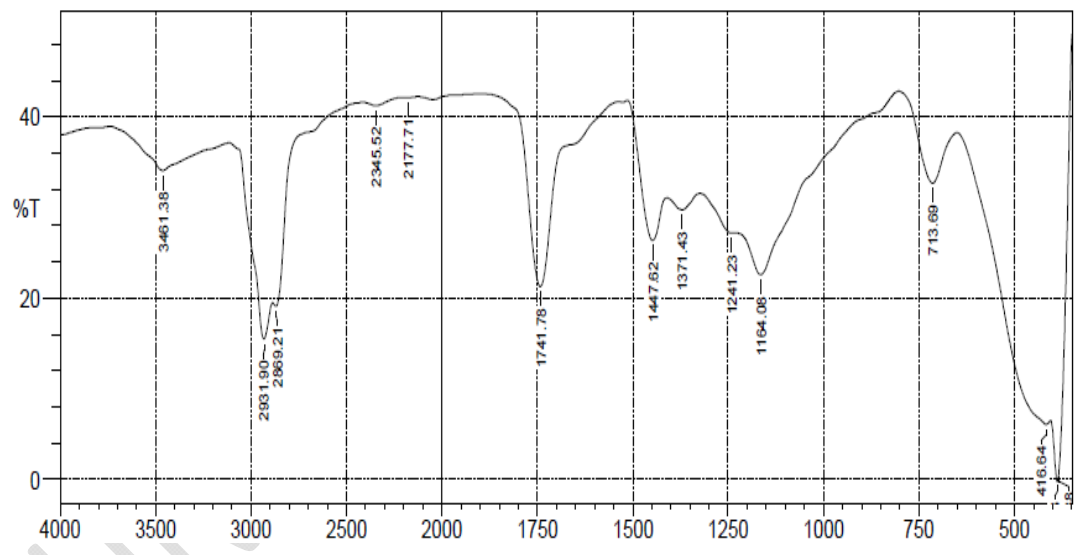
119 **Brown sesame seed oil**



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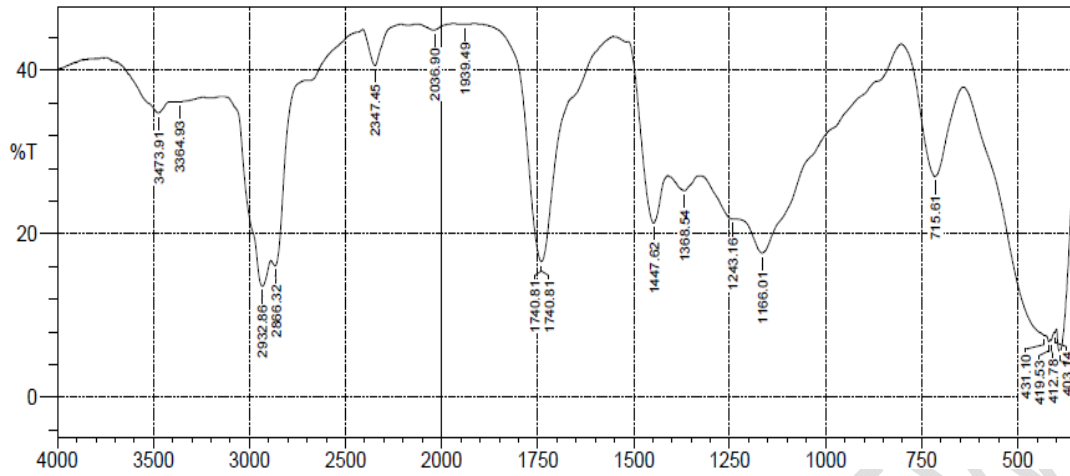
121 *Jatropha curcas* seed oil

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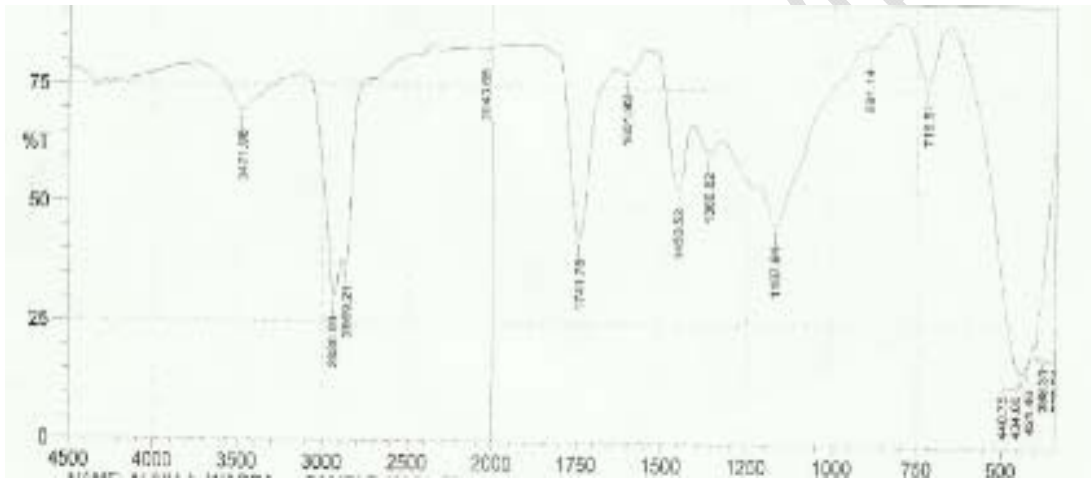
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124 *Allium cepa* seed oil



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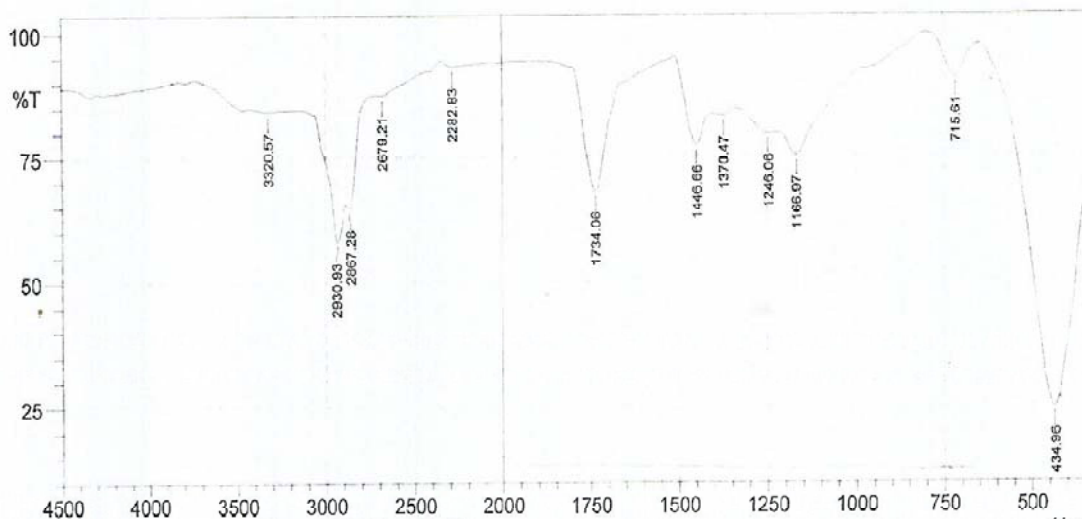
126 **Cucumis melo seed oil**



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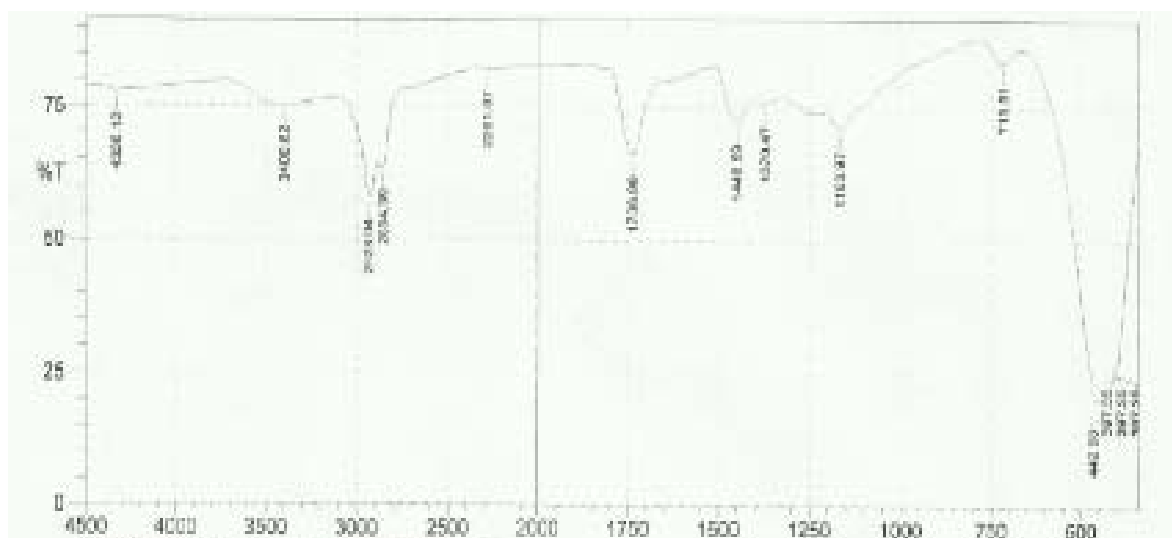
128 ***Lannea microcarpa* seed oil**

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131 ***Lagenaria Vulgaris* seed oil**



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134 **White sesame seed oil**

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137 **FT-IR Spectra of the selected seed oils analysed**

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139 **Table 1: Some common illegitimate absorption bands in infrared spectrum**

Wave no (Cm-1)	Compound	Source
3700-3500	H ₂ O	Any Source
3450	H ₂ O	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

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144 **Table 2: IR Region of the seed oils**

Region	Band	Characteristics wave number	Remark
I	4500-3000	4334.19,4265.11,4337.09, 4336.12, 4259.93,4335.16, 4336.12	Aliphatic C-H bond
		3471.98,3438.23,3443.05, 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
II	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 -CH ₂ -, symmetric -CH ₃ and -CH ₂ - stretching vibrations
III	1800-1600	1740.81,1661.73,1737.92,1736.9 61735.03,1740.81,1631.83,1741. 78,1740.81,1741.78,1601.91734. 06,1736.96	-C=O stretching vibrations (e.g. in the -COOR /-COOH groups of aminoacids),= C - H s t r e t c h i n g vibrations, C - C s k e l e t a l vibrations
IV	1500-1300	1522,1449.55,1364.68,1444.73 1364.68,1445.09,1369.50,1447.6 21362.75,1449.55,1371.43,1447. 62,1366.54,1447.62,1450.5 1360.821370.47, 1446.66,1246.06,1370.47,1448.5	C-O vibrations (e.g. in the -COOR /aromatic -C=C stretching vibrations), -OH bending vibrations, -C-O-H in-plane bending vibrations, -CH ₃ out-of-plane bending vibrations, -CH ₂ -wagging and twisting vibrations
V	1300- 1230	9. 1166.97,1166.97,1166.97,1167.9 4,1166.01,1164.08,1166.01, 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,	C - O s t r e t c h i n g vibrations (e.g. in in triacylglycerols), t r a n s = C - H o u t - o f - p l a n e bending, t r a n s = C - H o u t - o f - p l a n e bending

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146 **Discussion**

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

159 As in table 2, Region 1 ($4500\text{cm}^{-1} - 4259\text{cm}^{-1}$) shows band for Castor oil, *Adansonia*
160 *digitata*, Wild castor seed oil, Castor bean seed oil, Brown sesame seed oil and white sesame
161 seed oil around this region but does not reveal any bands for Jatropha, Allium, Sesame

162 cucumis, lannea and lagenaria This band is not really useful in identification of oil it show
163 aliphatic C-H bond (RSC, 1992. 3471.98cm^{-1} - 3220.58cm^{-1} was observed for all the oils
164 which indicates the presence of stretching vibration of bonded and non-bonded -O-H groups
165 (R.S.C, 2016),double bond's stretching and deformation/ bending of other bonds (Vlachos,*et*
166 *al.*,2006) the band near 3008cm^{-1} assigned to the C-H stretching vibration of the cis - double
167 bond (=CH) is missing in all the oils. This band at 3008cm^{-1} - 3005cm^{-1} can be the index of
168 degree of unsaturation of edible oils and also used for their characterization.

169 Region II (2900cm^{-1} - 2800cm^{-1}) in this region IR spectrum of oils under study presents bands
170 around $2929.7\&2865$, $2929\ & 2864$, $2934.79\&2868.24$, $2929.97\&2866.32$, $2931.90\&2868.24$
171 and $2931.90\ & 2869.21\text{cm}^{-1}$, for Castor oil, *Adansonia digitata*, Wild castor oil, Castor bean
172 seed oil, *Sesamum indicum*, *jatropha curcas*, *Allium cepa*, *Cucumis melo*, *Lansea*
173 *microcarpa*,*Lagenaria Vulgaries* and White sesame seed oil respectively, which are
174 characteristics to symmetrical and asymmetrical stretching vibration of aliphatic CH₂ group
175 of triglycerides,those around 2900 (-CH₃ asymmetrical stretch). These bands are more
176 significant in vegetable oils.

177 Region III (1800 - 1600cm^{-1}) The bands at 1735cm^{-1} - 1741cm^{-1} and 1631 - 1661cm^{-1}
178 present in the spectra are concerned with double bond stretching. The band at 1735cm^{-1} -
179 1741cm^{-1} is C=O stretching vibrations e.g. in the -COOR /-COOH, spectral band at around
180 1631 - 1661cm^{-1} was found in all the oils and these corresponds to the double C=C link,C - H
181 stretching vibrations, C - C skeletal vibrations and may be related to the polyunsaturated
182 fatty acids. It is found in spectra of edible and medicinal oil .

183 Region IV (1500 - 1300) corresponds to deformation and bending vibrations e.g -C-O-H in-
184 plane bending vibrations,-CH₃ out-of-plane bending vibrations, -CH₂-wagging and twisting
185 vibrations. The spectra of oilsof the present investigation reveal the following bands
186 1522 , 1449.55 , 1364.68 , 1444.73 , 1364.68 , 1445.09 , 1369.50 , 1447.62 , 1362.75 , 1449.55 , 1371.43 ,

187 1447.62,1366.54,1447.62,1450.5, 1360.821370.47, 1446.66,1246.06, 1364.47, 1448.59 for
188 *Adansonia digitata*, Wild castor oil, Castor bean seed oil, *Sesamum indicum*, *Jatropha*
189 *curcas*, *Allium cepa*, *Cucumis melo*, *Lannea microcarpa*, *Lagenaria Vulgaris* and White
190 sesame seed oil respectively.1450 cm⁻¹-1444 is related to bending vibration of CH₂, and
191 CH₃; (cis =C–H bending)and at 1360 - 1370 cm⁻¹ is concerned with the bending vibrations
192 CH₂ groups. These bands can be used to determine total unsaturation. Region V (1300- 1230)
193 corresponds to C (O)–O stretching vibrations and –OH in plane vibrations (e.g. in aromatic
194 ethers) which is found in all the oils.

195 Region VI (1230-400) bands were found near 750-400 cm⁻¹ for all the oils indicating C – O
196 stretching vibration (e.g. in triacylglycerol), (cis –CH=CH– bending out of plane) and CH₂
197 rocking. The results obtained shows that all the oils are unsaturated because there is no band
198 around 3005- 3009 which is the band that normally determine the index of degree of
199 unsaturation and Also that IR spectroscopy is an important technique for identification,
200 analysis, determination of degree of saturation of fatty acids in Oils [16].

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

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212 **REFERENCES**

- 213 1. Warra, A.A. 2014. Cosmetic Potential of oil Extracts from Seeds and Nuts Commonly
214 Found in Nigeria. Vol 1. Ahmadu Bello University Press Limited, Zaria, Nigeria.
215
- 216 2. Annongu, A.A., Joseph, J.K.. 2008. Proximate Analysis of Castor Seeds and Cake. J. Appl
217 Sci. Environ Manage. 12(1): 39 – 41
218
- 219 3. Prasad, M.N.V., Kiran, B.R. 2017. *Ricinus communis* L. (Castor bean), a potential multi-
220 purpose environmental crop for improved and integrated phytoremediation. The
221 EuroBiotech J. 1(2):1-16
222
- 223 4. Ahaotu, E.O., Osuji, C.N., Nwabueze, E.U., Ibeh, C.C. 2013. Benefits of *Jatropha*
224 *gossipifolia* in Nigeria. International J. Agric. Biosci, 2(6): 349-355.
225
- 226 5. De Caluwé, E., Kateřina Halamová, K., Van Damme, P. 2010. *Adansonia digitata* L.
227 – A review of traditional uses, phytochemistry and pharmacology. *Afrika focus* (3)1: 11-51
228
- 229 6. Warra, A.A., Sahabi, M.S., Abubakar, A. 2015. Physico- Chemical, GC-MS Analysis
230 and Cold Saponification of Baobab (*Adansonia digitata* L., Malvaceae) seed oil.
231 International J. Innovative Studies Sci. Eng. Technol. 1 (1):21-25
232
- 233 7. Minocha, S. 2015. An overview on *lagenaria siceraria* (bottle gourd). J. Biomedical
234 Pharma. Res. 4(3): 04-10
235
- 236 8. Warra, A.A., Umar, R.A., Atiku, F.A., Nasiru A. and Gafar M.K. 2012. Physical and
237 Phytochemical Characteristics of seed Oils from Selected Cultivars Grown in Northern
238 Nigeria. *Research and Reviews: J. Agric. Allied Sci* 1(1):4-8
239
- 240 9. Essien, E.E. Antia, B.S. and Peter, N.S. 2015. *Lagenaria siceraria* (molina) standley.
241 total polyphenols and antioxidant activity of seed oils of bottle gourd cultivars.
242 World J. Pharma. Res. 4(6): 274-2854,
243
- 244 10. Sharma, E., Shah, T., Khan, F. 2014. A review enlightening genetic divergence in
245 *Sesamum indicum* based on morphological and molecular studies. International J.
246 Agric. Crop Sci 7 (1):1:9
247
- 248 11. Nigerian Export Promotion Council. 2018. Expanding Nigeria's exports of sesame seeds
249 and sheanut/butter through improved SPS capacity building for private and public sector.
250 Accessed 12/12/2018 Available from
251 <https://aflasafe.com/wp-content/uploads/projects/16/Expanding%20Nigerias%20exports.pdf>
- 252 12 Gunstone, F.D. 2004. The chemistry of oils and fats sources, composition, properties
253 and uses. Blackwell Publishing Ltd, UK.. p54.
254
- 255 13 Warra, A.A., Sheshi, F. 2015. Physico-Chemical, GC-MS Analysis and Cold
256 Saponification of Onion (*Allium cepa* L.) Seed Oil. *American J.f Chem. Appl.*
257 (2) 5: 108-113.
258

14. Warra, A.A., Sheshi F., Ayurbami H.S, Abubakar, A .2015. Physico-chemical, GC-MS analysis and cold saponification of canary melon (*Cucumis melo*) seed oil. Trends in Ind. Biotechnol Res. 1(1): 10-17.
15. Vlachos, N., Skopelitis, Y., Psaroudaki, M., Konstantinidou, V., Chatzilazarou, A., Tegou, E. 2006 .Applications of Fourier transform-infrared spectroscopy to edible oils, Elsevier Journal of Analytica Chimica Acta .573–574 ,459–465
16. Nazima, S., Adeel, A. 2013. Infra res spectroscopic studies on Edible Oils and Medicinal oils, International J. Sci. Environ Technol.. 6 (2), 1297-1306

259

260 17. Kerenkan, A.E., Béland, F., and Trong-On Do, T (2016). Chemically catalyzed
261 oxidative cleavage of unsaturated fatty acids and their derivatives into valuable products
262 for industrial applications: a review and perspective. : Catalysis Sci Technol 6, 971

263 18. Warra, A.A (2018). Castor (*Ricinus communis* L.) Plant: Medicinal, Environmental and
264 Industrial Applications. International J. Agric. Environ Sci 3(5): 78-91.

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UNDER PEER REVIEW