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PREPARATION AND CHARACTERIZATION OF ADSORBENT FROM WASTE SHEA (*Vitellaria paradoxa*) NUT SHELL

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ABSTRACT

The activated carbons (adsorbents) prepared in this study, were from Vitellaria paradoxa nut shell following the two steps of carbonization and activation processes using H_3PO_4 and KOH as activating agents and were labelled as HVP for H_3PO_4 modified adsorbent and KVP for KOH modified adsorbent. The activated carbons were characterized using standard methods of analysis. Bulk density (g/cm^3) of the carbons (HVP and KVP) were 0.72±0.03 and 0.69±0.05, and attrition (%) values of 40.76±0.16 and 42.37±0.23 were obtained for HVP and KVP respectively. Results obtained also depict the presence of carboxyl O-H and C-O groups, and the N-H of amine on the surfaces of the activated carbons. The values of pH point of charge zero (pH_{zpc}) obtained for HVP and KVP were 6.40 and 6.60 and were less than the pH values of 6.78±0.00 and 7.18±0.01 for HVP and KVP respectively. SEM results revealed surfaces with pores with different dimensions in both HVP and KVP. Results of the study suggest that the prepared adsorbents can be effectively applied in wastewater treatment.

Keywords: Adsorbent, Agricultural wastes, Porosity, Bulk density, Wastewater

INTRODUCTION

The use of adsorbent (activated carbon) produced from agricultural wastes for the remediation of the environment has attracted significant consideration amongst environmental scientists. Wastes of agricultural produce are becoming attractive for the preparation of adsorbent owing to their ability to reduce the amounts of pollutants in solutions. Agricultural wastes signify a huge resource that is unused and are environmentally friendly and readily available (Ahmad *et al*., 2014). Several agricultural materials have been utilized to detoxify wastewater that contain elements which could be toxic to human and the environment. Amongst all adsorbents, activated carbon remains one of the eminent and cost-effective adsorbents. Activated carbon (AC) is a carbonaceous material whose internal porosity is highly developed and has a large capacity for adsorption (Shaba *et al*., 2021). This adsorbent is known to consist of interconnected micro crystallites which are irregular and each consist of hexagonal ring of carbon fused in similar structure to that of a graphite. AC is one of the most important discoveries ever made that has been extensively used to treat polluted water (Mathew *et al*., 2023a; Ahmad *et al*., 2016).

The discovery of carbon structured materials has generated great interest among researchers due to the exceptional textural properties which include highly organized pores and well-defined morphology. These properties have made AC an attractive adsorbent for different kinds of applications which include selective removal of adsorbates and immobilization of bio-molecules. In addition, AC has been reported to be used in the purification of water, air and food. It has also been used to treat industrial wastewater and oil spill through sorption (Mathew *et al*., 2023b; Bernard *et al.*, 2013). Lately, the application of AC obtained from agricultural wastes has been emphasized as it is cheap and do not form sludge or precipitate. *Vitellaria paradoxa* nut shell are common agricultural wastes that can be converted to useful material.

This study seeks to prepare and determine selected properties of activated carbon from *Vitellaria paradoxa* nut shell for application in wastewater treatment.

MATERIALS AND METHODS

Sample collection and treatment

The sample (*Vitellaria paradoxa* nut shell) used for preparing the activated carbon was collected from local oil mill site in Minna, Niger State, Nigeria. The *Vitellaria paradoxa* nut shell which was a bye product of shea butter oil extraction was collected into clean polyethylene bag. The dried nut shell of *Vitellaria paradoxa* was washed thoroughly with distilled water. This was done to remove dirt from the sample before sun drying. The dried sample was crushed to reduce the size and kept in dry air-tight container ready for preparation of activated carbon.

Production of the activated carbon (AC)

The method used for production of the AC in this study is the to step method, which involved carbonization of the crushed *Vitellaria paradoxa* nut shell followed by activation of the charcoal at high temperature (Shaba *et al*., 2021). Five (5.0) grams of thecrushed *Vitellaria paradoxa* nut shell was heated at 400 ^oC for 30 mins in a muffle furnace to carbonized. It was then washed thoroughly with plenty of warm distilled water to remove the ash before dying. The carbonized sample was then divided into two portions of 5.0 g. Each portion was macerated separately in 5.0 cm^3 H_3PO_4 and KOH, allowed to stand for 2 hours, then activated in a furnace at 800 ^oC (Shaba *et al.*, 2021; Musah *et al*., 2016; Rahman *et al.*, 2014). The activated carbon was labelled HVP (H_3PO_4 modified activated carbon). The same process was repeated using 1.0 moldm^{-3} KOH as activating agent and labelled KVP (KOH modified activated carbon).

Measurement of pH

The method reported by Okeimen *et al.* 2004 was employed to determine the pH of the activated carbon (AC). In this method, 1.0 g of the sample was weighed and transferred into a beaker followed by the addition of 20 cm^3 distilled water. The mixture was stirred with glass rod until the AC became uniformly wet. Distilled water was added to raise the volume to 100 cm^3, it was then stirred for 30 seconds, then covered for one hour. The mixture was further stirred before 10.0 cm^3 of the extract was decanted into a clean, dried beaker before pH value was measured using pH meter.

Carbon Hardness

Attrition value was determined according to the method of Rao *et al*. (2003), where 100 cm^3 of acetate buffer was added to 2.0 g of the AC sample in a 150 cm^3 beaker. The mixture was stirred continually for 4 hrs at 25 ^oC then poured onto a 0.35 mm screen (mesh size 45) to filter. The resultant residue was washed thoroughly with distilled water and then transferred into an aluminium weighing dish (pre-weighed) and dried in an oven for 4 hrs at 90 ^oC. The sample was removed and cooled in a desiccator and then weighed. The AC hardness (% attrition) was calculated as:

$$\% Attrition = \frac{(Wo-W1)}{Wo} \, x \, 100 \tag{1}$$

Where: $Wo \; and \; W1$ the initial and final weights of AC

Bulk density

Three (3.0) g of the prepared adsorbent was packed into a 25 cm^3 measuring cylinder, the tapped on a bench for 2 mins to enable AC compact in the cylinder until the volume stopped decreasing. The volume displaced was recorded and the AC bulk density was determined according to equation 2 (Sugumaran *et al.*, 2012):

$$P = \frac{mass \; of \; AC \; immersed}{Volume \; displaced} \tag{2}$$

Surface area, pore size and pore volume

Surface area, total pore volume and pore volume were determined using NOVA 4200e QuantHVProme Instrument analyser according the method describe by Sugumaran *et al.* (2012). About 300 mg of the *Vitellaria paradoxa* nut shell activated carbon was degassed at 150 ^oC for four (4) hours under nitrogen gas flow to remove water moisture and impurities. The nitrogen gas was used as the adsorbate and analysis was performed at -196°C(Sugumaran et al., 2012).

Burn off

Burn off was calculated as described by Ioannidou and Zabaniotou (2006). It was calculated as percent of the difference in weight between the precursor and the AC divided by the weight of the char:

$$\% Burn \; off = \frac{(Wo-W1)}{Wo} \tag{3}$$

Where: *Wo* is weight of precursor; *Wi* is weight of AC

Yield of Activated Carbon

Yield of the prepared AC was determined as a ratio of the mass of the resultant AC to the mass of the precursor as expressed in eqn. (4) (Musah *et al.,* 2011; Yulu *et al*., 2003)

$$\% Yield = \frac{Wix100}{Wo} \tag{4}$$

Wo is weight of the precursor; *Wi* is weight of AC

pH zero point of charge (pHzpc)

The zero point of charge of the surface of the AC was obtained using the salt addition method by described by Cardenas-Pena *et al.* (2012). In the method, 2.0 g of each AC was weighed and transferred into 10 different 50.0 cm^3 beakers followed by the addition of 20.0 cm^3 distilled water to each beaker. pH was adjusted with either 0.1 moldm^{-3} of HCl or 0.1 moldm^{-3} of NaOH to attain pH values 2.0, 3.0, 4.0, 5.0, 6.0, 7.0, 8.0, 9.0, 10.0 and 11.0. They were left to stand for 4 days with intermittent stirring, then the pH of solution in each beaker was measured and recorded as pH$_i$. Then 0.5 cm^3 2 mol/dm^3 NaCl solution was added with continual stirring for 3 hrs. Thereafter another pH value was obtained and recorded as pH$_f$. for each beaker. Change in pH was calculated as ΔpH = pH$_f$ - pH$_i$ and a plot of ΔpH versus pHi was used to determine the point of zero charge as the point where ΔpH equals zero.

Surface morphology

Scanning electron microscopy (SEM) was used in the determination of the morphology of the AC surface. Sample was prepared for SEM analysis by suspending about 0.02 mg in 1 cm^3 of methanol. The solution was sonicated for 10 minutes and 2 drops was coated with gold by a gold sputtering device to obtain a good visibility of the surface morphology. SEM image of the AC was then obtained on a Joel JEM 100S SEM operating at 80 kV (Musah *et al*., 2016; Sugumaran *et al*, 2012).

Determination of functional group

Sample quantity of 0.1 g of the AC was mixed with 1.0 g of KBr and hydraulic press was used to compressed the sample to obtain a pellet disc of 1.0 mm thick before Perkin-Elmer Fourier transform infrared spectrometer was employed to analyse the sample and the spectra was recorded within the range of 450 – 4000 cm^{1}. (Mathew *et al.,* 2023a).

Particle size determination

The particle size of the AC was determined using Zetasizer Nano S at scattering angle of 173^o operating at a temperature of 25 ^oC with equilibrating time of 10.0 sec/run. One (1.0) mg of the AC was dispersed in 10.0 mL of water by means of sonication (Misonix 3000; power: 1 W/mL, pulse on: 3.0 sec, pulse off: 3.0 sec, time: 1.0 min) and a centrifugation (5.0 min at 3000 rpm). The supernatant (without large aggregates) was transferred into a cuvette using syringe with 0.23 μm filter, then placed in the Zetasizer Nano S cuvette holder for analysis (Musah *et al.*, 2018).

RESULTS AND DISCUSSION

Results of the physico-chemical Properties of HVP and KVP are presented in Table 1. pH values obtained for HVP and KVP were 6.78±0.00 and 7.18±0.01 respectively. Bulk density values for the activated carbons were slightly different, with the value for HVP (0.72±0.03 g/cm^3) been higher than that obtained for KVP (0.69±0.05 g/cm^3). Attrition values of HVP and KVP were 40.76±0.16 and 42.37±0.23 % respectively.

Table 1: Physico-chemical Properties of the Activated Carbons

Parameter	HVP	KVP
pH	6.78±0.00	7.18±0.01
Bulk density (g/cm^3)	0.72±0.03	0.69±0.05
Attrition (%)	40.76±0.16	42.37±0.23
Yield (%)	21.37±0.15	23.17±0.37
Burn off (%)	78.63±0.19	76.83±0.24
Pore size (nm)	2.13±0.02	2.02±0.01
Pore volume (cm^3/g)	0.13±0.01	0.14±0.00
Surface area (m^2/g)	265.63±0.00	276.32±0.00

Key: HVP = activated carbon modified with H_3PO_4; KVP =activated carbon modified with KOH.

Bulk density of activated carbons to a large extent is dependent on the nature and type of material used and the preparation process. This property is employed to ascertain the capacity of AC for liquid retention and compressibility (Hebert, 2011). Results indicate that HVP had higher (0.72±0.03 g/cm^3) bulk density than KVP (0.69±0.05 g/cm^3). Values were greater than the 0.34±0.00 g/cm^3 and 0.33±0.00 g/cm^3 reported for ACs prepared from *Delonix regia* fruit pod and banana empty fruit bunch respectively (Sugumaran *et al.*, 2012) but comparable to the 0.72 g/cm^3 reported for AC prepared from *Balsamodendron caudatum* wood (Sivikumar *et al.*, 2012).

Attrition, a measure of hardness was found to be 40.76±0.16% for HVP and 42.37±0.23% for KVP indicating that KVP is harder than HVP. These activated carbons were slightly harder than adsorbent prepared from pulp (40.00%) reported by Kale (2019). Percentage yield of 21.37±0.15% and 23.17±0.37% were obtained for HVP and KVP after activation. The yield for HVP was lower than that of KVP and both yields were lower than the 31.9% reported for *Eucalyptus camaldulensis* Dehn Bark AC (Kongsuwan *et al*., 2006). Very low burn off value is an indication that the prepared AC may have low capacity for adsorption of target adsorbate (Senthilkumaar *et al*., 2010). Burn offs of 78.63±0.19 % and 76.839±0.24% were obtained for HVP and KVP respectively. These values were higher than the 69.78±0.15 % reported palm kernel shell AC but lower than 85.1% obtained for *Carica papaya* AC (Musah *et al*., 2011; Karthikeyan and Ilango, 2008).

The results obtained for surface area, pore volume and pore size of HVP and KVP are presented in Table 1. These parameters are important and known to influence the suitability and applicability of any AC. According to the classification of pore dimensions by IUPAC, pores can be grouped into three groups based on the sizes their pores; micropores has pore size of less than 2.0 nm, mesopores with pore size between 2.0 and 50.0 nm while the size of macropores are greater than 50.0 nm. Pore size distribution of HVP and KVP studied indicate that there was no significant difference in the pore sizes of HVP (2.13±0.02 nm) KVP (2.02±0.01 nm). Activating agents used during production seem to have similar influence in the size of pores created. Although mesoporous, these pores were slightly smaller than 2.76 nm reported for pomegranate peel AC (Ahmed *et al*., 2014). Smaller pore is considered suitable for the sorption of ions from solutions. Values of pore volumes obtained for HVP and KVP were 0.13±0.01 and 0.14±0.00 cm^3/g respectively. These values were lower than 0.1714 obtained for functionalized acid modified gold coast bombax AC but higher than 0.0580 cm^3/g reported for AC prepared brewer's spent grain (Musah *et al*., 2016; Mussato *et al*., 2010). The results indicated that HVP had a surface area (265.63±0.01 cm^2/g) that was similar to 276.32±0.03, cm^2/g obtained for KVP. Although values obtained were higher than the 111.75, 94.264 and 183.89 cm^2/g reported for Acs prepared from date tones, orange peels and pomegranate peels respectively but were lower 459.66 cm^2/g obtained for coconut shell activated carbon (Hussain *et al*., 2023; Madojemu *et al*., 2020). Higher surface area value indicates the carbon's ability to cover wider surface.

Figure 1 present the point of zero charge (pHzpc) for HVP and KVP respectively. The pHzpc of a sorbent is the pH at which the AC surface show net electrical charge of zero when submerged into electrolyte (Sumithra *et al*., 2014). pH value is used to describe the zero point of charge (zpc) only for systems were H^+/OH^- are the potential determining ions. When the pzc value is greater than pH value, the surface of the AC is positively charged and will attract negatively charged ions (anions). When pzc value is lower than the pH value, the surface of the AC is negatively charged hence increasing the electrostatic attraction between cations and the AC (Dawodu and Akpomie, 2014).

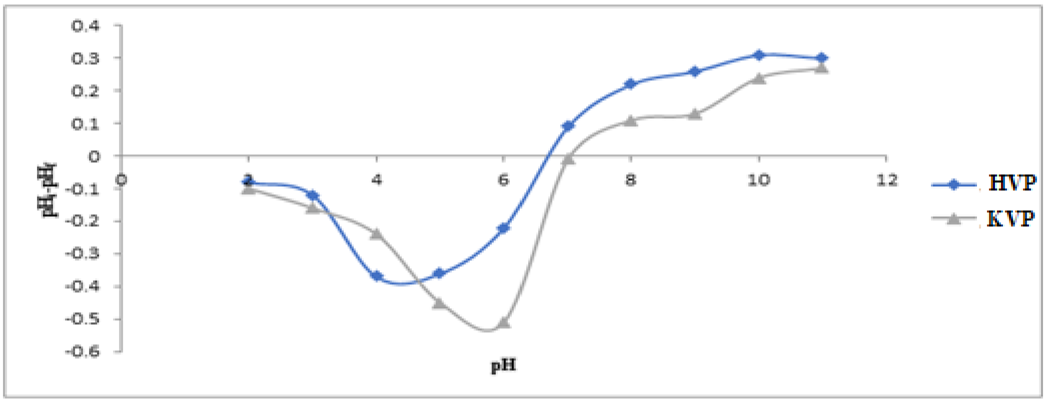

Figure 1: pH Point of Zero Charge for H_3PO_4 modified (HVP) KOH modified (KVP) activated carbons

Values of pHpzc obtained for HVP and KVP were 6.60 and 6.90 respectively. These can be interpreted as pHpzc < pH for HVP and KVP (6.60 < 6.78 and 6.90 < 7.18). The surfaces of the ACs were negatively charged as result of the hydroxyl (OH^-) group present in HVP and KVP. The pHzpc values (6.60 and 6.90) obtained are similar to the 6.8 reported for activated carbon prepared from agro-waste reported by Njewa *et al*. (2022).

To determine particle size of the activated carbons, values obtained for particle size determination using Zetasizer Nano S for HVP and KVP were employed to developed correlation charts (Figures 2-3) where the average aspect ratios, diameters and lengths of HVP and KVP were obtained (see Table 2). The correlation charts presented in Figures 2-3 indicate low aspect ratio value of 127.95 for HVP compared to 186.57 obtained for KVP. The diameter of HVP (38.90 nm) was higher than the 18.73 nm obtained for KVP; similarly, higher length value (4977.11 nm) was observed for HVP compared to the 3494.46 nm obtained for KVP. These values suggest smaller particle size for KVP adsorbent. The aspect ratio of activated carbon generally refers to the ratio of its particle size, specifically the ratio of its diameter to its length or width.

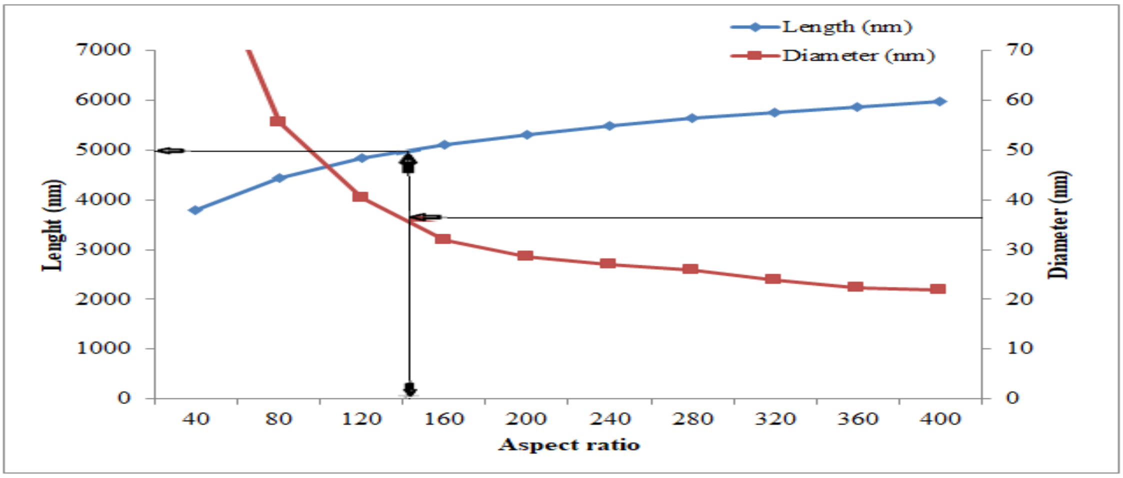

Figure 2: Aspect ratio correlation of length and diameter of HVP

From Figure 2, the aspect ratio, length and diameter of HVP are 127.95, 4977.11 and 38.90 respectively.

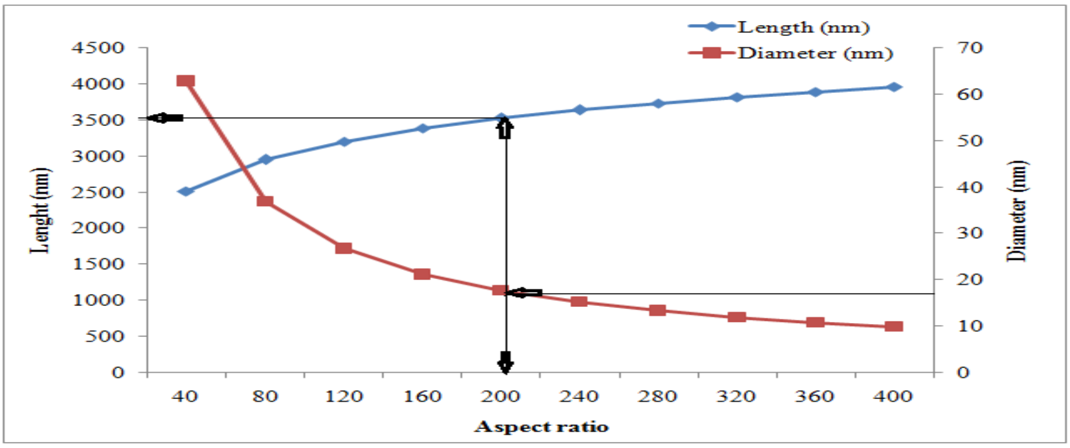

Figure 3: Aspect ratio correlation of length and diameter of KVP

Correlation charts depicted in Figures 1 and 2 indicated that value of aspect ratio obtained for HVP (127.95) was lower than the 186.57 for KVP but diameter (38.90 nm) and length (497.11 nm) were higher than 18.73 nm for diameter and 3494.46 nm for length obtained for KVP. These values corroborate the values of aspect ratios for HVP and KVP.

Table 2: Aspect ratio, length and diameter activated carbons

Parameter	HVP	KVP
Aspect ratio	127.95	186.57
Length (nm)	4977.11	3494.46
Diameter (nm)	38.90	18.73

The various forms and shapes of activated carbons are indications that they can have different aspect ratios based on their manufacturing process. Aspect ratio may be considered an important factor in cases where the physical shape of the activated carbon (AC) particle could impact the AC's effectiveness.

The FTIR spectra and band assignment of HVP and KVP are depicted in figures 4 - 5. The bands around 3439.19-3280.1cm^{-1} and 33287.5 cm^{-1} in HVP and KVP are associated to the O-H of carboxyl group; while spectra at 1736.9 cm^{-1}and 1744.4 cm^{-1} are ascribed to C=O of ester group in HVP and KVP ACs. The bands related to S=O group of sulphate were observed at 364.2 – 1420.1 cm^{-1} (figure 4) and 1375.4 – 1457.4 cm^{-1} (figure 5) for the acid modified (HVP) and base modified activated carbons respectively. The absence of peaks around regions that are associated to the presence of hydroxyl functional group in HVP and KVP is an indication of their absence and this could be due to the heat and chemical reactions that took place on the AC surface during the process of preparation. (Auta and Hameed, 2011).

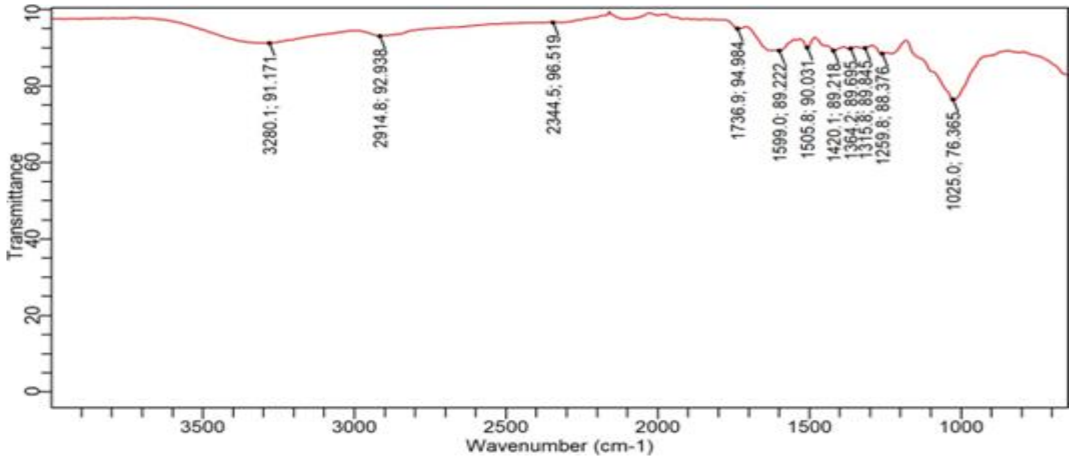

Figure 4: FTIR Spectra of HVP

The spectrum for HVP showed peaks at different wave numbers that were not similar to those in KVP: 1259.8cm^{-1} and 1535.39cm^{-1} which indicate the presence of C-O of carboxy and P-H of phosphine. This could be due to the activating agent used in the preparation of HVP The C-N functional group of amines was detected at bandwidth of 1025.0 cm^{-1} and 1017.6 cm^{-1}, 1505.8 and 1513.3 5 cm^{-1} also indicates the N-H of amine functional group in the HVP and KVP respectively. Similar band was reported at 1000-1300 cm^{-1} for C-O stretching of carbon in jute stick activated carbon (Nurul *et al*., 2022). The presence of band at 2914.8 cm^{-1} and 2922.2 cm^{-1} carboxyl for HVP and KVP respectively are ascribed to the presence of O-H in carboxyl group.

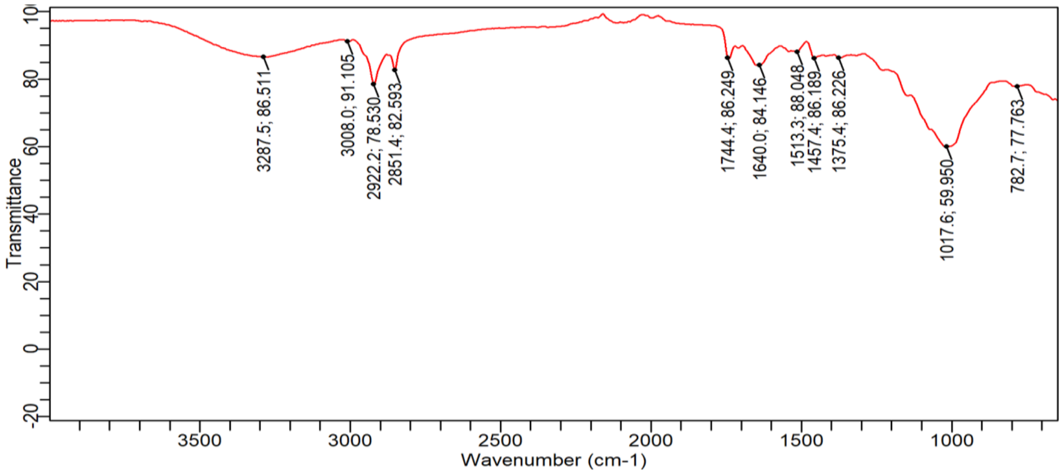

Figure 5: FTIR Spectra of KVP

Surface morphologies of HVP and KVP as revealed by their SEM micrographs are presented in plates I and II. The images. The SEM micrographs of HVP and KVP showed uneven, rough and flaky surfaces with pores of different dimensions and forms present within the activated carbons.

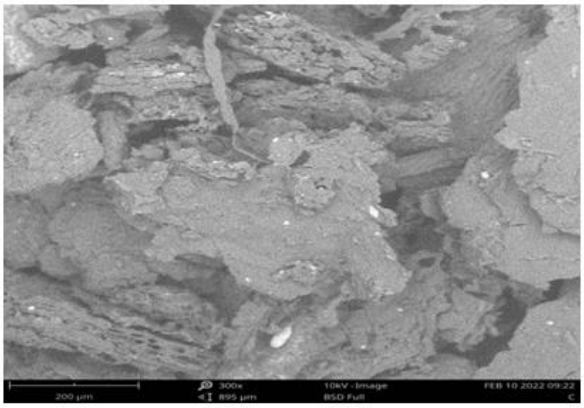

Plate I: SEM image of HVP

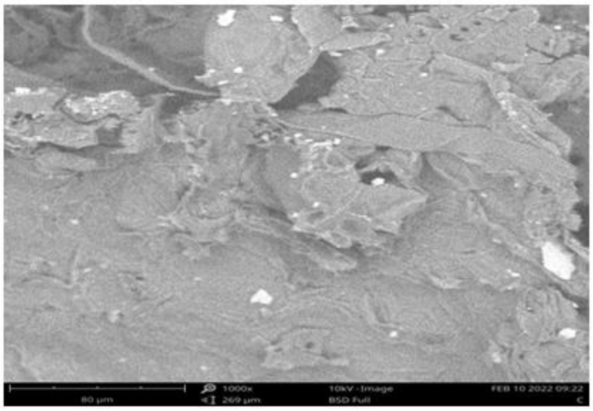

Plate II: SEM image of KVP

The presence of cave-like openings observed were prominent on the surface of HVP. Similar uneven, rough and fractured surfaces were observed in the SEM micrographs of activated carbon prepared from spent coffee material impregnated with phosphoric acid (Ziezio *et al*., 2020).

CONCLUSION

Modification of *Vitellaria paradoxa* nut shell with H_3PO_4 and KOH improved properties such as bulk density and its ability to withstand abrasion during sorption. The low values of pHzpc compared to the pH, high surface area, pore volume, the presence of organized graphitic structure in the adsorbents are indications of the suitability of the prepared activated carbon for use in the treatment of wastewater.

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