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Abstract

Activated carbon prepared from vitellaria paradoxa (shea nut) shell using otho-phosphoric acid has been used for the removal of Cd(II) ion from aqueous solution. The adsorbent sample was characterized by some physicochemical and spectroscopic parameters such as pH, point of zero charge (PZC), moisture content, iodine number, loss on ignition, bulk density, XRF, SEM and TEM. The pH and the PZC of the sample were found to be 4.0 and 5.8 respectively. The adsorbent sample has moisture content of 5.22 ± 0.1%, iodine number of 456.50 ± 59%, loss on ignition 10.71 ± 0.18% bulk density of 0.84 ± 0.09%. The XRF analysis indicated that Ca, Si and Fe were major constituents in the adsorbent sample. The SEM and TEM analysis results indicated good adsorptive characteristics of the adsorbent. Result from the FTIR analysis indicate presence of some important components such as C=O, O-H, C-O N-H, and P-H which responsible for high adsorptive capacity of the adsorbent. The equilibrium adsorption of & G , LRQ GDWD ZDV ZHOO ¿ WWHG ZLWK / DQJP XLU DQG) UHXQGOLFK LVRWKHUPV UHVSHFWLYHO\ 7KH NLQHWLF VWXG\ UHYHDOHG WKDW SVHXGR VHFRQG RUGHU ¿UVW RUGHU NLQHWLF 7KHUPRG\QDPLF H[SHULPHQW LQGLFDWHG WKDW WKH DGV .- PRO DQG "6 HTXDOV .- PRO.

Vitellaria paradoxa) is found in areas with 400-

1800 mm rainfall per year. It is a multi-purpose tree daily used by rural African communities. e fruit when very ripe is either eaten as a snack, but it is also a famine food. It can be eaten raw or slightly cooked. e pulp can be processed into juice. According to McAllan et al. [14], the pulp could also be removed by fermentation. e nuts are

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was 204.2 and 256.2 respectively (Table 1). e result indicated that the activated sample prepared by acid activation possesses good adsorptive capacity. Higher iodine number re ects better development of the microporous structure and higher adsorption abilities [29,30]. It was clear that the iodine number of acid activated adsorbents is high due to the phosphoric acid destroying the aliphatic and aromatic species present in plants therefore swi ly removing the volatile matters during the carbonization process as reported by EI-Hendawy [31].

Scanning electron microscopy (SEM)

e surface morphology of the adsorbent was analyzed by scanning electron microscope (SEM). e results of SEM analysis are shown in the micrographs Figures 1-3. e raw sample in Figure 1 showed rough areas of surface of the carbon and very minute micropores.

It was generally observed that the pore structure development is in uenced by many factors, such as inorganic impurities and the initial structure of the carbon precursor [32]. It can clearly be seen in Figure 2 that chemical activation resulted in a porous structure and the opening of pores on the surface of the activated sample.

SEM analysis a er adsorption of Cd in Figure 3 however showed a reduced pore sizes as compared to that of the acid activated sample.

Transmission electron microscopy (TEM)

Transmission electron microscope (TEM) was used to determine the internal morphology of the adsorbent. e results of TEM analysis of the adsorbents, raw sample, activated sample and sample a er adsorption are shown in Figures 4-6.

e internal pore size of the raw sample Figure 4 was between 0.94-2.90 m, those of the activated sample Figure 5 ranged between 5.27-5.92 m. is improvement in pore size of the activated adsorbent could be due to the carbonization in the presence of acid.

However, a er adsorption, the internal pore size of the Cdembedded adsorbent reduces between 2.43-2.86 m Figure 6. is observation indicated that the adsorbent e ective on Cd adsorption,

Figure 1: SEM Micrograph of raw sample.

Figure 2: SEM Micrograph of activated sample.

Figure 3: SEM Micrograph of activated sample after adsorption.

Figure 4: TEM Micrograph of raw sample.

Figure 5: TEM Micrograph of activated sample.

since the internal pore sizes reduced Cd-embedded adsorbent. is implies that Cd ions occupied spaces in adsorbent.

XRF elemental analysis

e elemental analysis of the adsorbent as revealed by XRF (Table 2) result showed that Ca, Si and Fe are present as major constituents

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the modi ed and metal-loaded adsorbents. ese groups might have been destroyed or evaporated during carbonization or converted into another functional group.

e sharp stretch vibrations found within band range 2284-2400 cm⁻¹ appeared to represent functional group of phosphorus belonging to phosphine (P-H). e presence of phosphine group is expected to increase the adsorptive capacity of the adsorbents.

e stretch vibrations of C=O in ketones and aldehydes were found in the range 1700-1735 dr[35]. is peak appeared in raw and Cd-loaded adsorbent but absent in activated adsorbent. is observation may be attributed to the loss of \mathcal{C} Ouring carbonization process and do not reappear a er activation.

Na, Ti and Zn are minor constituents while Mg, Al, P, Mn, Co, Ni, Cu and Cd are in traces.

e point of zero charge of activated sample was determined by potentiometry method. e graph of the pH of sample solution and that of the blank solution were plotted as shown in Figure 7. It could be observed that the pH drop for the sample solution and the blank solution intercepted at pH 5.8, this shows that the **phra**s found to be 5.8 for vitellaria paradoxa. Cation adsorption becomes enhanced at higher pH than pHpzc, while adsorption of anions equally enhanced at pH less than pHpzc [33].

FTIR Results of the raw sample, activated sample and a er adsorption

e comparisons of the Fourier transform infra-red result spectra of raw, modi ed and a er adsorption of the vitellaria paradoxa were given in gures. e FTIR spectrum of the raw reveals complex nature of the adsorbent as evidenced by the presence of a large number of peaks. e band between 3000-3500 chas been assigned to free and intermolecular bonded hydroxyl (O-H) group of alcohol, phenol or carboxyl [34]. ese bonds were present in raw, modi ed and metalloaded adsorbents; indicating participation of the (O-H) functional group in the metal binding. e peaks at 2918.40 chr2046.54 cm ¹, 1633.76 cm and 1427.37 cm have been assigned to C-H, C AC, C AN and C=C respectively in the raw but were completely absent in

e bend vibrations of N-H at band range 1450-1550 $\dot{\mbox{ cm}}$

trend could be as a result of the increase in the electrostatic interaction mained constant at pH above 6. e maximum percentage removal between the Cd(II) the absorbent active sites. Moreover, this can beCd(II) was 95.62%.

explained by the fact that more adsorption sites were being covered as the metal ions concentration increases [37]. In the case of low metal ions concentration, the ratio of the initial number of moles of metal ions to the available surface area of the adsorbent is large and

subsequently the fractional adsorption becomes independent of initial pigure he prst stTd 7 Tw rapi6(csoraseions penn 75%sed ncreased, concentration. However, at higher concentrations, the available sites of adsorption become fewer and hence the percentage removal of metal

ions which depends upon the initial concentration decreases [38]. It could however be deduced from the result above the equilibrium

concentration is at 200 mg/L.

E ect of pH: e e ect of initial solution pH on the adsorption capacity at equilibrium condition was studied. e result for the adsorption of Cd(II) is shown in Figure 8 using 200 mg/L as initial metal concentration. It could be seen that the removal concentration and percentage removal is pH dependent. It was clear that the degree of metal ions adsorption onto the adsorbent increased from 50.48 mg/L to maximum of 57.37 mg/L of Cd(II) when the solution pH was increased from 2-6. However, at the adsorption capacity of the adsorbent

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deposited metal ions penetrated to the interior of the adsorbent through intra-particle di usion which was slower process. is is in accordance with the observation of other similar studies (Figure 10) [40,41].

E ect of temperature: s (Figuen.86

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 $\Delta G = \Delta H - T \Delta S$

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