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## Comparative Investigation on Fourier Transform Infrared Spectra of Unmodified, Extract and Modified *Cola Lepidota* seed

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**Abstract** This study examined the spectra of the seed of *cola lepidota* in the unmodified form, the acetone extract and the acetone extract modified with oxalic acid. The comparison of the spectra data obtained using Fourier Transform Infrared (FTIR) was carried out to resolve the functional groups present in *cola lepidota* seed. The seeds of *cola lepidota* were air dried, pulverized to particle size of 150 $\mu$ m and stored at 28°C. The acetone extract was obtained using soxhlet extraction method while the acetone extract was reacted with oxalic acid in the modification process. The unmodified, acetone extract and the oxalic acid-modified extract were analyzed using FTIR in the wave number range of 650-4000  $\text{cm}^{-1}$ . Result analyses revealed the likely presence of =C-H, C-O, C-O-C, O=C-O-O, C=C, C=O and O-H functional groups. The characteristic peaks at 748.41 and 840.99  $\text{cm}^{-1}$  for the spectrum of unmodified *cola lepidota* seed reflects bond deformation, while that at 3279.10 and 3387.11  $\text{cm}^{-1}$  for the unmodified and extract respectively which disappeared in oxalic acid-modified *cola lepidota* seed corresponds to the formation of new compound. The formation of oxalic acid-modified *cola lepidota* seed extract was indicated by the variation of percentage transmittance with wave number (103.0-111.5 %, 86.0-111.6 % and 86.0-109.8 % for the unmodified, acetone extract and extract modified with oxalic acid respectively), as well as differences in absorption band intensities in relation to functional groups per molecule.

**Keywords** Fourier transform infrared spectrum, cola lepidota seed, absorption bands, oxalic acid, functional group

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### Introduction

*Cola lepidota* is an indigenous fruit found in West and Central African forests. It is commonly called monkey cola; and belongs to the family of sterculioidae [1-3]. *Cola lepidota* has the following local names in Nigeria: achicha or ochicha by the Ibos, ohihia by the Ikweres, ndiya by the Ibibios, tuwo-biri by the hausas, etc. The nutritive values of *cola lepidota* has been examined, and the findings reported that every 100g of the edible part of *cola lepidota* is composed of the following average contents: moisture content 82.60 g, fibre ash 1.58 g, energy value 55 kcal, fats 0.00 g, carbohydrate 25.8 g, crude protein 1.75 g, vitamin C 11.28 mg, vitamin A 0.25 IU, phosphorus 0.09 mg, potassium 0.19 mg, calcium 0.42 mg and magnesium 0.09 mg [4]. *Cola lepidota* is among the indigenous crops that are underused and long neglected [5]. In order to gain from its existence and prevent it from wasting away, there is need to develop processes that will fully utilize the plant and its residue. *Cola lepidota* has not been commercially cultivated and there is no competition as regards its cultivation and consumption till date. This can be a wealth creating opportunity for the unemployed who are interested in farming by cultivating the plant on commercial basis and incorporating proper irrigation system to make the fruit available all year round. Furthermore, the plant could serve as source of revenue if the government can cultivate the tree on a large scale and export its produce to other nations. This will also help to ensure the



constant supply of the fruit as raw material, as well as the sustenance of the use of *cola lepidota* seed for manufacturing industrial and domestic sorbents, etc.

Investigations in recent years have made conscious efforts to use biological materials, agricultural by-product and waste for remediation of heavy metal ions contaminated systems, because they are less expensive, available, biodegradable, sustainable, simple in operation and non-hazardous to the environment [6-9]. Basically, the following functional groups: hydroxyl, carbonyl, phenyl, carboxyl, etc. found in agricultural waste are responsible for the sorption of heavy metals from waste water. The seed of *cola lepidota* is the agro-waste of interest in the present work.

Studies on the spectra of unmodified *cola lepidota* seed, acetone extract of *cola lepidota* seed and oxalic acid-modified *cola lepidota* seed resolved using Fourier Transform Infrared (FTIR) spectrometer was undertaken. The FTIR spectrometer is an instrument that operates via Beer-Lambert principle, where the amount of electromagnetic radiation absorbed is directly proportional to the concentration usually represented as percentage transmittance at a particular wavelength range. The wavelength of absorption and intensity of a particular compound (usually the unknown) is compared with that of the reference in which the spectrum was also obtained under similar condition [10].

This research is aimed at analyzing the seed of *cola lepidota* in the unmodified form, the acetone extract and the acetone extract modified with oxalic acid using the FTIR spectrometry; and to apply the data in evaluating the functional groups present in the *cola lepidota* seed which could in modified or unmodified state exhibit tendency for metal binding affinity in the remediation of heavy metal laden effluent.

## **Experimental**

### ***Collection and Preparation of sample***

Samples of *cola lepidota* fruit were bought from the mile-1 market in Port Harcourt, Nigeria for the purpose of this study. The fruit of *cola lepidota* was identified by the Herbarium, Department of Plant Science and Biotechnology, University of Port Harcourt, Nigeria. The samples of *cola lepidota* fruit were washed with deionised water, the seeds removed from the edible part, sliced into little pieces and air dried for three weeks. The air dried seed sample was ground with an electric milling machine, sieved to particle size of 150  $\mu\text{m}$  and stored in tightly covered plastic container at 28 °C.

### ***Extraction Procedure***

The process of extraction was performed by placing 2550 g seed (150  $\mu\text{m}$  size) of *cola lepidota* in soxhlet extractor, with acetone as the extracting solvent. The extracted sample was recovered by expelling acetone from the acetone-extract mixture using rotor evaporator.

### ***Modification of extract***

The modification was carried out by weighing 15 g of the extract into distilled water and stirred for 30 mins at 28 °C. 0.9 g sample of oxalic acid was added to the mixture with constant stirring for 10 mins at 50 °C. 25ml iodine solution (5% w/v) was poured into the mixture with continuous stirring for 80 mins at 50 °C, and then cooled to 28 °C. Thereafter, 40 ml of sodium thiosulphate was added to the mixture, and allowed to stand for 24 hours; followed by the formation of organic and aqueous layers. The organic phase was separated, washed with distilled water, dried and stored in a corked bottle at 28 °C. Reagents used are of analytical grade.

### ***Spectroscopic studies***

The characteristic functional groups present in the molecular structure of the unmodified, acetone extract and oxalic acid-modified *cola lepidota* seed samples were resolved using the Fourier Transform Infrared spectrometer (Shimadzu FTIR 8400S) with potassium bromide pressed disc method in the frequency range of 650-4000  $\text{cm}^{-1}$ .

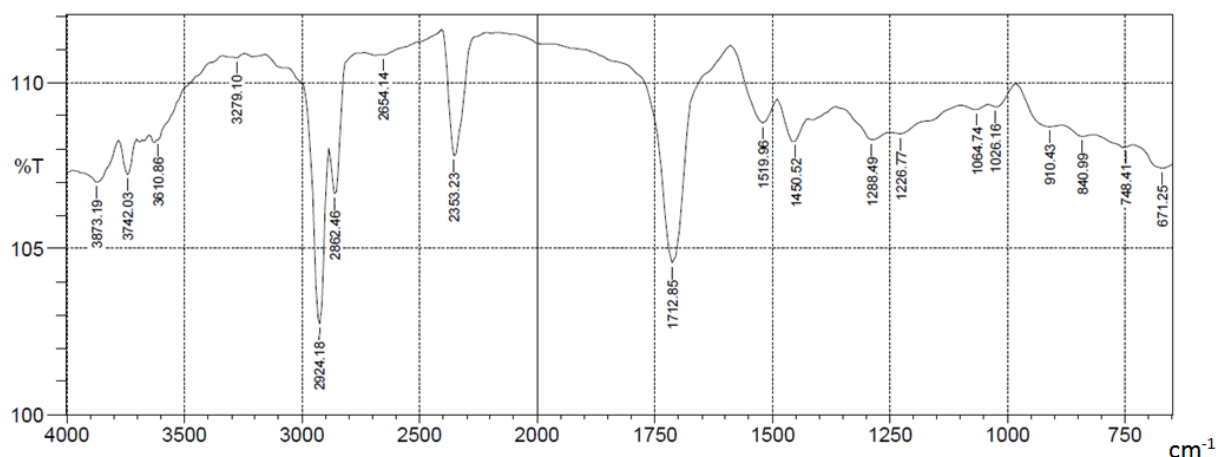
## **Results and Discussion**

The results of the FTIR analyses are reported at the percentage transmittance for wave numbers of 650-4000  $\text{cm}^{-1}$ . The spectra for unmodified, acetone extract and oxalic acid-modified *cola lepidota* seed are depicted in Figs. 1, 2, 3 respectively, while their absorption bands and possible functional group assignments are listed in Table 1 [11-14].



**Table1:** Functional group assignments for the FTIR Absorption bands

Possible functional group assignment	Absorption bands (cm <sup>-1</sup> )		
	Unmodified <i>cola lepidota</i> seed	Extract of <i>cola lepidota</i> seed	Oxalic acid-modified <i>cola lepidota</i> seed
=C-H bend	671.25	709.83	717.54
C-H bend	748.41, 840.99	-	-
=C-H bend	910.43	925.86	933.58
=C-O stretch	1026.16, 1064.74	1080.17	1103.32
=C-O-C &	1226.77, 1288.49	1234.48	1234.48 , 1280.78
O=C-O-O stretch			
C=C stretch	1450.52	1450.52	1450.52
C=C stretch	1519.96	1643.41	1519.96
C=O stretch	1712.85	1705.13	1712.85
O-H stretch	2353.23, 2654.14, 2862.46, 2924.18, 3279,1	2345.52, 2669.57, 2854.74, 2924.18, 3387.11	2353.23, 2669.57, 2854.74, 2924.18
O-H stretch	3610.86, 3742.03, 3873.19	3734.31, 3857.76	3742.03, 3880.91

Figure 1: Percentage transmittance versus wave numbers for unmodified *cola lepidota*

The FTIR spectrum of unmodified *cola lepidota* (Fig.1) displayed a medium peak at 671.25 cm<sup>-1</sup> due to the possible presence of a =C-H bending of cis-alkene (RCH=CHR') in the region of 665-730 cm<sup>-1</sup>. The strong peaks at 748.41 and 840.99 cm<sup>-1</sup> could be ascribed to C-H out-of-plane ring bending (735-770 cm<sup>-1</sup> region) in various substituted benzene rings. Medium absorption band at 910.43 cm<sup>-1</sup> reflects a =C-H bending of alkenes which probably have no geometric isomer. The medium absorption bands at 1026.16 and 1064.74 cm<sup>-1</sup> reveals the likely presence of symmetric ether (Ar-O-R) in the 1020-1075 cm<sup>-1</sup> region. Medium bands at 1226.77 and 1288.49 cm<sup>-1</sup> could be assigned to aromatic ethers (1200-1275 cm<sup>-1</sup> that are asymmetric in nature (=C-O-C). Also, the bands which fall into the region of 1250-1310 cm<sup>-1</sup> is possibly indicating the presence of O=C-O-C stretching of aromatic esters. The strong absorption peak at 1450.52 cm<sup>-1</sup> is assigned to the C=C stretching within the aromatic ring (1450-1600 cm<sup>-1</sup>) involving skeletal vibration. The very strong peak at 1712.85 cm<sup>-1</sup> reveals the presence of C=O stretching of ketone (R'RC=O) and that of six member cyclic compound. The strong absorption peaks at 2353.23, 2654.14, 2862.46, 2924.18 and 3279.1 cm<sup>-1</sup> indicate the presence of O-H stretching of carboxylic acid in the region of 2500-3500 cm<sup>-1</sup>. The spectrum showed strong absorption bands at 3279.10, 3610.86, 3742.03 and 3873.19 cm<sup>-1</sup> which indicate the presence of O-H stretching of alcohol arising from the substitution pattern.



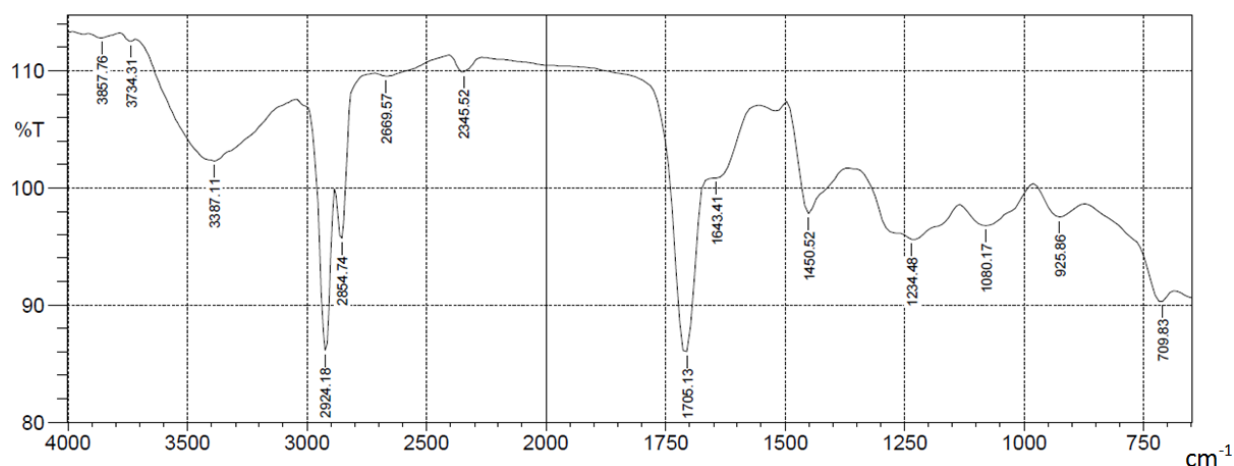


Figure 2: Percentage transmittance versus wave numbers for extract of cola lepidota seed

The FTIR spectrum of the extract of *cola lepidota* seed (Fig. 2) exhibits a medium to strong band at  $709.83\text{cm}^{-1}$  which suggest the presence of cis-alkene ( $\text{RCH}=\text{CHR}^1$ ) in the region of  $665\text{-}730\text{cm}^{-1}$ . The medium to strong absorption band at  $925.86\text{cm}^{-1}$  could be associated with  $=\text{C-H}$  bend of alkenes ( $\text{RCH}=\text{CH}_2$ ) in the  $910\text{-}990\text{cm}^{-1}$  region. It could also be as a result of asymmetrical ring stretching of C-C bond due to the C-O bond contraction in the region of  $810\text{-}950\text{cm}^{-1}$ . Strong band at  $1080.17\text{cm}^{-1}$  corresponds to the C-O stretching of primary alcohol ( $\text{RCH}=\text{CH}_2\text{-OH}$ ) or  $\text{C}=\text{C-CH(R)-OH}$  within the region  $1050\text{-}1085\text{cm}^{-1}$ . The medium to strong absorption peak at  $1234.48\text{cm}^{-1}$  is in the C-O stretching region ( $1050\text{-}1300\text{cm}^{-1}$ ) of alcohols, carboxylic acids and ethers. The sharp peak at  $1450.52\text{cm}^{-1}$  indicates the presence of C=C stretching of aromatic ring in the region of  $1450\text{-}1600\text{cm}^{-1}$ , while the very weak band at  $1643.41\text{cm}^{-1}$  could be ascribed to C=C stretching of alkenes in the  $1640\text{-}1670\text{cm}^{-1}$ . The obvious peak at  $1705.13\text{cm}^{-1}$  may belong to the C=O stretching of aldehyde ( $\text{C}=\text{C-CH=O}$  or  $\text{Ar-CH=O}$ ) in the  $1685\text{-}1710\text{cm}^{-1}$  region. Very strong peaks at  $2345.52$ ,  $2669.57$ ,  $2854.74$  and  $2924.18\text{cm}^{-1}$  could be due to the O-H stretching of carboxylic acid ( $2500\text{-}3500\text{cm}^{-1}$  region). Broad band at  $3387.11$ ,  $3734.31$ , and  $3857.76\text{cm}^{-1}$  suggesting the likelihood of O-H stretching of alcohol, apparently as a result of bond interactions within the molecular structure.

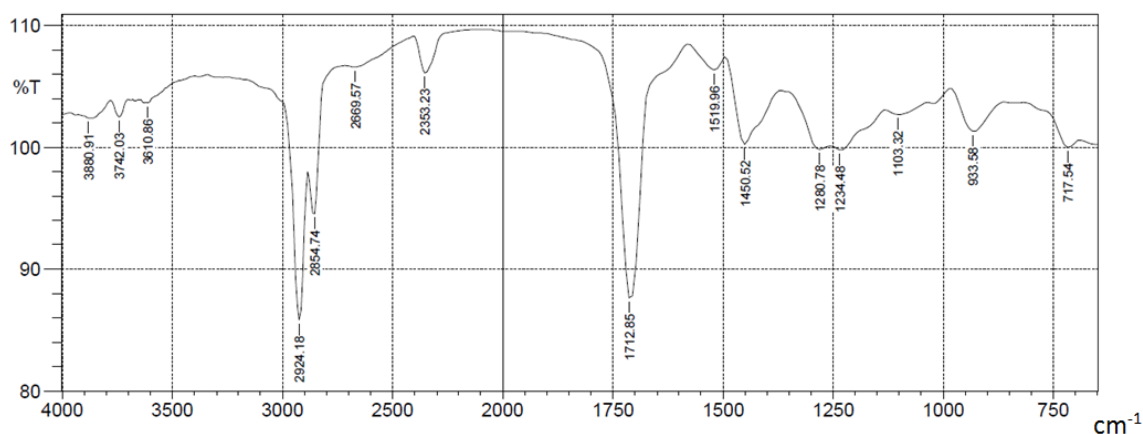


Figure 3: Percentage transmittance versus wave numbers for modified cola lepidota seed

The FTIR spectrum of modified *cola lepidota* (Fig. 3) showed a strong peak at  $717\text{cm}^{-1}$  which reveals the presence of  $=\text{C-H}$  bending of cis-alkenes ( $\text{RCH}=\text{CH R}^2$ ) in the region of  $665\text{-}730\text{cm}^{-1}$ . The medium to strong band at  $933.58\text{cm}^{-1}$  corresponds to  $=\text{C-H}$  bending of alkene ( $\text{RCH}=\text{CH}_2$ ) in the region of  $910\text{-}990\text{cm}^{-1}$ . Absorption band at  $1103.32\text{cm}^{-1}$  could be assigned to the C-O stretching of secondary alcohol  $\text{RR}'\text{CH-OH}$  or  $\text{C}=\text{C-CRR}'\text{-OH}$  found in the region of  $1085\text{-}1125\text{cm}^{-1}$ . The broad band at  $1234$  and  $1280\text{cm}^{-1}$  may be due to the C-O stretching region ( $1050\text{-}1300\text{cm}^{-1}$ ) of alcohols, carboxylic acids and ethers. Strong band at  $1450.52\text{cm}^{-1}$  is attributed to the aromatic ring C=C stretching in the  $1450\text{-}1600\text{cm}^{-1}$  region. The very strong peak at  $1712.85\text{cm}^{-1}$  indicates the C=O stretching of ketone ( $\text{RR}'\text{C}=\text{O}$ ) or six member cyclic ring in the region of  $1710\text{-}1720\text{cm}^{-1}$ .



$\text{cm}^{-1}$ . It could also be due to the C=O stretching of carboxylic acid [R-C(O)-OH] in the region of 1710-1715  $\text{cm}^{-1}$ . The O-H stretching of carboxylic acid (2500-3500  $\text{cm}^{-1}$  region) could be responsible for the strong peaks at 2353.23, 2669.57, 2854.57 and 2924.18  $\text{cm}^{-1}$ . O-H stretching of alcohol at absorption peaks of 3742.03 and 3880.91  $\text{cm}^{-1}$  suggests changes in O-H bonding interactions (Silverstein *et al.*, 1991; Uzoukwu, 2009; Lazar *et al.*, 2014; Ibezim-Ezeani *et al.*, 2010; Ibezim-Ezeani and Akaranta, 2017).

However, some observed differences in the FTIR spectra bands of unmodified, acetone extract and oxalic acid-modified *cola lepidota* seed could be attributed to variations in the structural characteristics, bond behavior, functional group geometry, intra- and intermolecular forces. The obvious peaks at 748.41 and 840.99  $\text{cm}^{-1}$  for the spectrum of unmodified *cola lepidota* seed was non-existent in that of extract and modified forms, suggesting that the bonds bearing the ortho- and para- substituent may have been affected during the extraction process. Another prominent peak at 3279.10 and 3387.11  $\text{cm}^{-1}$  for the unmodified and extract of *cola lepidota* seed was conspicuously missing in the spectrum for oxalic acid-modified *cola lepidota* seed, revealing the formation of a new compound with substituent and bond interaction different from that of extract and unmodified moieties. Again, the percentage transmittance between wave numbers of 650 and 4000  $\text{cm}^{-1}$  for spectra of unmodified (103.0-111.5 %) and acetone extract (86.0-111.6 %) are higher than that of oxalic acid-modified (86.0-109.8 %) *cola lepidota* seed. This is apparently as a result of a new product formed and also that the band intensity is influenced by the number of functional groups per moiety.

### Conclusion

This study has evaluated the FTIR spectra of unmodified, acetone extract and oxalic acid-modified extract *cola lepidota* seed in the wave number region of 650-4000  $\text{cm}^{-1}$ . Spectra analyses indicated the presence of =C-H, C-O, C-O-C, O=C-O-O, C=C, C=O and O-H functional groups. Prominent peaks at 748.41 and 840.99  $\text{cm}^{-1}$  for the spectrum of unmodified *cola lepidota* seed indicate bond forces at play; while that at 3279.10 and 3387.11  $\text{cm}^{-1}$  for the unmodified and extract respectively which is absent in oxalic acid-modified *cola lepidota* seed reveals the presence of a new moiety. The variation of percentage transmittance as a function of wave number appeared between 103.0 and 111.5 %, 86.0 and 111.6 %, and 86.0 and 109.8 % for the unmodified, acetone extract and extract modified with oxalic acid respectively.

Comparison of the data based on structural and bond properties confirmed the successful modification of the *cola lepidota* acetone extract with oxalic acid. The utilization of this seed will bring about an alternative eco-friendly adsorbent. It will also lead to the cultivation of the plant in commercial quantity, provide employment opportunity and serve as potential source of foreign exchange earnings.

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