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PHYSICOCHEMICAL CHARACTERIZATION OF OKRA AND POTATO WASTES FOR THE PRODUCTION OF BIOCRUDE OIL

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Abstract: A significant amount of agricultural and food waste is not properly or optimally utilized to produce value-added materials; instead, some are simply incinerated, contributing to environmental pollution challenges. Effective utilization of agricultural waste for producing value-added requires thorough feedstock characterization. In light of this, the present study focused on the characterization of okra and sweet potato wastes to evaluate their suitability as feedstocks for biocrude oil production via hydrothermal liquefaction (HTL). The characterization analyses conducted include proximate and ultimate analyses, thermogravimetric analysis (TGA), and Fourier transform infrared (FTIR) spectroscopy. The proximate analysis revealed that sweet potato waste has a low ash content (9.0%) and a high fixed carbon content (60.66%), while okra waste also showed a low ash content (9.67%) and an even higher fixed carbon content (66.00%). These low ash and high fixed carbon values suggest that both feedstocks have strong potential for biocrude oil production via HTL. Additionally, the ultimate analysis results for both wastes were found to be encouraging. The TGA results indicated a nearly constant weight loss within the temperature range of 450 °C–550 °C for both feedstocks, attributed to thermal degradation. The FTIR spectra further confirmed the presence of functional groups such as O–H, C=C, C≡C, C–C, and C–O at various stretching bands. The presence of C=C and other functional groups suggests the likely presence of organic compounds and chemicals in the feedstocks. In conclusion, the characterization results demonstrate that okra and sweet potato wastes are promising feedstocks for the production of biocrude oil and other valuable chemicals through the hydrothermal liquefaction process.

Keywords: Potato, Okra, Proximate, Ultimate, Hydrothermal

INTRODUCTION

Physical and chemical study of a substance is an analytical procedure that is very useful for characterising materials for fuel production. The aforementioned, is the field of chemistry dealing with the interrelation between the composition and properties of matter (Kalkan et al., 2020). The Food and Agriculture Organization reports that approximately 1.6 billion tons of food is wasted globally each year, accounting for approximately one-third of total food production (Wang et al., 2023). Luo et al. (2024) reported that okra is prone to issues such as lignification, browning, softness, and nutrient loss during storage, all of which negatively impact its flavor. In addition, these challenges have reduced the nutritional value of okra and affect the development of the industry. Proximate analysis is the process of determination of the compounds contained in an organic substance such as biomass.

Furthermore, outcome of an ultimate analysis gives the percentage composition of the samples in terms of oxygen, nitrogen, hydrogen, carbon and sulphur on an as-received basis. The aim of the study is the evaluation of the proximate and ultimate analysis. Waste-to-energy (WtE) is the process of generating energy in the form of electricity and/or heat from the primary treatment of waste, or the processing of waste into a fuel source (Ondachi et al. 2023*)*. Using waste to generate energy is classified as a source of renewable energy (De-Campos et al., 2022); this is because using WtE will lead to commensurate reduction in energy generation using fossil fuels which are non-renewable and pollute the atmosphere by relasing carbon dioxide and other greenhouse gases (GHG) to the atmosphere. Steamed/Boiled chunks are also very common in many African homes used as a simple, healthy snack; sweet potato chips can be sliced, fried, and eaten just like potato chips or French fries. In addition, raw sweet potatoes can be eaten as well, usually as a chip; in previous studies, sweet potato was processed into different confectioneries (Meludu, 2010).

Fourier transform infrared (FTIR) is one of the tools to represent molecular structure based on atomic vibration. It can denote a specific property of the chemical bond and molecular structure in the material, especially when analyzing specimens relating to plastics, polymers, and organic components (Nandiyanto et al., 2023). Information from FTIR spectra can help to identify a molecule's backbone or the functional groups connected inside the material, including the type of functional groups which might reveal insight on the available compounds in the material (Sukamto & Rahmat, 2023).

Therefore, this study focused on the characterization of the okra and sweet potato wastes for the production of biocrude oil via hydrothermal liquifaction process.

MATERIALS AND METHODS

Sample Collection

Sweet potato waste was obtained from the local market in Bauchi and dried Okra stalk powder was collected from farm land after harvest in Bauchi. The samples were brought to Department of Chemical Engineering ATBU Bauchi prior to delivery for sample characterization In Jos-Plateau State and Katsina State.

Preparation of Samples

The sweet potato waste tubers were thoroughly sorted to remove bad ones from the lot. The sorted tubers were washed to remove adhering soil particles, weighed accordingly. The tubers after weighing were thereafter peeled and sliced into small pieces, transferred into motar pestle where it ws grounded to powder, small amount of the grounded powder was then taken in polythene bag for proximate and ultimate characterization. The dried Okra stalk obtained from the farm land was grounded to powder form; small amount of the powder was then taken in polythene bag for proximate and ultimate characterization.

Proximate Characterization of the Samples

Physical properties of the Okra and sweet potato wastes were determined for parameters such as moisture content, ash content, fixed carbon and volatile matter in order to know the suitability of the feedstocks for the biocrude production through hydrothermal liquefaction. Detail procedure on the steps followed for the determination of the aforementioned procedure is as follows:

Determination of moisture content

The procedure for moisture content determination was conducted following ASTM-E871 standards as reported in literature (Tursunov & Abduganiev, 2019; Anguruwa & Oluwadare, 2019). Equation 1 was used for the calculation of percentage (%) moisture content.

$$\%M_C = \frac{M_1 - M_2}{M_1} \times 100 \quad (1)$$

Where M_1 is the initial mass of sample and M_2 is the mass of dry sample.

Percentage volatile matter determination:

The amount of volatile matter present is equal to loss in weight after heating the sample to 600 °C and is calculated using the formula as presented in Equation 2 (Rominiyi et al., 2017):

$$\%Vm = \frac{M_d - M_2}{M_i} \times 100 \quad (2)$$

Where M_i = Initial mass of the sample, Md= mass of dry sample, Mb = final constant mass of the sample before oxidation.

Percentage ash content determination: The amount of residual matter is determined by difference, as presented in Equation 3.

$$\%\, ash\ content = \frac{M_a - M_c}{M_i} \times 100 \quad (3)$$

Where M_i = Initial mass of the sample, M_a is the mass of crucible plus ash, Mc is the mass of empty crucible.

Percentage fixed carbon determination: The amount of fixed carbon was evaluated using the formula presented in Equation 4:

$$\%\, Fixed\ carbon = 100 - (\% M_c + \%Vm + \%\ ash\ content) \quad (4)$$

Ultimate Characterization of the Samples

Chemical composition of the samples was determined in order to ascertain the elements such CHNS/O in the materials. Detail on the procedure of the individual elements determined is presented below:

Determination of carbon and hydrogen in solid waste

The procedure adopted is in accordance with ASTM D: 5868-10a as reported by Rominiyi et al., (2017); *Ondachi et al.* (2023). Per cent hydrogen and elemental carbon contents in the samples were then calculated using Equations 5 and 6, respectively:

$$\%\, H_2 = \frac{2Y}{18X} \times 100 \quad (5)$$

$$\%\, C = \frac{12Z}{44X} \times 100 \quad (6)$$

Where: Y is the increase in the weight of $CaCl_2$ tube; Z is the increase in the weight of KOH tube; X is the weight of waste sample taken for the analysis.

Determination of nitrogen content in waste sample

Nitrogen content is estimated using Kjeldahal's method as outlined by Rominiyi et al. (2017); detail information on the model equation used is shown in Equation 7.

$$\%\, N_2 = \frac{V_{acid \times N}}{M_s} \times 1.4 \quad (7)$$

Where V_{acid} is the volume of sulphuric acid used and N is the normality of acid. Also, Ms is the mass of the sample used.

Determination of sulphur in the waste sample

Determination of sulphur in the waste samples were in compliance with ASTM D-5868-10a as also reported by Rominiyi et al, (2017); Equation 8 was used for the calculation of % S.

$$\%\, S = \frac{32W}{233X} \times 100 \quad (8)$$

Where X is the weight of waste sample taken; W is the weight of $BaSO_4$ precipitate formed.

Determination of Oxygen content in the waste sample

The percentage oxygen content was determined by subtracting the total percentages of carbon, hydrogen, nitrogen and sulphur from hundred as presented in Equation 9 (Atiku et al., 2024; Adekunle et al., 2015).

$$O\% = (100 - (C + H + N + S) \quad \ldots (9)$$

RESULTS AND DISCUSSION

Proximate Properties of Disposed sweet potatoes waste and dried okra stalk powder

This is to ascertain the suitability of the sweet potatoes and okra waastes (feedstocks) for the production of biocrude through hydrothermal liquefaction process. The result of the proximate analysis for the substrate (sweet potato waste) revealed a moisture content of 16.67%, which is above the values of 5.1% reported by Anukan *et al*. (2017), variation in these values of the moistures in the experimental samples may be due to the source of the substrates considered in the respective studies; high moisture significantly lower carbon burn rate. The sweet potato waste used in this work was characterized by ash content of 9.0 % which is above the value of 2.70% reported by Sulaiman *et al*. (2019). In addition to the aforementioned explanation, the sweet potato waste was characterized by 8.33% volatile matter and 60.66% fixed carbon. Furthermore, Okra waste revealed ash content of 9.67% which is also above the value reported by Sulaiman *et al*. (2019); higher fixed carbon of 66.00% was recorded which implies the potential of the feedstock as carbonaceous material for biocrude oil production via hydrothermal liquefaction process. Detail information on the Proximate Characterization of Coarse potatoes and Okra Powder is shown in Table 1.

Table 1: Proximate Characterization of Potato and Okra waste

I.D	Ash (%)	Moisture (%)	Volatile Matter (%)	Fixed Carbon (%)
A (Potato waste)	9.0	16.67	8.33	60.66
B (Okra waste)	9.67	20.00	9.67	66.00

Ultimate Characterization of the Samples

The oxygen content was obtained by difference as presented in Table 2, lower values of nitrogen (1.90%) and sulfur (0.08%) implied lower emission during combustion process. However, the high carbon content of 53.85% relative to percentage oxygen of 43.11.03% and hydrogen of 1.32% in the disposed waste coarse potatoes powder hence, revealed the potential of sweet potato waste as feedstock for biocrude oil production through hydrothermal liquefaction. Also, lower proportion of the oxygen (43.11%) and hydrogen (1.32%) in comparison with carbon content (53.59%) increases the energy value of a fuel; this explanation is in compliance with the literature by Kaur et al. (2024); Sulaiman *et al*. (2019) and Anukam *et al*. (2017). Furthermore, lower values of nitrogen (1.93%) and sulfur (0.106%) implied lower emission during combustion process. However, the high carbon content of 68.85% relative to percentage oxygen of 27.56% and hydrogen of 1.55% in the dried okra stalk powder revealed the potential of dried okra stalk as feedstock for biocrude oil production through hydrothermal liquefaction. The aforementioned explanation is in agreement with the literature by Atiku et al. (2024); Adekunle et al. (2015). Detail result on the ultimate characteristic of potatoes and Okra wastes is presented in Table 2.

Table 2: Ultimate Characterization of Potato and Okra wastes

I.D	C(%)	N (%)	H (%)	S(%)	O (%)
A (Potato waste)	53.59	1.90	1.32	0.08	43.11
B (Okra waste)	68.85	1.93	1.55	0.106	27.56

Thermogravimetric Analysis (TGA) of Waste Samples

Thermogravimetric analysis (TGA) using PerkinElmer Instrument is widely used to characterize and verify materials. TGA is applicable to most industries for the thermal analysis of materials. Environmental, food science, pharmaceutical, and petrochemical applications are the mainstay of thermogravimetric analysis and evolved gas analysis. Potato waste sample was run in static air from 30 °C to 950 °C. Scanning rate was 10 °C/minute. The red curve is the weight loss curve and its 1st derivative is the blue curve as shown in Figure 1. A better understanding of when thermal events occur enables a process to be optimized. A TGA thermal curve is displayed from left to right. The descending TGA thermal curve indicates a weight loss occurred. A 14.209 mg sample of Coarse potatoes powder was analyzed. Temperature Program = Heat from 30 °C to 950 °C @ 10 °C/minute in Nitrogen atmosphere with a purge rate of 20 mL/minute. As the temperature reaches 150 ^{o}C to 450 ^{o}C there was a corresponding weight loss due to thermal degradation of the sample as depicted in Figure 1. It was observed from Figure 1 that after 450 ^{o}C changes in the weight loss due to temperature effect was almost constant.

Furthermore, okra waste sample was run in static air from 30 °C to 950 °C. Scanning rate was 10 °C/minute. The red curve is the weight loss curve and its 1st derivative is the blue curve as shown in Figure 2. A better understanding of when thermal events occur enables a process to be optimized. A TGA thermal curve is displayed from left to right. The descending TGA thermal curve indicates a weight loss occurred. A 14.226 mg sample of Coarse potatoes powder was analyzed. Temperature Program = Heat from 30 °C to 950 °C @ 10 °C/minute in Nitrogen atmosphere with a purge rate of 20 mL/minute. As the temperature reaches 150 °C to 550 °C there was a corresponding weight loss due to thermal degradation of the sample as depicted in Figure 2. It was observed from Figure 2 that after 550 °C changes in the weight loss due to temperature effect was almost constant.

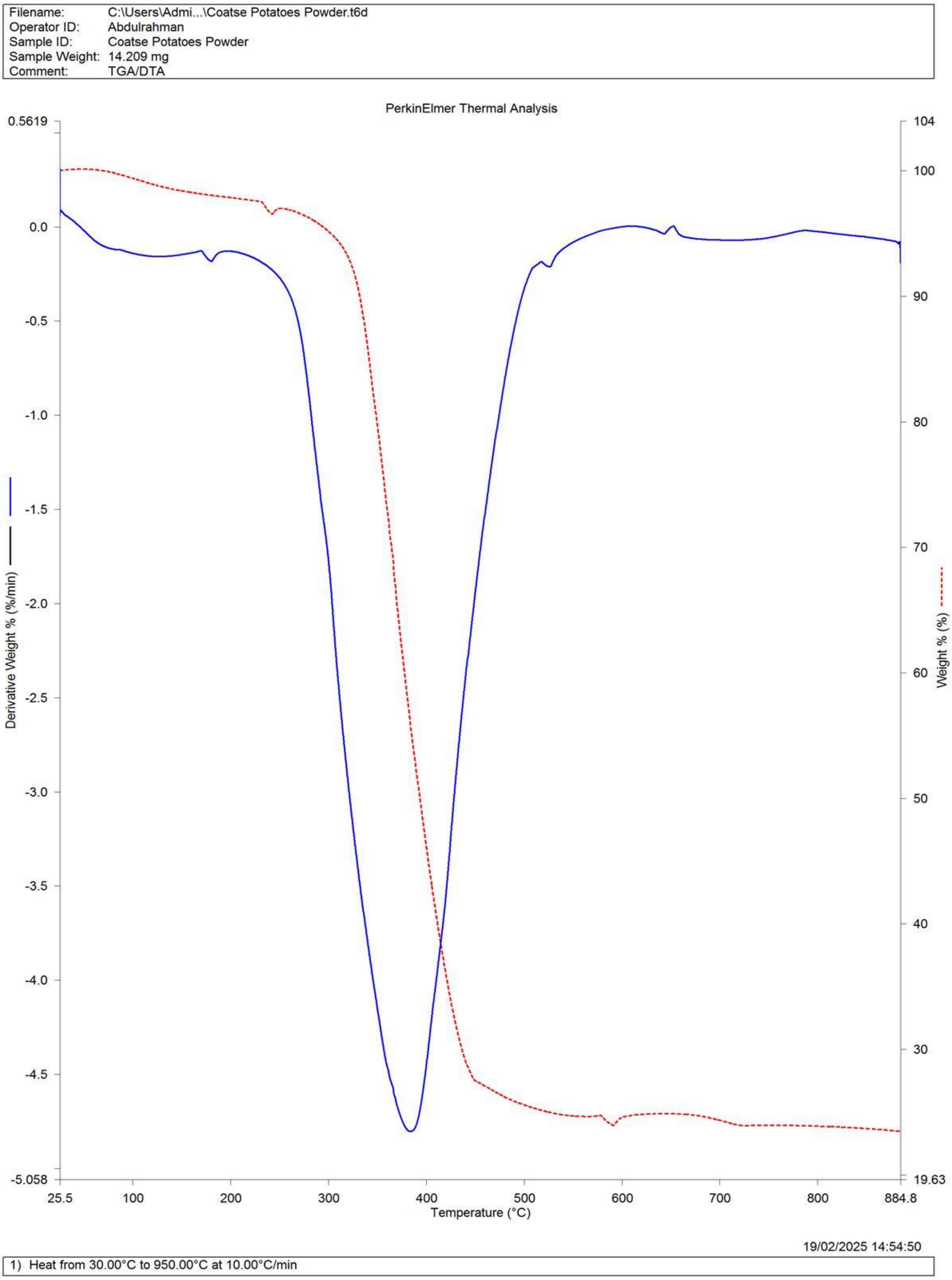

Figure 1: Thermogravimetric Analysis Curve for the Waste Potato Sample

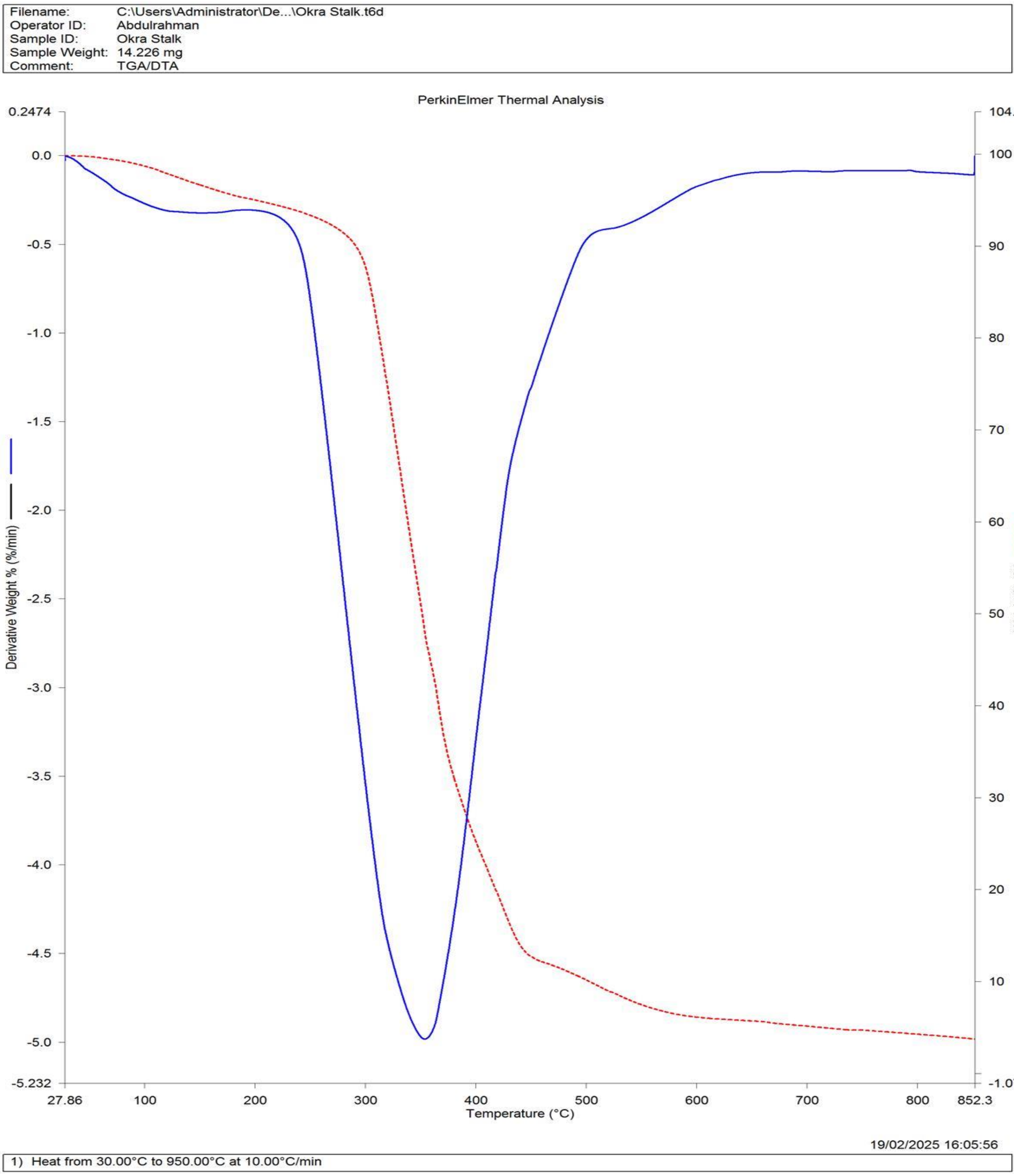

Figure 2: Thermogravimetric Analysis Curve for the Okra waste

Characterization via FTIR spectra

The infrared spectra of the Waste Potato were measured between 500 and 4000 cm^{-1} as presented in Figure 3.The absorption peak at 10213.814 cm^{-1} is assigned to C-O stretching band, as shown in Figure 3. The peak absorbance of the OH stretching band observed in the wide region of 3324.8 cm^{-1} is identified as OH stretch, while the peak at 1625.1 cm^{-1} is assigned to C = C stretching band; the peak at 775.3 cm^{-1} is assigned to C-C stretching band, and the result obtained in the present study is in compliance with the literature reported by Atiku et al. (2024) and Nisha & Vidya (2024).

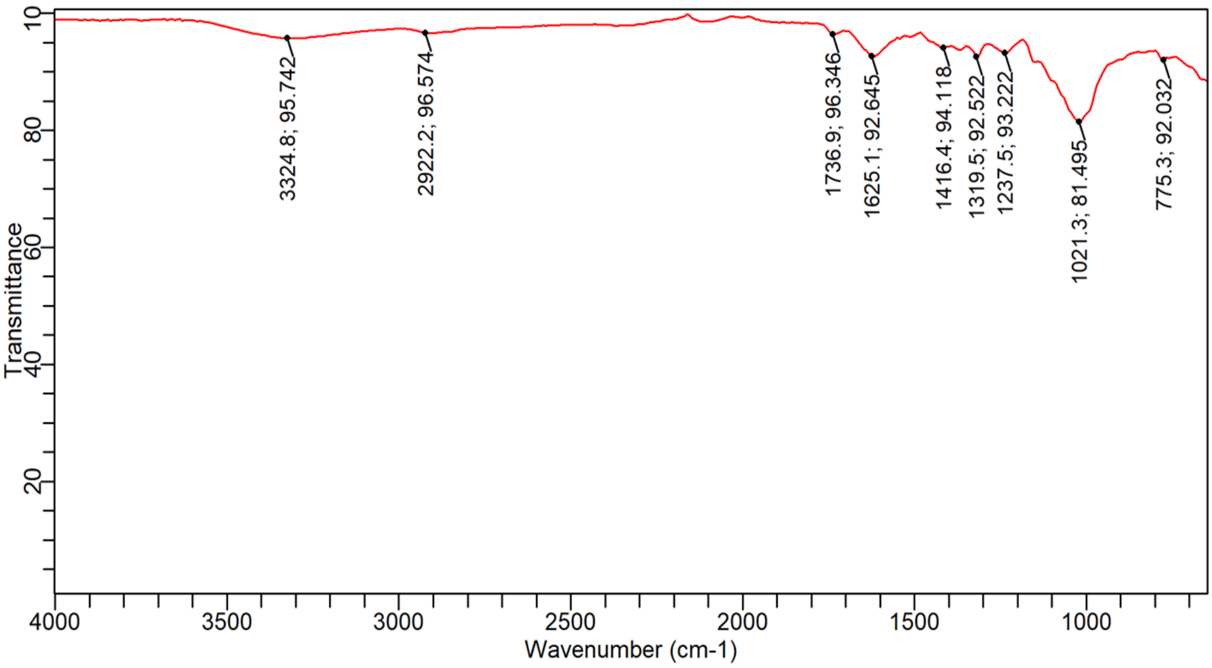

Figure 3: FTIR Spectra for the Waste Potato Powder

However, the infrared spectra of the okra waste were measured between 500 and 4000 cm^{-1} as presented in Figure 4.The absorption peak at 2102. 2 cm^{-1} is assigned to C≡C stretching band, as shown in Figure 4. The peak absorbance of the OH stretching band observed in the wide region of 3280.1 cm^{-1} is identified as OH stretch, while the peak at 998.9 cm^{-1} is assigned to C-C stretching band, and also, the result obtained in this study is in agreement with the literature reported by Atiku et al. (2024) and Nisha & Vidya (2024).

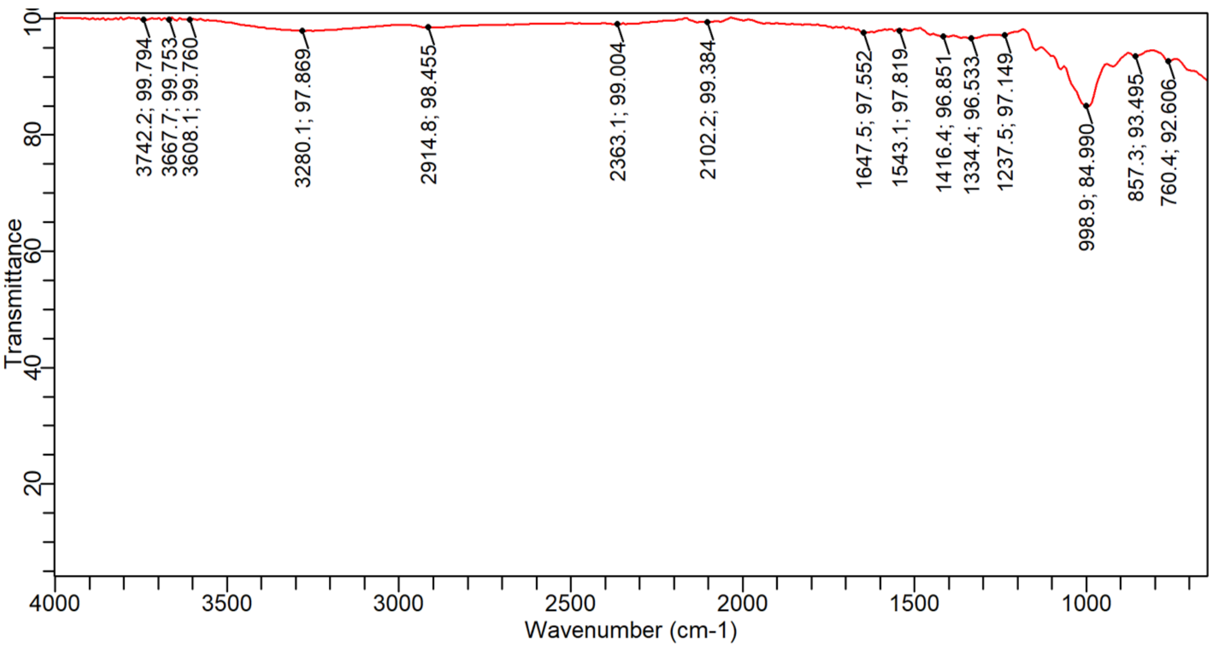

Figure 4: FTIR Spectra Okra waste

CONCLUSION

The characterization, via proximate analysis, for the sweet potato waste revealed ash content of 9.0 % 8.33% volatile matter and 60.66% fixed carbon hence, indicates the potential of the feedstocks for biofuel production. Also, Okra waste revealed ash content of 9.67%, higher fixed carbon of 66.00% was recorded which also implies the potential of the feedstock. For the ultimate analysis. In the same vein, the sweet potato waste revealed lower values of nitrogen (1.90%) and sulfur (0.08%) implying lower emission during combustion process; the okra waste revealed lower values of nitrogen (1.93%) and sulfur (0.106%) implied lower emission during combustion process. In addition, it was observed from the thermogravimetric analysis (TGA) result for feedstocks in the temperature range of 450 °C-550 °C, changes in the weight loss due to thermal degradation as a result of temperature effect was almost constant. Furthermore, the fourier transform infrared (FTIR) spectra revealed the presence of OH, C = C, C≡C, C-C and C-O functional groups at different stretching band C = C. Based on the aforementioned, the sweet potato and okra wastes can be used for the production of biocrude for fuel and other chemicals through hydrothermal liquefaction process.

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