

## PRODUCTION AND CHARACTERIZATION OF ALKALI EXTRACTS FROM PLANTAIN PEEL, COCOA POD HUSK, AND CASSAVA PEEL ASH FOR INDUSTRIAL APPLICATIONS

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### ABSTRACT

Growing demand for low-cost, eco-friendly substitutes for inorganic alkalis have driven interest in agricultural wastes as feedstocks. In this study, alkali extracts were produced and characterized from three agricultural residues: plantain peel, cocoa pod husk and cassava peel with the aim of developing sustainable substitutes for conventional alkalis while adding value to waste materials. To achieve this, the biomass samples were subjected to proximate analysis to determine their moisture and ash contents, while the derived alkali extracts were evaluated for pH, molarity, and elemental composition using standard analytical techniques. In addition, one-way ANOVA and regression analysis were employed to examine statistical relationships among the alkali properties. The results revealed significant variations in moisture content ( $70.3 \pm 0.78^c$  -  $79.8 \pm 0.49^d$  %) and yield of ash ( $5.1 \pm 0.23^d$  -  $8.8 \pm 0.20^d$  %) across the samples. Similarly, the final alkali solutions exhibited wide differences in molarity ( $0.01 \pm 0.00^b$  -  $0.80 \pm 0.01^f$  mol/dm<sup>3</sup>) and pH (9.77–11.46). Notably, plantain peel yielded the most effective alkali solution, attributable to its higher potassium and sodium contents, with cocoa pod husk ranking next, whereas cassava peel produced the lowest yield. FTIR results showed that all the volatile organic component are largely burnt off, and what remains is predominantly inorganic ash. These findings demonstrate that agricultural waste-derived alkali, particularly from plantain peel and cocoa pod husk can serve as low-cost and environmentally sustainable alternative to commercial alkalis, with practical applications in soap production and decomposition nitrocellulose-based propellants.

**Keywords:** Agricultural Wastes, Biochar, Potassium Hydroxide, FTIR, AAS, FES

### INTRODUCTION

The management of agricultural wastes has transitioned from a burden of disposal to frontier of green chemistry and industrial sustainability. In many developing economies like Nigeria, the accumulation of biomass residues such as plantain peels (*Musa paradisiaca*), cocoa pod husk (*Theobroma cacao*) and cassava peels (*Manihot esculenta*) poses environmental challenges if left to rot in open dumpsites. This research aim to develop sustainable substitutes for conventional alkalis while adding value to waste materials by characterizing alkali production from plantain peel, cocoa pod husk, and cassava peel. By evaluating physico-chemical parameters such as moisture content, ash content, pH, and alkali molarity, this study seeks to identify the most potent bio-alkali candidates for high-value industrial applications such as soap making, energy and explosives, food processing and pharmaceutical industry among others. Furthermore, it explores the correlation between the physical structure of the solid residues and the chemical behavior of their liquid extracts. However, these materials are rich in lignocellulosic structures and mineral components, specifically potassium and sodium, which can be harnessed to produce alkali (Ajayi et al., 2026; Eric et al., 2025; Ighashio & Obasuyi, 2022; Okunola et al., 2019; Pratama et al., 2024). According to Oliver-Simancas et al., (2026) the production of alkali from these local sources offers a renewable and cost-effective alternative to imported synthetic caustic soda and potash used in soap, detergent and textile industries. Agricultural by-products often constitute more than 70% of the total fruit weight, generated in massive quantities as waste for food processing. The transformation of these peels and husk into industrial alkali involves a two-stage process of combustion and leaching. During combustion, the

organic matter is oxidized, leaving behind a mineral-rich ash. The alkali primarily in form of potassium hydroxide (KOH) or sodium hydroxide (NaOH) is then extracted by leaching the ash with water (Komba et al., 2025, Okunola et al., 2019). Laverde-Albarracin et al, (2025) indicates that the quantity of the alkali produced are heavily dependent on the carbonization temperature study on comparative study of thermochemical valorization of cocoa shells. Characterizing these extracts is essential to determine their suitability for industrial applications. By valorizing these wastes, we not only mitigate environmental pollution but also support a circular economy where waste becomes a primary raw material for industrialization.

### MATERIALS AND METHODS

#### Materials

The materials and equipment used in this study included laboratory instruments for sample preparation, physicochemical analysis and instrumental characterization. The major equipment and their manufacturers are presented in Table 1. The primary materials used in this study included three agricultural wastes, namely: plantain peel, cocoa pod husk and cassava peel.

#### Reagents

The chemical reagents used for the extraction, and physico-chemical analysis for this study included 0.1M potassium hydrogen phthalate (KHP 99.5% purity), 60% perchloric acid (HClO<sub>4</sub> 99.7% purity), 60% nitric acid (HNO<sub>3</sub> 99.5% purity) and distilled water were sourced from Emmaco Chemicals, Kaduna State. All reagents were of analytical grade and used without further purification.

**Table 1: Equipment**

Name	Model	Manufacturer
Fourier Transform Infrared Spectrometer	PerkinElmer 175X	PerkinElmer, Inc.
Atomic Absorption Spectrometer	Thermo Finnigan	Thermo Fisher Scientific
Flame Emission Spectrometer	PerkinElmer	PerkinElmer, Inc.
Analytical sieve	ISO 3310-!ASTME1, 125mm	Gilson Company Inc.
Analytical balance	Mettler Toledo ME04E	Mettler-Toledo International Inc.
Heating mantle	Cole-Parmer HM100C	Cole-Parmer Instrument Company, LLC
Oven dryer	Jacket-heating, 20V 60 Hz	Thermo Fisher Scientific
Muffle furnace	Thermolyne small bench	Thermo Fisher Scientific
pH Meter	Mettler Toledo PH/ION S220	Mettler-Toledo International Inc.

**Sample Collection**

In this research, plantain peel and cassava peel were collected from Sabo market at Chikun LGA of Kaduna State, while cocoa-pod husk was collected from Oke Oja local market of Osun State, Nigeria. The study was executed in the Chemistry Department lab Kaduna State Polytechnic.

**Sample Preparation**

The collected samples were manually sorted to remove foreign materials such as stones, soil particles, plastics, and other contaminants. The samples were washed thoroughly with distilled water and allowed to oven-drying at 105 °C 2 hours. to remove surface moisture. The dried samples were crushed and ground using a mortar and pestle to obtain a fine powder. The plantain peel was labeled as (SP<sub>1</sub>), cocoa pod husk (SP<sub>2</sub>) and cassava peel (SP<sub>3</sub>).

**Determination of Moisture Content (MC)**

Moisture content of the samples was determined using the oven-drying method according to standard procedures recommended by the Association of Official Analytical Chemists (AOAC, 2020). Exactly 5 g of samples were weighed using Mettler Toledo analytical balance and recorded as w<sub>1</sub>, moisture is removed from the sample by drying at 105 °C 2 hours until constant weight is obtained and recorded as w<sub>2</sub>. The loss in mass during drying represents the moisture present in the sample and calculated from the equation below: (Khanolkar et al., 2024).

$$\% \text{ moisture} = \frac{w_1 - w_2 \times 100}{\text{Weight of sample}} \quad 1$$

Where

W<sub>1</sub> = Initial weight of crucible + Sample (g)

W<sub>2</sub> = Final weight of crucible + Sample (g)

Weight of sample = 5 g.

**Determination of Ash Yield**

The ash yield of the samples were determined using the dry ashing method in a muffle furnace according to standard analytical procedures (Mauer, 2024). A mass of 5 g was weighed into crucibles and recorded as w<sub>1</sub>. The crucible with the samples were placed in a muffle furnace and heated gradually to 550 °C. The samples were allowed to ash for 4 hours until the residues turned light grey, indicating complete combustion of organic matter. The crucibles were carefully removed from the furnace using crucible tongs and placed in a desiccator to cool to room temperature. After cooling, the crucibles containing the ash were weighed and recorded as W<sub>2</sub>. The experiment was conducted in triplicate and the average value was reported. Ash content was calculated using the equation:

$$\text{Ash Content (\%)} = \frac{w_1 - w_2 \times 100}{\text{Weight of sample}} \quad 2$$

Where

W<sub>1</sub> = Initial weight of crucible + Sample (g)

W<sub>2</sub> = Final weight of crucible + Sample (g)

Weight of sample = 5g

**Extraction of Alkali from Ash Samples**

This method is based on water-leaching of ash, which dissolves water-soluble alkali salts present in biomass ash (Uzor et al., 2024). Due to furnace capacity limitations, biomass sample were ashed in batches of 5 g. The ashing process was repeated multiple times, and the resulting ash samples were pooled to obtain the required quantity equivalent to 50 g of the original biomass for alkali extraction. Exactly 50 g of each sieved ash samples were weighed using an analytical balance and transferred into a 500 cm<sup>3</sup> conical flask and labelled SP<sub>1</sub>, SP<sub>2</sub> and SP<sub>3</sub>. A volume of 300 cm<sup>3</sup> distilled water was added to the ashes. Then the mixtures were heated gently on an electric heating mantle at 100 °C for 2 hours. Heating continued for 2 hours until dissolution of soluble alkali compounds. Eventually, mixtures were allowed to cool slightly and filtered using Whatman No. 44 filter paper and a funnel. The filtrates were collected in a clean 500 cm<sup>3</sup> beakers. The alkali extracts solution was transferred into a labeled reagent bottle and store for further pH determination, alkalinity determination and elemental analysis.

**Alkali Molar Content of Extracts (AMC)**

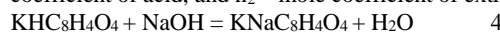
The alkali molar content of a biomass samples was determined by using the extracted alkali solutions and standardizing the alkali extract solutions. The standardization of the ash extracts was carried out according to Zauro et al. (2016). Exactly 5 cm<sup>3</sup> of 0.1 M KHP solution was accurately measured in a clean conical flask and 2 drops of phenolphthalein indicator was added. Each ash extracts were filled in the burette and titrated against 5 cm<sup>3</sup> of 0.1 M KHP solution in a 250 cm<sup>3</sup> conical flask until the first faint pink colour appeared. The titration was repeated two more times using a fresh sample of 5 cm<sup>3</sup> of 0.1 M KHP solution and the average titre values were determined. Molarity of each alkali extracts were determined from the titration:

$$\frac{C_1 \times V_1}{n_1} = \frac{C_2 \times V_2}{n_2} \quad 3$$

Where

C<sub>1</sub> = concentration of acid (mol/dm<sup>3</sup>)

V<sub>1</sub> = volume of acid (cm<sup>3</sup>), C<sub>2</sub> = concentration of extract (mol/dm<sup>3</sup>), V<sub>2</sub> = volume of extract (cm<sup>3</sup>), n<sub>1</sub> = mole coefficient of acid, and n<sub>2</sub> = mole coefficient of extract



**Determination of pH of Ash Extracts**

The pH of each ash extracts were determined on a Benchtop Mettler Toledo pH/MV meter according to Zauro et al. (2016). Each of the 10 cm<sup>3</sup> ash extracts were measured in a 250 cm<sup>3</sup> beaker. A calibrated pH meter electrode was dipped into the samples. The pH of the alkali extracts were recorded.

**Test for the Alkalinity of the Ash Extract**

An alkalinity test was done by the use of a red litmus paper (Igbashio & Obasuyi, 2022). A strip of red litmus paper was dipped into each of the ash extracts, the colour change was observed.

**Determination of Metal Content**

Metal concentrations of ash samples (K, Na, Fe, Mg, Mn, Zn, Cu, Ni, Co, Pb, and Cd) were determined using Atomic Absorption Spectroscopy (AAS) and Flame Emission Spectroscopy (FES) after wet digestion. AAS was carried out with using a flame atomic absorption spectrometer equipped with hollow cathode lamp specific to each element within the region 190-900 nm with a recovery standard of 100±5%. FES analysis was performed at characteristics emission wavelength of Na, 589 nm and K, 766.5 nm. Exactly 1.0 g of the powdered agricultural waste sample was taken in digesting glass tube and 12 cm<sup>3</sup> of nitric acid HNO<sub>3</sub> was added to the samples and mixture was kept for overnight at room temperature. Then 4.0 cm<sup>3</sup> perchloric acid (HClO<sub>4</sub>) was added

to the mixture and kept in the fumes block for digestion. The temperature was increased gradually, starting from 50°C and increasing up to 250-300°C. The digestion was completed in about 80 mins as indicated by the presence of white fumes. The wet digested solution were transferred to plastic bottles and used for metal determination (Odiko et al., 2024).

**Statistical Analysis**

All experiments were conducted in triplicate. One-way ANOVA and Tukey post hoc tests were applied (p < 0.05) to identify moisture content, ash yield, alkali molar content, and pH group differences. Correlation and regression analyses evaluated relationships between alkali properties.

**RESULTS AND DISCUSSION**

The results revealed clear variations in moisture content, ash yield, alkali molarity, and pH across the agricultural residues, demonstrating that feedstock type strongly influences alkali extraction potential. Statistical analysis of the parameters revealed significant differences (p<0.05).

**Table 2: Results of Proximate Analysis of Agricultural Wastes**

Organic waste	MC (%)	Ash Yield (%)	AMC (mol/dm <sup>3</sup> )	pH	Alkalinity
Plantain peel	79.8 ± 0.49 <sup>d</sup>	5.1 ± 0.23 <sup>d</sup>	0.80 ± 0.01 <sup>f</sup>	11.46	Blue
Cocoa pod husk	71.5 ± 0.85 <sup>c</sup>	8.6 ± 0.17 <sup>c</sup>	0.22 ± 0.01 <sup>e</sup>	10.99	Blue
Cassava Peel	70.3 ± 0.78 <sup>c</sup>	8.8 ± 0.20 <sup>d</sup>	0.01 ± 0.00 <sup>b</sup>	9.77	Blue

Plantain peel exhibited the highest moisture content (79.8 ± 0.49%), confirming its highly perishable nature and the need for prompt processing after collection. While this value aligns broadly with previously reported ranges 74% and 84% Jumare, et al., (2022) and 78.4 % by Umoh, (2024) the consistency across studies suggests that plantain peel inherently retains high water content regardless of source. In contrast, cocoa pod husk and cassava peel showed slightly lower but comparable moisture levels respectively, indicating that high moisture is a common characteristic of these biomass types. However, Irma et al., (2024); Laverde-Albarracin et al., (2025) reported the much lower values (25.63 ±0.30 %; 12.71%) highlight the strong influence of drying conditions, geographical origin, and post-harvest handling on moisture determination. Ash yield showed an inverse trend, with cassava peel recording the highest value, while plantain peel had the lowest. Rather than simply reflecting compositional differences, this suggests that cassava peel contains a higher proportion of inorganic minerals, which could enhance its suitability for ash-based applications. However, the relatively lower ash content of plantain peel does not diminish its performance, indicating

that alkali quality depends more on the type of mineral constituents than on total ash yield alone. Study by Esan et al., (2023) recorded 6.1% which conform to the observed value. Particularly, plantain peel demonstrated the highest alkali molarity and pH, significantly exceeding those of cocoa pod husk and cassava peel. This finding highlights its superior capacity for alkali generation and suggests a higher concentration of active alkaline metals such as potassium and sodium. Unlike previous studies that primarily reported comparable values, the present work establishes a clearer performance hierarchy among the biomass types, with plantain peel consistently outperforming the others. This strong alkalinity confirms its suitability for industrial applications. The consistent alkaline behavior of all extracts, as confirmed by the change of red litmus paper to blue, further validates their potential as alternative alkaline materials, although their effectiveness varies significantly depending on the biomass type.

**Metal Composition**

The metal composition of the biomass samples is shown in Table 4.

**Table 4: Results of Metal Content**

Samples	Plantain Peel	Cocoa Pod Husk	Cassava Peel
K (mg/kg)	1499.1 ± 13.91 <sup>c</sup>	1679.8 ± 38.13 <sup>f</sup>	259.8 ± 6.00 <sup>b</sup>
Na (mg/kg)	997.9 ± 9.19 <sup>f</sup>	1499.0 ± 18.51 <sup>g</sup>	149.5 ± 1.85 <sup>c</sup>
Mg (mg/kg)	158.1 ± 1.06 <sup>f</sup>	384.3 ± 2.56 <sup>g</sup>	71.3 ± 0.72 <sup>b</sup>
Fe (mg/kg)	430.3 ± 1.06 <sup>f</sup>	788.3 ± 4.36 <sup>g</sup>	63.9 ± 0.70 <sup>b</sup>
Cu (mg/kg)	11.9 ± 0.36 <sup>b</sup>	69.0 ± 0.86 <sup>e</sup>	11.0 ± 0.35 <sup>b</sup>
Zn (mg/kg)	72.1 ± 0.15 <sup>c</sup>	176.3 ± 1.21 <sup>f</sup>	58.5 ± 0.47 <sup>d</sup>
Pd (mg/kg)	60.2 ± 0.21 <sup>e</sup>	65.9 ± 0.33 <sup>f</sup>	20.1 ± 0.18 <sup>b</sup>
Ni (mg/kg)	5.1 ± 0.12 <sup>d</sup>	27.0 ± 0.25 <sup>g</sup>	9.9 ± 0.09 <sup>f</sup>
Mn (mg/kg)	259.1 ± 0.12 <sup>f</sup>	485.0 ± 0.79 <sup>g</sup>	67.1 ± 0.15 <sup>c</sup>
Cd (mg/kg)	1.0 ± 0.03 <sup>d</sup>	2.1 ± 0.09 <sup>e</sup>	0.2 ± 0.06 <sup>b</sup>
Co (mg/kg)	5.0 ± 0.06 <sup>c</sup>	10.8 ± 0.20 <sup>e</sup>	7.9 ± 0.06 <sup>d</sup>

Values are Presented as Mean ± SEM and Values with Different Superscript (Letter) Within the Same Column (Between Groups) Indicate Statistically Significant Difference (p<0.05) at 95% Confidence Interval

Potassium and sodium were the predominant elements in all biomass samples, confirming their key role in alkali formation. Cocoa pod husk and plantain peel showed higher concentrations of these alkali metals, which explains their superior alkali extraction performance compared to cassava peel. The observed metal composition followed the trend  $K > Na > Fe > Mg > Mn > Zn > Cu > Pb > Ni > Co > Cd$ . Although elemental distributions generally agree with literature as reported by Gopal et al., (2025) and slightly different from  $K > Ca > Mg > Si > S > Al > Mo > Ba > Co > Pd$  as reported by Kone et al., (2020). Observed variations reflect differences in biomass origin, composition, and processing conditions. The high potassium content is attributed to its accumulation in plant tissues, particularly in fruit peels, during growth (Kouadio et al., 2025). Generally, the low levels of heavy metals indicates all agricultural wastes used in this study are environmentally friendly and high alkali-forming potential highlight these agricultural wastes, especially cocoa pod husk and plantain peel as promising, safe, and sustainable sources of industrial alkali.

**FTIR Characterization of Dried and Ash Sample**

The FTIR spectra of the dried biomass samples reveal characteristic functional groups typical of lignocellulosic materials as shown in Figure 1, 3 and 5. Broad bands observed around 3223–3324  $cm^{-1}$  are attributed to O–H stretching vibrations of hydroxyl groups present in alcohols, phenols, and carbohydrates, while peaks at 2922–2818  $cm^{-1}$  correspond to aliphatic C–H stretching of  $-CH_2$  and  $-CH_3$  groups. The strong absorption in the range 1722–1702  $cm^{-1}$  indicates C=O stretching associated with esterified pectin and

other carbonyl-containing compounds, whereas bands near 1602–1605  $cm^{-1}$  and 1461–1315  $cm^{-1}$  reflect carboxylate groups, aromatic structures, and carbohydrate backbone vibrations. The fingerprint region (1200–945  $cm^{-1}$ ) further confirms the presence of polysaccharides through C–O–C and C–O stretching, collectively indicating that the raw biomass is rich in cellulose, hemicellulose, and pectin-based components. While these spectral features are consistent with previous studies by (Fabricia et al., 2024; Kaur et al., (2022); Lei et al., (2025); Sathesh et al., 2020) and their simultaneous occurrence across all samples reinforces the typical lignocellulosic nature of the selected agricultural wastes.

After ashing at 550 °C, the FTIR spectra show a marked reduction in organic functional groups and a shift toward inorganic signatures as revealed in figure 2, 4 and 6. The diminished intensity of O–H and C=O bands, alongside the persistence of weaker C–H signals, indicates substantial thermal degradation of organic constituents such as cellulose, hemicellulose, lignin, and proteins, an observation which align with literature (Chamorro et al., 2024; Kawiya, et al 2021; Promraksa & Rakamak 2020). In contrast, bands observed within 1461–1327  $cm^{-1}$  and 860–801  $cm^{-1}$  regions are associated with metal oxides, carbonates, phosphates, and silicate structures, confirming the transformation of biomass into predominantly inorganic ash. This transition highlights the effectiveness of the ashing process in removing volatile organic matter and concentrating mineral components, which are essential for alkali formation. Overall, the FTIR results provide clear evidence of the structural shift from organic-rich biomass to mineral-dominated ash, supporting the observed alkali properties of the samples.

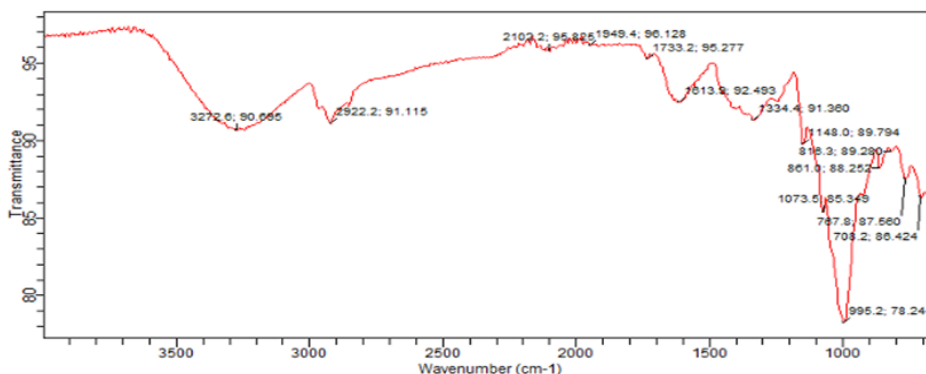


Figure 1: FTIR Spectrum of Dried Plantain Peel Sample

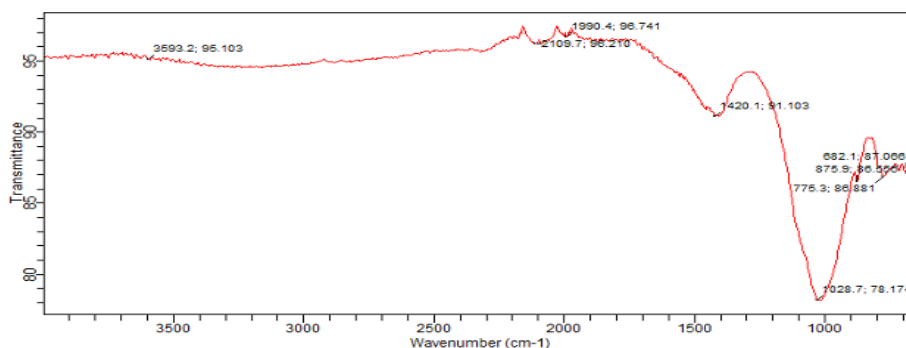


Figure 2: FTIR Spectrum of Plantain Peel Ash

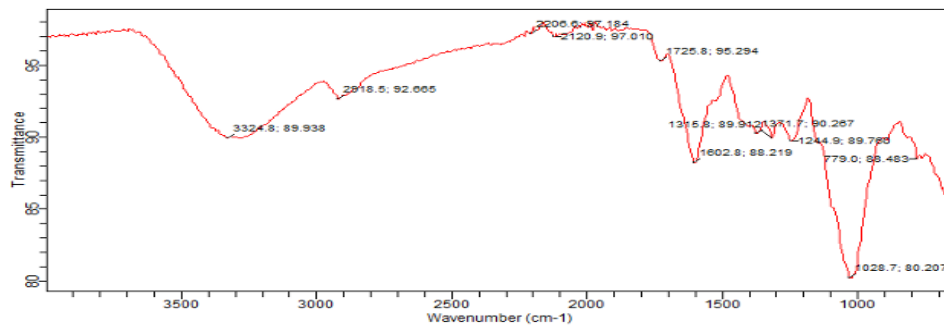


Figure 3: FTIR Spectrum of Dried Cocoa Pod Husk

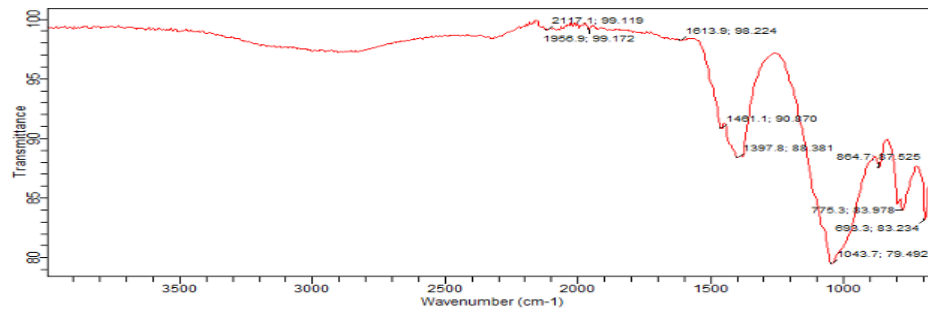


Figure 4: FTIR Spectrum of Cocoa Pod Husk Ash

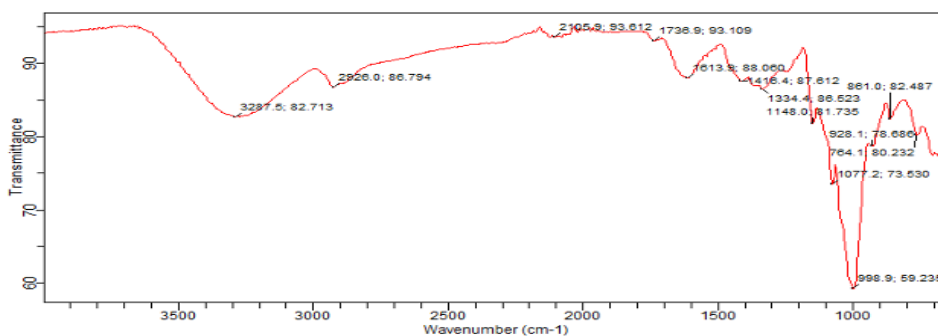


Figure 5: FTIR Spectrum of Dried Cassava Peel Sample

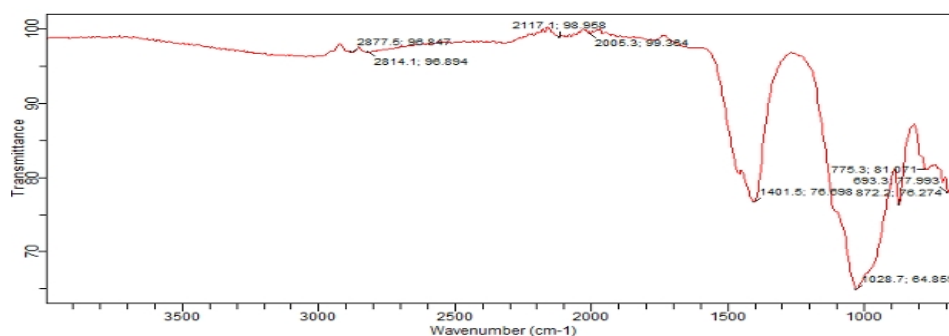


Figure 6: FTIR Spectrum of Cassava Peel Ash

**Correlation and Regression**

Strong positive correlations were observed between AMC and pH ( $r > 0.80$ ). Regression models confirmed alkali content as a significant predictor of potential use for industrial purposes.

**CONCLUSION**

This study demonstrates that agricultural residues like plantain peel and cocoa pod husk are viable, sustainable sources for industrial alkali production. Characterization revealed high potassium and sodium concentration levels, with plantain peel recording the highest value ( $0.80 \pm 0.01$

mol/dm<sup>3</sup>) confirming their potency for applications such as soap making and safety testing. Utilizing these wastes supports a circular economy, offering a cost-effective, eco-friendly alternative to synthetic alkalis.

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