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# EVALUATION OF SOME EDIBLE WET SEASON CROPS USING FOURIER TRANSFORM INFRARED (FTIR) SPECTROSCOPY AT GALENA MINING AREA OF NAHUTA, ALKALERI LOCAL GOVERNMENT AREA, BAUCHI STATE, NIGERIA

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### Abstract

Fourier Transformation Infra-red spectroscopy analysis investigated the presence of sulphur compounds in wet season edible crops (Beans, Maize, rice, Okro and tomatoes) .stretching bonds of absorbed wavelength intensities showed dissolved sulphur compounds in form of ions from the soil into their shoot system revealed the following range of bands.C-S (800-600 cm<sup>-1</sup>);  $SO_3$  (1260-1150 cm<sup>-1</sup>);  $SO_2$  symmetric (1160-1120 cm<sup>-1</sup>); S=O (11060-1040 cm<sup>-1</sup>)

Keywords: Edible crops, wet season, Sulphur compounds,

## 1. Introduction

Activities of chemical weathering cause Galena as a deposit minerals undergo dissolution of, heavy metals and metalloids are continually released into the environment at various rates and concentrations from natural materials. (Gordon *et al.*, 1999). Major constituents of Galena are Pb and S, the study of FTIR spectroscopy revealed the absorption band of sulphur compounds such as C-S, SO<sub>2</sub>, SO<sub>3</sub> S=O present in soil sediments dissolved form are absorbed by fish and some edible crops grown at the Galena mining area.

FTIR Spectrometers adopted help in organic synthesis, polymer science, petrochemical engineering, pharmaceutical industry and food analysis. In addition, FTIR spectrometers can be used in chromatography, of chemical reactions and the detection of unstable substances can be investigated with such instruments. The range of Infrared region is  $12800 \sim 10 \text{ cm}^{-1}$  and can be divided into near-infrared region ( $12800 \sim 4000 \text{ cm}^{-1}$ ), mid-infrared region ( $4000 \sim 200 \text{ cm}^{-1}$ )

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and far-infrared region (50 ~ 1000 cm<sup>-1</sup>). FTIR Spectroscopy method can used to determine the structures of molecules with characteristic absorption of infrared radiation and most widely used methods to identify the chemical constituents and functional groups of compound structures.(Pavan *et al.*, 2018). Most molecules are infrared active except for several mononuclear diatomic molecules such as  $O_2$ ,  $N_2$  and  $Cl_2$  due to the zero dipole change in the vibration and rotation of these molecules. Infrared absorption spectroscopy is capable of analyzing all gas, liquid and solid samples. The common used region for infrared absorption spectroscopy is between 4000 ~ 400 cm<sup>-1</sup> because the absorption radiation of most organic compounds and inorganic ions is within this region as stated by (Nancy *et al.*, 2014).

## 2.0 Materials and Methods

#### **2.1 Plants Samples Analysis**

Edible plants and vegetable samples collected from different locations were washed with water to remove impurities. Succulent's samples such as Okro and tomatoes was cut and sliced for easily drying while hard grains like rice and maize was chaffed that removed pericarp and the endocarp was then grinded into powder form using mortar and pestle. The samples was dried at constant weight at 90 °c in an oven and quartered to obtain a 100 g of representative fraction for analysis as described by (Titus *et al.*, 2012)

#### **3.0 Sampling and Sample Preparation**

## **3.1 Fourier Transformation Infra-Red Spectroscopy Analysis**

Fine-grained powders of sample was prepared using mechanical vibrators, or mills, or with a mortar and pestle. 0.7 to 2g of samples was ground under acetone or ethanol to avoid damaging crystals or absorbing water during the grinding process. The sample was mixed with an IR carrier such as a mulling agent known as trans missive salt .Petroleum jelly and isopropanol were used which immediately activate a random orientation by polarization that help transmission of IR radiation and this was achieved which turned the mixture into a pellet or by touching the sample through window. Many carrier used was advantage in order to obtain spectra over a wide spectral range without signatures from the carrier. Typically, this approach results in lightly mixing about 100-300 mg of dried KBr solution at temperature  $400^{0C}$  in 6 hours then placing the samples in a pre-heated oven at temperature of 100°C, in 20 minutes under a heat lamp. The material was pressed in a cylindrical die under a vacuum for 2 to 5 minutes duration at 10 000 kg/cm<sup>2</sup> to produce a transparent sample disc. The disc was used to interpreted IR wavelength but must not be cloudy due to water incorporated in the salt carrier and it may not be possible to properly interpret the 3000-3600 cm<sup>-1</sup> region (O-H stretching) and 1610 to 1640 cm<sup>-1</sup> range (H-O-H bending) of the sample. Scattering may occur as surface scattering and volume scattering the radiation enters and exits the sample dependent on the absorption coefficient and particle size. To avoid scattering problems in transmission analysis the powder must have a grain size smaller than the wavelength of the radiation as adopted by (Kings *et al.*, 2007)

### 4.0 Results and Discussion

#### 4.1 FTIR Spectra of Wet Season Beans Samples

Intensities of FTIR on Figures 4.47 showed Br 1 and all the five samples analyzed contain the same saturated compounds of C-N, C-O, C-H and O-H with peak intensities range of 1000-1350cm<sup>-1</sup>, 1050-1150cm<sup>-1</sup>,3000-3100 cm<sup>-1</sup> and 3200– 3400 cm<sup>-1</sup> as compared to (Pavan *et al.*, 2018). All the wet season beans samples from Galena mining area at Nahuta contain the same compounds of unsaturated groups of C=C, C=N and S=O, only as sulphur oxides. However, C-S, SO<sub>2</sub> and SO<sub>3</sub> are absent in the wet season grown beans at Galena mining area this indicated that low or small percolation of some dissolved sulphur compounds are not directly observed by these beans plants roots due to low suction capacity by some plants or deficient of such compounds as reported by (Mario, 2018).



#### 4.2 FTIR Spectra of Wet Season Maize Samples

Figure 2a showed FTIR spectra with of wet season maize Mr1 samples grown at galena mining vicinity showed only one sulphur compounds of C-S having 800-600 cm<sup>-1</sup> as stated by (Valeriy *et al.*, 2016). Compounds of C-N, C-O, C-H and N-H with peak intensities of 1000-1350cm<sup>-1</sup>, 1050-1150cm<sup>-1</sup>, 3000-3100 cm<sup>-1</sup> and with intensity range of 3100-3500 cm<sup>-1</sup> as reported by (Pavan *et al.*, 2016) Unsaturated compounds are absent in Mr1 sample of wet season maize.



Figure 1a FTIR Spectra of Mr1 Wet season Maize Sample

Figures 2b showed FTIR intensities bands of wet season maize Mr2 sample grown within the vicinity of Galena mining area which showed only one sulphur compound of  $SO_3$  symmetric stretching with values of 1160-1150 cm<sup>-1</sup> as stated by (Valeriy *et al.*, 2016).Compounds of C-H, N-H and unsaturated, C=N with absorption peaks of 3000- 3100 cm<sup>-1</sup>, 3100-3500 cm<sup>-1</sup>, and 2850- 3000 cm<sup>-1</sup> occurred as compared to (Pavan *et al.*, 2016).



Figure 2b FTIR Spectra of Mr2 Wet season Maize Sample

Figures 2c showed FTIR intensities of wet season maize Mr3 sample grown at the vicinity of Galena mining area showed sulphur compounds of C-S,  $SO_3$  asymmetric stretching and  $SO_2$  symmetric stretching with absorption range of 800-600 cm<sup>-1</sup> and 1160-1150 cm<sup>-1</sup>. Other compounds of C-O, C-H, and unsaturated, S=O with absorption peaks of 1050- 1150 cm<sup>-1</sup>, 1260-1150 cm<sup>-1</sup>,1060- 1040 cm<sup>-1</sup> 1160-1120 cm<sup>-1</sup> are present as stated by (Pavan *et al.*, 2016).



Figure 2c FTIR Spectra of Mr3 Wet season Maize Sample

Figures 2d showed FTIR intensities bands of irrigated maize Mr4 sample grown within the vicinity of Galena mining area at Nahuta which showed only one sulphur compound of  $SO_2$  symmetric stretching with range values of 1160-1120 cm<sup>-1</sup>. Other compounds of C-H, N-H and unsaturated, S=O with absorption peaks of 3000- 3100 cm<sup>-1</sup>, 3100-3500 cm<sup>-1</sup>, and 1060- 1040 cm<sup>-1</sup> occurred as compared to (Pavan *et al.*, 2018).



Figure 2d FTIR Spectra of Mr4 Wet season Maize Sample

Figures 2e showed FTIR intensities bands of irrigated maize Mr5 sample grown within the vicinity of Galena mining area showed only one sulphur compound of  $SO_3$  symmetric stretching with values of 1160-1150 cm<sup>-1</sup> as compared to (Valeriy *et al.*, 2016).Compounds of C-H, N-H and unsaturated, S=O with absorption peaks of 3000- 3100 cm<sup>-1</sup>, 3100-3500 cm<sup>-1</sup>, and 1060-1040 cm<sup>-1</sup> occurred as stated by (Pavan *et al.*, 2018). Compounds of sulphur SO<sub>3</sub> was present in Mr2, Mr3and Mr5 samples, while S=O appeared in Mr4 and Mr5 samples SO<sub>2</sub> was present was

in Mr3 and Mr4. These compounds confirmed that sulphur existed in ores of Galena which was observed into maize samples at Galena mining area. Also C-S was present, S-H and S=O were absent in the wet season maize grown at vicinity of Galena mining area.



Figure 2e FTIR Spectra of Mr 5 Wet season Maize Sample



Figures 3a showed FTIR spectra's of wet season Okro Or1 samples grown at Galena mining area, contain only one sulphur compounds of  $SO_3$  asymmetry stretching with value range 1260-1240 cm<sup>-1</sup> as stated by(Valeriy *et al.*, 2016).Compounds of C-N, C-O, C-H and N-H functional group with intensities range of 1000-1350cm<sup>-1</sup>, 1050-1150cm<sup>-1</sup>, 3000-3100 cm<sup>-1</sup> and 3100-3500 cm<sup>-1</sup> are present as reported by (Pavan *et al.*, 2018). C=N Unsaturated compounds was present in the Okro1.



Figure 3a FTIR Spectra of Wet season Okro Or 1 Sample

Figures 3b showed FTIR spectra's of wet season Okro Or2 samples grown at Galena mining area, contain only one sulphur compounds of SO<sub>3</sub> asymmetry with value range of 1260-1240 cm<sup>-1</sup> as stated by(Valeriy *et al.*, 2016).Compounds of C-N, C-O, C-H and N-H groups with peak intensities of 1000-1350cm<sup>-1</sup>, 1050-1150cm<sup>-1</sup>, 3000-3100 cm<sup>-1</sup> and 3100-3500 cm<sup>-1</sup> are present as stated by (Pavan *et al.*, 2018). C=N Unsaturated compounds was present in the Okro Or2 samples.



Figure 3b FTIR Spectra of Wet season Okro Or 2 Sample

Figures 3c showed FTIR spectra's of wet season Okro Or3 samples grown at Galena mining area, contain only one sulphur compounds of SO<sub>3</sub> asymmetry stretching with value of 1260-1240 cm<sup>-1</sup> as stated by(Valeriy *et al.*, 2016) .Compounds of C-N, C-O, C-H and N-H with intensities range of 1000-1350cm<sup>-1</sup>, 1050-1150cm<sup>-1</sup>, 3000-3100 cm<sup>-1</sup> and 3100-3500 cm<sup>-1</sup> are also present. C=N Unsaturated compounds was present in the Okro Or3 samples.



Figure 3c FTIR Spectra of Wet season Okro Or 3 Sample

Figures 3d showed FTIR spectra's of wet season Okro Or4 samples grown at Galena mining area, contain sulphur compounds of  $SO_3$  asymmetry stretching with value range 1260-1240 cm<sup>-1</sup> .Compounds of C-N, C-O, C-H and N-H with peak intensities range of 1000-1350cm<sup>-1</sup>, 1050-1150cm<sup>-1</sup>, 3000-3100 cm<sup>-1</sup> and 3100-3500 cm<sup>-1</sup> are also present. S=O Unsaturated compounds was present in the Okro Or4 samples.



Figure 3d FTIR Spectra of Wet season Okro Or 4 Sample

Figures 3e showed FTIR spectra's of wet season Okro Or5 samples grown at Galena mining area, have sulphur compounds of C-S and SO<sub>3</sub> asymmetry stretching with values 800-600 cm<sup>-1</sup> and 1260-1240 cm<sup>-1</sup>. Compounds of C-N, C-O, C-H and N-H functional group intensities of 1000-1350cm<sup>-1</sup>, 1050-1150cm<sup>-1</sup>, 3000-3100 cm<sup>-1</sup> and 3100-3500 cm<sup>-1</sup> are present as reported by (Pavan *et al.*, 2018). Analysis showed C=N was present in Okro Or5 samples. While SO<sub>3</sub> was common to all samples, S=O appeared in Or4 sample, while C-S was only present in Or5. Compounds of sulphur revealed by the FTIR analysis was due to their absorption band in wet season Okro.



Figure 3e FTIR Spectra of Wet season Okro Or 5 Sample

#### 4.4 FTIR Spectra of Wet Season Rice Samples

Figures 4a showed wet season Rice R1 grown at the vicinity of Galena mining area showed sulphur compounds of SO<sub>2</sub> symmetric stretching and unsaturated sulphur compounds with 1160-1120 cm<sup>-1</sup>.and 1060-1040 cm<sup>-1</sup> as stated by (Valeriy *et al.*, 2016). Compounds of C-O, C-H and O-H with intensities of 1050-1150 cm<sup>-1</sup>,3000-3100 cm<sup>-1</sup> and 3200– 3400 cm<sup>-1</sup> were also present as reported by (Pavan *et al.*, 2018).



Figure 4a FTIR Spectra of Rr 1 Wet season Rice Sample

Figures 4b showed wet season Rice R2 grown at the vicinity of Galena mining area showed sulphur compounds of SO<sub>3</sub> symmetric stretching sulphur compounds only with 1050-1010 cm<sup>-1</sup> as reported by (Valeriy *et al.*, 2016).Compounds of C=C, C-O, C-H and O-H with intensities range of 1500- 1680 cm<sup>-1</sup> 1050-1150 cm<sup>-1</sup>,3000-3100 cm<sup>-1</sup> and 3200– 3400 cm<sup>-1</sup> as reported by (Fedi *et al.*, 2016).



Figure 4b FTIR Spectra of Rr 2 Wet season Rice Sample

Figures 4c showed wet season Rice R3 grown at the vicinity of Galena mining area of Nahuta showed sulphur compounds of SO<sub>2</sub> symmetric stretching, SO<sub>3</sub> symmetric stretching and unsaturated sulphur compounds of C=C, C=N with 1160-1120 cm<sup>-1</sup> 1050-1010 cm<sup>-1</sup>...and 1060-1040 cm<sup>-1</sup> 1640-1690 cm<sup>-1</sup> as stated by (Valeriy *et al.*, 2016).Compounds of C-O, C-H and O-H with intensities of 1050-1150cm<sup>-1</sup>,3000-3100 cm<sup>-1</sup> and 3200– 3400 cm<sup>-1</sup> occurred.



Figure 4c FTIR Spectra of Rr 3 Wet season Rice Sample

Figures 4d showed wet season Rice R4 grown at the vicinity of Galena mining area of Nahuta showed sulphur compounds of  $SO_2$  symmetric stretching,  $SO_3$  symmetric stretching and unsaturated sulphur compounds of C=N with 1160-1120 cm<sup>-1</sup> 1050-1010 cm<sup>-1</sup>..and 1640-1690 cm<sup>-1</sup> as stated by (Valeriy *et al.*, 2016).Other compounds of C-O, C-H and O-H with intensities range of 1050-1150cm<sup>-1</sup>,3000-3100 cm<sup>-1</sup> and 3200– 3400 cm<sup>-1</sup> appeared.



Figure 4d FTIR Spectra of Rr 4 Wet season Rice Sample

Figures 4e showed wet season Rice R5 grown at the vicinity of Galena mining area showed sulphur compounds of SO<sub>2</sub> symmetric stretching, SO<sub>3</sub> symmetric stretching and unsaturated sulphur compounds of C=N with 1160-1120 cm<sup>-1</sup> 1050-1010 cm<sup>-1</sup>..and 1640-1690 cm<sup>-1</sup> as stated by (Valeriy *et al.*, 2016). Other compounds of C-O, C-H and O-H with intensities range of 1050-1150 cm<sup>-1</sup>,3000-3100 cm<sup>-1</sup> and 3200- 3400 cm<sup>-1</sup> are also present in sample as compared to R4

are related to (Pavan *et al.*, 2018). Analysis revealed that R2, R3, R4 and R5 samples of rice showed major compounds of sulphur  $SO_3$  and  $SO_2$  was present in due to percolation of some dissolved sulphur compounds, which are not directly observed by these rice plants roots due to low suction capacity by some plants or deficient of such compounds.



Figure 4e FTIR Spectra of Rr 5 Wet season Rice Sample

#### 4.5 FTIR Spectra of Wet Season Tomatoes Samples

Figures 5a showed FTIR spectra's of wet season Tomatoes samples T1 grown at galena mining area contain sulphur compounds of C-S and SO<sub>3</sub> asymmetric stretching with intensities of 800- $600 \text{ cm}^{-1}$  and 1260-1150 cm<sup>-1</sup> as reported by (Valeriy *et al.*, 2016).Compounds of C-N, C-O, C-H and N-H functional group with intensities of 1000-1350cm<sup>-1</sup>, 1050-1150cm<sup>-1</sup>, 3000-3100 cm<sup>-1</sup> and 3100-3500 cm<sup>-1</sup> are present as reported by (Pavan *et al.*, 2018). Unsaturated groups of only C=O also occurred in T1 tomatoes.



Figure 5a FTIR Spectra of Tr 1 Wet season Tomato Sample

Figures 5b showed FTIR spectra's of wet season Tomatoes samplesT2 grown at galena mining area contain sulphur compounds of C-S and SO<sub>2</sub> symmetric stretching with intensities range of 800-600 cm<sup>-1</sup> and1165-1150 cm<sup>-1</sup> as compared to (Valeriy *et al.*, 2016).Compounds of C-N, C-O, C-H and N-H with peak intensities of 1000-1350cm<sup>-1</sup>, 1050-1150cm<sup>-1</sup>, 3000-3100 cm<sup>-1</sup> and 3100-3500 cm<sup>-1</sup> are present (Pavan *et al.*, 2018). Unsaturated compounds of C=O and C=N with intensities of 1640-1710 cm<sup>-1</sup> and 1640-1690 cm<sup>-1</sup> also exist in the T2.



Figure 5b FTIR Spectra of Tr 2 Wet season Tomato Sample

Figures 5c showed FTIR spectra's of wet season Tomatoes samplesT3 grown at galena mining area contain sulphur compounds of C-S and S=O unsaturated with intensities range of 800-600 cm<sup>-1</sup> and11060-1040 cm<sup>-1</sup> as reported by (Valeriy *et al.*, 2016).Other compounds of C-N, C-O, C-H and N-H with peak intensities range of 1000-1350cm<sup>-1</sup>, 1050-1150cm<sup>-1</sup>, 3000-3100 cm<sup>-1</sup> and 3100-3500 cm<sup>-1</sup> are present as stated by (Pavan *et al.*, 2018). Unsaturated functional groups of only C=C with intensities range of 1500-1680 cm<sup>-1</sup> also exist in the Tr3.



Figure 5c FTIR Spectra of Tr 3 Wet season Tomato Sample

Figures 5d showed FTIR spectra's of wet season Tomatoes samples T4 grown at galena mining area showed only one sulphur compounds of C-S with intensities of 800-600 cm<sup>-1</sup> as stated by(Valeriy *et al.*, 2016).Compounds of C-O and C-H with peak intensities of 1050-1150 cm<sup>-1</sup> and 3000-3100 cm<sup>-1</sup> are present as reported by (Pavan *et al.*, 2018). Unsaturated groups of only C=O with intensities range of 1640-1710 cm<sup>-1</sup> also exist in the T4.



Figure 5d FTIR Spectra of Tr 4 Wet season Tomato Sample

Figures 5e showed FTIR spectra's of wet season Tomatoes samples T5 grown at galena mining area showed only sulphur compounds of C-S with intensities range of 800-600 cm<sup>-1</sup> as reported by (Valeriy *et al.*, 2016).Compounds of C-O and C-H functional group with peak intensities range of 1050-1150cm<sup>-1</sup> and 3000-3100 cm<sup>-1</sup> are present as stated by (Pavan *et al.*, 2018). Unsaturated groups of only C=O with intensities range of 1640-1710 cm<sup>-1</sup> also exist in the T5 sampled tomatoes. Compounds of sulphur such as S-H stretching and S-O stretching, in the FTIR analysis of wet season tomatoes are absent in all sample of tomatoes spectra's.



Figure 6e FTIR Spectra of Tr 5 Wet season Tomato Sample

#### Conclusions

Wet season Beans, showed only S=O (1060-1040 cm<sup>-1</sup>), other samples of beans indicated that  $SO_2$  (1160-1120 cm<sup>-1</sup>),  $SO_3$  symmetric (1050-1010 cm<sup>-1</sup>) sulphur compounds and C-S (800-600 cm<sup>-1</sup>) were absent in beans as compared to spectra other samples. Rice showed major compounds of sulphur of SO<sub>3</sub> and SO<sub>2</sub> present, Okro showed SO<sub>3</sub> was common to all samples, S=O appeared in Or4 sample, while C-S was only present in Or5 and Wet season Tomatoes showed sulphur compounds of C-S,  $SO_2$ ,  $SO_3$  and S=O However Wet season maize sample showed sulphur compounds of C-S,  $SO_2$ ,  $SO_3$ , S=O, and C=O compared to wet season beans.

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