

Preparation and Characterization of Adsorbent Derived from Maize Cob

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DOI: <https://doi.org/10.51244/IJRSI.2025.120500020>

Received: 02 May 2025; Accepted: 07 May 2025; Published: 29 May 2025

ABSTRACT

This study focuses on the preparation and characterization of adsorbent derived from maize cob, emphasizing its potential for removing contaminants from aqueous solutions. Maize cobs were collected, dried, prepared, and subjected physiochemical and elemental analysis. Characterization was done using scanning electron microscopy and Electron Dispersive X-ray spectrometer (SEM/EDX) and Fourier-transform infrared spectroscopy (FTIR). The results indicate that the elements carbon and oxygen had greater values (42.69% and 40.22%, respectively), whereas the elements sodium, calcium, and nitrogen had lower values (5.64%, 2.56%, and 1.28%, respectively). The maize cob material showed a unique pH of 6.5 which firmly indicates that this corncob material might be employed as an effective adsorbent for purifying water. Also, the high fixed carbon content (65.50%) revealed that the maize cob material is of good quality, and appropriate for adsorption. Moreover, the FTIR peaks validate the absorptive properties of maize cob due to the presence of adsorbents' functional groups; -OH, C=N, and C=O, which control the adsorption mechanism in addition to their behavior during adsorption. On the other hand, the micrograph images of the material made of maize cobs display the aggregation of the adsorbent particles and the observed closed pores following adsorption resulted in a more distinct and transparent adsorbent structure for maize cobs.

Keywords: Maize cob, Adsorption, Micrographs, Adsorbent, FTIR

Maize is one of the most important crops worldwide with a total production of around 1060 million tons in 2018 (IGC - International Grain Council, 2018). In Nigeria, maize serves as a major ingredient for the production of various types of food and can also be consumed directly when boiled or roasted. (Lala *et al.*, 2023) After the maize is consumed, the residue is known as the maize cob. In other words, maize cob is the hard thick cylindrical central core of harvest and unharvested maize or corn, where the maize grains are borne. (Ojedele and Ahaneku, 2015)

Furthermore, it is estimated that maize cob accounts for 40–50 percent of total maize production, and it's considered an agriculture waste (by-product of sweet maize). (Islam *et al.*, 2023) Moreover, maize cob is one of the natural wastes from maize plant which causes high level of environmental pollution if not properly handled. Lala *et al.* (2023), even reported that the ratio of maize grain to the cob is given as 100:18, which may result in useless accumulation of the cobs worldwide and particularly in Nigeria in case of not being properly utilized.



Figure 1: Maize cob

According to Zhang *et al.* (2012), about 150 g of cobs are generated in order to produce 1 kg of dry maize grains, which would have resulted, over the years, in approximately 159 million tons of Maize Cob Waste (MCW). The cobs are usually left in the field as part of the maize stover for soil conditioning. This lignocellulosic bio-waste can be considered a good precursor for adsorbents like activated carbon with relatively high surface area. This is due to its high carbon content (around 45% w/w) and low percentage of ashes (about 2% w/w). (Surra *et al.*, 2019)

There are several components that comprise the agricultural waste, maize cob. These components include; the hemicellulose content of maize cobs which comprises 41.4%, the cellulose content is 40%, the lignin content is 5.8%, the crude protein content is 2.5%, the starch content is 2.1%, the ash content is 1.8%, the water-soluble carbohydrate content is 1.1%, and the crude fat content is 0.7% (Kaliyan & Morey, 2010). Although, the insoluble dietary fibre made up more than 60% of the maize cob, the cellulose is the main component, while hemicelluloses come second (Lau, 2018). Additionally, according to inorganic elemental analysis, maize cobs contained silicon (5.33 g kg^{-1}), phosphorus (1.11 g kg^{-1}), potassium (10.8 g kg^{-1}), magnesium (0.55 g kg^{-1}), inorganic sulphur (0.14 g kg^{-1}), aluminium (0.18 g kg^{-1}), barium (0.11 g kg^{-1}), calcium (0.23 g kg^{-1}), and traces of strontium and barium (0.11 g kg^{-1}), (Mullen *et al.*, 2010).

It has always been a challenge for scientists to produce effective adsorbents required for the elimination/removal of heavy metal ions. However, adsorbents have proven to possess many benefits over traditional chemical sorbents in water treatment systems: biodegradability in natural/ environmental settings, high abundance in nature, high surface-area, a greater tendency to adsorb such metal ions, appropriate pore dimension and volume, more mechanical strength, compatibility, they are easily available, they are easily renewable, they have a cheap cost, their eco-friendliness, they have easy manufacturing methods, and they are more specific in nature. (Markovic *et al.*, 2015)

By definition, adsorption process is a unit operation where fluid concentration is altered by passing it through a porous material called adsorbent. (Lala *et al.*, 2023) Adsorption among other methods of waste water treatment is widely accepted due to its effectiveness, simplicity, possibility of using different low-cost materials, and also because the method is highly environmentally friendly (Osasona *et al.*, 2018; Yusuff *et al.*, 2019).

He *et al.* (2014) discovered that structures of these adsorbents have a high surface area to volume ratio and multiple active binding sites on their surface, enabling heavy metals to be maintained effectively under some conditions (e.g., $-\text{COOH}$, $-\text{NH}_2$, $-\text{OH}$, $-\text{SH}$ groups). Also, the effectiveness of an adsorbent depends on its method of preparation and the functional groups present within (Herrera-Barros *et al.*, 2018) Studies have shown that carboxyl, hydroxyl and amide groups which are in favour of the heavy metal adsorption are present maize cob (Herrera-Barros *et al.*, 2018). Furthermore, adsorbents such as activated carbon and zeolite have been utilized for removing trace metals in wastewater. (Osasona *et al.*, 2018), zeolite (Kussainova *et al.*, 2018) A study of Al_2O_3 nanoparticles modified maize cob (Herrera-Barros *et al.*, 2018) showed 86% removal of nickel from aqueous solution. This is higher than those obtained using maize cob particles.

Adsorption has certain advantages over conventional methods such as minimizing chemical and biological sludge, low cost, high efficiency, regeneration of adsorbents, and the possibility of metal recovery. A large number of natural adsorbents were synthesized for the extraction of heavy metal ions. (Gupta *et al.*, 2021)

The bark is the outermost layer of stems and roots of wooden plants, and is obtained as a by-product of the wood manufacturing units. (Gupta *et al.*, 2021) In bark, tannin is present in a large amount and this organic substance is very efficient for the extraction of heavy metal ions. Tannin contains polyhydroxy polyphenol moieties which help in the adsorption of metal ions. (Gupta *et al.*, 2021) A study by Randall *et al.* (1978) reported in Gupta *et al.*, (2021) investigated the elimination of heavy metal ions like cadmium, lead, copper, and mercury by using bark, formaldehyde polymerized bark, and peanut skins. Adsorption capacities for bark have been reported to be in the range of 1.5–27.6 mg/g for various tannin-containing materials. (Gupta *et al.*, 2021) However, the problem of discoloration of water arose due to the presence of soluble phenols. Chemical pre-treatment of bark with acid, base, or acidified formaldehyde has been shown to overcome the problem of discoloration in water without appreciably affecting ion removal capacity. (Gupta *et al.*, 2021) Thus, the bark

is helpful in the elimination of toxic ions from water bodies and is the main possible option due to economics along with its large accessibility.

Lignin is derived from black liquor and used as a devastating ingredient in paper processing units. It is a very cheap adsorbent as compared to activated carbon for the elimination of toxic ions. One major factor responsible for the sorption competence of lignin is polyhydric phenols and additional functionality present on the surface. The ion exchange method was used to remove heavy metal ions by the use of lignin. (Mishra and Wimmer, 2017)

Celik and Demirba, (2005) synthesized a natural sorbent from improved lignin material for the extraction of lead, cadmium, copper, and zinc metal solutions prepared in a lab with an initial concentration of 50 mg/L. The maximum adsorption value was found to be 11.3 mg/g for Zn (II), 17.5 mg/g for Pb (II), 7.7 mg/g for Cd (II) by the lignin. The time period to attain the highest adsorption was 4 h for lead ions with 96.7% adsorption capacity and for Zn (II) ions 10 h with 95.0% sorption efficiency. Its worthy to note that adsorption values can improve with pH indicating that metal ions undergo ion exchange with lignin. (Siddiqui et al., 2020) Lignin based-adsorbents were reviewed recently with the focus on lignin, its modification, and carbon materials derived from this abundant feedstock. (Supanchaiyamat *et al.*, 2019)

In recent times, different industrial wastes and biomass have been used in preparing activated carbon (Reza *et al.*, 2020; Heidarinejad *et al.*, 2020). Activated carbon (AC) has been recognised as an essential sorbent for a long time. These adsorbents were synthesised by heat treatment of carbon substances followed by steam or chemical methods that drastically enhance the specific surface area and permeable character of the adsorbent. (Gupta *et al.*, 2021) Several adsorbents of activated carbons of various permeable sizes were obtained using various methods and were used for various applications in the extraction process. Coarse-activated carbon has also been used for the expulsion of harmful metals from groundwater. Walnut shell-based granular enacted carbon has additionally been discovered for evacuation and adsorption of color dissolved organic, heavy metals from wastewater. (Singh and Lal, 2008) Eeshwarasinghe et al. (2009) studied the removal of polycyclic aromatic hydrocarbons such as acenaphthylene, phenanthrene, and heavy metals like cadmium, copper, zinc by granular activated carbon.

Activated carbon from pecan nutshell was used for the elimination of Zn (II), Cd (II), Ni (II), and Cu (II), from a binary system of metal ions. (Aguayo-Villarreal et al., 2017). Dong et al. (2018) examined exhausted activated carbon for the extraction of heavy metal ions; the exhausted activated carbon was collected from a biological activated carbon water treatment plant of southern China. It was found that activated carbon had a great sorption value of 95% and 86% for Pb^{2+} and Cd^{2+} at very little concentration of 200 ppb, respectively. Phosphoric acid impregnated activated carbon (DPAC) was synthesized from the seeds of date pits via single-step activation. Phosphoric acid is introduced into the seeds of date pits through 12-hour soaking. (Krishnamoorthy *et al.*, 2019).

Kulmedov and Mohammed (2023) investigated the use of maize cob carbon powder as an adsorbent for the removal of zinc (II) and chromium (VI) ions from industrial effluent. In this study, maize cob carbon powder was chosen as the adsorbent due to its low cost, non-toxicity, and availability. A batch experimental approach was utilized to investigate the effects of temperature, pH, adsorbent loading, contact time, and initial metal ion concentration on the adsorption process. The results of the study indicate that maize cob carbon powder can be effectively used as an economical adsorbent for the treatment of contaminated water. (Kulmedov and Mohammed, 2023)

Melese et al., (2020) prepared Corncob adsorbent through environmentally friendly process without any additional chemical treatment. The prepared material was characterized by X-ray diffract meter (XRD), Fourier transform infrared spectroscopy (FTIR) and pH points zero charge techniques. The XRD diffracted pattern of corncob adsorbent did not shows well-defined sharp peaks instead it shows pumps peaks; this indicates that the diffraction patterns of the corncob adsorbent have low crystallinity (amorphous). FTIR characterizations of corncob indicate the presence of functional groups. The point zero charge of corncob adsorbent was found to be 7.05; it can be basic modification of the adsorbate which gave a negative (basic) surface charge for the adsorbent. The effect of corncob dosage, contact time and solution pH were studied at

optimum values for removal of Pb, Cd, Cr and Cu metals using Batch method. Adsorption equilibrium data fitted well with the Freundlich isotherm for Pb, Cd and Cu metal ions but Langmuir isotherm for Cr(VI) metal ion. (Melese *et al.*, 2020)

In addition, the adsorption performance of maize cob-derived activated carbon (MCAC) for the removal of nickel (II) from solution was investigated by (Lala *et al.*, 2023). The MCAC was prepared by adsorbent chemical activation method. Afterwards, the MCAC was characterized and utilized as the adsorbent for batch adsorption process. Initial adsorbate concentration, contact time and adsorbent dosage were considered as the independent variables for the adsorption process. With the use of MATLAB, they were able to simulate 97.113% removal efficiency of nickel (II) on experimental validation at optimum conditions of 9.75 mg/L initial nickel (II) concentration, 120 min contact time and 0.803 g adsorbent dosage. This result correlates with CCD that predicted 97.6154% nickel (II) removal efficiency. Hence, it was concluded that MCAC is highly effective for the removal of Ni (II) from aqueous solution and the highly generated agricultural waste (Maize cob) can be easily modified as a adsorbent. (Lala *et al.*, 2023).

MATERIALS AND METHODS

Sample Collection

Corn cob samples were collected from a farm stead in Otuoke, Ogbia LGA Bayelsa state of which fertilizer and other chemicals have not been used on farmland for growth of plant.

Preparation of Corn cob Adsorbent

The collected corncobs were washed with tap water several times to remove dirty dust and later with distilled water to remove for further purification. The washed corncobs were dried in sun light for six hours. The dried corncobs were cut into small pieces then put in drying oven at 110 °C for 24 h and finally dried mass was ground using a grinder and sieved with 250 µm porous sieve to have the same particle sizes, then the samples were packed for adsorption experiments.

Adsorbent Characterization

pH Value

3 g of the grounded sample was weighed and soaked into 30ml of boiling deionized water for 24 hrs. The pH readings were observed with a digital pH meter, Jenway 3520 (ASTM: D 3838). The pH of carbon is important to the adsorption of pollutant in solution.

Determination of moisture content

Three crucibles were cleaned with ethanol, dried, labelled and pre-weighed using an analytical weighing balance. 2 g of the maize cob was weighed in each Petri dish. The sample was dried in the vacuum oven at a temperature of 500 °C for 3 hours, cooled in desiccators and weighed. The drying and weighing was repeated twice until constant weight was achieved. The moisture content was achieved following the method of AOAC, (2019). (Onawumi *et al.*, 2021)

$$\% MC = \frac{W_{fs} - W_{ds}}{W_{fs}} \times 100 \quad (1)$$

Where W_{fs} = weight of fresh sample; W_{ds} = weight of dry sample

Volatile matter

Volatile matter content was determined according to standard method (ASTM: D 2974-2014, Boadu *et al.*, 2018). 1 g of samples was taken in a pre-dried crucible and covered with lid, the heated in a Gallenkamp muffle furnace regulated at 9500 °C for 30 minutes. After heating, the plate was quickly covered, cooled in desiccators and weighed. The amount weighed was taken as volatile matter.

Determination of ash content

Ash content was determined according to standard method (ASTM: D 2974-2014, Boadu *et al.*, 2018). 5 g of dried samples were weighed into a crucible of a known weight and heated in a Gallenkamp muffle furnace for 6hrs at 600 C. When constant weight was achieved, the crucibles was allowed to cool in desiccators. The mass of the ashed carbon was determined. The weight of ashed carbon is expressed as the percentage weight of the original carbon sample.

$$\% \text{ Total Ash} = \frac{D-C}{D-B} \times 100 \quad (2)$$

Where: B = Weight of the crucible (g); C = Weight of crucible + original sample (g); D = Weight of crucible + ashed sample (g)

Determination of fixed carbon

The fixed carbon was determined by subtracting the sum of the moisture content, ash content and volatile matter content in percentage composition from hundred. The value of fixed carbon content obtained is expressed in percentage

$$FC = 100\% - (MC+AC+VM) \% \quad (4)$$

The value for the fixed carbon content should be equal or greater than 65% for a good activated carbon (Olayiwola *et al.*, 2015)

Bulk density

The standard procedure used in analyzing bulk density was from Akpapunam and Markakis (1981). 5 g of the sample was placed into a pre-weighed 5ml measuring cylinder (w_1). The cylinder was gentle tapped to eliminate air spaces within the samples in the cylinders to give a possible close pack (PBD). The volume occupied by the samples and the added weight in the cylinders were determined using analytical weighing balance and will be recorded as (w_2). The bulk density is expressed as:

$$\text{Bulk Density} = \frac{W_2 - W_1}{\text{Volume of cylinder}} \quad (3)$$

Where: W_2 = weight of samples and cylinder (g); W_1 = weight of measuring cylinder (g)

Particle size: Particle sizes of the ground samples alone was determined. The samples were prepared using electric blending machine after which a sieve analysis was carried out using CONTROLS MILAND-ITALY D402-01 MATR 84000 109 sieve shaker at rotation of 10-15 min with (2-36 mm, 1.18 mm, 0.6 mm, 212 μm) sieves (ASTM D-2862-97).

$$\% C = \frac{\text{Weight of carbon after sieve}}{\text{Total weight of carbon}} \quad (4)$$

Fourier Transform Infrared Spectroscopy (FTIR) Analysis

FTIR is an instrument used in determining the surface functional group of a material. The FTIR spectroscopy method was employed in this study in order to observe the functional groups of the different agricultural waste and probably deduce their surface chemistry and hence their structures. It also gives information on the possibilities of the functional groups of chemicals activated adsorbents. FTIR spectra was obtained with dried powder of different agro-waste samples under consideration. 100 mg of potassium bromated (KBr) was weighed on a sensible weighing balance and mixed with 2.1mg of adsorbents powder in a mortar and pestle. The mixture was compressed in a compressor machine until the sample was compacted. Samples were placed in a cell before fixing it in a Parkin Elmer FT-IR system BX spectrum and spectra reading will be taken. (Onawumi *et al.*, 2021).

Scanning Electron Microscopy/ Energy Dispersive X-ray Spectroscopy (SEM/EDS) Analysis.

The surface morphology of the adsorbent can be demonstrated by SEM photograph, using a JSM_7610F (Tokyo, Japan). SEM is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that can be detected and that contain information about the sample's surface topology and composition. The electron beam is generally scanned in a raster scan pattern, and the beam's position is combined with the detected signal to produce an image. SEM can achieve resolution better than one nanometer. Specimen can be observed in high vacuum, low vacuum, wet conditions (in environmental SEM), and at a wide range of cryogenic or elevated temperature. (Onawumi *et al.*, 2021)

Table 1. Elemental composition of maize cob

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
8	O	Oxygen	42.69	39.25
6	C	Carbon	40.22	27.76
7	N	Nitrogen	5.64	4.54
15	P	Phosphorus	4.71	8.39
11	Na	Sodium	2.56	3.39
40	Zr	Zirconium	2.02	10.59
20	Ca	Calcium	1.28	2.95
12	Mg	Magnesium	0.55	0.78

Table 2: Physicochemical properties of maize cob

S/no	Parameters	Mean
1	pH	6.50
2	Moisture content (%)	14.20
3	Volatile matter (%)	10.20
4	Ash content (%)	8.10
5	Fixed carbons (%)	65.50
6	Bulk density (g/cm ³)	0.440
7	Surface area (m ² /g)	650
8	Particle size (μm)	250

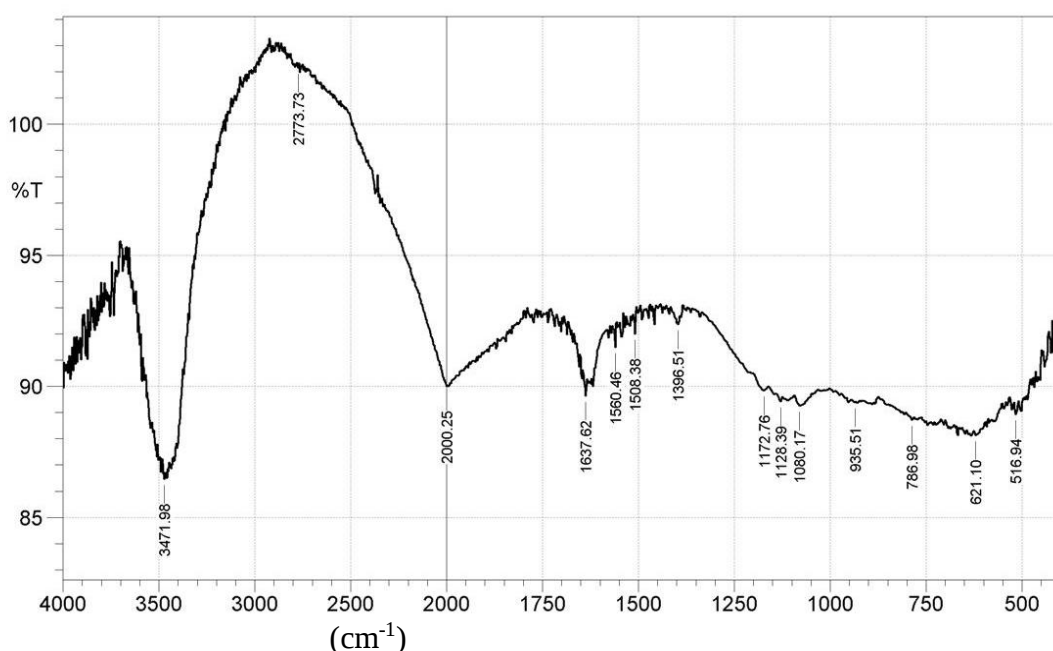


Figure 2: FT-IR spectrum of maize cob



Figure 3: SEM image of maize cob

DISCUSSION

Table 1 displays the elemental composition of the maize cob absorbent. The results indicate that the elements carbon and oxygen had greater values (42.69% and 40.22%, respectively), whereas the elements sodium, calcium, and nitrogen had lower values (5.64%, 2.56%, and 1.28%, respectively). Lala *et al.* (2023) state that biomass having a high concentration of carbon, oxygen, and hydrogen works well during the adsorption process.

The physicochemical characteristics of the produced maize cob absorbent are shown in Table 2. In comparison to the pH of MCAC (7.8) reported by Lala *et al.* (2023), the reported pH (6.5) is more acidic. Furthermore, the observed pH value, which is near to neutral, indicates that this corncob adsorbent might be employed as an effective adsorbent for purifying water (Igwegbe *et al.*, 2020). The moisture content reported is greater, at 14.20%, than the corresponding values for the egg shell activated carbon samples, which are $12.90 \pm 0.110\%$. According to reports, activated carbon with less moisture content has a higher adsorption effectiveness (Elelu *et al.*, 2019). The examined maize cob material's volatile matter, ash content, and fixed carbon content were reported to be 10.20%, 8.10%, and 65.50%, respectively. The result suggests a lesser value of fixed carbon contents (Onawumi *et al.*, 2021). Nevertheless, the high fixed carbon content revealed that the maize cob material is of good quality. Olayiwola *et al.* (2015) state that adsorbents having a fixed carbon content of at least 65% are thought to be appropriate for adsorption. Furthermore, only samples with a high carbon content can be effective adsorbents based on the carbon content in the removal of pollutants, according to Xiong *et al.* (2013) as stated by Bello *et al.* (2017). The bulk density of 0.440 g/cm³ that was obtained is marginally less than the value of 0.482 g/cm³ reported by Lala *et al.* (2023). The investigated maize cob adsorbent is suitable for adsorption-based in the range of values obtained. Given its effectiveness as an adsorbent, the chosen particle size of the produced maize cob material (250 μm) seemed appropriate. Additionally, the adsorption effectiveness is increased by the larger adsorbents caused by the growth of microporous structure, which has a notable surface area of 650 m²/g. (Lala *et al.*, 2023)

The examined maize cob material's FT-IR spectrum is displayed in Figure 2. The FT-IR spectrum's peaks were categorized into different functional groups based on the wave numbers that corresponded to them. A sharp band detected at 3471.98 cm⁻¹ is indicative of the alcohol or carbonyl group's -OH bonding group. The peak at 2773.73 cm⁻¹ is associated with amines' C≡N stretch vibration. The bands that correspond to C=O stretching acid halide are located at 2000.25 cm⁻¹ and 1637.62 cm⁻¹. Adsorbents' functional groups control the adsorption mechanism in addition to their behavior during adsorption (Onawumi *et al.*, 2021). On the other hand, the micrograph images of the material made of maize cobs following adsorption are shown in figure 3. The

aggregation of the adsorbent particles and the observed closed pores following adsorption resulted in a more distinct and transparent adsorbent structure of maize cobs.

CONCLUSION

Adsorbent derived from the maize cob was successfully prepared and characterized. Maize cob adsorbent showed a unique pH of 6.5 which firmly indicates that this corncob material might be employed as an effective adsorbent for purification of contaminated water. Also, the high fixed carbon content (65.50%) revealed that the maize cob material is of good quality, and appropriate for adsorption since only samples with a high carbon content are effective adsorbents in the removal of pollutants. In addition, the FTIR peaks validate the absorptive properties of maize cob due to the presence of adsorbents' functional groups; -OH, C≡N, and C=O, which control the adsorption mechanism in addition to their behavior during adsorption. Furthermore, the micrograph images of the adsorbent made from maize cobs displayed a regular and heterogenous pore size which enhance better adsorption.

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