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Characterization and Application of Activated banana stalks (Musa spp) as an adsorbent for Zn2+ and Fe2+ removal from Marble Mining Industrial Wastewater (MMIWW)

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Abstract
Adsorption process is the adhesion of metals ions from wastewater to the surface of a material which have proven to be effective for treatment of wastewater before discharge. Wastewater from marble mining industry contains toxic metals like Zn, Fe etc. which causes several health problems in human beings, aquatic animals and plants, the use of activated banana stalks as an adsorbent is adopted in the adsorption of Zn and Fe from MMIWW. The initial and final concentrations of Zn2+ and Fe2+ in the wastewater were determined using Atomic Adsorption Spectrometry (AAS). The banana stalks obtained from LAUTECH Teaching and Research Farm in Ogbomoso town, were washed with distilled water, oven-dried at 1050C, Ground and sieved, then carbonized in a furnace at 550oC. Phosphoric acid (H3PO4) was used to modify the carbonized banana stalk to catalyze the adsorption rate. The wastewater was treated with the adsorbent dosages (0.2, 0.4, 0.6, 0.8 and 1.0 g), contact time (20, 40, 60, 80, 100 and 120 mins), agitation rate of 150 rpm, pH of 6.8 and temperature 40 0C, using One-Factor -At- a -Time (OFAT) method. The Activated Banana Stalks (ABSs) were characterized using Batch experiment to determine the effect of adsorbent dose, contact time and agitation speed also, Fourier Transform Infrared (FTIR), X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) experiments were performed on ABSs.. The initial concentration of Zn2+ and Fe2+ were 1.32 and 0.36 mg/L respectively while respective final concentrations of Zn2+ and Fe2+ were 0.008 and 0.023 at 120 mins and 1.0g. The adsorbent revealed a significant distribution of well-developed porous, rough surfaces with cracks characterized by different functional groups for the efficient adsorption process. The respective dosage treatment efficiency order for Zn and Fe were (83, 92. 97, 97 and 99 %) and (58, 81, 89, 94 and 98 %) respectively. ABS offers efficient and cost-effective removal of Zn2+and Fe2+ from marble mining wastewater,

Keywords: Adsorption, Banana Stalk (Musa spp), Heavy metals and Marble Mining wastewater

Introduction
Generation and emission of toxic wastes that are pollutant in nature is one of the negative consequences of industrialization and industrial production (Emerson, 2024). Wastewater from numerous industries such as mining operations, paints and pigments, glass production, metal plating, and battery manufacturing processes are known to contain contaminate such as heavy metal. Heavy metals such as Pb, Cd, Cr, Ni, Zn, Cu and Fe are present in industrial wastewater. These heavy metals in wastewater are not biodegradable and their existence in receiving lakes and streams causes bioaccumulation in living organisms, which leads to several health problems in animals, plants and human beings such as cancer, kidney failure, metabolic acidosis, oral ulcer, renal failure and damage in stomach of the rodent (Emerson, 2024; Adetoro et al., 2022) Adsorption and desorption of Carica papaya stem activated with zinc chloride for mining waste water treatment.

Mining involves the deposition of a thin protective layer (usually metallic) onto a prepared metal surface, using electrochemical processes. The process involves pretreatment (cleaning, degreasing, and other preparation steps), plating, rinsing, passivating, and drying. The cleaning and pretreatment stages involve a variety of solvents (often chlorinated hydrocarbons, whose use is discouraged) and surface stripping agents, including caustic soda and a range of strong acids, depending on the metal surface to be plated. The use of halogenated hydrocarbons for degreasing is not necessary, as water-based systems are available. In the

plating process, the object to be plated is usually used as the cathode in an electrolytic bath. Plating solutions are acid or alkaline and may contain complexing agents such as cyanides (Kumar and Singh, 2023)
Effluents from various processing industries such as mining industries is reported to contain high amounts of heavy metal ions, such as nickel, iron, lead, zinc, chromium, cadmium and copper. (Ajala and Ojoawo (2022)). The presence of Zinc and Iron in industrial wastewaters is of serious oncern because they are highly toxic, nonbiodegradable, carcingen, and continuous deposition into receiving lakes, streams and other water sources within the vicinity causes bioaccumulation in the living organisms. These perhaps, could lead to several health problems like cancer, kidney failure, metabolic acidosis, oral ulcer, renal failure and many more (Ajala and Ojoawo (2022)).
Banana stalk is an agricultural residue generated from agricultural activities. It is chosen due to the waste generated is rising dramatically which can risk both human and ecosystem (Oladeji et al, 2013). This waste can be converted into useful products through recycle which subsequently minimize the level of pollution caused by agricultural burning activities. From previous studies, agricultural wastes are potentially low-cost adsorbent as they are unexploited resources, widely available, environmentally friendly and also natural materials that exist in large quantities (Adetoro et al; (2022)). Many research had been conducted such as the study of the removal of Pb (II) by citrus pectin and adsorption of Fe (II) using Cow Bone Char (Olaoye et al, 2021). The optimum adsorption process was identified as a function of pH, contact time and dosage.

Materials and Methods

The Study Area

Igbori Marble Mining Site is located at Igbori along Igbeti-Igboho road in Orire Local Government, Ogbomoso, Oyo State. The site is a tropical savanna wet land and between latitude 8o 37’59’’N and 4o 10’ 59’’ E. Agricultural activities are carried out in the environment and buildings are far from the industry and scattered from each other. Figure 1 shows the map of Oyo State showing the local Government of where Igbori belongs to.

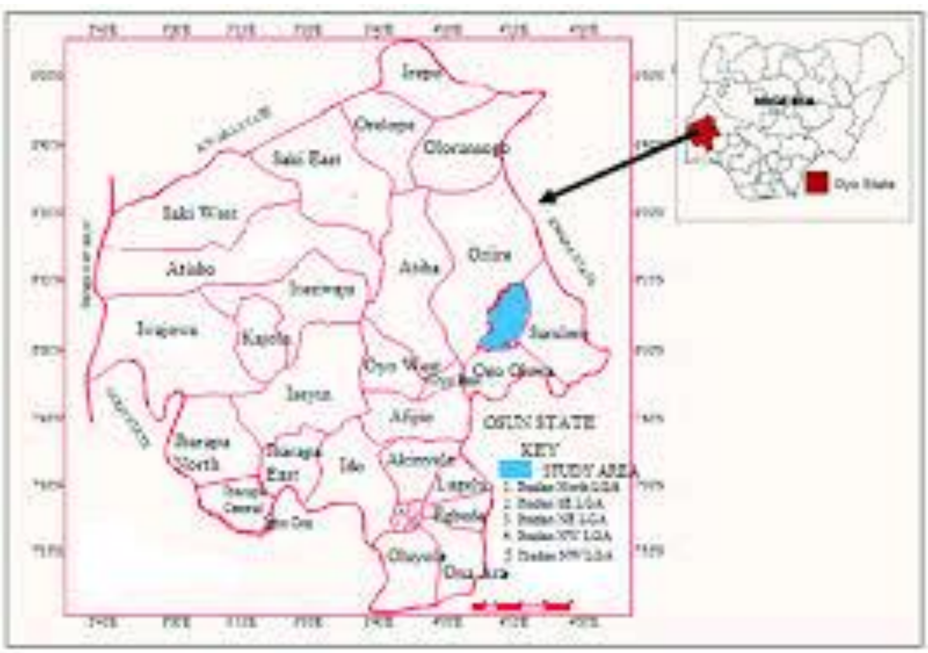

Figure 1: Physical Map of Ogbomoso, Oyo State. Nigeria.

Preparation and Characterization Marble Mining Wastewater

The mining wastewater used for this project was collected from Igbori Marble Mining Industry along Igbeti–Igboho road, Oyo State , Nigeria in a sterilized air-tight 25-litre plastic keg and immediately corked and transferred to Pure and Applied Chemistry Laboratory, Ladoke Akintola University of Technology, Ogbomoso. The mining wastewater and blank samples (250 mL each) were put into two different heating bottles (one for each) and 20mL of nitric acid (HCl-HNO3 – 3:1, v/v) was gently mixed with the contents, covered for 10 mins. at room temperature, heated on an electric hot plate at 80oC until clear solutions were obtained, cooled, then filtered into a transparent flask using glass funnels and Whatman No. 42 filter papers. The filtrates were increased to 100 mL with deionized water (distilled water) and put into transparent tightly corked bottles in preparation for the AAS reading. The filtrates were then analyzed for the concentrations of selected metal ions (i.e. Zn2+and Fe2+) using AAS. (Adetoro and Ojoawo, 2020).

This procedure was utilized for metal ions analyses of the marble mining wastewater prior to and after treatment with the adsorbent using Perkin Elmer Analyst 200 Atomic Absorption Spectrometer (AAS) in Research Laboratory, University of Ibadan, Oyo state, Nigeria, was used to read were converted and recorded, according to Ajala and Ojoawo. (2022). The results were converted to mg/l.

Preparation and characterization of Banana stalk

Banana Stalks (BSs) were obtained from Ladoke Akintola University of Technology. Fresh banana stalks were cut into 2cm size to facilitate drying and further processing, and carefully washed with tap water and deionized water in order to get rid of impurities present in it. The BSs were oven-dried at 105°C for 24hrs and then ground into powder and sieved with sieve mesh No.200 to obtain the fine powdered. The fines passing through the sieve was collected and kept inside a sealed nylon to prevent external moisture (Olaoye et al,2020).

To prepare the adsorbent: 50g of dried sample was measured inside a beaker, distill water was added to and of Phosphoric acid (H3PO4) was added as an activating agent in accordance with modified methods from Ajala and Ojoawo (2022). The mixture inside the beaker was placed on a magnetic stirrer to agitate the mixture thoroughly, then filter with Whatman No. 42 filter paper and washed with distilled water, filtered again, then pH of the filtrate was checked and the procedure was repeated severally until pH of the filtrates (wash water) was neutral, that is (6.8) was achieved. The residue (blackish in colour) was oven dried at 70oC (adsorbent) and kept inside sealed nylon for further analysis as shown in the plates 1,2,3 and 4;

Plate 1: Washed Banana Stalk in the oven

Plate 2**: Ground and seived Banana Stalk**

Plate 3: Wet Activated Banana Stalk **Plate 4: DriedActivated Banana Stalk (blackish in colour)**

Characterizations of banana stalks: Characterizations of banana stalks were performed using Fourier transform infrared spectrometer, FTIR (Perkin Elmer) to determine the characteristics of the functional groups on the adsorbent surface while X-ray Diffraction (XRD) to determine the crystalline structure of materials and Scanning Electron Microscope (SEM) to employed to determine the surface morphology of the adsorbent.

Batch Adsorption Experiment

The batch experiment was performed in accordance to the steps outlined in previous studies. Batch adsorption experiment at room temperature was adopted to find the effects of adsorbent's dosage (0.2, 0.4, 0.6, 0.88 and 1.0 g) at constant agitation rate (150 rpm), contact time (20, 40, 60, 80, 100 and 120 mins.) and pH (6.8) on the remediation of colour, Zn2+ and Fe2+ from marble mining industrial wastewater (Olaoye et al, 2020; Petrov et al, 2020; Kumaraiah, 2021).

50 mL of the mining wastewater sample was poured into a 100 mL conical flask and varying dosages of the prepared adsorbent was added, placed on a Magnetic Stirrer to rotate for different contact time at 150 revolutions per minute (rpm) at 60oC temperature.

Agitation rate, pH and Temperature were constant while adsorbent dosage and time vary throughout the experiments. All the suspensions from the experiments were filtered using Whatman No.42 filter paper and the filtrates subjected to AAS analysis as explained above (Adetoro and Ojoawo, 2020).

Tests carried out on the banana stalks and activated banana stalks

Fourier Transform Infrared (FTIR) characterization of the adsorbent:

The functional groups of banana stalks and adsorbent were determined by FTIR using Infrared Spectrophotometer at the Central Research Laboratory of Amadu Bello University, Zaria, Nigeria. The typical functional groups present on the adsorbent's surface (i.e. the chemistry of surface of the raw Banana Stalks (BSs) and Activated Banana Stalks (BSs- H3PO4)) were determined through FTIR spectroscopic analysis using Infrared Spectrophotometer. The spectra range required to capture the needed functional groups was estimated at 4 000 to 400 cm-1. The dried sample (1 mg) and KBr (500 mg) (Merck, for spectroscopy) were blended in a transparent mortar and the resulting blend was subsequently pressed at 10 000 kg cm-2 for 15 min under vacuum. The FTIR spectra revealed the functional groups typical of the raw and ACBSs-H3PO4 adsorbent surfaces (Bello et al., 2019).

X-Ray Diffraction (XRD): This is used to determine the crystal structure and Phase composition of materials. The X-ray interact with the samples atoms, causing Diffraction (scattering) of the X-rays.

Scanning Electron Microscopy (SEM): This was prepared by carbon taping, it was stuck on the carbon tape plate and subjected to detail-obscuring conducive coating, gold coating using Auto-fine coater equipment JEOL, JEC-1600 to improve the conductivity of specimen since it is a non-conductive sample. The carbon-taped sample was transferred into JEOL fine coater at pressure of 30Pa and allowed to be pressurized to a value <5Pa, creating a vacuum inside. After which certain rays were displayed, coating the specimen and the timing count-down was from 60 to 0 sec. Coated specimens were immediately transferred into the Specimen Stage of the SEM (Analytical Scanning Electron Microscope) after its cooling system has been topped with liquid nitrogen coolant. The surface morphology of the specimens was then examined by SEM and monitored on the attached Personal Computer (PC) and the elements present in the specimens were identified with their concentration levels.

Results and Discussion

Concentration of Toxic Metals Zinc and Iron in the Marble Mining Wastewater Samples

The toxic metals' concentration in the marble mining wastewater samples before treatment is shown in Table 1. All the selected toxic metals were more than the required standard (according to WHO, (2022)) before treatment, which portrayed possibility of environmental pollution of the study area by the marble mining wastewater. Both Zinc and Iron are drastically reduced after treated by activated banana stalk with Phosphoric acid, H3PO4

Table 1: Concentrations of the Selected Heavy Metals in the Marble Wastewater Samples prior to treatment.

Metals	Initial Concentration, Co,(mg/l)	WHO Specification for drinking water
$Zinc^{2+}$	1.32	3
$Iron^{2+}$	0.36	0.3
$Cadmium^{2+}$	0.64	0.003
$Lead^{2+}$	0.10	0.01
$Copper^{2+}$	0.37	1

Characterization of the produced BSs –H3 PO4 adsorbent

FTIR characterization results of the adsorbent: The FTIR spectra of the pulverized banana stalk and its activated carbon as shown in Figure 2 and Figure 3reveal significant structural and chemical transformations during the activation process. The precursor displayed broad and intense O–H absorption between 3809–3324 cm^{-1}, strong C–O stretching, and distinct carbonyl and aromatic bands, confirming the presence of cellulose, hemicellulose, and lignin typical of lignocellulosic materials (Amadu Bello University Laboratory). In contrast, the activated carbon showed a notable reduction in the intensity of these oxygenated bands, indicating effective removal of hydroxyl, carbonyl, and ether functionalities through dehydration and decarboxylation reactions. The aromatic C=C bands shifted from approximately 1578 cm^{-1} in the precursor to around 1615 cm^{-1} in the activated carbon, reflecting increased aromatic condensation and graphitization. The weakened C–H and C–O bands suggest breakdown of aliphatic and polysaccharide structures, leaving behind a predominantly carbonaceous framework. The emergence of new bands between 896–669 cm^{-1} signifies aromatic C–H out-of- plane bending, characteristic of polyaromatic or graphitic structures formed during carbonization. These spectral modifications confirm that activation transformed the raw banana stalk from a highly functionalized organic material into a stable, aromatic, and porous carbon matrix. The reduction in oxygenated groups and enhancement of aromaticity indicates improved thermal stability and surface chemistry, making the activated carbon more suitable for adsorption and catalytic applications The profiles of the respondents and organizations were investigated according to the opinions of the respondents and presented in Table 1.

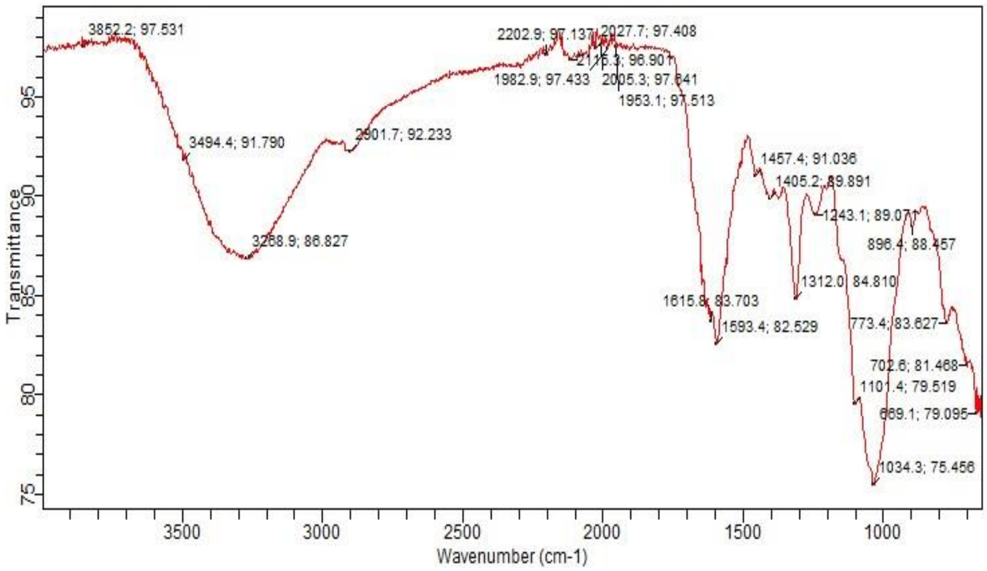

Figure 2: FTIR analysis Results on the Banana Stalk

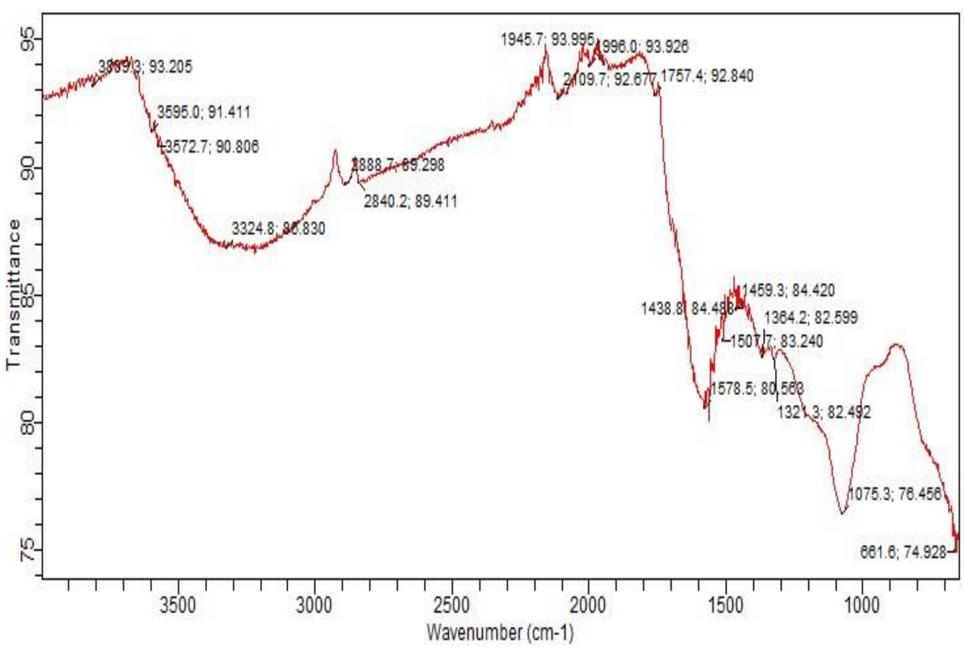

Figure 3: FTIR analysis Results on the Activated Banana Stalk

SEM analysis

The surface morphology of BS-H_3PO_4 is shown in Figure 4a and Figure 4b the surfaces were rough, uneven and many pores had formed, due to the modification of the material using the activating agent. A pore structure with several rough cavities distributed over the adsorbent's surface resulted from the breakdown of lignocellulose material at high temperature. The evaporation of volatile compounds usually result in materials with well-developed pores. The increase in the rate of reaction during the activation process resulted in carbon 'burn off', and developed good pores on the precursor. There was an increase in the porosity of the material and creation of new pores as a result of carbon loss via CO and CO_2 forms. The physiochemical treatments thus resulted in porous materials, as expressed by Adetoro and Ojoawo (2020b). The surface of the ACBS has many large pores in a honeycomb shape, indicating the effectiveness of the activating agent (i.e. H_3PO_4) in creating heterogeneous and well-developed pores on the precursor's surface, leading to ACBS with a large porous surface structure. These pores provided suitable surfaces for metals to be trapped and adsorbed.

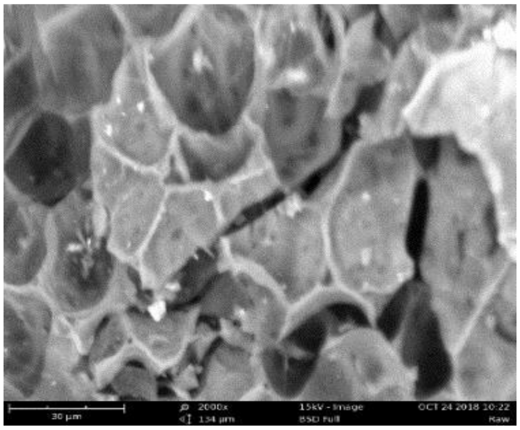

Figure 4a: SEM micrograph on Raw Banana Stalk.

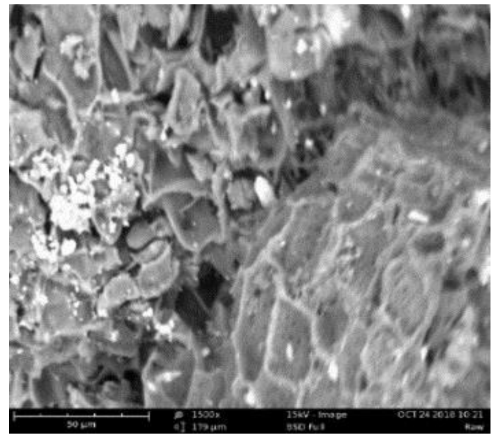

Figure 4b: SEM micrograph on Activated Banana Stalk

*XRD Analysis***:** Figure 5a and 5b show the analysis of X-ray refraction of raw banana stalk and activated banana.

The XRD pattern of the raw banana stalk in figure 5a shows a broad diffraction peak around, which is characteristic of amorphous cellulose typically found in lignocellulosic biomass materials.

The activation process (thermal and chemical) in figure 5b removes volatile organic components such as hemicellulose and lignin, resulting in the breakdown of the amorphous cellulose matrix and the formation of more ordered carbon structures. The identified phases include quartz, hematite (Fe_2O_3), hydroxyapatite, silicon oxide, and other phosphate-related compounds depending on the activating agent used. The raw banana stalks and activated banana stalks clearly demonstrates that activation significantly alters the structural composition of the banana stalk. While the raw sample is predominantly amorphous due to its high lignocellulosic content, the activated sample exhibits enhanced crystallinity and more pronounced mineral phases. However, the presence of a broad background hump in both patterns indicates that the material still retains partial amorphous characteristics, which is typical of activated carbon materials.

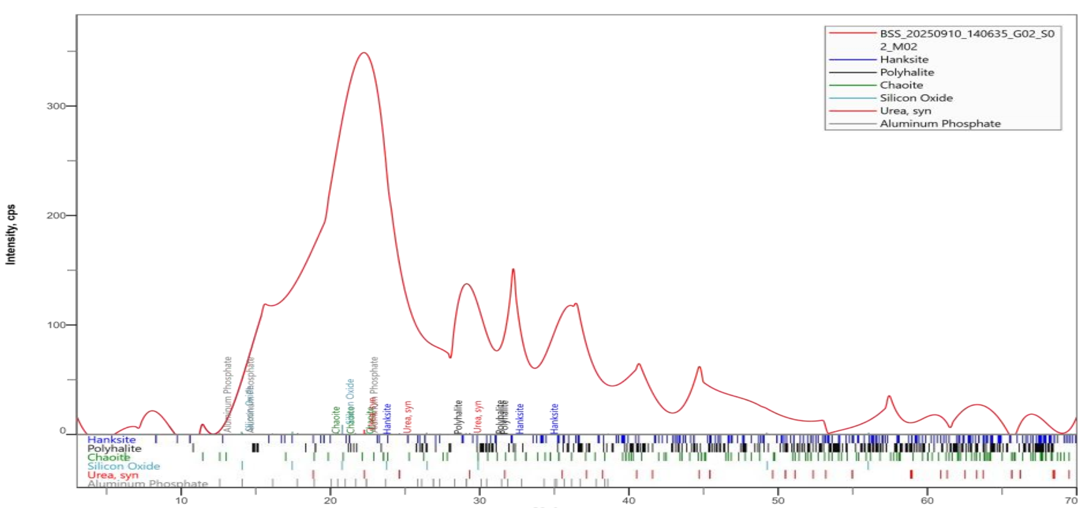

Figure 5a: Result of XRD of Raw Banana Stalk

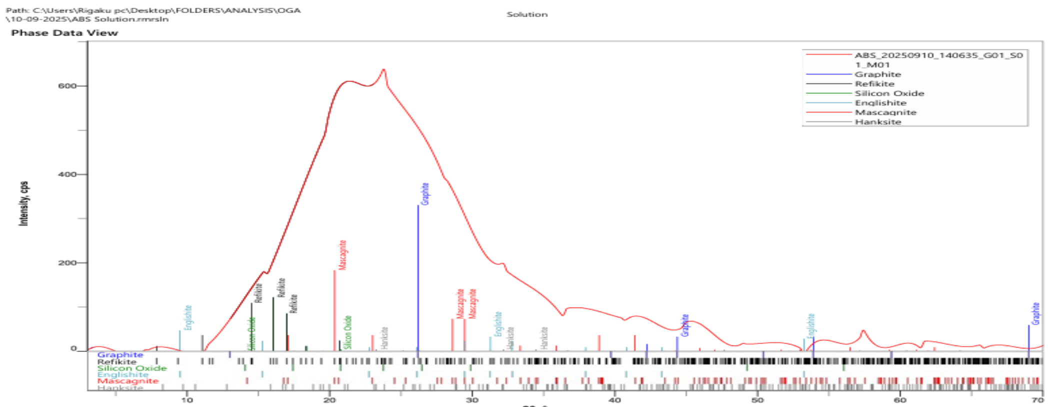

Figure 5b: Result of XRD on the Activated Banana Stalks

Conclusion and Recommendation

Activated Banana Stalk (ABS), an agro waste shows significant adsorption capacity for the remediation of Zinc and Iron from marble mining wastewater. Moreover, the activated banana stalk was also most efficient in the removal of Zn2+ and Fe2+ under standard parameters and suitable experimental conditions. The study concludes that:

i. Activated banana stalk is highly effective in the adsorption of Zn2+ and Fe2+ from marble mining wastewater. Banana stalks being readily available at low cost can be activated or carbonized and serve as a suitable cheap and environmentally friendly adsorbent in metal ions' removal from marble mining wastewater

ii Moreover, the study outcome can also be valuable for designing and devising a frugally low-cost treatment process plant for the adsorption of metals from marble mining wastewater from industries.

iii. Banana stalk has high carbohydrate (82.25%) and low moisture (3.10%) contents, which indicated that it is a good adsorbent for the removal of toxic metals from wastewater. It has some functional groups and metabolites, which give it its toxic metal binding ability.

Activated banana stalk (ABS) is therefore recommended as an adsorbent for marble mining wastewater's treatment before discharging into the environment. It could be employed as a substitute to the costly commercial adsorbents for metals' removal.

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