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## FTIR and GC-MS analyses of raw materials produced from food wastes used in synthesis of biofilms

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### Abstract

The present study carried out the GC-MS and FTIR analyses of raw materials (ripe plantain peel powder, cassava starch, glycerol, acetic acid (vinegar) and eggshell powder) produced from food wastes that are used in production of biofilm. The GC-MS results revealed the presence of pentadecane, bis (2-methylpropyl) ester and benzoic acid, 3-(2-Hydroxy-6-methylphenyl)-4(3H)-quinazolinone). The FTIR analysis revealed 17 peaks for glycerol, 16 peaks for vinegar, 15 peaks for cassava starch, 16 peaks for eggshell; representing the functional groups of ether, ethene, amine, carboxylic acid, nitriles, methylene cyclic ester, primary, secondary and tertiary alcohols common to hydrocarbons. The presence of carboxylic acid was important for enhancing the mechanical and chemical properties of biofilms thereby making them viable alternatives to petroleum-based plastics. Amines contributed to the flexibility, strength, and thermal degradation resistance of biofilms, thus aiding in their biodegradability. In conclusion, the study showed that the raw materials possessed biological activities and their functional groups indicated that the raw materials are safe to humans and the environment.

**Keywords:** Biofilms; FTIR; GC-MS analyses; Raw materials from food wastes

### 1. Introduction

Due to the excellent properties of plastics such as lightweight, tough, moderately low-priced, and durable. Plastics has become an indispensable polymer utilized in every household. They come in different forms such as tables, chairs, kitchen utensils (spoons, plates, forks) and are also employed in different industries as packaging materials, carrier bags etc. The vast utilization of this polymer has posed environmental threats to the ecosystem. Every year, about 300 million tonnes of plastics which are utilized once and disposed of after use [19].

However, this polymer when discarded as waste may take hundreds to thousands of years to decompose [17]. This is because of the durability and low degradability of these polymers. Besides, when plastics are produced, only 7% is recycled, while about 8% is incinerated and the residual land filled [6].

In 1975, The Public Foundation of Sciences deduced that about 14 billion pounds of trash was released yearly which was, either covered underground or covered in the seas. This makes the water bodies or the terrestrial habitat to be filled with plastic waste.

As a matter of fact, in excess of 10 million tons of plastic waste is unloaded in the seas alone, so most of anthropogenic garbage littering the seas is made out of human-made plastics. Reports propose that plastics can now be utilized as a

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geographical stratigraphic sign of the anthropogenic period [21, 6]. This anthropogenic debris threatens ocean safety, integrity, and sustainability [17]. Overall, the environmental problems caused by plastic waste is yet unsolved.

The environmental threats caused by continuous disposal of plastics waste nowadays has raised the quest for substitutes.

Bioplastics and biofilms, which are eco-friendly but in practice are similar to their synthetic counter part have been found to be a promising material to resolve these issues. Bioplastics and biofilms are 'plastics' produced from biomass such as cellulose, starch, chitosan etc.

These 'plastics' can be biodegradable (e.g. polycaprolactone or polybutylene succinate (PLC or PLB) or non-degradable (a blend of biomass and petroleum based materials). For example, biopolyethylene(Bio-PE) and biopropylene(Bio-PP) [3; 10]. This study reports the GC-MS and FTIR analyses of raw materials produced from food wastes that are used in production of biofilm.

## 2. Material and methods

### 2.1. Collection of plantain peels, palm oil and cassava roots, pineapples and egg shells

The ripe plantain peels (*African Rhino horn species*) were obtained from Buka 9 in Federal University of Technology, Owerri (FUTO) Market, Nigeria (Longitude 6°59'E and Latitude 5°23'N). The palm oil (produced from the *Elaeis guineensis specie*) was purchased from Ihiagwa Market, Nigeria (Longitude 7°01'E and Latitude 5°24'N). The cassava roots (*TNE 419 specie*) were harvested from a farm in FUTO Market (Longitude 6°59'E and Latitude 5°22'N). The fresh eggshells from chickens of *Leghorn specie* were obtained from Lala Meshai Spot in FUTO (Longitude 6°59'E and Latitude 5°23'N). The pineapple peels (*Abacaxi specie*) were collected from roadside fruit sellers at Ihiagwa Market (Longitude 7°01'E and Latitude 5°24'N).

### 2.2. Treatment of plantain peels

The plantain peels were treated using a modified method as described by [12].

### 2.3. Preparation of crude glycerol

The crude glycerol was produced according to the method described by [25].

### 2.4. Preparation of cassava starch

The cassava starch was produced according to the method described by [13].

### 2.5. Preparation of acetic acid (vinegar)

The vinegar was produced following the modified method of [8].

### 2.6. Treatment of eggshells

The eggshells were treated according to the modified method described by [24].

### 2.7. Fourier transform infrared spectroscopy analyses of the raw materials

Zero point five grammes (0.5 g) of the sample was mixed with 0.5 g of potassium bromide (KBr), after which 1.0 ml of nujol (a solvent for preparation of sample by Buck M530 IR-spectrophotometer) was introduced into the sample with the aid of a syringe to form a paste before introducing it into the instrument sample mould and allowed to scan at a wavelength of 600-4000 nm to obtain its spectra wavelength [18].

### 2.8. Gas chromatography-mass spectrometry (GC-MS) analyses of the raw materials

The GC-MS analyses of the produced raw materials was conducted on Agilent Technologies 6890N Network GC System and Agilent Technologies 5973 network mass selective detector coupled with 7683B series injector. During the analysis, the oven temperature was maintained at 50 °C for 5 min. The injector and detector temperatures were 120 °C and 160 °C, respectively. The flow rate for carrier gas (Helium) was set at 1.2 ml/min and the injection volume were 1 NL [15]. The inlet temperature was maintained at 230 °C. The oven was programmed again to increase to 300 °C at a rate of 10 °C, ending in 25 min and this temperature was held for 15 min. The total runtime was 45 min. The mass spectrometry

(MS) transfer line was maintained at a temperature of 250 °C. The source temperature was also maintained at 230 °C and Ms quad at 150 °C. The ionisation mode to be used was electron ionization mode at 70 Ev. Total ion count (TIC) was used to evaluate for compound identification and quantification. The spectrum of the separated compound was compared with the database of the spectrum of known compounds saved with the database in the NIST02 Reference Spectra Library [13].

### 3. Results and discussion

#### 3.1. Fourier transform infrared spectroscopy

Fig. 1 shows the FTIR results for crude glycerol were, peak values around 1102.833  $\text{cm}^{-1}$  and 1298.699  $\text{cm}^{-1}$  was assigned to CO stretching vibration of the ether compound. The absorbance of 1393.905  $\text{cm}^{-1}$  was assigned to C=C antisymmetric vibration of alkene compound. The Medium band of 1620.021  $\text{cm}^{-1}$  was assigned to N-H stretching vibration of 1<sup>o</sup> amine compound. Spectra height of 1837.330  $\text{cm}^{-1}$  corresponds to CO stretching vibration of the cyclic ester compound. Wavelength of 2045.241  $\text{cm}^{-1}$  and 2277.092  $\text{cm}^{-1}$  were assigned to COO stretching vibration of carboxylic acid compounds, respectively. The peaks around 2451.037  $\text{cm}^{-1}$  and 2594.904  $\text{cm}^{-1}$  were assigned to CN stretching vibration of nitrile compound, respectively. The weak bands around 2682.750  $\text{cm}^{-1}$  and 2919.177  $\text{cm}^{-1}$  were both assigned to C-H stretching vibration of methylene compound, respectively. Strong bands around 3024.229  $\text{cm}^{-1}$ , 3165.812  $\text{cm}^{-1}$ , 3275.510  $\text{cm}^{-1}$ , 3558.926  $\text{cm}^{-1}$ , 3704.989  $\text{cm}^{-1}$  and 3704.989  $\text{cm}^{-1}$  were assigned to OH stretching vibration of 1<sup>o</sup> and 3<sup>o</sup> alcohols, respectively.

Fig. 2 shows FTIR spectra of vinegar, the absorbance of 11021.292  $\text{cm}^{-1}$  was assigned to CO stretching vibration of ether compound. The peak values around 1322.171  $\text{cm}^{-1}$  and 1437.724  $\text{cm}^{-1}$  were assigned to C=C stretching vibration of ethene compound, respectively. The medium band at 1626.265  $\text{cm}^{-1}$  was assigned to N-H stretching vibration of 1<sup>o</sup> amine compound. The peak value at 1889.889  $\text{cm}^{-1}$  was assigned to CO stretching vibration of cyclic ester compound. Absorbance at 2026.817  $\text{cm}^{-1}$  and 2113.309  $\text{cm}^{-1}$  were assigned to COO stretching vibration of carboxylic acid, respectively, whereas the peak located at 2201.730  $\text{cm}^{-1}$  was assigned to CO stretching vibration of carbonyl compound. The peak at 2449.360  $\text{cm}^{-1}$  was assigned to CN stretching vibration of a nitrile compound. The weak peak values located at 2619.551  $\text{cm}^{-1}$ , 2742.866  $\text{cm}^{-1}$  and 2911.437  $\text{cm}^{-1}$  were all assigned to C-H stretching vibration of methylene compound, respectively. Strong band wavelengths located at 3184.373  $\text{cm}^{-1}$ , 3374.118  $\text{cm}^{-1}$ , 3575.131  $\text{cm}^{-1}$  and 3803.590  $\text{cm}^{-1}$  were assigned to OH stretching vibration of 1<sup>o</sup>, 2<sup>o</sup> and 3<sup>o</sup>, respectively. The N-H group comes from biogenic amine formed by microbial decarboxylation of amino acids present in vinegar. Formation of biogenic amines is influenced by bacterial strain, level of carboxylase activity and availability of amino acids as substrates. The presence of these groups signified that the fermented product was vinegar [4, 19].

Fig. 3 shows FTIR spectra for cassava starch. The peak values at 1058.841  $\text{cm}^{-1}$  was assigned to CO stretching vibrations of the ether compound while the peak values at 1360.767  $\text{cm}^{-1}$  was assigned to C=C stretching vibration of ethene compound. The medium bands at 1624.580  $\text{cm}^{-1}$  and 3405.882  $\text{cm}^{-1}$  were assigned to N-H stretching vibration of 1<sup>o</sup> and 2<sup>o</sup> amine compounds, respectively. The peak value at 1885.184  $\text{cm}^{-1}$  was assigned to CO stretching vibration of cyclic ester compound. Absorbance values at 2054.885  $\text{cm}^{-1}$  and 2163.940  $\text{cm}^{-1}$  were assigned to COO stretching vibration of carboxylic acid, respectively, whereas the peaks located at 2624.921  $\text{cm}^{-1}$  and 2929.717  $\text{cm}^{-1}$  were both assigned to C-H stretching vibration of methylene compounds. The peak values located at 3054.420  $\text{cm}^{-1}$ , 3175.895  $\text{cm}^{-1}$  and 3516.919  $\text{cm}^{-1}$  were all assigned to OH stretching vibration of 1<sup>o</sup>, 2<sup>o</sup> and 3<sup>o</sup> alcohols, respectively.

Fig. 4 shows the FTIR spectra of eggshell, the peak values at 1383.668  $\text{cm}^{-1}$  and 1467.005  $\text{cm}^{-1}$  were assigned to C=C stretching vibrations of the ethene compound. The medium bands at 1623.465  $\text{cm}^{-1}$  and 3457.769  $\text{cm}^{-1}$  were assigned to N-H stretching vibration of 1<sup>o</sup> amine compound. The peak values at 1830.269  $\text{cm}^{-1}$  and 1988.089  $\text{cm}^{-1}$  were assigned to CO stretching vibration of cyclic ester compound respectively. Absorbance at 2120.197  $\text{cm}^{-1}$  was assigned to COO stretching vibration of carboxylic acid, respectively, whereas the peaks located at 2746.925  $\text{cm}^{-1}$  and 2904.522  $\text{cm}^{-1}$  were both assigned to C-H stretching vibration of methylene compounds. The peak values located at 3008.660  $\text{cm}^{-1}$ , 3242.667  $\text{cm}^{-1}$ , 3345.205  $\text{cm}^{-1}$ , 3700.921  $\text{cm}^{-1}$ , and 3839.897  $\text{cm}^{-1}$  were all assigned to OH stretching vibration of 1<sup>o</sup>, 2<sup>o</sup> & 3<sup>o</sup> alcohols respectively. The C=C that correspond to the body of ethene may be due to 2-Methyl-1-tetradecene, 1-Hexene, 3,4-dimethyl-1-Pentene, 2-methyl- and 1-Hexene, 3,4-dimethyl-(Z)-Hex-2-ene or 5-methyl-2,4-Dimethyl-1-hexene.

Fig. 5 shows FTIR spectra of plantain peels. The peak values at 1328.304  $\text{cm}^{-1}$  and 1433.162  $\text{cm}^{-1}$  were assigned to C=C stretching vibration of ethene compound. The medium bands located at 1620.894  $\text{cm}^{-1}$  and 1667.273  $\text{cm}^{-1}$  were assigned to N-H stretching vibration of 1<sup>o</sup> amine compound, respectively. The peaks at 2024.931  $\text{cm}^{-1}$ , 2086.796  $\text{cm}^{-1}$  and 2214.360  $\text{cm}^{-1}$  were all assigned to COO stretching vibration of carboxylic acid, respectively, whereas the peaks located

at 2513.176  $\text{cm}^{-1}$  was assigned to CN stretching vibration of nitrile compound. The weak bands located at 2627.409  $\text{cm}^{-1}$ , 2750.631  $\text{cm}^{-1}$  and 2849.984  $\text{cm}^{-1}$  were all assigned to C-H stretching vibration of methylene compounds. The strong bands located at 3083.774  $\text{cm}^{-1}$ , 3190.627  $\text{cm}^{-1}$ , 3350.071  $\text{cm}^{-1}$  and 3661.209  $\text{cm}^{-1}$  were all assigned to OH stretching vibration of 1<sup>o</sup>, 2<sup>o</sup> and 3<sup>o</sup> alcohols, respectively. The samples consist of alkene, alcohol indicating the presence of hydrocarbons in the plantain peel powder. This is likely as a result of the various vibration modes that are found in carbohydrates and lignin [2].

From the result of the study, important functional groups were detected. These include, ether, ethene, amine, carboxylic acid, nitriles, methylene cyclic ester, primary, secondary and tertiary alcohols common to hydrocarbons, respectively. The presence of carboxylic acid is important for enhancing the mechanical and chemical properties of biofilms, making them a viable alternative to petroleum-based plastics. Amines contribute to the flexibility, strength, and thermal degradation resistance of biofilms, thus aiding in their biodegradability [23].

### 3.2. GC-MS analysis

Fig. 6 shows the GC-MS chromatogram of plantain peels. Fifteen compounds were identified in the GC-MS analysis of ripe plantain peel powder. Amongst the compounds identified, the most prominent compounds were N4-Phenethylmorpholine-4-carbothioamide, dichloroacetaldehyde, 1,16-cyclocorynan-17-oic acid and 19,20-didehydro-, methyl ester.

Fig. 7 shows the GC-MS chromatogram of cassava starch with 15 peaks. The peaks indicated the presence of fifteen compounds. The most abundant compounds were identified as dihexyl monoselenide, 2-amino-oxazole, trichloroacetic acid, decyl ester (11.54%), dihexyl monoselenide 3-trifluoroacetoxypentadecane, pentadecane, 8-methylene- (11.34%), 1 H-1,2,4-triazole, 3-methyl-5-(methylthio)-Dihexylmonoselenide, 2-t-butyl-5-methyl-10H-acridin-9-one (9.98%) and methaqualone. Other compounds were detected in minimal concentrations.

Fig. 8 shows the GC-MS chromatogram of eggshell with fifteen compounds identified. Amongst the compounds detected, 1,2-benzenedicarboxylic acid, monobutyl ester, dibut-3-enyl phthalate, phthalic acid and monodecyl ester were identified to be the most prevailing bioactive compounds (11.62%), followed by 1-hexene, 3,4-dimethyl-(Z)-Hex-2-ene, 5-methyl-2,4-dimethyl-1-hexene (9.26%), N-[3-aminophenyl]-1 piperidinecarbothioamide, 5-methyl-6-nitro-2-phenyl-1H-indol, 3-(2-hydroxy-6-methylphenyl) 4(3H)quinazolinone (8.37%). Other compounds were detected in minimal concentrations.

Fig. 9 showed GC-MS chromatogram of acetic acid (vinegar) with fifteen peaks. The peaks indicated the presence of fifteen compounds. Amongst the compounds identified, bis(2-methylpropyl) ester, 1,2 -benzenedicarboxylic acid, phthalic acid, 8-bromooctyl isobutyl ester, 1,2-benzenedicarboxylic acid, butyl 2-methylpropyl ester were found to have higher concentrations (19.70 %) followed by benzoic acid (17.04 %). N-Isopropyl-N-methyl aminoethyl-2- chloride, N-methyl-3-piperidinecarboxamide, ethyl oxachloride had lower concentrations (0.93 %). The benzoic acid serves as a preservative to biofilms; helping to prolong its shell life [5]. Phthalic acid serves as a plasticizer; helping to make biofilms flexible and elastic [5].

Fig 10 shows GC-MS chromatogram of crude glycerol with thirty compounds identified. Amongst the compounds identified, the most prevailing compounds identified included 5-hydroxymethylfurfural, 5-hydroxymethylfurfural and 4-mercaptophenol were identified to have the higher concentrations (18.23 %).

Amongst the compounds identified in the synthesized raw materials (glycerol, cassava starch, plantain peel, eggshell, acetic acid), few of the compounds possessed certain biological activities. These include 2-butenedioic acid and (Z)-, dibutyl ester have been reported to have antioxidant and antiinflammatory activity; [18] reported the antiinflammatory, analgesic and antipyretic properties of pentadecane. Bis (2-methylpropyl) ester and benzoic acid possess antioxidant and antimicrobial activity. Also, methaqualone, a substituted quinazolines, has been reported to be a sedative, hypnotics and possesses antispasmodic properties [8, 19]. Likewise, dibut-3-enyl phthalate was reported by [22] to exhibit antimicrobial activity. Likewise, 1, 2-benzenedicarboxylic acid has been reported to have antimicrobial activity [1]. The 3-(2-hydroxy-6-methylphenyl)-4(3H)-quinazolinone was reported to possess anticancer properties [1]. Furthermore, the biological activities of diosgenin include anticancer, cardio- protective, antiasthmatic, anti-diabetic, [14, 16, 23]. Furthermore, 1, 2-benzenedicarboxylic acid has been reported to have antimicrobial activity according to [1]. The 3-(2-Hydroxy-6-methylphenyl)-4(3H)-quinazolinone was reported to possess anticancer properties [1]. Dibut-3-enyl phthalate was reported to exhibit antimicrobial activity [22]. The compounds identified here were completely different from previous reports by other authors [18]. The difference in composition of these extracts could be due to the treatment of the peel before extraction. In the previous reports, the banana peels were dried

for two weeks at atmospheric conditions, powdered, and extracted with ethanol or methanol. However, in this study, the plantain peels and eggshells were sun dried for one month, powdered before being analysed. The difference may also have been attributed to the species of the plantain [18]. The cassava starch was sun dried after extraction and then analysed. Ethanol or methanol was not employed.

The compounds identified by the GC-MS differed from those in the previous report. This could be due to differences in the method of treatment and differences in the species of plantain used.

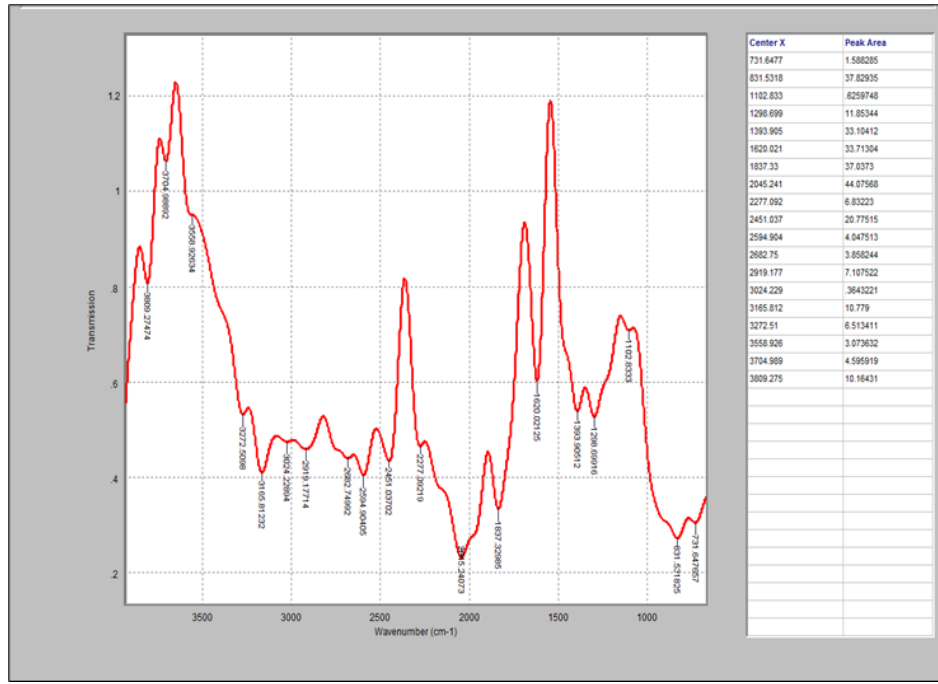


Figure 1 FTIR spectra of glycerol

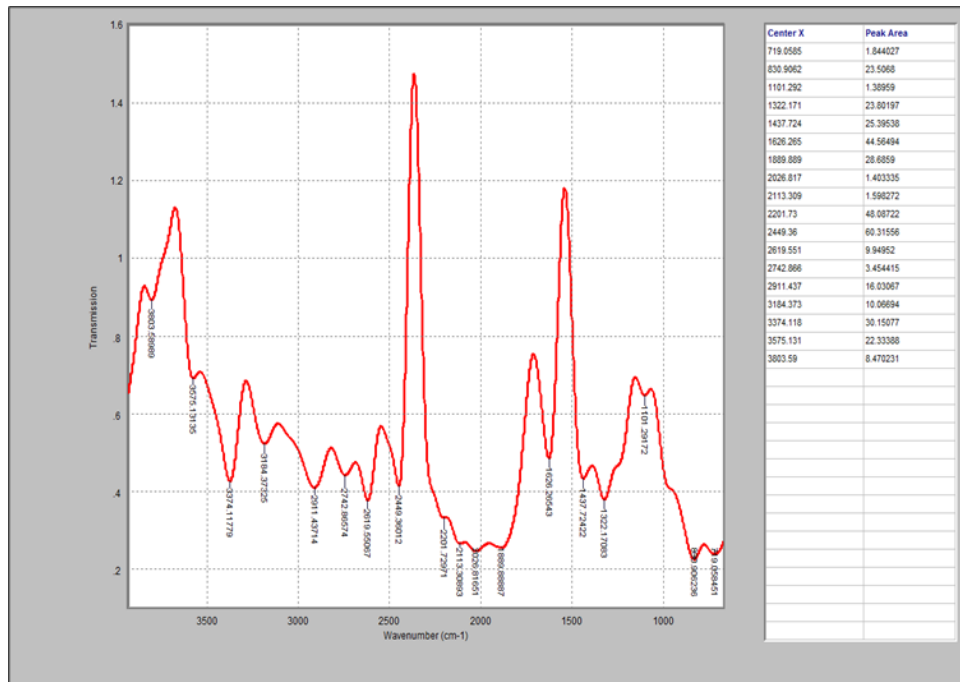


Figure 2 FTIR spectra of vinegar

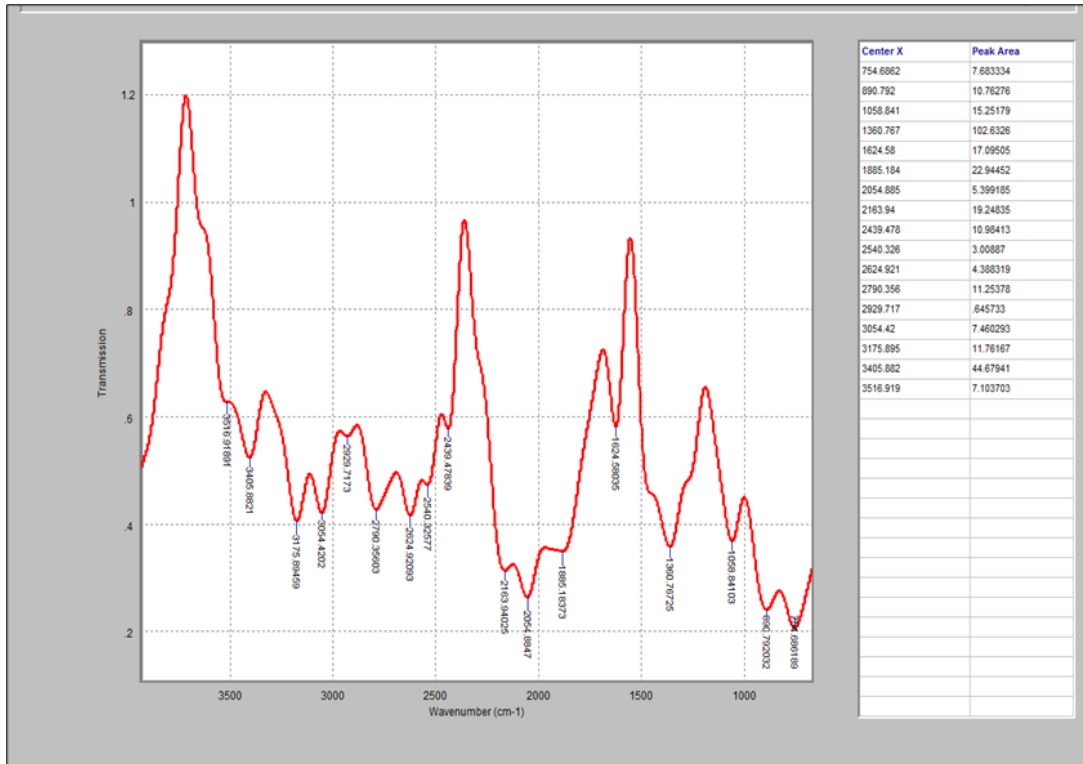


Figure 3 FTIR spectra of cassava

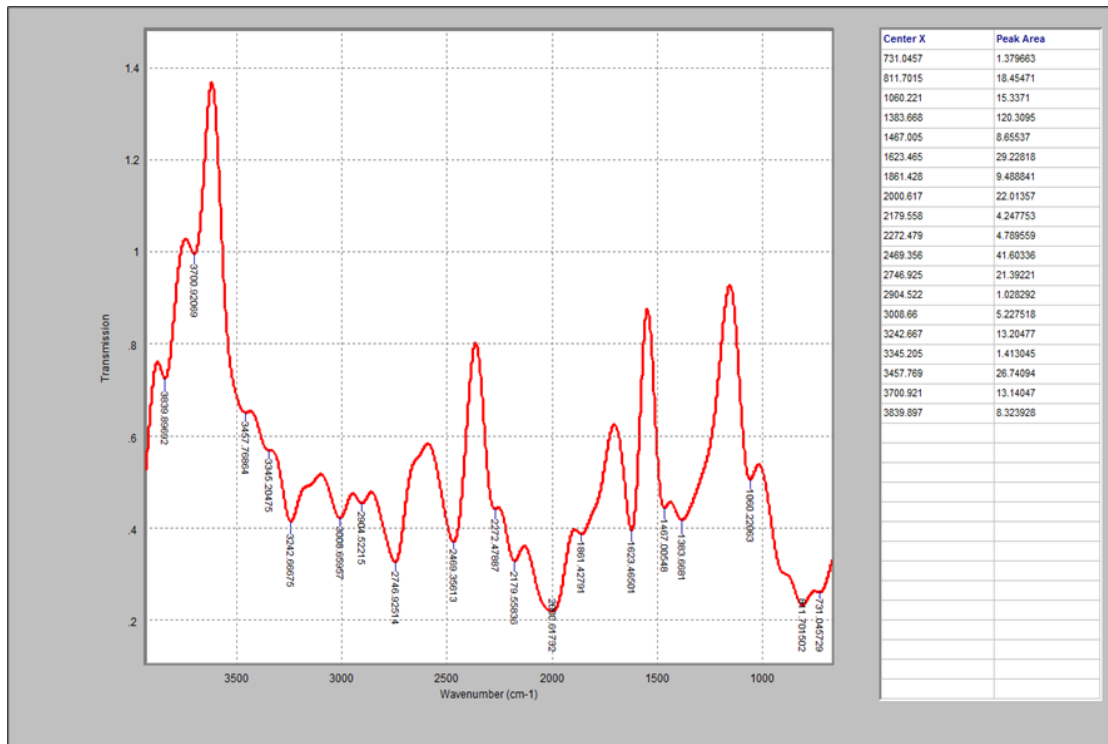


Figure 4 FTIR spectra of egg shell

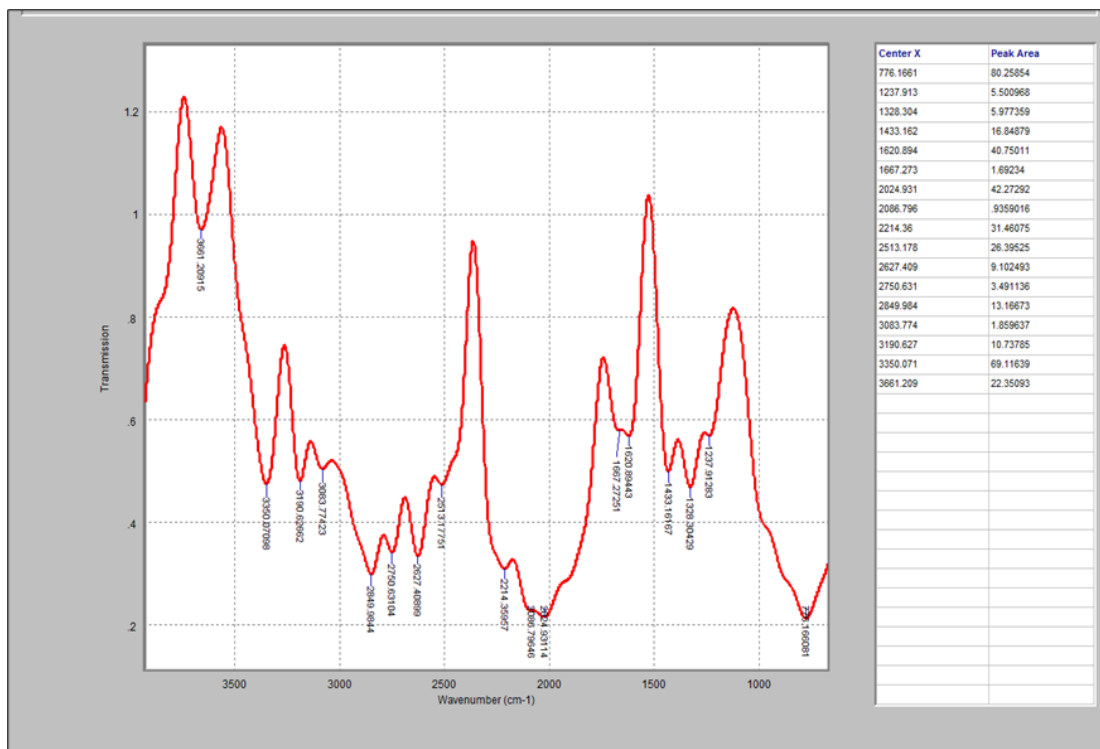


Figure 5 FTIR spectra of plantain peels

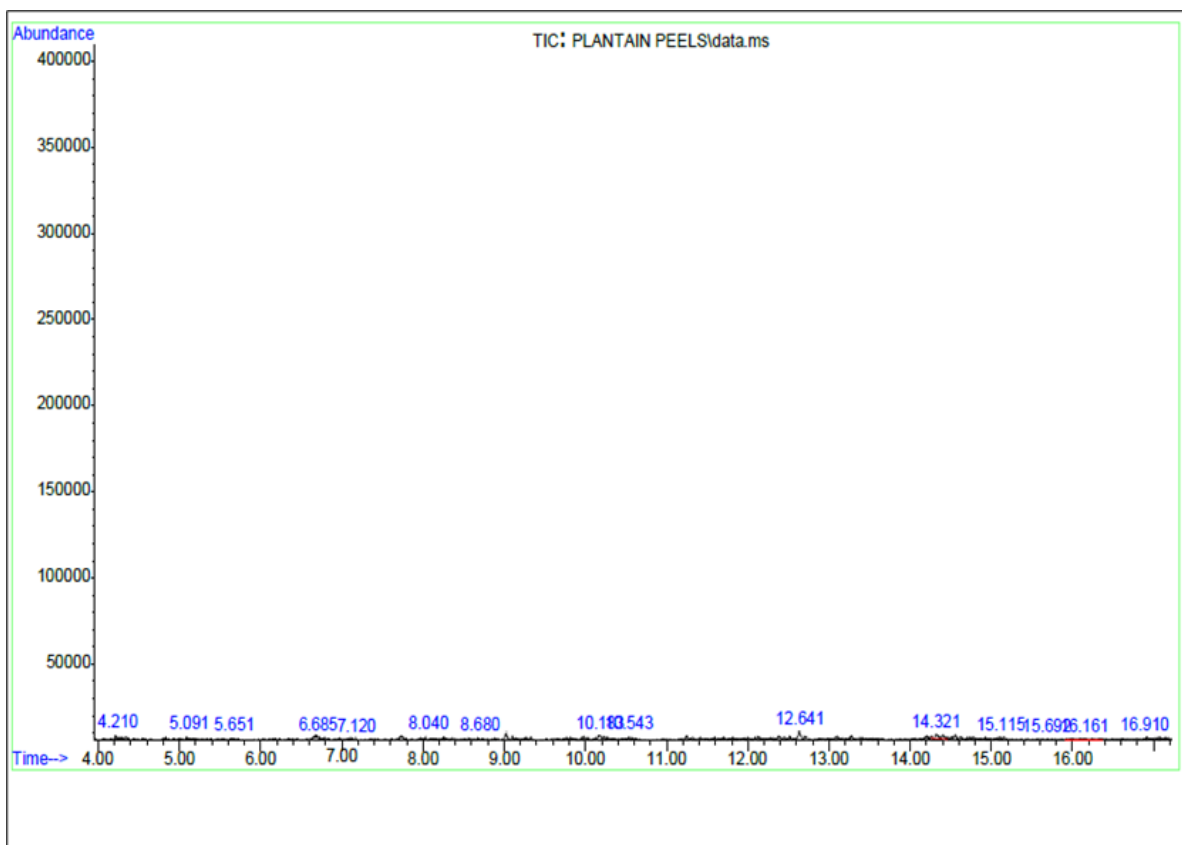


Figure 6 GC-MS chromatogram of plantain peels

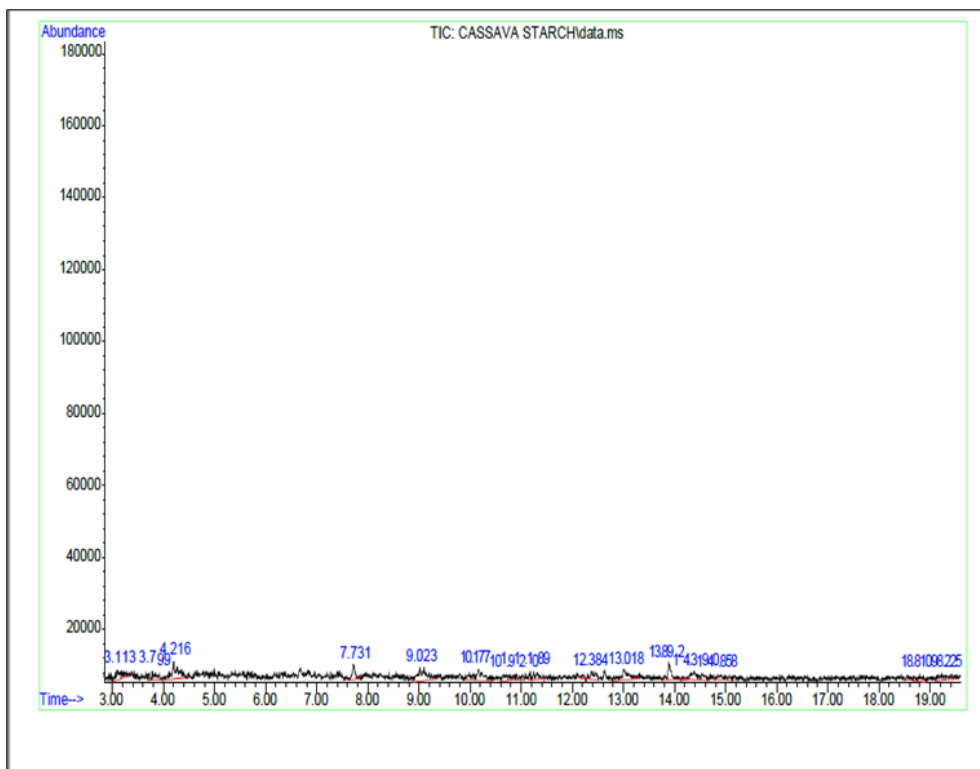


Figure 7 GC-MS chromatogram of cassava starch

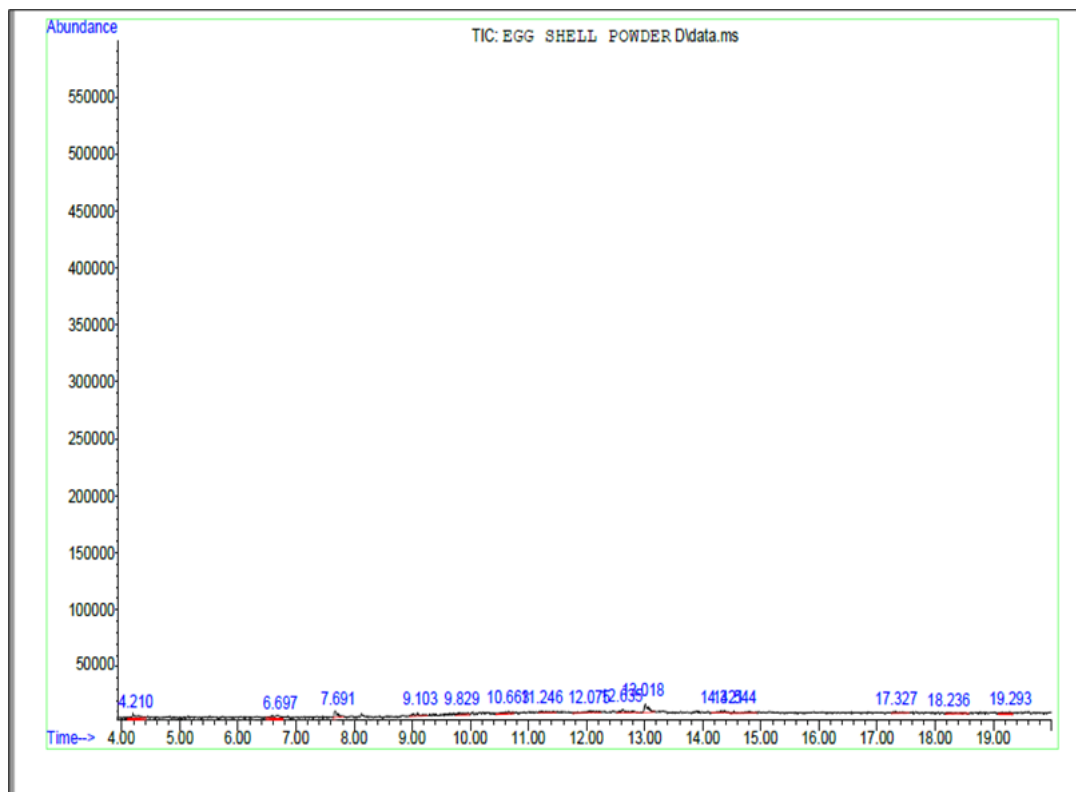


Figure 8 GC-MS chromatogram of egg shell



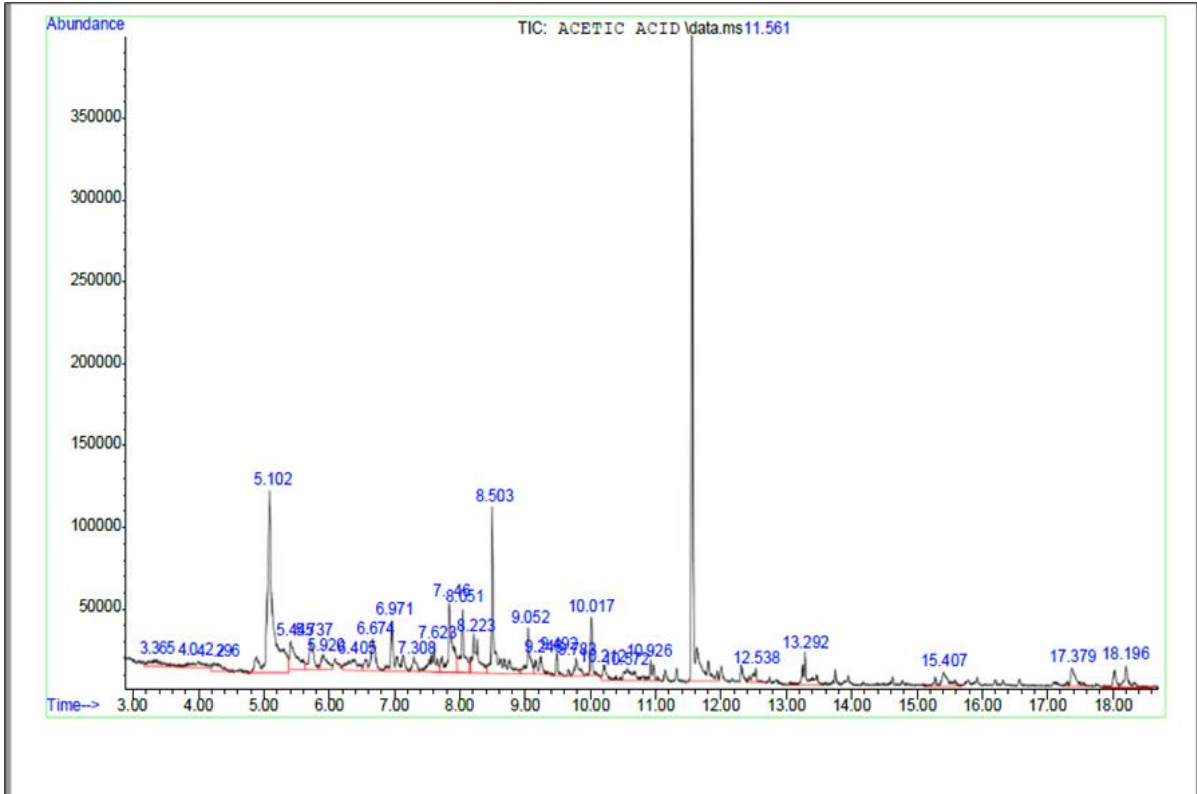


Figure 9 GC-MS chromatogram of acetic acid (vinegar)

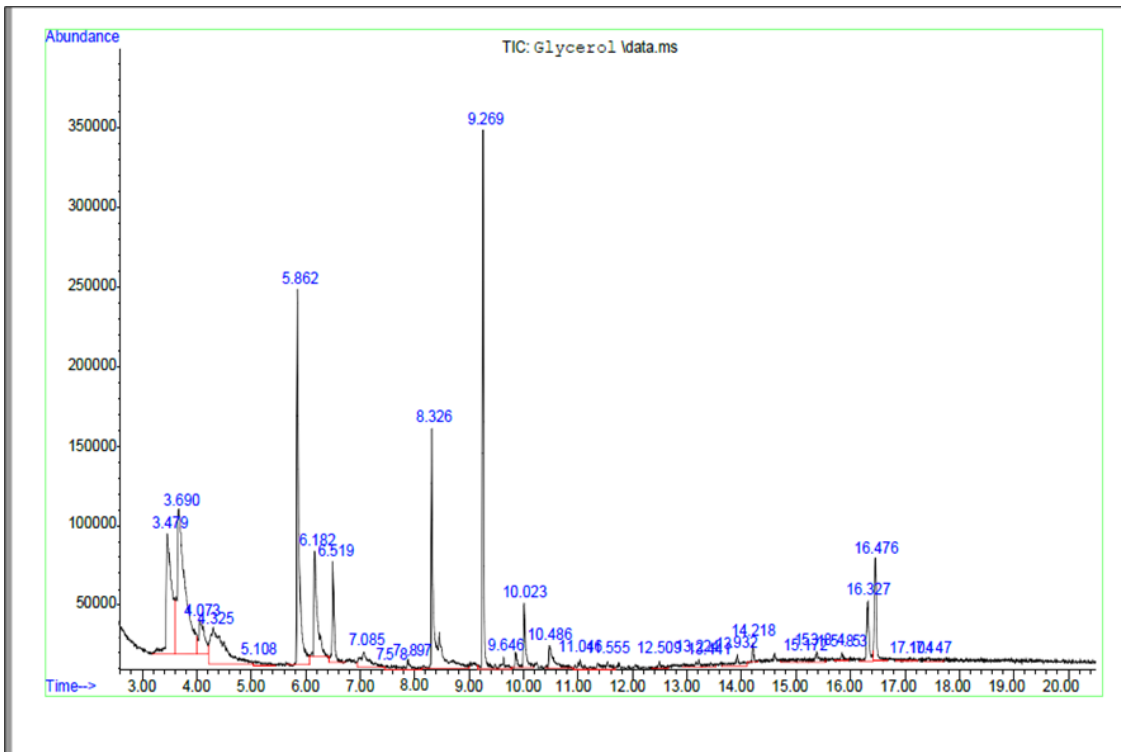


Figure 10 GC-MS chromatogram of crude glycerol

#### 4. Conclusion

The result of the present study showed that the composition of the raw materials differed from those in previous research, which could be due to differences in the species and method of pretreatment. However, the raw materials were found to be safe for human and environmental use. This implies that the raw materials can be used to produce an eco-friendly bioplastic.

#### Compliance with ethical standards

##### *Disclosure of conflict of interest*

We declare that no conflict of interest.

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