

Research Paper

Influence of adsorption factors on the removal of Cr (VI), Mn (II) and Cd (II) ions from solutions using modified *Bombax buonopozense* calyx

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The potential of chemically modified *Bombax buonopozense* calyx to adsorbed Cr⁶⁺, Mn⁶⁺ and Cd⁶⁺ ions from aqueous solutions are prime intent in batch experiments. In this study, activated carbon (AC) prepared from *Bombax buonopozense* Calyx by chemical modification using H₂SO₄ (ACH) and KOH (ACK) for adsorption process is reported. The variations in the percentage removal of Cr⁶⁺, Mn²⁺ and Cd²⁺ ions were established using the effects of time, adsorbent dosage, initial concentration and pH. The activated carbon modified H₂SO₄ (ACH) showed higher percentage removal

of Cr⁶⁺, Mn²⁺ and Cd²⁺ ions than ACH at 120 min contact time, 0.8 g adsorbent dosage, 10 mg/dm³ initial concentration and pH between 4 and 6, while the corresponding trend order of adsorption were reported to be Cr⁶⁺ > Mn²⁺ > Cd²⁺ on the adsorbents.

Key words: Activated carbon, adsorption, aqueous solutions, initial concentration, batch experiments

INTRODUCTION

Different functional groups including carboxyl, hydroxyl and amino groups on the surface of the activated carbons are known to have high affinity towards cations (Patil et al., 2012; Allwar, 2012). Adsorption of cations onto the surface of the adsorbents results from electrostatic attraction between carboxylate ion (-COO⁻) and/or oxyanion (-O⁻) on the adsorbent surface and the cation (Yakout and Elsherif, 2010). In elastic physisorption, the attraction exists between the cationic adsorbates and the negative poles of partial polar bonds of CO, OH and NH groups of the activated carbon involved. Adsorption can also be due to ion exchange between hydroxyl (-OH) or carboxyl (-COOH) groups on the adsorbent surface and cationic adsorbate, thereby releasing protons (H⁺) into the solution (Patil et al., 2012). The interaction of cation with the activated carbon surface can be illustrated thus: ('A' represents the adsorbent surface and M⁺ is the cationic adsorbate).

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cationic adsorbates and the negative poles of partial polar bonds of CO, OH and NH groups of the activated carbon involved.



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hydroxyl (-OH) or carboxyl (-COOH) groups on the adsorbent surface and cationic adsorbate, thereby releasing protons (H⁺) into the solution (Patil et al., 2012).



The study seeks to determine the suitability of adsorbent treated with H₂SO₄ and KOH in the removal of Cr (VI), Mn (II) and Cd (II) ions from aqueous solutions and pharmaceutical wastewater.

MATERIALS AND METHODS

Production of activated carbon

The production of activated carbon from dried raw materials involved two stages: carbonization of the *Bombax buonopozense* calyx followed with the activation of the carbonized sample at a high temperature.

Carbonization

Five grams of *Bombax buonopozense* calyx were weighed and introduced into six different pre-weighed and clean crucibles, and then put into muffle furnace and carbonized at 400°C for 20 min (resident time) after which it was quenched in iced water before washing with distilled water. This procedure was repeated until large amount of carbonized sample is obtained. The carbonized samples were washed with 0.1 M HCl to eliminate ash from the surface, then with hot water, followed by washing with distilled water to further remove acid residue. The washed samples were dried in an oven at 105°C for 60 min (Rahman et al., 2014).

Preparation of activated carbon

Five grams of the carbonized sample were mixed with 5 cm³ of activating agent (1 M H₂SO₄). The procedure was repeated using 1 M KOH. The samples were allowed to stand for 120 min and then heated at 600°C for 20 min (resident time) in a furnace. The activated samples were removed and quenched in iced water then rinsed thoroughly with 0.1 M HCl to eliminate ash from the surface of the activated carbon, then with hot water and subsequently rinsed with plenty of distilled water to get rid of acid residue in the sample. Washing of the samples was completed when pH of the supernatant was between 6-8 and then dried in oven at 105°C overnight before being grounded and stored in air tight containers. The same process of activation was repeated using 1 M KOH as activating agent.

Sorption experiment

The batch adsorption method was used to study the effects of contact time, adsorbent dose, concentration and pH of solution on adsorption of chromium, manganese and cadmium ions from solutions.

Contact time

The effect of contact time on adsorption of chromium, manganese and cadmium ions was studied using the batch method. In the experiment, 0.2 g of the adsorbent was interacted with 20 cm³ of solution and interacted at 200 rpm for 30, 60, 90, 120 and 150 min. After which the adsorbent was removed through filtration and the filtrates were analyzed for chromium, manganese and cadmium ions using atomic absorption spectrophotometer (Variant AA240Fs-Fast Sequel Atomic Absorption Spectrometer) (Patil et al., 2012).

Adsorbent dosage

The effect of adsorbent dose on adsorption was determined by changing the masses of adsorbent from 0.2 to 1.0 g while the volume of the solution, concentration and rate of agitation per minute (rpm) were maintained at 20 cm³, 10 mg/L and 200 rpm. After interaction, the adsorbent and solution were separated by filtration of the mixture using whatman filter papers (No.42) and the filtrates were kept in sample bottles for analysis.

Initial metal ion concentration

Known mass (0.2 g) of each adsorbent was interacted separately with 20 cm³ chromium solution having initial metal ion concentrations of 10, 20, 30, 40 and 50 mg/L for 60 min at 200 rpm and 28±2 °C. The adsorbent and solution were separated by the use of whatman filter papers (No.42). The procedure was then repeated for solutions of manganese and cadmium.

pH of solution

The initial pH of the solutions containing the selected heavy metals was adjusted to 2, 3, 5, 6, 7 and 8 using dilute solutions of HCl and NaOH. Interaction time, adsorbent dosage, concentration of solution, temperature and rate of agitation were maintained at 60 min, 0.2 g, 10 mg/L, 28±2°C and 200 rpm respectively. The solution and adsorbent were separated by whatman filter paper (No. 42) after interaction and the filtrates were analyzed using atomic absorption spectrophotometer (Variant AA240 Fs-Fast Sequel Atomic Absorption Spectrometer).

RESULTS AND DISCUSSION

Effect of contact time

As previously mentioned, ACH and ACK are the H_2SO_4 and KOH modified activated carbon respectively. The relationship between time of contact (30, 60, 90, 120 and 150 min) of adsorbates with the adsorbents (ACH and

ACK) and the percent removal of Cr^{6+} , Mn^{2+} and Cd^{2+} from solution is presented in (Figure 1). It is evident from (Figure 1) that the percentage adsorption of Cr^{6+} , Mn^{2+} and Cd^{2+} increased at first as contact time increases and adsorption equilibrium was reached at 120 min for the removal of Cr^{6+} , Mn^{2+} and Cd^{2+} by ACH and ACK adsorbents after which there was no further increase in sorption as contact time increases. At this point, the

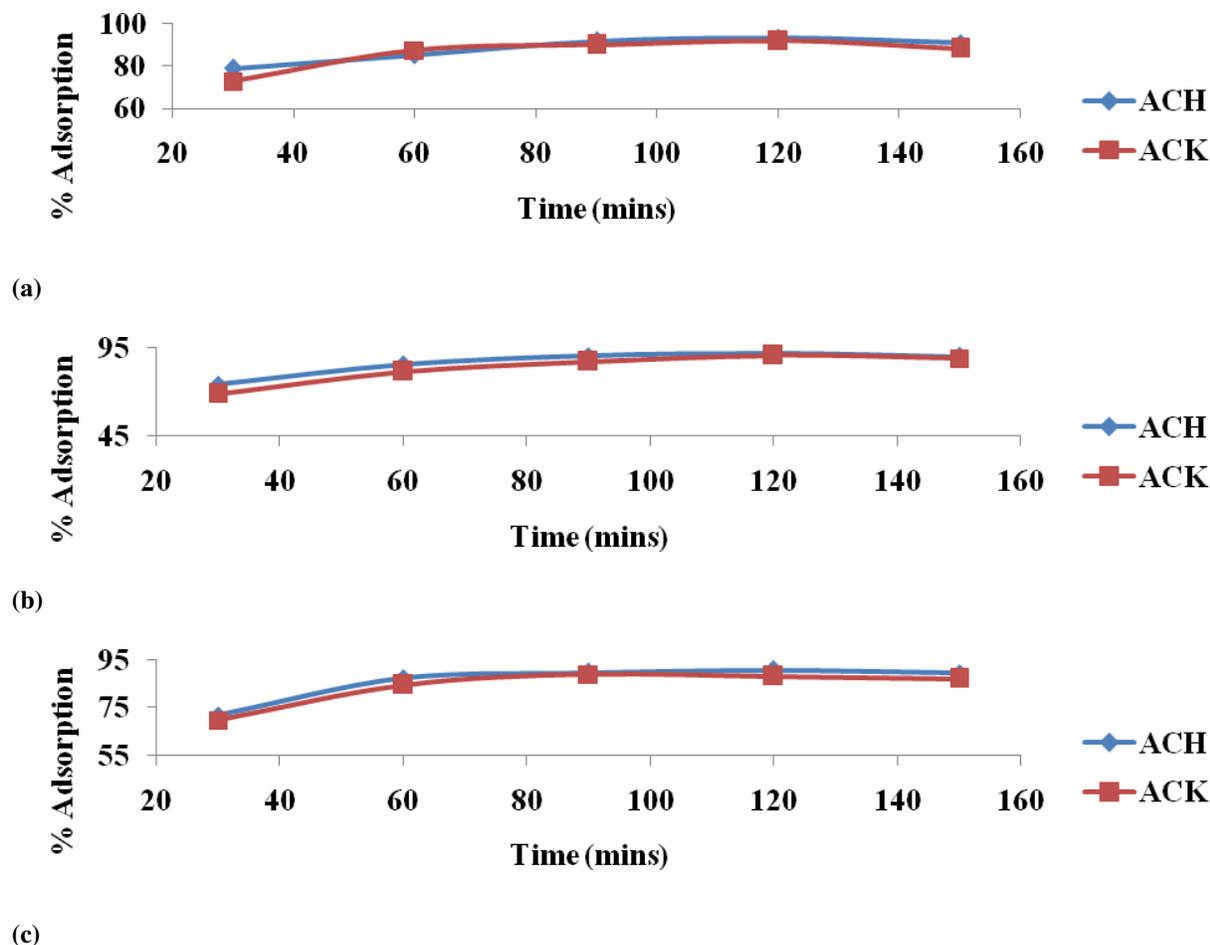


Figure 1. Effect of time on percentage adsorption of (a) Cr^{6+} (b) Mn^{2+} (c) Cd^{2+} .

adsorbents have reached saturation state and a further increase in contact time between adsorbate and adsorbent will lead to desorption of the metal ions (adsorbates) back to the solution. The rapid increase in the adsorption of metal ions at the beginning of the process is due to vacant binding sites in the adsorbents and high gradient of solute concentration (Farghali et al., 2013).

Maximum of 92.14, 91.75 and 89.97% of Cr^{6+} , Mn^{2+} and Cd^{2+} were removed by ACH while 91.97%, 90.97%

and 88.23% of the metal ions were adsorbed by ACK. The trend observed in adsorption is $\text{Cr}^{6+} > \text{Mn}^{2+} > \text{Cd}^{2+}$. This differential in adsorption could be due to the difference in ionic radii as Cr^{6+} has an ionic radius of 58 pm, Mn^{2+} has 80 pm while Cd^{2+} has an ionic radius of 92 pm (Ingatiev, 2002); the smaller the ionic size the higher is the ion's affinity to the binding site. The difference in properties of the adsorbents like surface area and pore size may also have contributed to the adsorption pattern observed.

Effect of adsorbent dosage

The availability of binding or adsorption sites and their accessibility is controlled by adsorbent dosage. The effect of adsorbent type and dosage on adsorption is presented in (Figure 2). Adsorbent dosage was varied between 0.2 to 1.0 g at a concentration of 10 mg/dm³, agitation of 200 rpm and temperature of 28±2°C. The maximum percent (%) adsorption of Cr⁶⁺, Mn²⁺ and Cd²⁺ was attained at an adsorbent dose of 0.8 g after which further increase in adsorbent dosage did not produce any significant increase in the percent adsorption of metal

ions. ACH showed more effectiveness in the removal of Cr⁶⁺, Mn²⁺ and Cd²⁺ with maximum adsorption of 95.13, 94.31 and 92.76% compared to the 93.64%, 91.87% and 91.86% obtained when ACK was used. From (Figure 2), the percent of metal ions adsorbed increases as adsorbent dose increases but the amount of metal ions that are adsorbed per unit mass of adsorbent decreased. This is inferred from the result (Figure 2) where the trend in percent adsorption showed sharp increase as adsorbent dose increased from 0.2 to 0.6 g but further increase in adsorbent dosage (0.8 -1.0 g) did not show

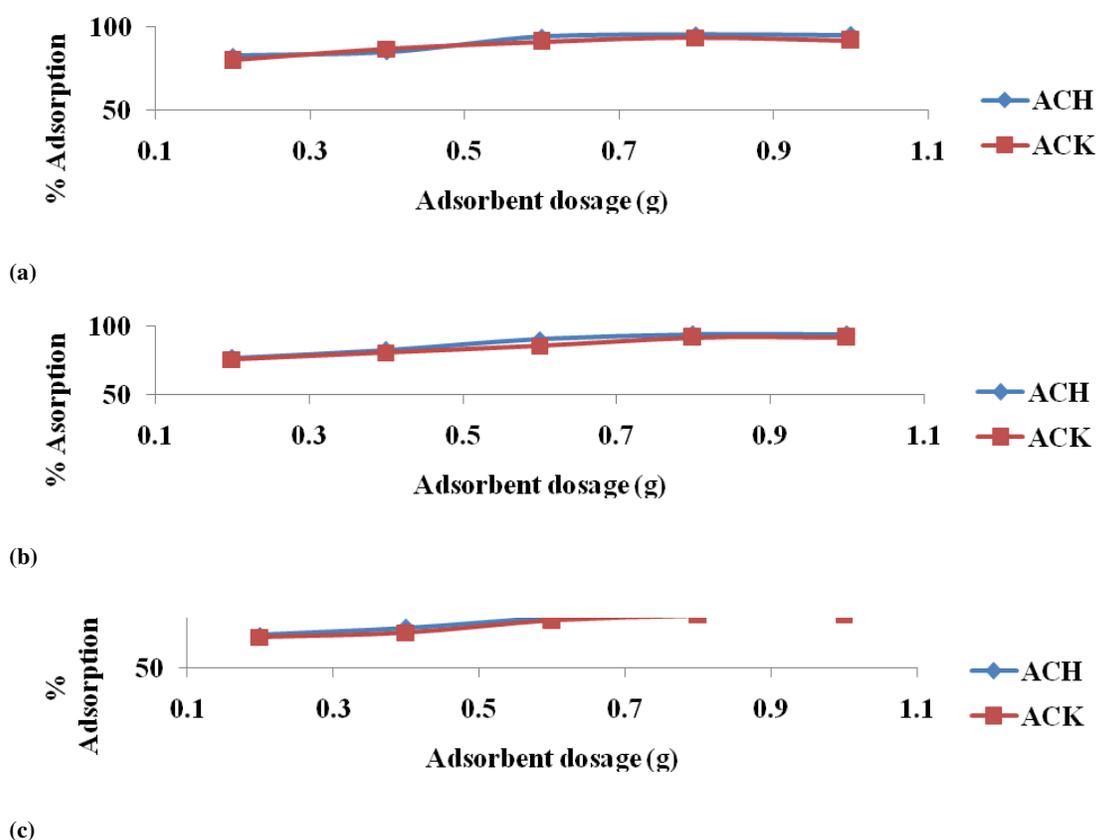


Figure 2. Effect of dosage on percentage adsorption of (a) Cr⁶⁺ (b) Mn²⁺ (c) Cd²⁺.

such a sharp increase in adsorption. The decrease in adsorption density as adsorbent dosage increases may be due to saturation of the binding sites as adsorption progresses (Patil et al., 2012; Malik et al., 2006).

Effect of initial concentration of metal ion

The effect of initial concentration variation of simulated wastewater (10, 20, 30, 40 and 50 mg/dm³) on the

adsorption of Cr⁶⁺, Mn²⁺ and Cd²⁺ onto ACH and ACK at fixed adsorbent dosage (0.2 g) and rate of agitation per minute (200 rpm) is presented in Figure 3. Maximum adsorption was achieved at concentration of 10 mg/dm³ and the lowest adsorption was at 50 mg/dm³ for the adsorbents studied (Figure 3). At low concentrations, the metal ions in the solutions can easily interact with the adsorption sites and hence increase the amount of metal ions adsorbed, but at high concentration of adsorbate, low adsorption is observed. This is attributed to the

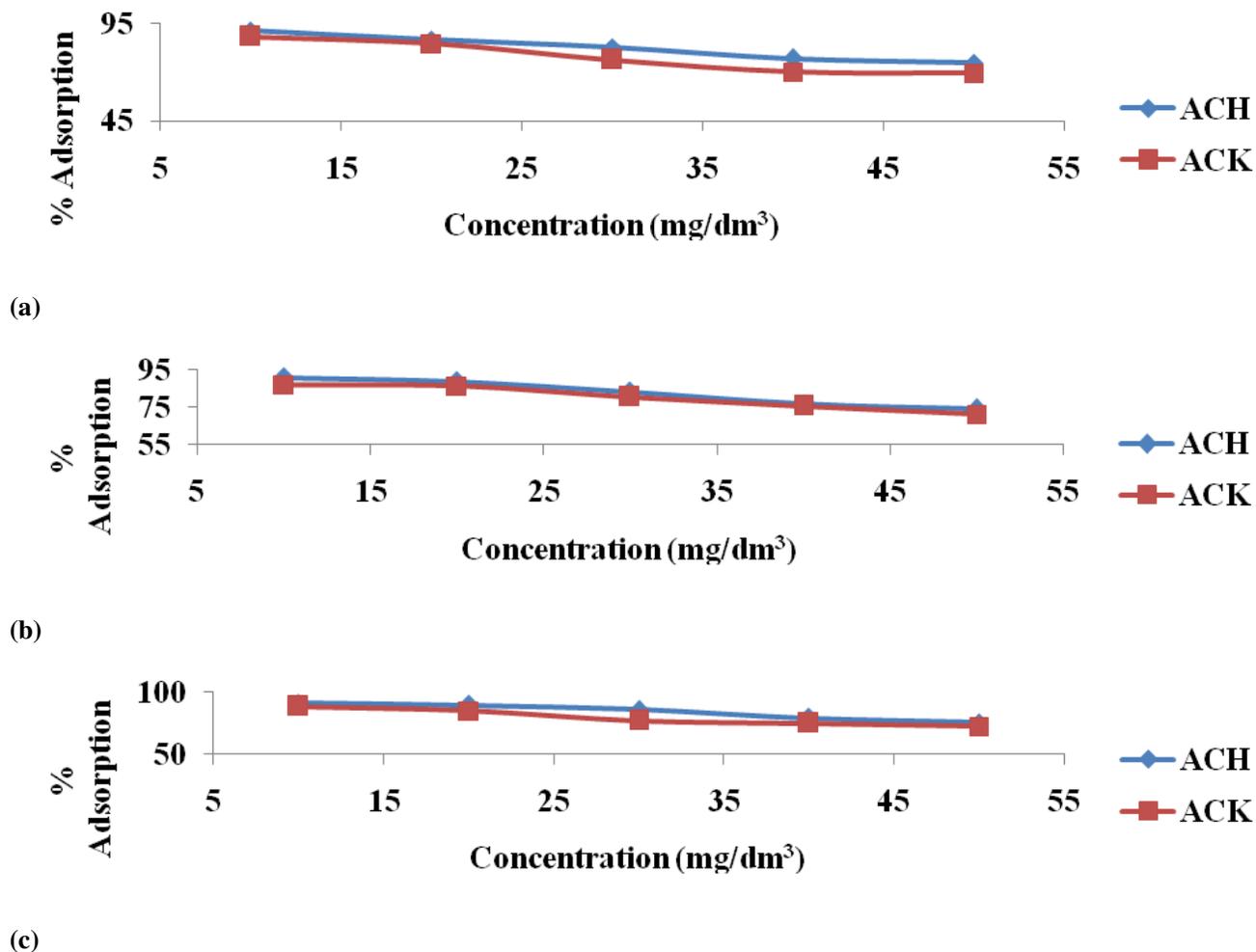


Figure 3. Effect of concentration on percentage adsorption of (a) Cr^{6+} (b) Mn^{2+} (c) Cd^{2+} .

increase in the number of ions that compete for the available adsorption sites and possible saturation of the sites at high concentration. Adsorption of metal ions also involves high energy sites on the adsorbent surface. As the initial concentration of ions is increased, the higher energy (more energetically favourable) sites gets saturated first before adsorption to the lower energy sites begins (Abdus-Salam and Adekola, 2005).

It can also be observed from Figure 3 that adsorption of Cr^{6+} , Mn^{2+} and Cd^{2+} onto the adsorbents was in the order: $\text{ACH} > \text{ACK}$. The amount of metal ions adsorbed is also a function of the affinity of metal ion for the adsorbent surface and possibly the type of complex formed on the adsorbent surface. A similar trend of high adsorption of metal ions at low concentration and low adsorption at high concentration were reported for adsorption of copper on pods of *Acacia nilotica* (Dar et al., 2013) and the removal of Cd (II) ions by *Eucalyptus globules* (Meera and Ganesan, 2015).

Effect of pH

The pH of solution regulates the surface charge of adsorbents and the degree of ionization of adsorbates present in solutions (Elkady et al., 2015). The effect of pH on adsorption was examined by varying the initial pH of solutions from pH 2 to 8 at a concentration of 10 mg/dm³, contact time of 60 min, adsorbent dose of 0.2 g and 200 rpm. Figure 4 present the effect of solution pH on the removal of Cr^{6+} , Mn^{2+} and Cd^{2+} by ACH and ACK.

The adsorption Cr^{6+} , Mn^{2+} and Cd^{2+} increased as pH of adsorbate solution increases. Maximum adsorption was attained between pH 4 and 6 after which adsorption began to decrease as pH of solution increases beyond 6. The observed adsorption order was $\text{Cr}^{6+} > \text{Mn}^{2+} > \text{Cd}^{2+}$ on the adsorbents with ACH adsorbing more. At low pH (below 3), adsorption of metal ions is low due to competition for the binding sites and electrostatic repulsion that exist between the hydrogen ion (H^+) in the

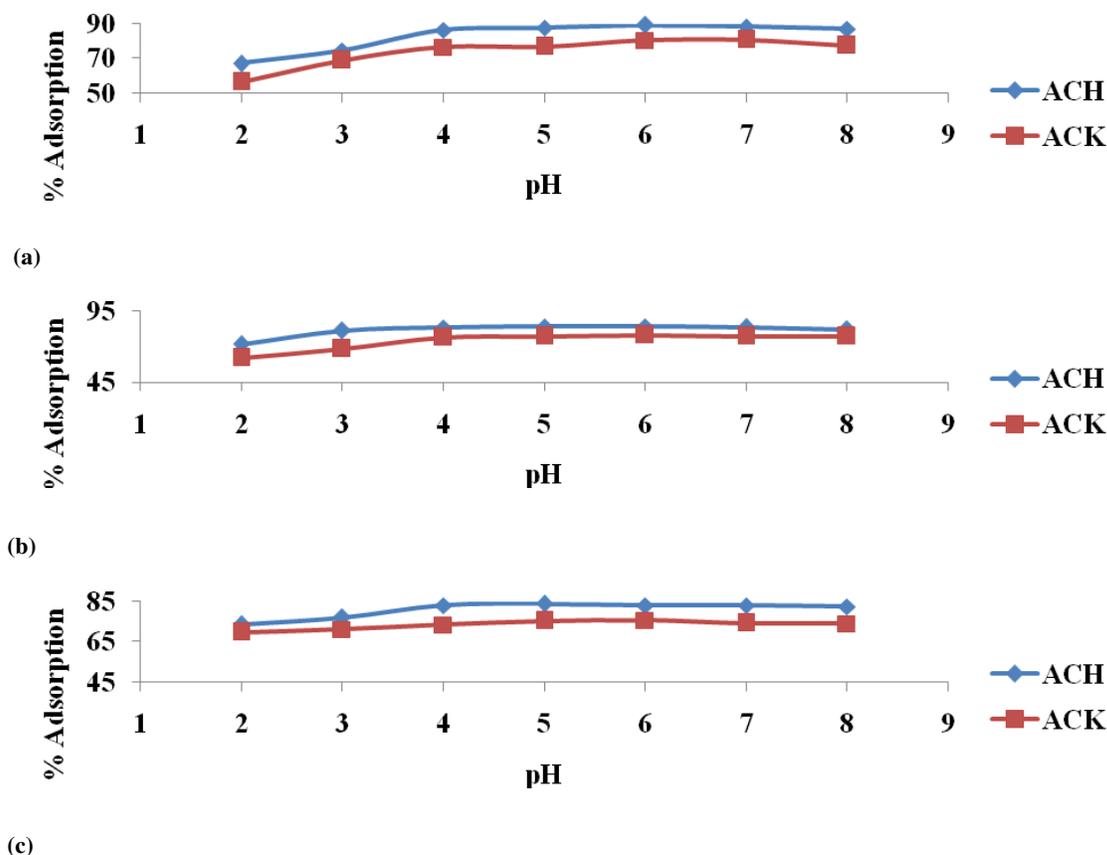


Figure 4. Effect of pH on percent adsorption of (a) Cr⁶⁺ (b) Mn²⁺ (c) Cd²⁺.

solution and the adsorbate cations (Dar et al., 2013). These limits access of metal ions to the adsorption sites on the adsorbents. At high pH, formation of hydroxylated complexes of the metal ions occurs and the resulting complexes also compete for adsorption sites in adsorbents, hence a decrease in adsorption of the metal ions occurs (Santuraki and Muazu, 2015). The pH of solution suitable for the adsorption of Cr⁶⁺, Mn²⁺ and Cd²⁺ onto ACH and ACK is higher than the 2 and 3 reported as optimum for the adsorption of Pb²⁺ from wastewater by Tehrani et al. (2013) but lower than pH 7 reported by Farghali et al. (2013) for the adsorption of Pb²⁺ from aqueous solution.

Variation of adsorption factors

Figures 5 – 8 are graphical presentations of how a change in two experimental conditions at a time can simultaneously affect the adsorption of ions. Figure 5 presents the effect of simultaneous variation in contact time and adsorbent dosage on adsorption of Cr⁶⁺ onto ACH and ACK while concentration and pH are kept

unchanged. At 30 min interaction time and 0.2 g adsorbent dosage with concentration and pH held at 10 mg/dm³ and 4.0 respectively, 83.49% of Cr⁶⁺ was adsorbed onto ACH; 93.11% was removed at 30 min and 1.0 g of adsorbent dose. The increase in adsorption is due to increase in the number of binding sites resulting from increased adsorbent dosage not the time of interaction. When time was increased to 150 there is a corresponding increase in adsorption (up to 96.53%) as dosage increased from 0.2 to 1.0 g, a similar trend was observed in the adsorption of Cr⁶⁺ onto ACK. These observations are similar to those reported for the removal Pb²⁺ (Tibet and Coruh, 2015).

Concentration of adsorbate is an important factor in adsorption. Varying concentration (10-50 mg/dm³) of solution and time of interaction (30 - 150 min) while keeping dosage and pH constant at 0.2 g and 4.0, significant adsorption of Cr⁶⁺ was observed when concentration was 10 mg/dm³ at 30 min as seen in Figure 8 (a). On the other hand, when concentration was increased from 10 to 50 mg/dm³ at 30 min interaction time, there was a decrease in the amount of Cr⁶⁺ adsorbed. This is due to increased competition for the

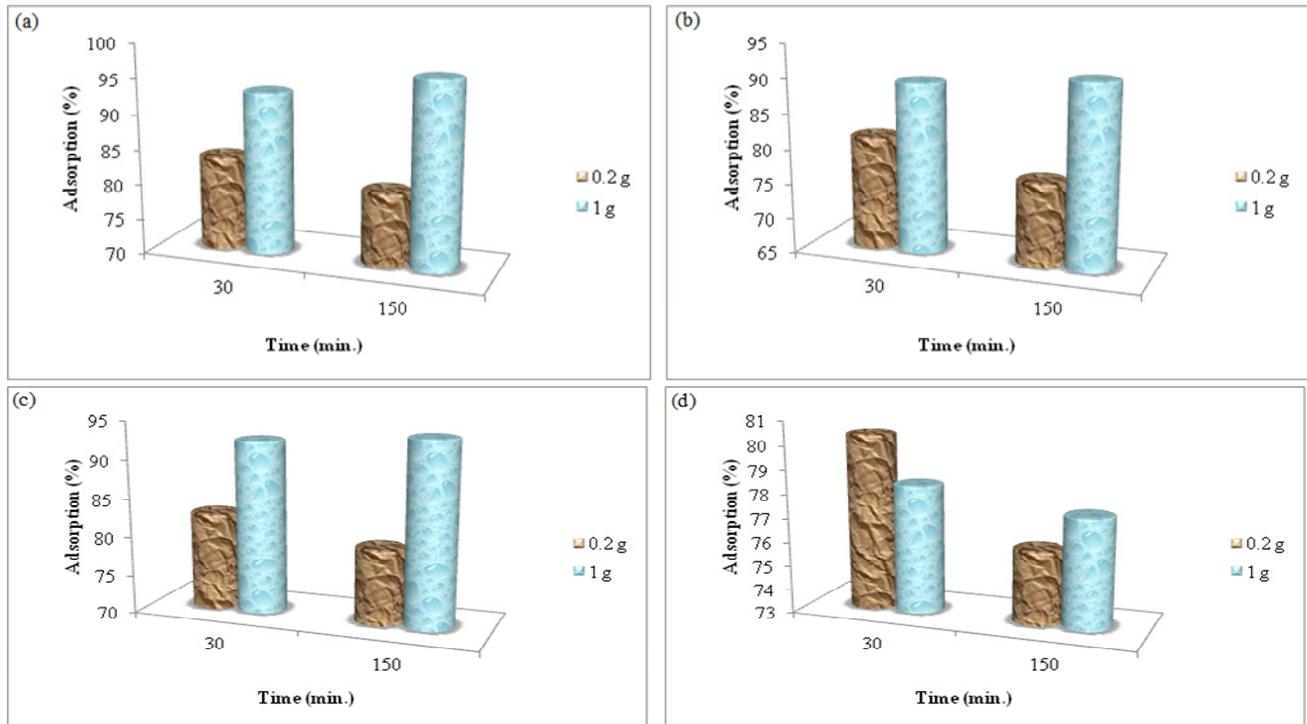


Figure 5. Effect of Time on ACH adsorption of Cr^{6+} (a) conc. of 10 mg/dm^3 and pH of 4, (b) conc. of 50 mg/dm^3 and pH of 4 (c) conc. of 10 mg/dm^3 and pH of 8 (d) conc. of 50 mg/dm^3 and pH of 8; at 200 rpm, $28 \pm 2 \text{ }^\circ\text{C}$

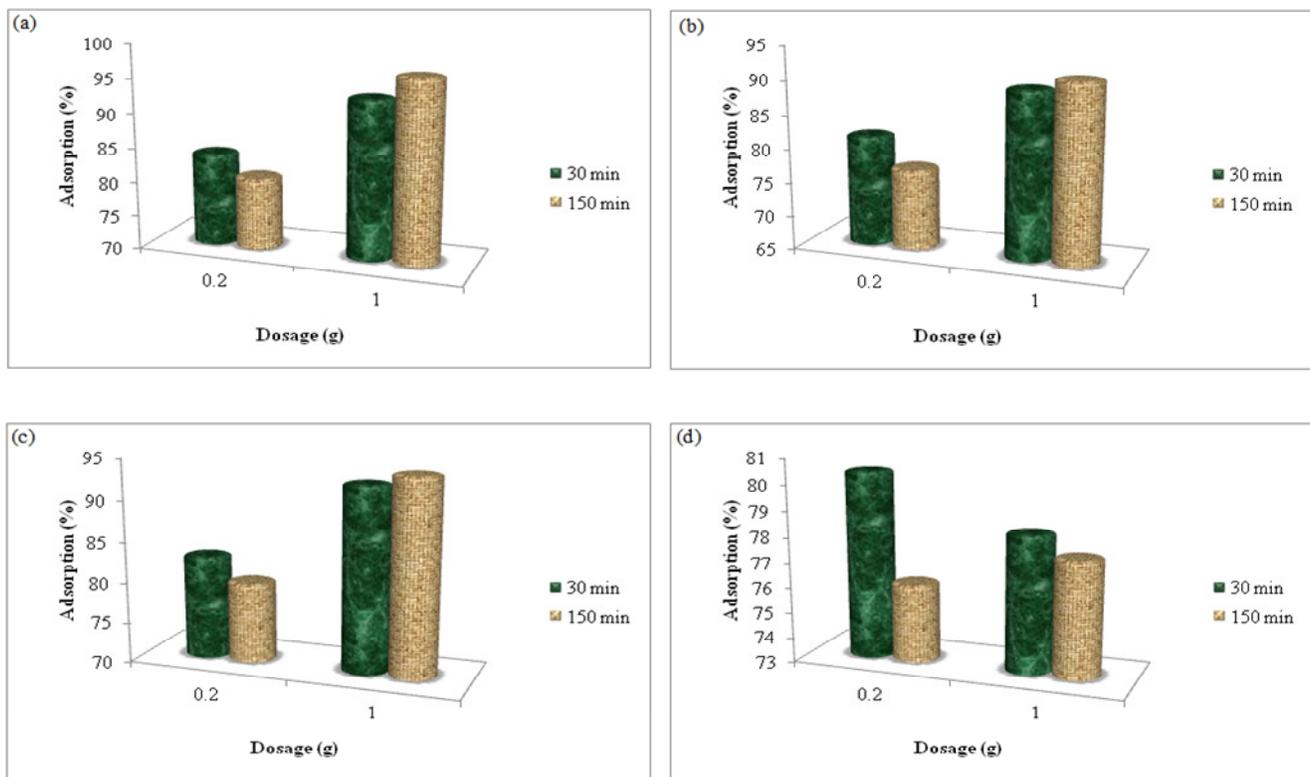


Figure 6. Effect of Dosage on ACH adsorption of Cr^{6+} (a) conc. of 10 mg/dm^3 and pH of 4, (b) conc. of 50 mg/dm^3 and pH of 4 (c) conc. of 10 mg/dm^3 and pH of 8 (d) conc. of 50 mg/dm^3 and pH of 8; at 200 rpm, $28 \pm 2 \text{ }^\circ\text{C}$.

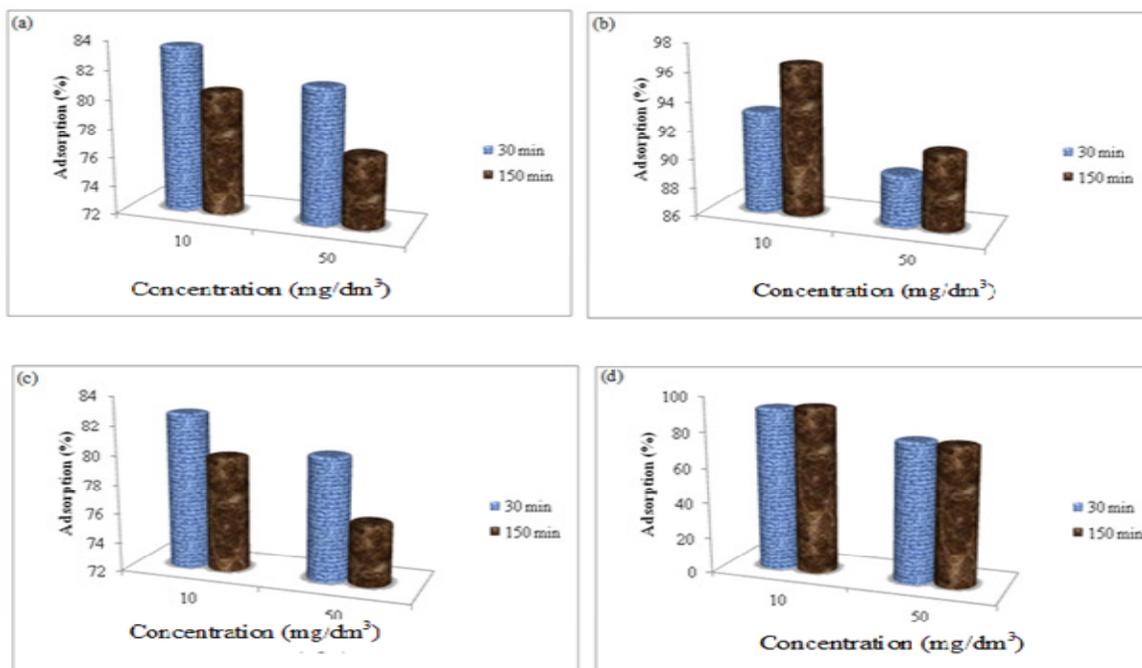


Figure 7. Effect of Concentration on ACH adsorption of Cr^{6+} (a) Dosage of 0.2 g and pH of 4, (b) Dosage of 1 g and pH of 4 (c) Dosage 0.2 g and pH of 8 (d) Dosage of 1 g and pH of 8; at 200 rpm, 28 ± 2 °C.

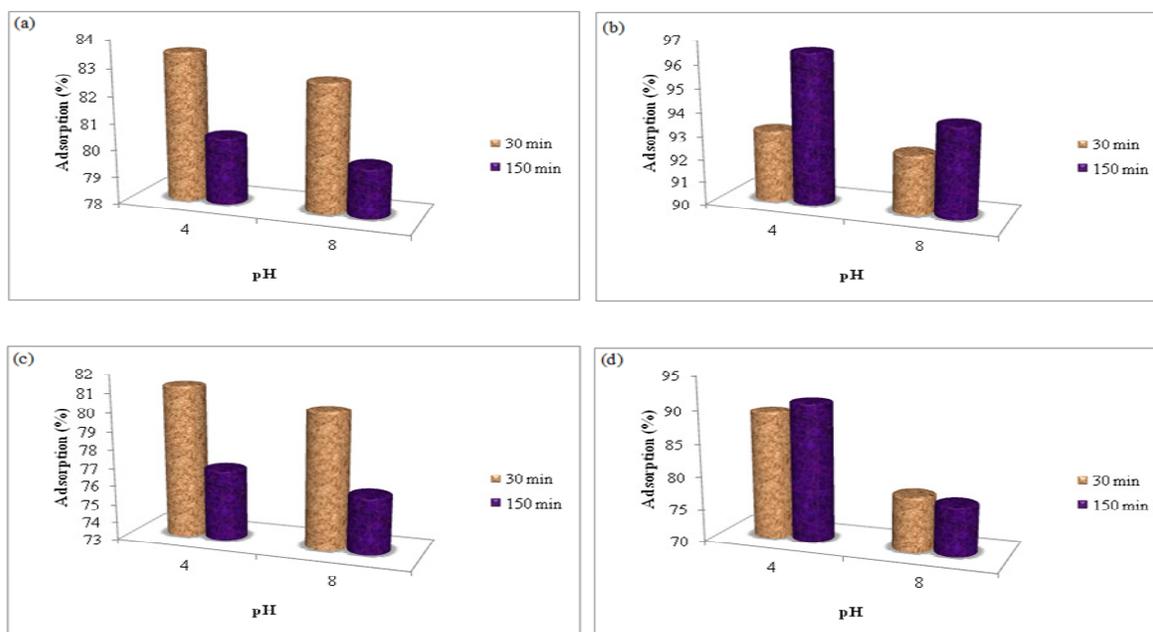


Figure 8. Effect of pH on ACH adsorption of Cr^{6+} (a) conc. of 10 mg/dm^3 and Dosage of 0.2 g (b) conc. of 50 mg/dm^3 and Dosage of 0.2 g (c) conc. of 10 mg/dm^3 and Dosage of 1 g (d) conc. of 50 mg/dm^3 and Dosage of 1 g; at 200 rpm, 28 ± 2 °C.

binding sites available on the adsorbent as more ions are present in 50 mg/dm^3 solution competing for binding sites available in 0.2 g. The amount of Cr^{6+} adsorbed at concentration of 50 mg/dm^3 and interaction time of 150

min when adsorbent dosage and pH are kept constant (0.2 g and 4.0) is lower than adsorption of 10 mg/dm^3 at 30 min. With limited binding sites and increased concentration, increase in time of interaction may not

significantly enhance the adsorption of Cr^{6+} from the solution as increase in adsorbent dosage would.

Adsorption of Cr^{6+} , Mn^{2+} and Cd^{2+} from pharmaceutical wastewater

Colour of water is a quality property that is generally used to assess wastewater condition. Light brown colour of wastewater is an indication that the wastewater is less than (\leq) six hours old (Al-tukmani, 2014). Dark gray or black colour suggests the wastewater is septic and have undergone decomposition by the action of bacteria under anaerobic condition (Al turkmani, 2014). These may depend on the type of activities taking place where the wastewater is generated.

Table 1 presents the colour and heavy metal concentration of pharmaceutical wastewater before and after interaction with the activated carbons and percent removal Cr^{6+} , Mn^{2+} and Cd^{2+} are presented in (Figures 9 – 11). The colour of pharmaceutical wastewater studied was light brown at the point of collection, suggesting the effluent was about six hours. The presence of colour also indicates that the wastewater was not properly treated before being discharged. Upon interaction with ACH and ACK at 150 min, significant removal of colour was noticed. Colour was adsorbed into pores of the activated carbons. This observation is similar to report on the removal of colour and odour from wastewater using activated carbon prepared from bamboo wastes (Ademiluyi and Nze, 2016). Concentrations of Cr^{6+} , Mn^{2+} and Cd^{2+} in the wastewater before interaction were 0.105,

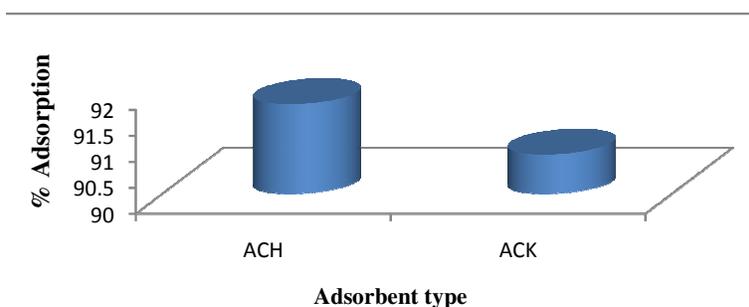


Figure 9. Removal (%) of Cr^{6+} from pharmaceutical wastewater onto ACH and ACK.

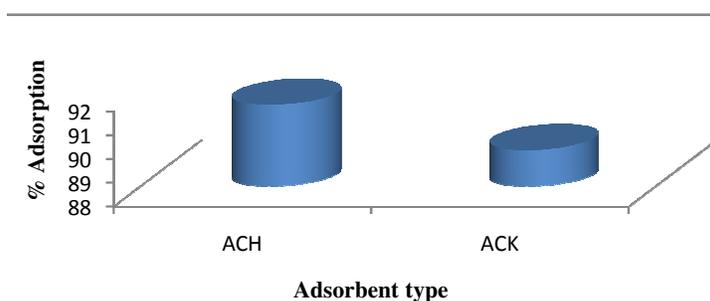


Figure 10. Removal (%) of Mn^{2+} from pharmaceutical wastewater onto ACH and ACK.

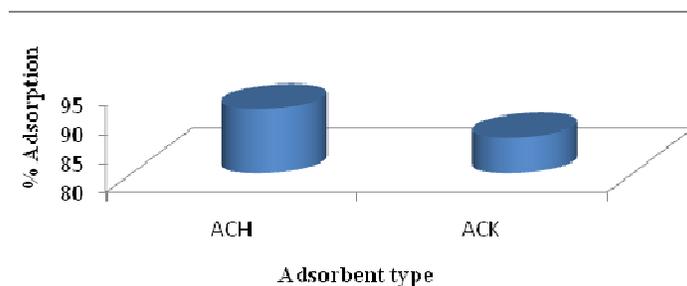


Figure 11. Removal (%) of Cd^{2+} from pharmaceutical wastewater onto ACH and ACK.

Table 1: Concentrations of Cr⁶⁺, Mn²⁺ and Cd²⁺ in pharmaceutical wastewater.

Parameter	Before Adsorption	After Adsorption	
		ACH	ACK
Colour	Light brown	Colourless	Colourless
Cr ⁶⁺ (mg/dm ³)	0.105	0.009	0.011
Mn ²⁺ (mg/dm ³)	0.616	0.051	0.057
Cd ²⁺ (mg/dm ³)	0.036	0.003	0.005

Table 2. Standards for cadmium, chromium and manganese in drinking and irrigation waters.

Metal	USEPA (mg/dm ³)		NSDWQ (mg/dm ³)		FAO (mg/dm ³)	
	Drinking water	Irrigation water	Drinking water	Irrigation water	Drinking water	Irrigation water
Cd	0.005	0.010	0.003	0.010	0.003	0.010
Cr	0.100	0.100	0.050	0.100	0.050	0.100
Mn	0.050	0.200	0.200	0.200	0.500	0.200

Source: Singh and Gupta (2016); Idris et al., (2013); Anyakora et al., (2011)

USEPA = United States Environmental Protection Agency

NSDWQ = Nigeria Standard for Drinking Water Quality

FAO = Food and Agricultural Organization

0.616 and 0.036 mg/dm³ respectively. These concentrations are higher than tolerable limits for drinking and irrigation water (Table 2). After interactions at 200 rpm, 28±2°C, 0.096 and 0.094 mg/dm³ of Cr⁶⁺ were adsorbed by ACH and ACK, which represent 91.72 % and 90.72 % adsorption (Figure 9) leaving 0.009 and 0.011 mg/dm³ in the solution. The concentration of Cd²⁺ remaining in the wastewater after interaction with ACK was 0.005 mg/dm³. This concentration is acceptable for irrigation water (Idris et al., 2013) but higher than acceptable limit for drinking water (Anyakora et al., 2011). ACH adsorbed Cd²⁺ better as only 0.003 mg/dm³ was remaining in the solution, a concentration within the tolerable limit for Cd²⁺. Similar observations were reported for the adsorption of Pb²⁺ and Cd²⁺ from industrial wastewater (Adie et al., 2012).

Conclusion

The present research shows that chemically modified *Bombax buonopozense* calyx is an effective adsorbent for removal of heavy metal ions from solutions. Percentage removal of heavy metal ions is greater than 90% in all cases. These results revealed that activated carbon produced from *Bombax buonopozense* calyx exhibit good ability to adsorb heavy metal ions from solutions, indicating that it can be employed as an alternative to the expensive commercial activated carbon (CAC) in waste water treatment.

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