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Preparation and Characterization of Organic Potash from Agricultural Wastes in Food and Fertilizer Applications: A Case Study of Corncob and Guinea Corn Husk

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ABSTRACT
Rapid growth in the world's population has caused a corresponding increase in the demand for food, shelter and social amenities. This in turn has encouraged the generation of huge quantities of agricultural wastes in the environment. The research is aimed at the production of organic potash from agricultural wastes ash via evaporation technology, and characterizing the same product using XRF, XRD and FTIR. Corncobs and guinea corn husks were pretreated with clean tap water and ashed in an incinerator to white ash. The white ashes freed from pieces of charcoal were soaked in clean tap water and then leached out the soluble components into an alkaline solution. The respective alkaline solutions were boiled to evaporation at 100 ^{o}C under continuous stirring and finally turned whitish or greyish/reddish brown or blackish powders on cooling. XRF analysis showed that the compositions of guinea corn husk and corncob potash is a potassium-based (K_2O) salt having 61.63% and 73.33% concentrations, respectively. The XRD analysis showed that the minerals present in the potash produced were mixtures of the chlorides of Na and K and oxides of Si, Mg and Ca with potassium having the highest chemical compositions of 31% and 66%, respectively. The FT-IR spectra analysis identified strong bands of potash with vital functional groups of O-H, C-H, N-H and C-O. Therefore, agricultural wastes are potential raw materials for the production of organic potash.

Keywords: Corncob, guinea corn husk, potash, food, fertilizer, XRF, XRD, FTIR

INTRODUCTION
Potash fertilizers play a dynamic role in present-day agriculture, providing essential nutrients such as potassium to crops for healthy growth and enhanced yields in a world with a growing population and shrinking available land. As the world population continues to grow, the demand for food production increases, thrusting the need for effective and sustainable fertilizer solutions.

Open air burning of agricultural wastes in open fields often constitutes serious pollution to the environment couple with further destruction on soil nutrients and microorganisms. Subsequently, mining of inorganic potash from the ground leads to serious problems such as; reduction in farm land, swamping and enormous cost of beneficiation which all results in high cost of potash. Therefore, production of organic potash from agricultural wastes ash (i.e. guinea corn husk and corncob) presents an alternate means of environmental hygiene, wealth creation and a good waste management disposal system.

The rapid growth in the world's population has culminated in increased demand for food, shelter and social amenities which in turn has led to the generation of large quantities of agricultural wastes in the environment. Agricultural waste are by-products of agricultural production following the different harvesting activities carried out within the period. Agricultural wastes are commonly available, nontoxic, renewable and almost free; hence they can be a potential material for value added products [1-4]. They constitute a significant percentage of environmental pollution worldwide, due to greenhouse gas effects on the environment and society at large. Researchers have been focused on the conversion of these agricultural wastes into value added products. Agricultural wastes include manure and animal carcasses (animal waste); corn stalks, corncob, rice and hungry rice stalks, wood, sugar cane bagasse, drops and culls from fruits and vegetables, and crop waste [1], crop residues (residual stalks, straw, leaves, roots, husks and shells) and animal waste [3]. By implication, an increase in agricultural production will significantly increase the amount of agricultural wastes in fields with an estimated value of about 998 million tonnes of agricultural waste produced yearly [2, 5].

Plates 1(a-d): shows images of corncob, corncob ash, guinea corn husks and guinea corn husk ash.

a. Corncob

b. Corncob ash

c. Guinea corn husks

d. Guinea corn husk ash

Plates 1(a-d): Shows images of corncob, corncob ash, guinea corn husk and guinea corn husk ash.

Generally, agricultural wastes are either burnt, incinerated or landfilled. Combustion incineration process still happens to be the most acceptable method of agricultural waste disposal and accounts for more than 95% of all agricultural waste handling today [2]. Most of these agricultural wastes are under-utilized, and left to rot or subjected to open-air burning with its accompanying environmental consequences such as greenhouse gas effects and degradation or loss of nutrients in soil [6, 7].

By employing suitable conversion technologies, agricultural wastes can be transformed into useful products like potash, fertilizer, biogas and animal feed. The use of firewood often results in the accumulation of wood ash in most rural homes. According to Kozicki [8], wood ash is a valuable asset used as manure (green fertilizer) to increase crop yield, food preservation, cooking food, wounds healing, explosives powder, pest control, black soap production, pottery, stain and odour removers, and even cosmetics.

Before the industrial revolution, potash was processed by leaching the filtrate from wood ash in a pot. The filtrate obtained was either used to produce black soap, stain and odour removal, and explosive powder. It was then evaporated by boiling to produce crystalline salt known as potash (K_2CO_3). According to Sarkar et al [9] in their study, discovered that organic potash is the best micronutrient for sustainability of soil health and crop yield rate which is far better than inorganic potash.

Organic potash, also known as botok ranrinya in (Koro), Kaun (Yoruba), Akanwa (Igbo), or Kanwa (in Hausa) does not only improve soil fertility but also increase crop output. Potash is a colourless and odourless crystalline residue obtained from burning and processing organic wastes matter. Potash has broad areas of applications ranging from house-hold uses (food seasoning and condiments) and has also been found as a relief to hypertensive patients. In the agricultural sector, potash is one of the sources of fertilizer and animal feed production; while in the industries, potash is used for the manufacture of soaps, glass, baked goods and gunpowder, brewing of beer, textiles, catalyst for synthetic rubber manufacturing, and pharmaceuticals [10]. Global potash consumption in 2021 for fertilizers production was estimated to have increased to 45 million tons from 44 million tons as recorded in 2020 [11]. This potash demand has been on the increase due to high population growth. Asia and South America are the leading consuming countries.

According to Koester [12], the composition of K_2CO_3 in wood ash is 14-19 %, also ash from other sources such as corn stalks, rice straws, potato weed, beanstalks, corncobs, vines, seaweed, reeds, hungry rice stalks are suitable for the production of organic potash. Potash fertilizers are progressing with an attention to sustainability, efficiency, and exactness in agriculture since the source of raw materials is renewable.

The increasing demand for food security and health challenges as a result of uptake of chemical fertilizers drives the need for effective organic fertilizers production, while technological advancements improve production processes. Sustainable agriculture practices promote the use of environmentally friendly potash fertilizers, and strict agriculture optimizes nutrient application. The growth of organic potash and the area of knowledge of fertilizers offer sustainable options for farmers, and the global expansion of potash production ensures a stable supply for growing economies. As these trends continue to influence the potash production from agricultural wastes in food and fertilizer applications in sustainable and efficient agriculture remains essential for feeding the world's growing population [13].

The novelty of the study is that the process for the production of organic potash from agricultural wastes incorporated a stimulant to enhance the process and the product output of organic potash. The aim of this study was to produce organic potash from agricultural waste materials (guinea corn husks and corncob) and to determine the chemical compounds.

MATERIALS AND METHODS

The equipment used for the research work include: incinerator, hot plate, oven, weight balance, and muffle furnace.

Material Sourcing and Preparation

The materials for this study are corn cobs, guinea corn husks, and tap water. These items were obtained from National Research Institute for Chemical Technology, (NARICT), Zaria, Kaduna State. The corncobs and guinea corn husks were pre-treated with tap water to remove foreign materials and sun dried for 48 hours and then ashed in an incinerator to white ash. The white ashes were freed from any pieces of charcoal and 140 g of the ash was recovered from 26 kg of corncobs, while that of the guinea corn husks ashes 128 g was recovered from 23 kg of guinea corn husks. Both powder were separately mixed with clean tap water and leached to dissolve out the soluble components into an alkaline solution. The method adopted for the research work was that of Sarkar et al [10]. The respective alkaline solutions were boiled to evaporation at 100 ^{o}C under continuous stirring until it solidified. Finally, the dried whitish solid potash weighed 85 g and 103 g for corncob and guinea corn husks, respectively. The potash produced from the ashes are hydroxides of alkali metals (Na or/and K) from XRF analysis results obtained, but in most cases, it contains other water-soluble non-alkali substances such as calcium, chloride, nitrate and sulphate salts [10]. The whitish powders were packed in a dry container and allowed to cool. In some instances, potash could be greyish/reddish brown or blackish in colour due to the presence of trace elements or other impurities [13, 14].

Plate 2 shows samples of organic potash from guinea corn husks and corncob.

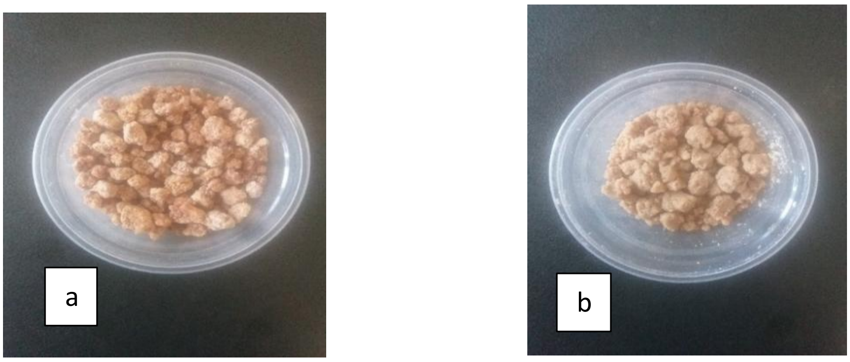

Plate 2 (a- b): Samples of organic potash produced from guinea corn husks and corncob

X-Ray Fluorescence (XRF) Analysis of Organic Potash derived from Guinea Corn Husks and Corn cobs

The XRF analysis of guinea corn husks and corn cobs potash was carried out using Genius IF – Xenemetrix XRF equipment. The samples were crushed and pulverized to 75 µm for XRF analyses. The pulverized aliquot was analyzed using the equipment. Duplicate analyses performed to obtain average values as shown in Table 1.

X-Ray Refractive Diffractometer (XRD) Analysis of Organic Potash obtained from Guinea Corn Husks and Corn cobs

The XRD analysis of guinea corn husks and corn cobs potash were carried out at National Steel Raw Materials Exploration Agency (NSRMEA), Kaduna, Nigeria. The samples were crushed and split into two equal halves. One crushed portion was pulverized to 100% passing through 75 µm sieve, split into two portions with one portion used for XRD analysis while the other half was kept as a reference sample. The pulverized fraction was analyzed using Rigaku Miniflex 600 XRD equipment employing Cu-Kα radiation at 2theta angle $2^o - 70^o$.

The results were presented as peak positions at 2θ and x-ray counts in the form of a spectra that reveals the compound's name, mineral name, chemical formula, empirical formula, and with the crystallographic parameters.

The crystalline phases, chemical formula and concentrations detected by XRD for guinea corn husks and corn cobs potash are as shown in Tables 2 and 3 with their quantitative values and accompanying diffractogram shown in Figures 5 and 6, respectively.

Fourier Transform Infra-Red (FTIR) Analysis of Organic Potash Derived from Guinea Corn Husks and Corn cobs

Fourier Transform Infrared spectroscopy (FTIR) is a technique that uses infrared light to observe properties of solid, liquid, or gas to detect functional groups and characterized bond formation in a sample. Analysis was carried out using Shimadzu (Japan) – 8400S equipment.

RESULTS AND DISCUSSION

Table 1 shows the results of XRF of potash from guinea corn husk and corncob. It revealed the potash produced from guinea corn husks and corncob was potassium-based potash haven the highest percentage concentrations of K_2O (61.63% and 73.33%).

Table 1: XRF results of guinea corn husks and corn cobs potash in percentage concentrations composition of elements (%).

S/N	Sample	SiO_2	V_2O_5	Cr_2O_3	Nb_2O_3	Fe_2O_3	CuO	K_2O	WO_3	MgO	Ag_2O	Al_2O_3	ZnO	Ta_2O_5	ZrO_2	Co_3O_4	MnO	SnO_2	Cl	SO_3	P_2O_5	BDL
1	A	3.51	0.01	0.01	0.05	0.13	0.06	61.63	0.01	10.01	7.59	4.15	0.04	0.01	0.01	0.01	0.01	6.76	4.42	0.89	-	0.69
2	B	4.21	0.003	0.01	0.01	0.19	0.05	73.33		3.52	0.02	3.84	0.01	0.01	0	0.02	0.01	4.19	4.89	0.80	4.53	4.76

A and B represent organic potash from guinea corn husks and corn cobs respectively. BDL represnts Below detection limit

Table 1 shows that potash produced from different sources contain varying levels of chemical composition. The XRF analysis of the compositions of guinea corn husk and corncob potash showed that it is a potassium-based salt having the highest percent concentrations in the two source agricultural wastes. The result obtained agreed with the findings of Shagal [15].

Tables 2 and 3 show the XRD results of guinea corn husks and corncob potash. The XRD determines the empirical formula, chemical formula and mineral type contained in the potash. It also revealed that the potash produced was potassium based (KCl) and had the highest percentage concentrations in both samples.

Table 2: Chemical formula of the mineral phases (%) as determined by XRD analysis of guinea corn husks potash

S/N	Mineral	Chemical Formula	Concentration (%)
1	Quartz	SiO_2	2.0
2	Periclase	MgO	12.0
3	Sylvine	KCl	31.0
4	Sodalite	$Na_4Al_3ClSi_3O_{12}$	28.0
5	Lime	CaO	27.0

Table 3: Chemical formula of the mineral phases (%) as determined by XRD analysis of corn cobs potash

S/N	Mineral	Chemical Formula	Concentration (%)
1	Quartz	SiO_2	10.0
2	Periclase	MgO	8.0
3	Sylvine	KCl	66.0
4	Sodalite	$Na_4Al_3ClSi_3O_{12}$	3.0
5	Lime	CaO	13.0

Figures 1 and 2 are the spectra for the chemical formula and compound names for the guinea corn husk and corncob potash. The spectra revealed that the potash produced after thermal combustion of guinea corn husk and corn cob in an incinerator into ashes under normal atmospheric pressure contains different phases of minerals.

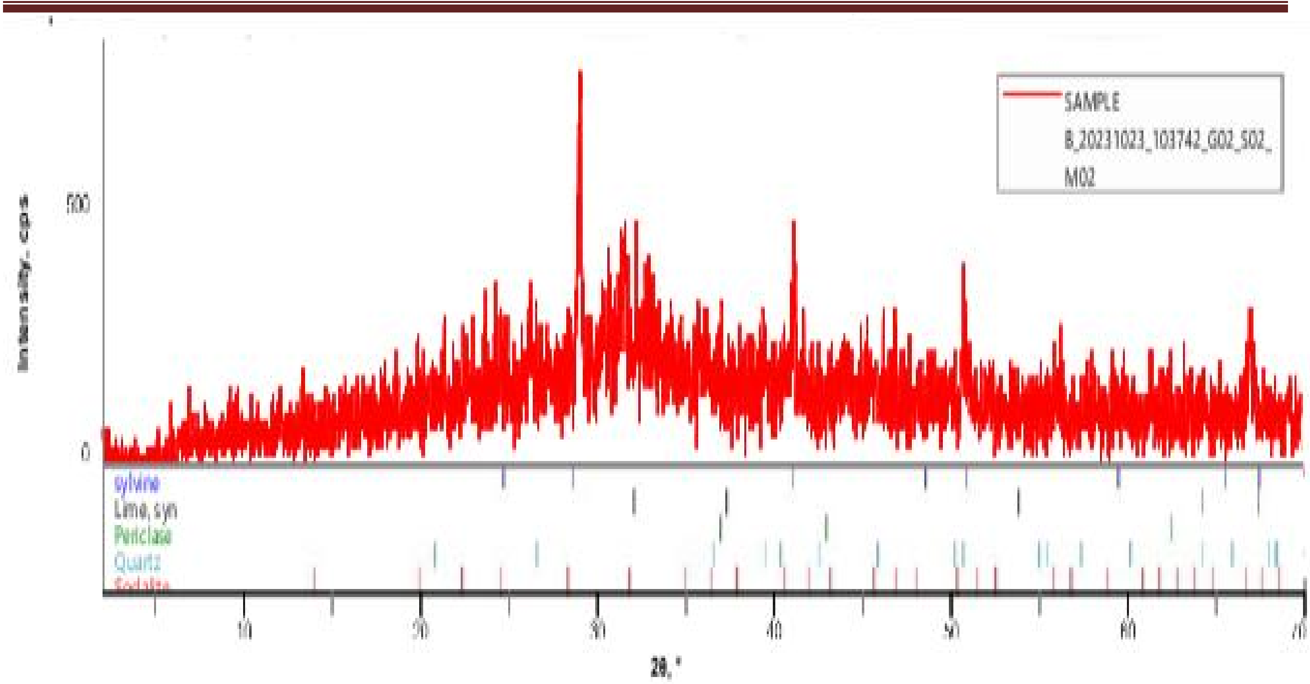

Figure 1: Guinea corn husk potash XRD spectrum

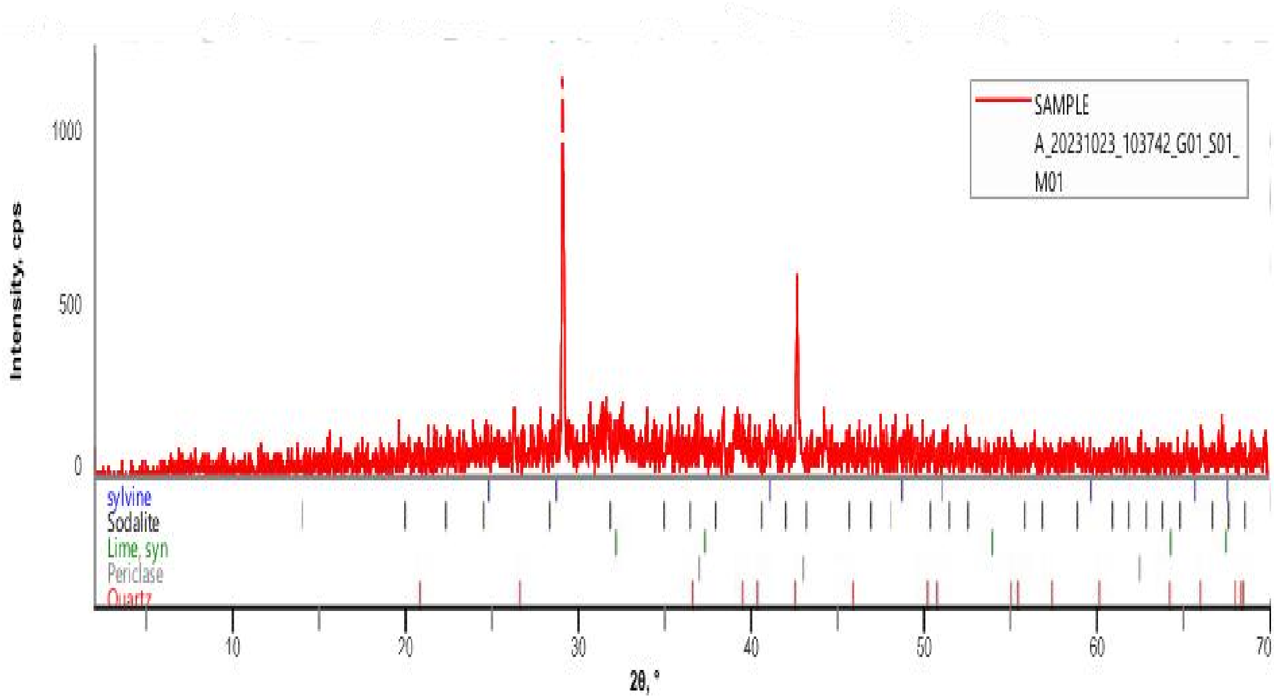

Figure 2: Corn cob potash XRD spectrum

From the minerals present in the guinea corn husk and corn cob potash are the oxides of silicon, magnesium and calcium and chlorides of potassium and sodium with the quantitative value of the minerals; SiO_2 is 2.0%, MgO is 12.0%, KCl is 31.0%, $Na_4Al_3ClSi_3O_{12}$ 28.0% and CaO is 27.0% for guinea corn husk, while that of corn cob is SiO_2 10.0%, MgO is 8.0%, KCl is 66.0%,

$Na_4Al_3ClSi_3O_{12}$ 3.0% and CaO is 13.0% as reported in Tables 2 and 3 above. Figure 5 shows the XRD for guinea corn husk: - The prominent peaks at 2θ approximately to 29.081° indicates sylvine (KCl) and peaks at 2θ approximately 42.572° indicates Periclase (MgO) and Quartz (Si_2O). Similarly, Figure 6 shows the XRD for corn cob; at 2θ approximately 29.01° indicates sylvine (KCl) and peaks at 2θ approximately 31.50° indicates the presence of Lime (CaO) and Sodalite (Na_4 Al_3 Cl Si_3 O_{12}), and other minor or trace compounds could be hardly indicating their presence at other 2θ.

It could be deduced that the minerals present in the guinea corn husk and corncob potash samples are mixtures of the chlorides of Na and K and oxides of Si, Mg and Ca.

Fourier Transform Infra-Red (FTIR) Analysis of Guinea Corn husk and corn cob potash

Figures 7 and 8 show below spectra of guinea corn husk and corn cob potash.

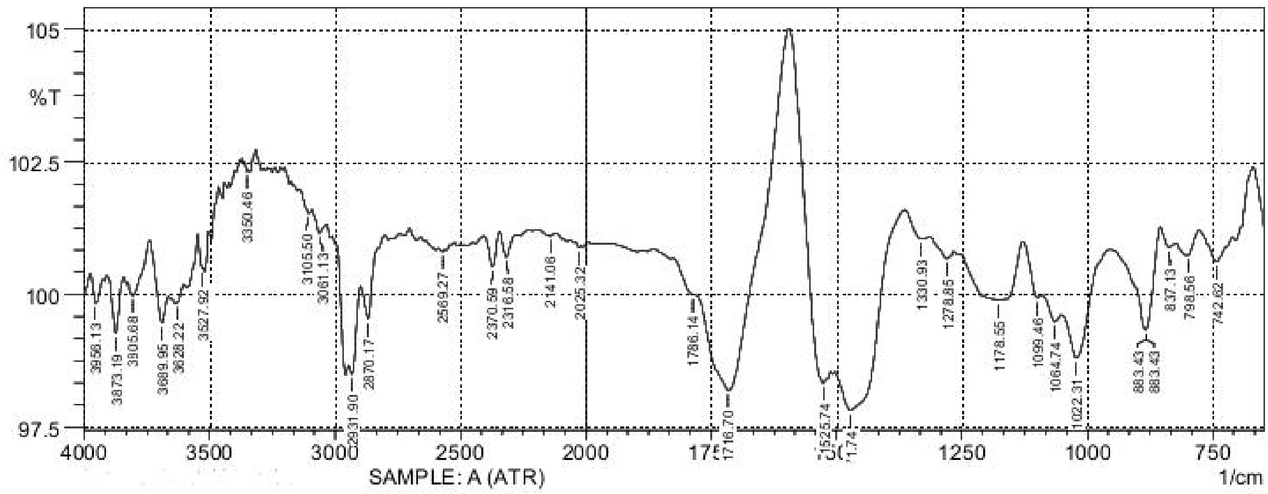

Figure 3: FTIR spectra of guinea corn husk potash

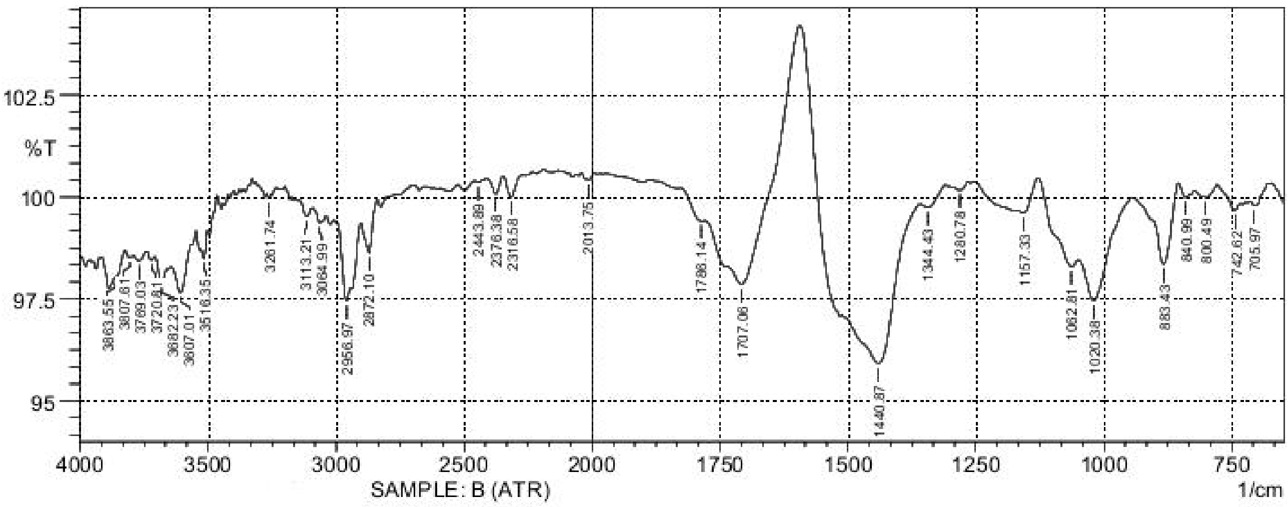

Figure 4: FTIR spectra of corn cob potash

The spectra analysis for guinea corn husk and corn cob potash are shown in Figures 7 and 8. One of the most vital functional groups present on the potash are; O-H, C-H, N-H and C-O. FTIR analysis shows absorption band's at 3873.19 cm^{-1}, 3863.55 cm^{-1}, 3805.68 cm-1, 3807.61 cm^{-1}, 3689.95 cm^{-1}, 3682.23 cm^{-1}, 3607.01 cm^{-1}, 3628.22 cm^{-1}, 3527.92 cm^{-1} and 3516.35cm^{-1} which are associated with N-H stretched groups in amines and amides; band's at 3350.46 cm^{-1} and 3261.74 cm^{-1} are associated with the stretched vibration of the O-H and N-H groups in carboxylic acids, amides, alcohols and phenols, while band's at 3061.50 cm^{-1} and 3113.21 cm^{-1} are associated with the stretch vibration of the O-H and N-H groups in carboxylic acids, phenols and primary amines, band's at 3061.13 cm^{-1} and 3064.99 cm^{-1}which are associated with the stretch vibration of the C-H and O-H groups in alkenyl and carboxylic acids. Also, band's at 2956.97 cm^{-1}, 2931.90 cm^{-1}, 2870.17 cm^{-1} and 2872.10 cm^{-1} are associated with the stretched vibration of the C-H groups in alkyl and aldehyde groups, band's at 2376.38 cm^{-1}, 2370.59 cm^{-1} and 2316.58 cm^{-1} are associated with the stretched vibration of the O-H groups in carboxylic acids, band's at 1716.70 cm^{-1} and 1707.06 cm^{-1} are associated with the stretched vibration of the C=O groups in carboxylic acids and ketones, band's at 1440.87 cm^{-1} and 1471.74 cm^{-1} are associated with the stretched vibration of the H-C-H groups in alkanes. Spectrum band's at 1344.43 cm^{-1} and 1330.93 cm^{-1} are associated with the stretched bend of N=O groups in nitromethane. Band's at 1278.85 cm^{-1} and 1280.78 cm^{-1} are associated with the stretched vibration of C-O groups in esters and ethers. Band's at 840.99 cm^{-1}, 837.13 cm^{-1}, 800.49 cm^{-1}, 798.56 cm^{-1} and 742.62 cm^{-1} are associated with the stretched of alkanes, alkynes and aromatic. While band's at 1178.55 cm^{-1}, 1157.33 cm^{-1}, 1099.46 cm^{-1}, 1062.81 cm^{-1}, 1064. 74. cm^{-1}, 1060.91 cm^{-1}, 1022.31 cm^{-1} and 1020.38 cm-1 are associated with stretches of C-O in esters and ethers. These functional groups determine the activities of the compounds or molecules present in the potash.

Comparison of the XRF results obtained showed the compositions of potash as a mixture of potassium and other elements, with K_2O accounting for 61.63% (guinea corn husks) and 73.33% (corncob) with that of [11] 50.75% (maize Stover ash potash), potassium chloride (KCl) 31% (guinea corn husks) and 66% (corncob) and that of [11] 29.10% (MSAP), respectively. The XRF results showed that the potash produced from guinea corn husks and corncob has unique compositions such as mixtures of Quartz, Periclase, Sylvite and Sodalite.

This study has shown that the XRF and XRD analyses of potash salt produced from the ash of guinea corn husks and corncob results in potassium salt for food and fertilizer applications. The likely limitations in the process technology are the incinerator and evaporator units which need an in-debt study on the temperature and time of ashing and evaporation. Over ashing and evaporation may either influence the product positively or negatively in terms of quality and quantity.

Source of raw materials for the production of organic potash is readily available and renewable, the process technology is simple and affordable.

CONCLUSIONS

The XRF results obtained from guinea corn husks and corncobs potash contain different percent concentrations of K_2O in corn cobs (73.33%) and guinea corn husks (61.63%) than the other constituents and it is in conformity with the reports of [7 and 11]. Also, the XRD results showed unique percent concentrations of mineral compounds present such as SiO_2; 10% corn cobs and 2% guinea corn husks, MgO; 8% corncobs and 12% guinea corn husks, KCl; 66% corn cobs and 31% guinea corn husks, $Na_2Al_3ClSi_3O_{12}$; 13% corn cobs and 28% guinea corn husks and CaO; 13% corncobs and 27% guinea corn husks and it is in conformity with the report of Ademola et al [7] and Ogunwede et al [11]. The potash produced from corncobs and guinea corn husks contains more of potassium oxides and potassium chlorides in the product. These results are very expedient for scale-up as an import substitution to inorganic potash. Therefore, agricultural wastes are potential raw materials for the production of organic potash that is beneficial to humans as a common food seasoning and condiments and to boost agricultural output and soil fertility. This calls for public awareness on the benefits of organic potash in food and fertilizer production which will reduce importation of inorganic potash, mining, and increase farm land.

RECOMMENDATIONS

The following recommendations are made in the light of this study:

1. To ascertain the percentage purity and concentrations of the potash produced
2. To ascertain the presence of heavy metals in the potash produced
3. To ascertain the anions, present in the potash

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