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# Equilibrium and Thermodynamic Studies on Adsorption of Hexavalent Chromium from Aqueous Solution onto Low Cost Activated Carbon

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## Abstract

The removal of hexavalent chromium [Cr(VI)] is a challenging task due to its acute toxicity even at low concentration. In the present study, a low-cost activated carbon (LAC) was prepared from desert date seed shell by chemical activation with  $H_3PO_4$  and utilized for the removal of hexavalent chromium from aqueous solution. Batch experiments were conducted to investigate the influence of operating variables such as pH, contact time, adsorbent dosage, initial concentration, co-existing ions and temperature. The amount of Cr(VI) adsorbed was found to vary with solution pH and maximum adsorption was observed at a pH value of 2.0. The extent of chromium uptake ( $mg\ g^{-1}$ ) was found to increase with increase in initial concentration and contact time. The applicability of the four isotherm models for the present equilibrium data follows the sequence: Freundlich > Temkin > Langmuir > Dubinin-Radushkevich. The mean free energy from the Dubinin-Radushkevich isotherm model hinted that the adsorption of Cr(VI) onto the adsorbent surface follows physisorption mechanism. Thermodynamic parameters related to adsorption, Gibbs free energy change ( $\Delta G^\circ$ ), enthalpy change ( $\Delta H^\circ$ ), entropy change ( $\Delta S^\circ$ ), were also calculated and the negative value of  $\Delta H^\circ$  indicates the exothermic nature of the adsorption process. The considerable adsorption capacity of  $99.09\ mg\ g^{-1}$  is a signifier of the suitability of the prepared adsorbent for commercial application. The findings implicated that the adsorbent can be employed in the treatment of Cr-bearing water and wastewater.

**Index Terms:** Activated carbon, Chromium(VI), Desert date, Co-existing ions, Modeling

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## **1. Introduction**

Heavy metal pollution of industrial effluent is a serious environmental problem due to their nonbiodegradability and acute toxicity. The problem becomes more worrying as our population soars and our industrialization becomes more vehement. Many chromium compounds are used in various industries such as electroplating, metal fabrication, dyeing and leather [1]. They also find applications in other industrial processes such as steel production, mining, petroleum refining, wood preservation, anodizing of aluminum, water-cooling, nuclear power plants, cement, ink, ceramic and glass [2]. Generally, industrial effluents contain both the trivalent Cr(III) and hexavalent Cr(VI) form of chromium. Among the two forms of chromium, the hexavalent specie is relatively more toxic and is deemed to be carcinogenic [3]. Therefore, it is mandatory to treat chromium-bearing effluents before disposal in to the recipient environment to avoid consequent health issues.

Treatment technologies for remediation of chromium-bearing wastewaters include membrane filtration, ultrafiltration, ion exchange, co-precipitation, electrolytic methods, photocatalysis and adsorption [4]. However, most of these established techniques are not capable of efficiently reducing chromium concentration to tolerable limit [5]. They also suffer from economic and technological drawbacks, such as intensive operation, energy-expensiveness, high processing/operational cost, high chemical consumption and the generation of colossal amount of secondary pollutants [6]. Most conventional techniques are therefore not profitable as industrial-scale method for removal of chromium from aqueous medium [7].

In an effort to enhance removal efficiency, considerable research efforts have been devoted to addressing the shortcomings associated with conventional treatment options, predominantly via adsorption. The use of adsorbents of natural origin has emerged in the recent years as one of the most viable options to established pollutant management strategies [8]. Many literatures have reported the use of various agricultural waste and byproducts such as maize cob [9], sawdust [10], hazelnut shell [11], groundnut hull [12], sugarcane bagasse [13], pea pod peel [14], avocado seed kernel [15], tea waste [16] and olive bagasse [17] as adsorbents for the removal of Cr(VI) from aqueous solution. However, most of the adsorbents have low adsorption capacity when utilized in their native form. To produce adsorbents with superior performance, the general trend lately has been that of modified adsorbents either by activation, grafting, changing the chemical form or through engineered composite materials [18].

Activated carbons derived from agricultural wastes have been reported to exhibit valuable properties commensurable to those of available commercial active carbon. Actually, wastes from different agro-sources can serve as suitable precursors for making low-cost activated carbons with good adsorption efficiency. Moreover, proper utilization of these wastes can help reduce the burden on the environment in terms of disposal problem. However, no published references were available on the uptake of Cr(VI) specifically by desert date seed shell activated carbon. Presently, the seed shell is nothing but a waste having no practical utility. Since it is lignocellulosic in nature, it would be highly desirable to utilize such unused agro-waste as an alternative raw material for making activated carbon for chromium removal from water and wastewater. In view of the above, the present work was initiated to make an active carbon from abundant and locally available lignocellulosic waste (desert date seed shell) with objective to employ it as adsorbent for the reclamation of chromium-bearing effluent. The prepared carbon was characterized using electron microscopic technique and pH point of zero charge (pHpzc). The influence of operating variables such as initial concentration, contact time, adsorbent dose, pH, co-existing ions and temperature were investigated. Equilibrium isotherm models were tested and thermodynamic quantities ( $\Delta G$ ,  $\Delta H$  and  $\Delta S$ ) were evaluated and discussed. Moreover, the adsorptive properties of the newly prepared activated carbon was compared with previously reported adsorbents of similar category.

## 2. Literature Review

Recently, the removal of hexavalent chromium from aqueous solution using an activated carbon derived from *Leucaena leucephala* seed pod was investigated. The prepared activated carbon was characterized by proximate analysis, BET surface area measurement, SEM-EDX and FTIR. The obtained results showed that 100 min contact time, 1.0 g adsorbent dose, 45°C temperature and solution pH of 6.0 resulted in maximum Cr(VI) uptake. The experimental data better fitted to the Langmuir isotherm with monolayer adsorption capacity of 26.94 mg g<sup>-1</sup>. The kinetic analysis revealed that pseudo-second-order model fitted well to the acquired experimental data. Thermodynamic parameters showed the process to be spontaneous and endothermic in nature [19].

In another study, modified biochar obtained after hydrothermal carbonization of *Lepironia articulata* was used for the removal of hexavalent chromium from aqueous medium. The performance of modified biochar was investigated through batch sorption experiments. Results revealed that removal (%) and the maximum adsorption capacity (q<sub>max</sub>) of Cr(VI) onto the adsorbent increased up to 98.9% and 28.75 mg g<sup>-1</sup> relative to 63.44% and 21.90 mg g<sup>-1</sup> in unmodified biochar at pH 2.0, 313 K, and 200 mg dm<sup>-3</sup> respectively. The sorption kinetics uptake data were best interpreted with pseudo-second-order model, and sorption isotherm was simulated with the Langmuir model. The thermodynamic parameters confirm the adsorption process to be endothermic and spontaneous [20].

Cucumis melo peel activated carbon was utilized for the removal of hexavalent chromium from aqueous solution. Results obtained showed that the maximum removal of Cr (VI) was found to be 98 % at pH 3.0 with 250 mg of adsorbent when the concentration of the metal solution was 100 mg dm<sup>-3</sup>. The kinetics was based on pseudo-second-order equation and the equilibrium data was better fitted to Langmuir and Freundlich models [21]. Also, sugarcane bagasse was utilized as a low cost adsorbent for the removal of Cr (VI) in aqueous solution. Result revealed 100 % removal at pH 1.5 where on increasing the pH to 3.0 the removal efficiency was reduced to 17% [22].

## 3. Materials and Methods

### 3.1 Chemicals

Potassium dichromate (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>), phosphoric acid (H<sub>3</sub>PO<sub>4</sub>), sodium nitrate (NaNO<sub>3</sub>), sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>), sodium chloride (NaCl), sodium hydroxide (NaOH) and hydrochloric acid (HCl) used were of analytical grade obtained from Sigma Aldrich. Deionized water was used in the preparation of all standard solutions. The Cr(VI) stock solution was prepared by dissolving 2.829 g of potassium dichromate in 1 dm<sup>3</sup> deionized water. The needed experimental concentrations were prepared by appropriate dilution of the stock with deionized water. 0.1 mol dm<sup>-3</sup> HCl and 0.1 mol dm<sup>-3</sup> NaOH were used to adjust the initial solution pH depending on the requirement.

### 3.2 Equipment

Microwave Plasma-Atomic Emission Spectrometer (4210 MP-AES; Agilent) was used for the determination of residual Cr(VI) concentration. Incubator shaker (Innova 4000; New Brunswick Scientific) was employed for the agitation of the sample solutions during batch experiments. pH meter (3510; Jenway) was used for pH measurement. Centrifuge (Centurion 4000 Series) was employed for centrifugation after each adsorption experiment. The thermal treatment during activated carbon preparation was carried out in a muffle furnace (SXL-1008). Surface morphology of the adsorbent was examined using scanning electron microscope

(PRO: X: Phenom World 800-07334).

### 3.3 Preparation of Activated Carbon

The preparation of the activated carbon was largely guided by the method described by [23], however with modification in the activating agent used ( $\text{H}_3\text{PO}_4$  instead of  $\text{KOH}$ ). The desert date seed shells were first washed thoroughly with water to get rid of surface adhered impurities. They were subsequently air dried for 48 h and then in an oven at  $105^\circ\text{C}$  overnight. The dried shells were ground and screened to pass through a 1 mm sieve to remove larger particles. They were subsequently carbonized at  $700^\circ\text{C}$  in a muffle furnace for 90 min. The carbonized sample was then mixed with solution of the activating agent such that the mass ratio of  $\text{H}_3\text{PO}_4$  to charcoal was 3:1. This was followed by second heating at  $750^\circ\text{C}$  for 90 min. The sample was cooled, washed first with  $0.1 \text{ mol dm}^{-3}$   $\text{HCl}$  and then thoroughly with deionized water until a neutral pH was attained. The desired activated carbon was obtained after drying in an oven at  $110^\circ\text{C}$  for 24 h.

### 3.4 Characterization of Adsorbent

#### 3.4.1 Scanning electron microscopy (SEM)/Energy dispersive spectroscopy (EDS)

The morphological features and elemental composition of the adsorbent were analyzed using SEM/EDS analysis. The samples were placed on brass stubs using adhesive tape on both sides. Scanning were done at an accelerating voltage of 15.00 kV and at x1000 magnifications.

#### 3.4.2 pH point of zero charge measurements (pHpzc)

The pH point of zero charge of the LAC was identified using the salt addition method described by [24]. In a series of  $50 \text{ cm}^3$  centrifuge tubes, 0.2 g of the adsorbent was added to  $40 \text{ cm}^3$  of  $0.1 \text{ mol dm}^{-3}$   $\text{NaNO}_3$  solution. The pH was adjusted using  $0.1 \text{ mol dm}^{-3}$   $\text{HCl}$  and  $0.1 \text{ mol dm}^{-3}$   $\text{NaOH}$  to obtain the pH range of 2-11. The initial pH values of the supernatant in each tube were denoted as  $\text{pH}_i$ . The contents were then shaken continuously for 24 h using an incubator shaker. After settling, the pH values of the supernatant in each tube were measured and denoted as  $\text{pH}_f$ . A graph between difference of final and initial pH ( $\Delta\text{pH}$ ) against  $\text{pH}_i$  was plotted to obtain the zero point value of pH (pHpzc).

### 3.5 Batch Adsorption Study

The influence of initial  $\text{Cr(VI)}$  concentration ( $50\text{-}250 \text{ mg dm}^{-3}$ ), contact time (20-240 min), adsorbent dose (0.1-0.4 g), pH (2.0-9.0), co-existing ions ( $0\text{-}0.2 \text{ mol dm}^{-3}$ ) and temperature ( $30\text{-}50^\circ\text{C}$ ) on the adsorption process were investigated using batch technique. Batch experiments were conducted by contacting a specified amount of adsorbent with  $100 \text{ cm}^3$  of different  $\text{Cr(VI)}$  solutions in a set of conical flasks. The initial solution pH was adjusted to the desired value and the contents of each flask were agitated in temperature controlled incubator shaker at a constant agitation speed of 150 rpm. After attainment of equilibrium, each sample was centrifuged at 1000 rpm for 5 min and the clear supernatant was decanted. The concentration of residual  $\text{Cr(VI)}$  was then analyzed by microwave plasma-atomic emission spectrometer (MP-AES). The amount of  $\text{Cr(VI)}$  adsorbed per gram of adsorbent at equilibrium ( $q_e$ ) and the removal percentage (R %) were evaluated using Eqs. (1)-(2), respectively:

$$q_e = \left( \frac{C_0 - C_e}{m} \right) V \quad (1)$$

$$R (\%) = \left( \frac{C_0 - C_e}{C_0} \right) \times 100 \quad (2)$$

where,  $C_0$  is the initial concentration of adsorbate ( $\text{mg dm}^{-3}$ ),  $C_e$  is the concentration of adsorbate at equilibrium ( $\text{mg dm}^{-3}$ ),  $V$  is the volume of adsorbate solution ( $\text{dm}^3$ ) and  $m$  is the mass of the adsorbent (g).

### 3.5.1 Influence of contact time

A specified amount of the adsorbent was added to a series of  $100 \text{ cm}^3$  of the Cr(VI) solution of fixed concentration ( $150 \text{ mg dm}^{-3}$ ). The contents were agitated for different time intervals (20, 40, 60, 80, 100, 120, 180 and 240 min) under room temperature ( $30 \pm 2 \text{ }^\circ\text{C}$ ). The mixture after each time interval were centrifuged immediately and subjected to analysis.

### 3.5.2 Influence of adsorbent dose

The influence of adsorbent dosage was investigated by agitating  $100 \text{ cm}^3$  of Cr(VI) solutions of fixed concentration ( $150 \text{ mg dm}^{-3}$ ) with varying amount of adsorbent (0.1, 0.2, 0.3 and 0.4 g) for a period of 240 min. After the completion of time, the mixture was centrifuged, and the residual metal ion in the supernatant was analyzed.

### 3.5.3 Influence of pH

The influence of pH on Cr(VI) adsorption by LAC was investigated over a pH range of 2.0-9.0. A  $100 \text{ cm}^3$  of Cr(VI) solution of fixed concentration ( $150 \text{ mg dm}^{-3}$ ) was contacted with 0.2 g of the adsorbent. The mixtures were agitated for 100 min at the aforementioned temperature. After centrifugation, the supernatant was analyzed for residual metal concentration.

### 3.5.4 Influence of co-existing ions

The influence of co-existing ions was investigated by agitating  $100 \text{ cm}^3$  Cr(VI) solutions with varying concentration of NaCl,  $\text{NaNO}_3$  and  $\text{NaNO}_2$  (0, 0.05, 0.1, 0.15, and 0.2  $\text{mol dm}^{-3}$ ) till equilibrium time. All other parameters were kept constant. The contents were then centrifuged and analyzed for residual metal concentration.

### 3.5.5 Equilibrium studies: influence of initial Cr(VI) concentration

The influence of initial concentration was studied at different Cr(VI) initial concentration ranging from 50-250  $\text{mg dm}^{-3}$ . The initial pH of all solutions was adjusted to optimized pH value (2.0) and then contacted with 0.2 g of the adsorbent. The contents were then agitated for predetermined equilibrium time of 100 min under room temperature ( $30 \pm 2 \text{ }^\circ\text{C}$ ). The mixture was centrifuged and the supernatant was analyzed for the concentration of unadsorbed metal ion.

### 3.5.6 Thermodynamic studies: influence of temperature

For the influence of temperature, 0.2 g of LAC was added into  $100 \text{ cm}^3$  of Cr(VI) solutions (150 and 200  $\text{mg dm}^{-3}$ ). Batch experiments were performed at different temperatures of 30, 40, and  $50^\circ\text{C}$  while keeping all other experimental parameters constant (equilibrium time, optimum pH and dosage). After each adsorption

experiment, residual concentrations of adsorbate were analyzed.

## 4. Results and Discussion

### 4.1 Characterization of the Adsorbent

#### 4.1.1 SEM/EDS

The morphological characteristics of adsorbent influence its performance in various applications. SEM is a very vital technique to probe the surface morphology of adsorbents. Fig. 1a displays the micrograph of the adsorbent before Cr(VI) adsorption. It is seen the adsorbent tend to possess highly rough heterogeneous surface with distinct crevices resulting from subjecting the material to thermal processes. However, after adsorption of Cr(VI), the surface of the LAC is relatively smooth (Fig. 1b). This is probably due to effective interaction of the adsorbate with the adsorbent's surface. EDS spectrum (Fig. 2) of the LAC revealed the peaks for C, O, N, Cl and P elements with a high C content (82.93 %).

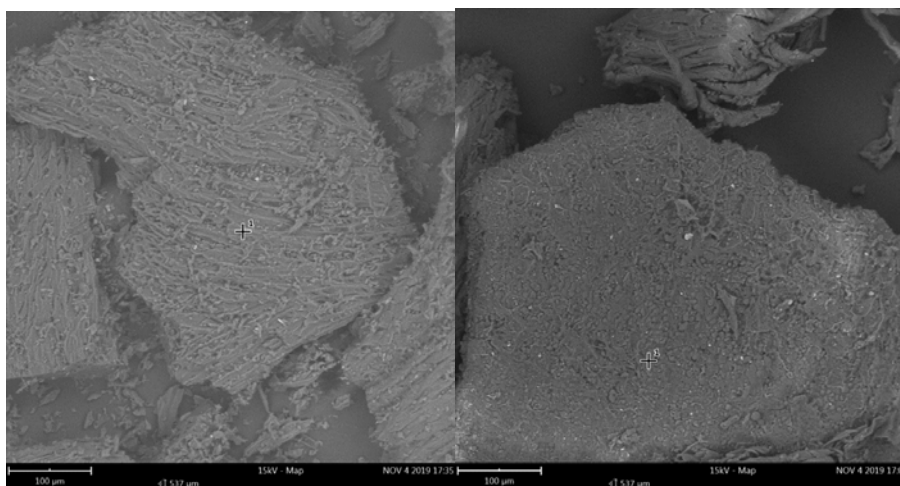


Fig. 1. SEM micrograph of LAC (a) before adsorption and (b) after adsorption

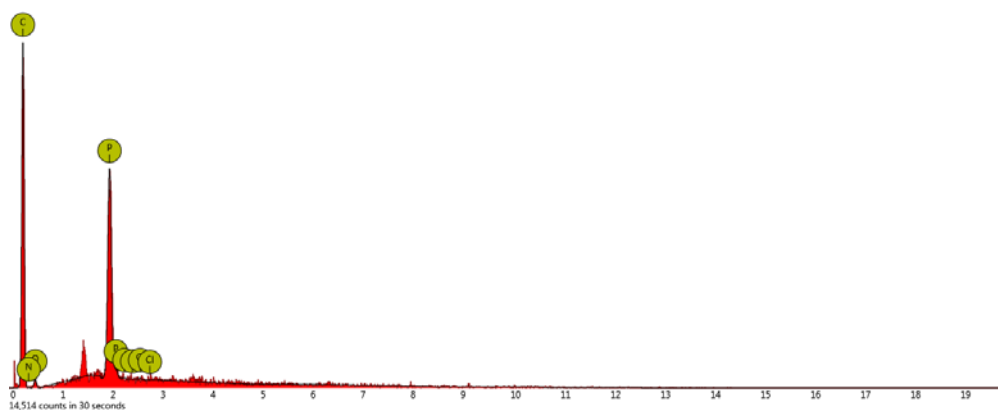


Fig. 2 EDS spectrum of LAC

#### 4.1.2 pH of point of Zero Charge $pH_{PZC}$

The determination of  $pH_{PZC}$  was carried out to determine the value of pH for which the surface of LAC bears a net charge of zero.  $pH_{pzc}$  plays vital role in surface characterization as it determines how easily an adsorbent can bind potentially harmful ions [25]. The zero point charge of the adsorbent is 6.4 (Fig. 3). The implication of this value is that at solution pH greater than 6.4, the surface of the adsorbent bears a net positive charge, thus favoring the sorption of cationic species. Conversely, for solution pH less than 6.4, the adsorbent surface is negatively charged and therefore promoting the adsorption of anionic species [26]. Since Cr(VI) exist in aqueous solution in different anionic forms, it can be suggested that the adsorption process will be favorable when the LAC surface is positive.

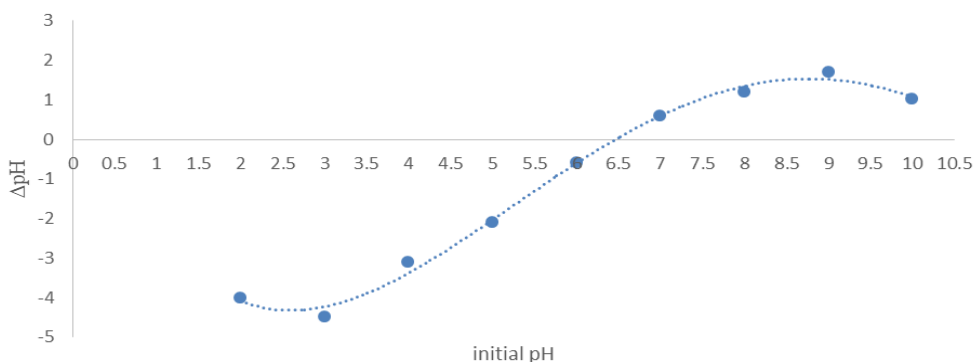


Fig. 3. Determination of  $pH_{PZC}$  of LAC

#### 4.2 Influence of contact time and adsorbent dose

The influence of adsorbent dose and contact time on the Cr(VI) uptake by LAC is illustrated in Fig. 4. It is seen that the extent of Cr(VI) adsorption increases with increasing contact time up to around 100 minutes and then tends to equilibrium thereafter. After attaining the equilibrium, further adsorption was insignificant as a function of contact time under the employed experimental conditions. In the early stages, there are substantial amount of vacant binding sites on the adsorbent accessible for adsorption. As the binding sites become occupied, the rate of adsorption decline and ultimately becomes nearly steady at equilibrium [27]. It is observed that the Cr(VI) adsorption capacity decreases as the dosage of the adsorbent increases. It is obvious from Fig. 4 that the adsorption capacities decreased from 62.1 to 30.9 mg g<sup>-1</sup> as the adsorbent dosage was increased from 0.1 to 0.4 g. It was also observed that the contact time required for attainment of equilibrium was dependent on the dose of the adsorbent used. For the adsorbent dose of 0.1, 0.2, 0.3 and 0.4 g, the time required for the adsorption to attain equilibrium were: 120, 100, 80 and 80 min, respectively. The decrease in Cr(VI) adsorption capacity with increase in dosage is attributed to unsaturation of many active sites of the adsorbent during the adsorption process [28]. A similar trend was highlighted in the adsorption of Cr(VI) using diphenylcarbazide-grafted macadamia nutshell powder[29].

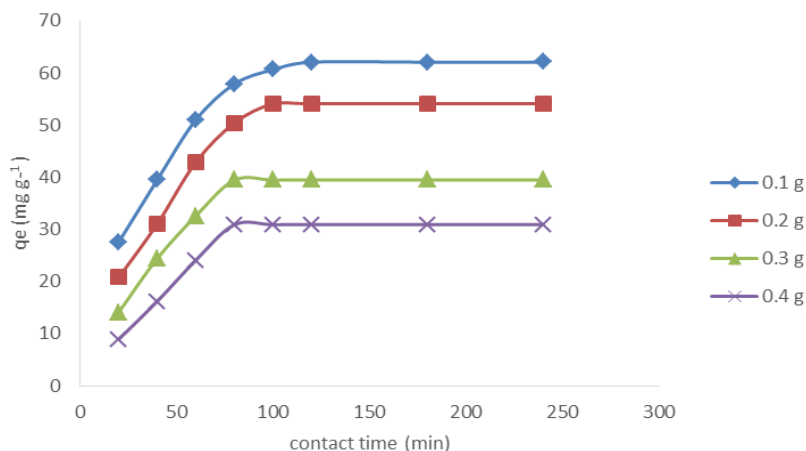


Fig. 4. Influence of contact time and adsorbent dose on Cr(VI) uptake (Conditions: initial concentration  $150 \text{ mg dm}^{-3}$ , temperature  $30 \pm 2^\circ\text{C}$ )

#### 4.3 Influence of pH

The pH of the aqueous medium is an important parameter that influence the adsorption process. It affects not only the surface charge of the adsorbent, but also the degree of speciation and ionization of the adsorbate during adsorption [30,31]. To investigate the effect of pH on the adsorption process, the pH solution was varied from 2.0 to 9.0 and the result was illustrated in Fig. 5. It can be observed that increasing the solution pH from 2.0 to 9.0 results in decrease in adsorption capacity from  $62.1$  to  $17.79 \text{ mg g}^{-1}$ ; so we can deduce that pH 2 is the optimum pH value for the adsorption of Cr(VI) on the adsorbent. This could be explained by the fact that Cr(VI) exist in the form of several species such as  $\text{HCrO}_4^-$ ,  $\text{Cr}_2\text{O}_7^{2-}$ ,  $\text{CrO}_4^{2-}$ ,  $\text{Cr}_3\text{O}_{10}^{2-}$ ,  $\text{H}_2\text{CrO}_4$ , etc depending on the aqueous solution pH and concentration [32].  $\text{HCrO}_4^-$  and  $\text{Cr}_2\text{O}_7^{2-}$  predominantly exist at solution pH within the range of 2.0-6.0. It is also established that  $\text{HCrO}_4^-$  is the dominant form of Cr(VI) at pH 2.0 [33]. Raising pH shifts the concentration of  $\text{HCrO}_4^-$  to other forms,  $\text{Cr}_2\text{O}_7^{2-}$  and  $\text{CrO}_4^{2-}$ . Therefore, maximum adsorption at pH 2 indicates that the adsorbent preferentially adsorbed the  $\text{HCrO}_4^-$  form of Cr(VI) anions [34]. In this context, it is imperative to comment that the  $\text{pH}_{\text{pzc}}$  of the adsorbent earlier determined in Fig. 3 was 6.4. This reflects that the favorable condition for adsorption of  $\text{HCrO}_4^-$  on LAC surface is the medium having pH less than 6.5. Hence, the observed higher adsorption at acidic medium can be ascribed to the strong electrostatic attraction between positively charged adsorbent and  $\text{HCrO}_4^-$ . Conversely, the reduced adsorption at higher pH may presumably be due to competitive adsorption between chromate and hydroxyl ions. Similar trend was highlighted in the adsorption of Cr(VI) from aqueous solution using various adsorbents [35-37].



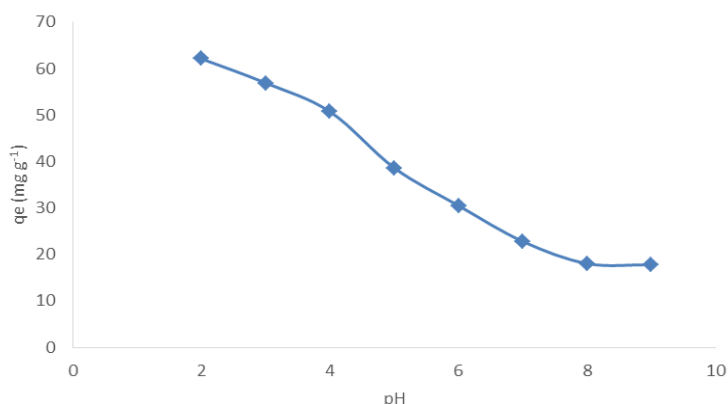


Fig. 5. Influence of pH on Cr(VI) uptake (Conditions: initial concentration  $150 \text{ mg dm}^{-3}$ , adsorbent dose  $0.2 \text{ g}$ , temperature  $30 \pm 2^\circ\text{C}$ )

#### 4.4 Influence of co-existing ions

The real chromium containing effluents are typically associated with the presence of ionic species such as  $\text{Na}^+$ ,  $\text{Cu}^{2+}$ ,  $\text{NO}_3^-$ ,  $\text{CO}_3^{2-}$ ,  $\text{SO}_4^{2-}$  and  $\text{Cl}^-$ . It is therefore essential to study the influence of these co-existing ions on the removal of Cr(VI) from aqueous solutions. Fig. 6 illustrate the effect of salt concentration on the adsorption of Cr(VI). It is seen that as the salt concentration increases from  $0.0$  to  $0.2 \text{ mol dm}^{-3}$ , the adsorption capacity of LAC reduces from  $61.9$  to  $45.5$ ,  $50.81$ ,  $53.9 \text{ mg g}^{-1}$  in the presence of  $\text{NaCl}$ ,  $\text{NaNO}_3$  and  $\text{Na}_2\text{CO}_3$  respectively. The decrease in amount of Cr(VI) adsorbed with an increase in salt concentration could be attributed to competition for available active sites between the  $\text{HCrO}_4^-$  and the negatively charged  $\text{Cl}^-$ ,  $\text{NO}_3^-$  and  $\text{CO}_3^{2-}$  being introduced. It is also observed that  $\text{Cl}^-$  ions reduce the uptake of Cr(VI) by LAC more than  $\text{NO}_3^-$  and  $\text{CO}_3^{2-}$  ions. This was attributed to the faster diffusion rate of  $\text{Cl}^-$  ions on the adsorbent, than  $\text{HCrO}_4^{2-}$ . Similar observations were previously reported on the removal of Cr(VI) by acid functionalized nanoporous carbon [38].

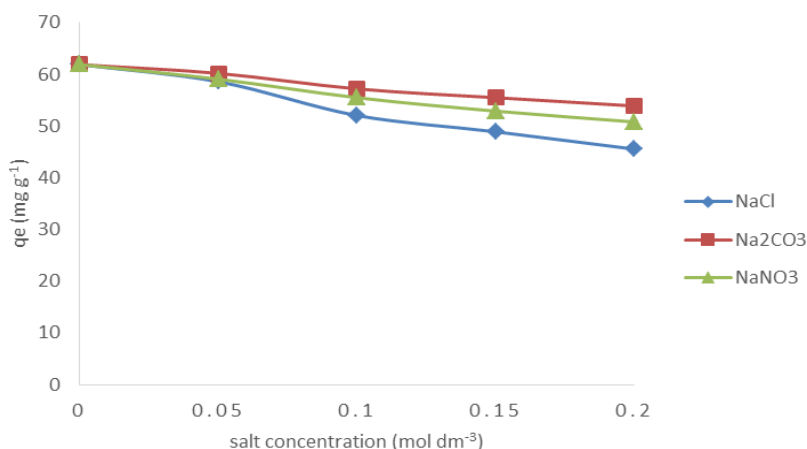


Fig. 6. Influence of co-existing ions on Cr(VI) uptake (Conditions: initial concentration  $150 \text{ mg dm}^{-3}$ , pH  $2.0$ , temperature  $30 \pm 2^\circ\text{C}$ )

#### 4.5 Influence of initial concentration

The variation of equilibrium amount of Cr(VI) adsorbed with initial concentration is depicted in Fig. 7. It is evident from the plot that the chromium adsorption capacity increased from 23.5 to 99.09 mg g<sup>-1</sup> with increase in initial adsorbate concentration from 50 to 250 mg dm<sup>-3</sup>. This happened due to availability of large number of adsorbate molecules in the aqueous phase enhancing the interaction between the metal ions and LAC. Furthermore, higher metal concentration provided increase in driving force of the concentration gradient to overwhelm all mass transfer resistance of Cr(VI) ions between liquid and solid phases. Consequently, the probability of collision between the adsorbate and adsorbent active sites increase and thus leading to higher Cr(VI) uptake [39,40]. These observations are consistent with previous reports [41,42].

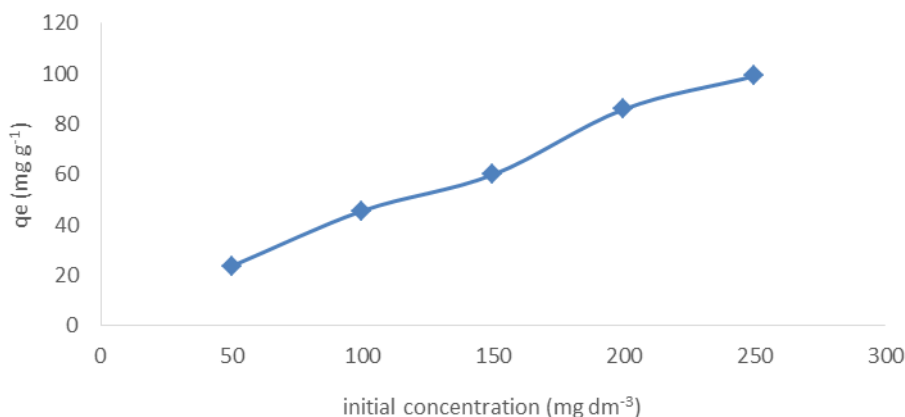


Fig. 7. Influence of initial concentration on Cr(VI) uptake (Conditions: adsorbent dose 0.2 g, pH 2.0, temperature 30 ± 2°C)

#### 4.6 Adsorption Isotherms

Isotherm models play significant role in describing the behavior of adsorbate-adsorbent interaction. Equilibrium isotherms, namely Langmuir, Freundlich, Temkin and Dubinin-Radushkevich were employed to analyze the adsorption data of Cr(VI) on the adsorbent, and their linear equations were expressed below, respectively [43]:

$$\frac{C_e}{q_e} = \frac{1}{K_L q_{max}} + \frac{C_e}{q_{max}} \quad (3)$$

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e \quad (4)$$

$$q_e = B_T \ln C_e + B_T \ln K_T \quad (5)$$

$$\ln q_e = \ln q_m - \beta \varepsilon^2 \quad (6)$$

where;  $q_e$  is the adsorption capacity (mg g<sup>-1</sup>) of the adsorbent at equilibrium,  $q_{max}$  is the maximum adsorption capacity (mg g<sup>-1</sup>) to form a complete monolayer coverage on the surface at high equilibrium adsorbate concentration ( $C_e$ ) (mg dm<sup>-3</sup>),  $K_L$  (L mg<sup>-1</sup>) is Langmuir constant related to affinity between the adsorbate and

adsorbent,  $B_T$  ( $\text{J mol}^{-1}$ ) and  $K_T$  ( $\text{L mg}^{-1}$ ) are Temkin constant and maximum binding energy respectively,  $\epsilon$  is the polyani potential,  $\beta$  is a constant related to the mean adsorption energy ( $\text{mol}^2 \text{kJ}^{-2}$ ),  $K_F$  ( $\text{L mg}^{-1}$ ) and  $n$  are Freundlich constants related with the adsorption capacity and adsorption intensity, respectively.

The fitted isotherm plots are shown in Fig. 8(a-d) and the parameters concerning the isotherms are summarized in Table 1. In the light of the values of coefficient of determination ( $R^2$ ), it can be observed that the experimental data exhibited better fit to the Freundlich model in comparison to the other three models. This implies that the removal of chromium occurred via multilayer adsorption on heterogeneous adsorption sites. The Freundlich model  $n$  value obtained was greater than 1.0 indicating that the adsorbate was adsorbed by LAC easily [44]. The maximum monolayer adsorption capacity of the adsorbent for Cr(VI) was found to be  $136.99 \text{ mg g}^{-1}$  at 303 K. The calculated value of  $R_L$  was in the range of 0-1 which indicated favorable adsorption of the adsorbate on the adsorbent. The maximum adsorption capacity,  $q_m$  obtained for the D-R isotherm model was  $77.44 \text{ mg g}^{-1}$  which is less in comparison to the Langmuir model ( $136.99 \text{ mg g}^{-1}$ ). The mean free energy of adsorption ( $E$ ) was obtained as  $0.5 \text{ kJ mol}^{-1}$  i.e lower than  $8.0 \text{ kJ mol}^{-1}$ . This indicates that the adsorption mechanism is governed by physical adsorption [45].

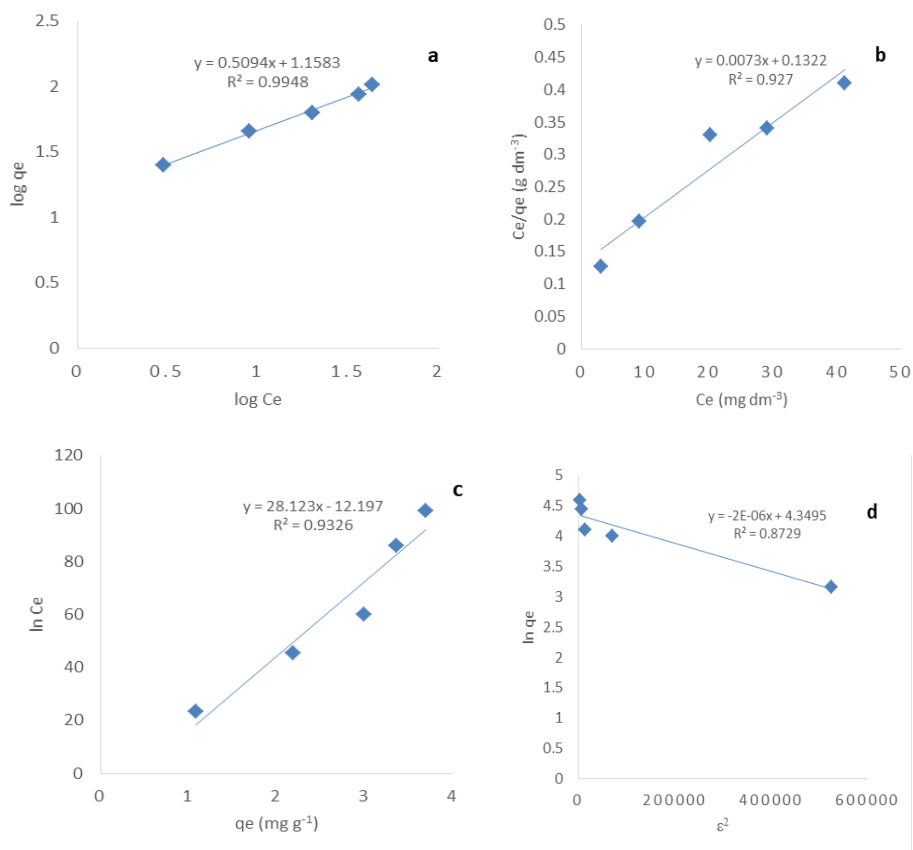


Fig. 8. The fitting results of (a) Freundlich model (b) Langmuir model (c) Temkin model, and (d) Dubinin-Radushkevich model

Table 1. Isotherm parameters for Cr(VI) adsorption onto LAC

Isotherm models	Parameters	Values
Langmuir	$q_{\max}$ (mg g <sup>-1</sup> )	136.99
	$K_L$ (dm <sup>3</sup> mg <sup>-1</sup> )	0.06
	$R_L$	0.25
	$R^2$	0.9270
Freundlich	$K_F$ (dm <sup>3</sup> mg <sup>-1</sup> )	14.40
	$n$	1.96
	$R^2$	0.9948
Temkin	$K_T$ (dm <sup>3</sup> mg <sup>-1</sup> )	0.65
	$B_T$ (J mol <sup>-1</sup> )	28.12
	$R^2$	0.9326
Dubinin- Radushkevich	$q_m$ (mg g <sup>-1</sup> )	77.44
	$\beta$ (mol <sup>2</sup> J <sup>-2</sup> )	$2 \times 10^{-6}$
	$E$ (kJ mol <sup>-1</sup> )	0.50
	$R^2$	0.8729

#### 4.7 Adsorption Thermodynamics

Thermodynamic parameters such as changes in enthalpy ( $\Delta H^\circ$ ), entropy ( $\Delta S^\circ$ ) and Gibbs free energy ( $\Delta G^\circ$ ) for the adsorption of Cr(VI) (at initial concentration of 150 and 200 mg dm<sup>-3</sup>) by LAC were calculated using the following equations [46], and their values are presented in Table 2.

$$K_c = \frac{C_{ADS}}{C_e} \tag{7}$$

$$\Delta G = -RT \ln K_c \tag{8}$$

$$\ln K_c = - \frac{\Delta H^\circ}{RT} + \frac{\Delta S^\circ}{R} \tag{9}$$

where,  $C_{ADS}$  and  $C_e$  (mg dm<sup>-3</sup>) are equilibrium concentration of Cr(VI) on the adsorbent and in the liquid phase, respectively,  $K_c$  is the equilibrium constant of adsorption,  $R$  is the gas constant (8.314 J K<sup>-1</sup> mol<sup>-1</sup>) and  $T$  is the temperature (K).

A plot of  $\ln K_c$  versus  $1/T$  according to Eq. 9 is shown in Fig. 9. From the plot, both the change in enthalpy ( $\Delta H^\circ$ ) and the entropy ( $\Delta S^\circ$ ) were determined from the slope and intercept respectively. The negative value of  $\Delta H^\circ$  is consistent with the exothermic nature of the adsorption process while the negative value of  $\Delta S^\circ$  reflects the affinity of the adsorbent towards the adsorbate [46]. From Eq. 8 the values of change in Gibbs free energy was evaluated and found to be negative in the temperature range studied. This indicates the spontaneity of Cr(VI) adsorption onto LAC. Furthermore, the  $\Delta G$  values become more negative with decreasing temperature of the system. This signifies that the adsorption process was more favorable at lower temperature. Similar trend in thermodynamics has been observed in the study of adsorption of Cr(VI) using functionalized chitosan [48].

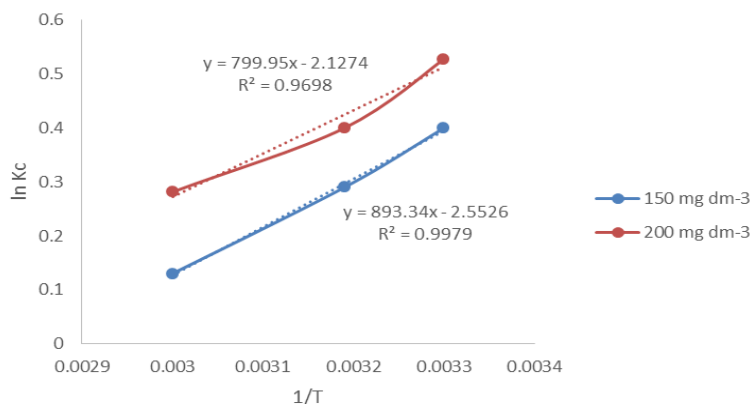
Fig. 9. Plot of  $\ln K_c$  versus  $1/T$  for Cr(VI) adsorption onto LAC

Table 2. Thermodynamic parameters for Cr(VI) adsorption onto LAC

$C_0$ (mgdm <sup>-3</sup> )	$\Delta H$ (kJ mol <sup>-1</sup> )	$\Delta S$ (kJ mol <sup>-1</sup> K <sup>-1</sup> )	$\Delta G$ (kJ mol <sup>-1</sup> )		
			303K	313K	323K
150	-7.427	-0.021	-1.005	-0.624	-0.349
200	-6.650	-0.018	-1.329	-0.948	-0.755

#### 4.8 Comparison of Cr(VI) adsorption capacity of LAC with other reported adsorbents

To justify the potential of LAC as an efficient adsorbent for chromium removal, a comparison of the maximum Cr(VI) adsorption capacity of the prepared adsorbent has been carried out with other reported adsorbents and the summary is presented in Table 3. It is pertinent to note that this comparison has a relative interpretation due to some factors, such as different experimental conditions (adsorbent dosage, pH, temperature, adsorbate concentration, agitation rate, shaking versus stirring), method (batch versus column) and type of biomaterial employed in each referenced study. Nonetheless, the maximum adsorption capacity of LAC for Cr(VI) is comparable and moderately higher than that of many of the reported sorbent materials of similar category. The implications were that LAC is a promising adsorbent for treatment of chromium-bearing water and wastewater.

Table 3. Comparison of Cr(VI) adsorption capacities of activated carbons from different sources

Activated carbon source	$q_{\max}$ (mg g <sup>-1</sup> )	Optimum pH	Max. Cr concentration used (mg dm <sup>-3</sup> )	Reference
Almond shell	165.70	2.0	1000	49
<i>Ziziphus jujuba</i> cores	196.38	2.0	500	50
Activated charcoal	2.60	4.0	50	51
Palmyra palm seed	32.14	1.0-2.0	200	52
<i>Juniperus procera</i> leaves	23.00	4.0	300	53
Rice husk	34.85	2.0	250	54
Peanut shell	8.31	2.0	100	55
Palm kernel shell	125.00	2.0	100	56
Walnut shell	6.12	2.0	100	57
Bamboo waste	59.23	2.0	150	58
Apple peels	36.01	2.0	50	59
Macadamia nutshell	22.30	2.0	150	60
Desert date seed shell	99.09	2.0	250	This study

## 5. Conclusion

There are numerous existing techniques for removal of chromium, each with their merits and limitations. Operational and cost effectiveness are the important characters that makes adsorption stands out among other conventional techniques. In the present work, a lignocellulosic waste is employed for making activated carbon which was successfully utilized for Cr(VI) removal from aqueous medium. The studied material is quite promising because of its abundance, low-cost and little processing. Operational variables like pH, contact time, initial concentration, coexisting ions, adsorbent dosage and temperature were found to influence the adsorption capacity of the prepared adsorbent significantly. The equilibrium data fit well to Freundlich isotherm model, indicating multilayer adsorption. The negative values of  $\Delta G^\circ$  confirmed the spontaneity of the adsorption process. The negative values of  $\Delta H^\circ$  and  $\Delta S^\circ$  indicated exothermic and enthalpy driven nature of Cr(VI) adsorption. The considerable adsorption capacity of 99.09 mg g<sup>-1</sup> is signifier of the suitability of the prepared adsorbent for commercial application. The present study concludes that LAC could be employed as an alternative low-cost adsorbent for the removal from of Cr(VI) from water and wastewater.

The expected contribution of this research include the following: (a) conversion of desert date seed shell into activated carbon will lessen the problem of disposal and management of this agro-waste, while providing valuable end-product for water treatment that could potentially expand the activated carbon market; (b) benefit industries especially those dealing with Cr(VI) compounds through the availability of cost-effective treatment approach; (c) contribution to existing data on wastewater treatment especially in chromium adsorption for future research. A follow-up research in the following direction is needed to substantiate the potential of activated carbon prepared in the current work for large scale application: (a) the feasibility of recovery and recycling of the exhausted carbon should be investigated; (b) dynamic/column adsorption studies should be conducted for design perfection purposes; (c) study of the adsorption behavior from quantum-chemical perspective for precise mechanistic insights.

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