



Adsorptive removal of chromium(VI) from aqueous solution unto groundnut shell

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Abstract

Chromium present in high levels in water poses a serious problem in many places around the world, sometimes in relation to natural sources and in other cases to anthropogenic ones. Adsorption process using natural materials is among the most effective techniques for removing many heavy metal ions from different types of water sources. In the present study, groundnut shell was used as a nonconventional adsorbent for effective removal of chromium(VI) from aqueous solutions using batch experiments. The adsorption of chromium(VI) unto groundnut shells was found to be dependent on contact time, pH, adsorbent dose, initial concentration and temperature. The adsorption of Cr(VI) was found to be optimum at a contact time of 120 min, pH 8.0, an adsorbent dose of 2.0 g/L, initial metal ion concentration of 25 mg/L and temperature of 41.5 °C. The experimental data were analyzed using three two-parameter isotherm models. The experimental data obtained for the adsorption of chromium(VI) ion fitted well to Temkin isotherm in comparison with the other isotherm models tested.

Keywords Adsorbent · Adsorption · Chromium · Groundnut shell · Isotherm

Introduction

Chromium is a major toxic pollutant, which entered the water streams through various industrial operations. Sources of chromium contamination include disposal of chromium-containing wastes and releases from electroplating processes. The potential sources of chromium(VI) wastes are effluents from metallurgy, electroplating, leather tanning, textile dyeing, paint, ink and aluminum manufacturing industries (Wuana and Okieimen 2011). Chromium is a toxic contaminant, even in very low concentrations. Chromium in its hexavalent form Cr(VI) is well known due to its health-related issues in humans including carcinogenic and mutagenic risks (Igwe and Abia 2006; Park and Jung 2001). More so, chromium can lead to cancer in the lungs and digestive tract. Excessive intake of chromium by humans leads to

hepatic and renal damages, capillary damage, gastrointestinal irritation and central nervous system (Wan Ngah and Hanafiah 2008). The conventional methods for treatment of chromium(VI) wastes include precipitation, adsorption with activated carbon, ion exchange, membrane processes, oxidation and reduction (Meunier et al. 2006). However, most of these methods resulted in incomplete removal of metal ions, low selectivity, high operational cost, high consumption of reagents, energy and generation of secondary pollutants (Wu et al. 2004; Eccles 1999). Besides, it was revealed that these techniques when applied, some of them are usually incapable of meeting the discharged standards limits for heavy metals concentrations ranging between 0.1 and 3 mg/L (García-Sánchez and Álvarez-Ayuso 2002). The adsorption technique still remains the most effective and common applicable technology widely used over other techniques in global environmental protection areas (Xunjun 2015). Adsorption has distinct advantages over the conventional methods which include reusability of biomaterial, low operating cost, selectivity for specific metal, short operation time and no chemical sludge (Rahmani et al. 2009; Shah et al. 2009). Several agricultural materials have been used for the adsorption of heavy metals as reported in many studies including plants seeds (Edokpayi et al. 2015); pomegranate peels (El-Ashtoukhy et al. 2008); and orange peel (Li et al. 2008).

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Groundnut shells are carbonaceous, fibrous solid agricultural waste that encounters disposal problem but potentially suitable for making low-cost adsorbent for adsorption of heavy metals from water and wastewaters due to its high carbon content (Idris et al. 2012).

The present research was conducted to utilize groundnut shell as efficient alternative adsorbent materials to remove chromium(VI) from aqueous solution using three two-parameter isotherm models. The influence of operating conditions such as contact time, pH, adsorbent dose, initial metal concentrations and temperature was studied.

Experimental methods

Collection and preparation of the adsorbent and adsorbate

All chemicals used for the study were of analytical reagent grade. Groundnut shells were collected from the local groundnut-processing center in Navrongo market.

The collected groundnut shells were washed repeatedly with distilled water to remove dust and insoluble impurities. The groundnut shells were then air-dried under room temperature and subsequently dried in an oven at 105 °C for 24 h to constant mass. The groundnut shells were ground and sieved to an obtained particle size less than 300 µm. The groundnut shells were used for the experiments without any physical or chemical treatments as an adsorbent (Abdel-Tawwab et al. 2017; Choudhury et al. 2012).

In the preparation of the stock solution of the adsorbate, Cr(VI) procedures specified in APHA, (1989) were followed. Specifically, a known mass (2.829 g) of potassium dichromate ($K_2Cr_2O_7$) was dissolved in de-ionized water and diluted to the 1000 mL mark using de-ionized water. Working concentrations ranged from 15 to 100 mg/L were prepared from the stock solution by diluting the stock solution with de-ionized water to the required concentrations. The Cr(VI) concentration was determined spectrophotometrically (Carry 60 UV–Vis spectrophotometer) by the diphenyl-carbazide method (Mahajan and Sud 2012).

Characterization of the adsorbent

The groundnut shell was characterized by using Fourier transform infrared spectrometer (Spectrum Two 94133 series) to determine the functional groups on the adsorbent. Characterization of the groundnut shell was done before and after the metal ion adsorption. Furthermore, the BET surface area and pore size distributions of the prepared adsorbent were carried out using Quantachrome analyzer.

Equilibrium adsorption studies

Effect of contact time

The investigation was done to determine the optimum time for adsorption of chromium(VI) using groundnut shell. In this study, 1.0 g of the adsorbent was contacted with 100 mL solutions of 50 mg/L Cr(VI) ion adjusted to pH 6. The solutions were transferred to 250-mL conical flasks and agitated at 120 rpm for different contact times (10–240 min). The mixture was filtered, and residual metal ions in the filtrate were analyzed using UV–Vis spectrophotometer (Carry 60).

Effect of pH

The effect of pH on metal ion adsorption was carried out in pH range of 2 to 10; 100 mL solutions of 50 mg/L Cr(VI) ion were transferred to 250 mL conical flasks agitated at 120 rpm for 120 min with 1.0 g adsorbent. The mixture was filtered and the filtrate analyzed for residual metal ion using UV–Vis spectrophotometer (Carry 60).

Effect of adsorbent dosage

The effect of the amount of groundnut shell used was investigated by equilibrating 100 mL solutions containing 50 mg/L of Cr(VI) ion with different masses of the adsorbent (0.5–2.5 g) agitated at 120 rpm for 120 min. The mixture was filtered, and the residual metal ion in the filtrate was analyzed using UV–Vis spectrophotometer (Carry 60).

Effect of initial metal concentration

The adsorption of chromium was carried out at different initial Cr(VI) ion concentrations ranging from 15 to 100 mg/L. Hundred milliliters of separate concentrations of metal ion was adjusted to optimized pH and taken into 250-mL conical flasks containing maximum adsorbent dose. The solutions were agitated at a fixed stirring speed of 120 rpm for a predetermined contact time of 120 min. The mixture was filtered and filtrate analyzed for residual metal ions.

Effect of temperature

The effect of temperature was performed at five different temperatures ranging from 25 to 65 °C in a thermostatic shaker machine while keeping all other parameters constant (optimum contact time, pH, adsorbent dose, and initial concentration). The adsorbent–adsorbate solutions were agitated with a fixed speed of 120 rpm, and the constancy of the temperature was

maintained with an accuracy of ± 0.5 °C. The mixture was filtered and filtrate analyzed for the residual metal ion.

The amount of adsorbate adsorbed at equilibrium, at a particular time and removal efficiency, was calculated using Eqs. (1–3).

The amount of adsorbate adsorbed at equilibrium (q_e),

$$q_e = \frac{C_0 - C_e}{m} \times V \tag{1}$$

The amount of adsorbate adsorbed at a particular time t (q_t),

$$q_t = \frac{C_0 - C_t}{m} \times V \tag{2}$$

Removal efficiency (RE),

$$RE = \frac{(C_0 - C_e)}{C_0} \times 100 \tag{3}$$

where q_e represents the equilibrium mass of the adsorbed substance per unit mass of adsorbent, q_t is the amount of metal ion adsorbed at time t , C_0 is the initial metal ion

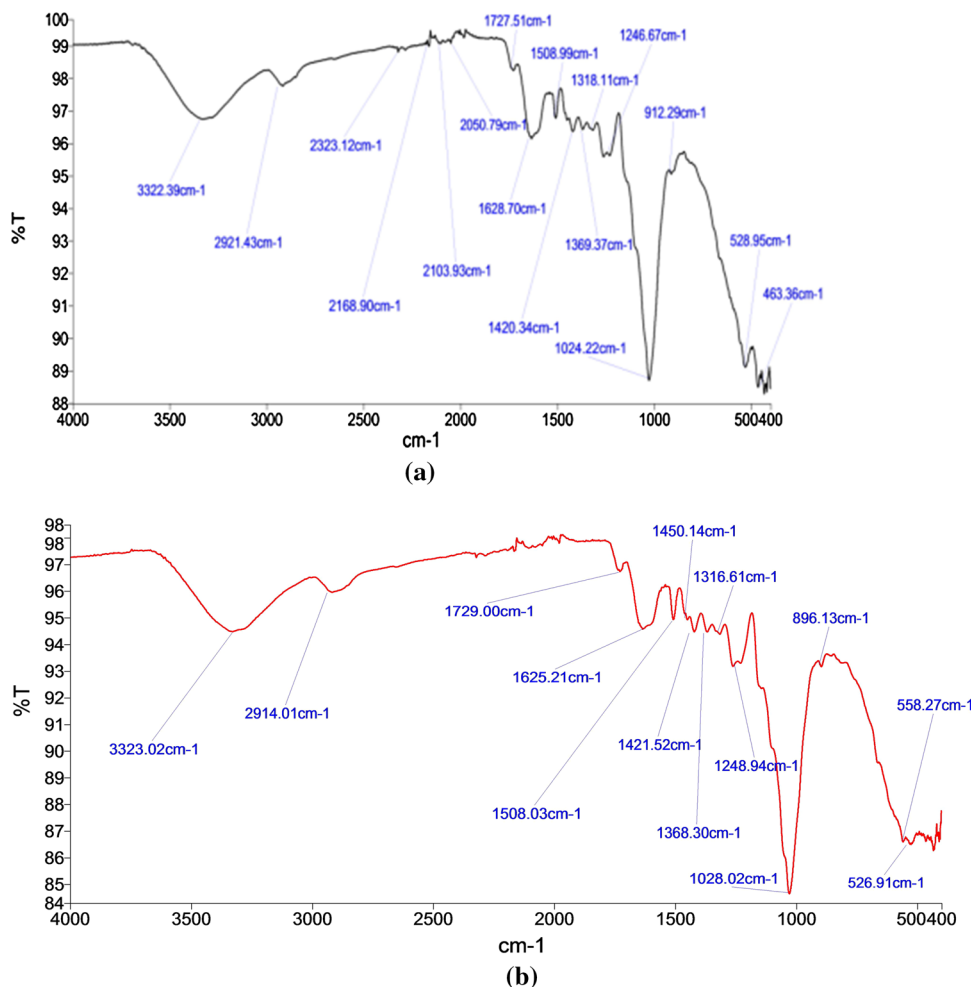
concentration (mg/L), C_e is the equilibrium metal ion concentration (mg/L), C_t is the metal ion concentration (mg/L) at time t , V is the volume of solution (mL) and m is the mass of the adsorbent (g).

Results and discussion

Characterization of groundnut shell

The FTIR spectra of groundnut shell before and after adsorption of Cr(VI) are shown in Fig. 1a, b, respectively. From Fig. 1a, the broad peak at 3322 cm^{-1} is an indicator of –OH and –NH groups. The stretching of the –OH groups bound to methyl radicals presented a signal between 2921 and 2169 cm^{-1} . The peaks located at 1728 and 1629 cm^{-1} are characteristics of carbonyl group stretching from aldehydes and ketones. The peaks at 1509 cm^{-1} are associated with the stretching in aromatic rings. The peaks observed at 1024 and 913 cm^{-1} are due to C–O and C–H bonds, respectively. The phenol (OH), amine (NH), carbonyl (C=O) and carboxylic (COOH) groups are important sorption sites

Fig. 1 FTIR spectra of groundnut shell before (a) and after (b) adsorption of Cr(VI) ion



(Kaiser et al. 2009). The presence of –OH group, coupled to carbonyl group, confirms the presence of carboxylic acid groups in the biosorbent (Chigondo and Nyamunda 2013). After the adsorption of Cr(VI), it was observed that there was broadening of OH peak at 3322 cm^{-1} and –C–O groups at 1024 cm^{-1} (Fig. 1b). The broadening of –OH and –C–O groups indicated the participation of –OH and –C–O groups in the adsorption of chromium(VI) ion. Similar spectra were obtained for bio sorption studies of metal ions using baobab fruit shells biomass (Onundi et al. 2010).

The BET surface area and pore size distributions of the prepared adsorbent before and after treatment with Cr(VI) were determined. The BET surface area of the prepared raw adsorbent without treating it with Cr(VI) was $32.96\text{ m}^2/\text{g}$ in comparison with activated carbon ($89.74\text{ m}^2/\text{g}$) reported by Yusuff (2018). However, it was found that the surface area ($2.48\text{ m}^2/\text{g}$) of the groundnut shell loaded with Cr(VI) ions decreased largely. More so, there was a significant decrease in the total pore volume of the adsorbent after the adsorption of Cr(VI) onto its surface. The total pore volume of the adsorbent was observed to decrease from 0.017630 to $0.00012\text{ cm}^3/\text{g}$ after the uptake of Cr(VI) ions onto its surface. This observation could be due to aggregation of adsorption sites implying the complete loading of Cr(VI) ions onto the surface of groundnut sample used as an adsorbent (Lim and Lee 2015).

Equilibrium studies on adsorption of Cr(VI)

Effect of contact time

As can be seen from Fig. 2, the adsorption process took place in two stages. The first stage was rapid, where chromium(VI)

removal efficiency (Fig. 2a) and adsorption capacity (Fig. 2b) were 72% and 3.60 mg/g , respectively, within the first 20 min. The second stage represented a slower progressive adsorption process. After the first 20 min, a gradual increase was observed and finally levels off after some 120 min. At the 120 min, there was no further increase or decrease in the adsorption process, which is indicative of equilibration due to saturation of adsorption sites. Rapid adsorption of metal ions during the initial stages was due to the large initial concentration gradient between the adsorbate in solution and the number of available vacant sites on the adsorbent surface (Chigondo and Nyamunda 2013). Hence, the maximum uptake of chromium(VI) was at 120 min with removal efficiency of 81.6% and adsorption capacity of 4.08 mg/g .

Effect of pH

From Fig. 3, the removal efficiency (Fig. 3a) and adsorption capacity (Fig. 3b) of Cr(VI) ion increased with an increase in pH attaining a maximum at pH 8.0. There was a rapid increase in removal efficiency and adsorption capacity when the pH was increased from 2.0 to 4.0 and later decreased at pH 6.0. A sharp increase was observed after pH of 6.0 and levels off after pH of 8.0. At pH 8.0, the groundnut shells were able to give chromium(VI) removal efficiency and adsorption capacity of 59.2% and 2.96 mg/g , respectively. The increase in metal adsorption with an increase in pH is due to a decrease in competition between hydrogen ions and metal ion for the surface sites. At lower pH, the surface of the adsorbent is surrounded by hydronium ions (H^+), thereby blocking metal ions from binding sites on the adsorbent. At pH of 8.0, there was maximum adsorption of Cr(VI) ion and

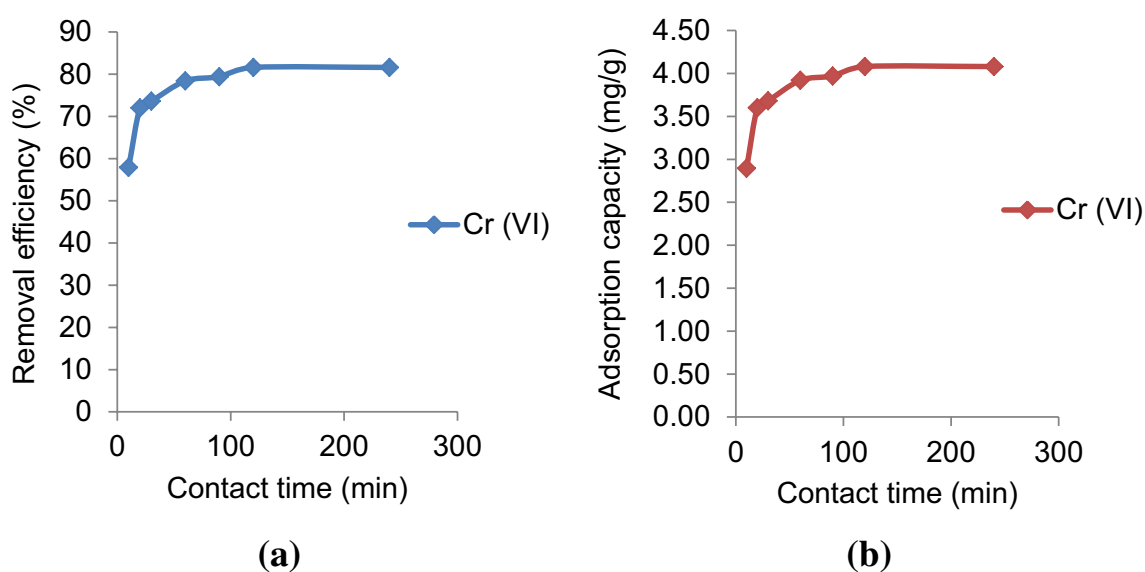


Fig. 2 Effect of contact time on the adsorption of Cr(VI) ion

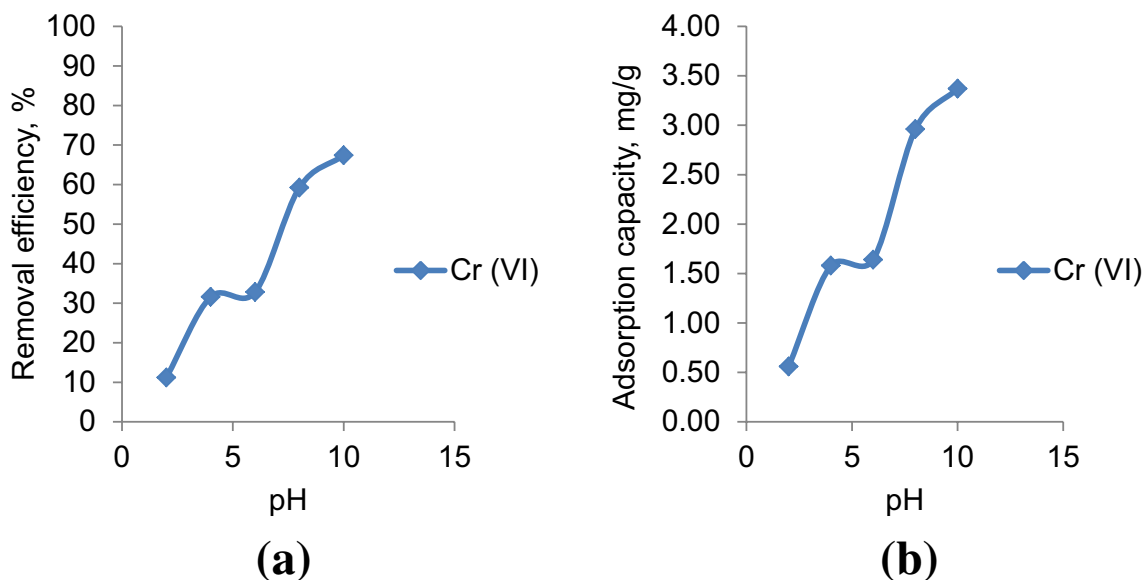


Fig. 3 Effect of pH on the adsorption of Cr(VI) ion

this is due to the fact that metal sorption tends to increase significantly by increasing the solution pH above pH_{zpc} of the adsorbent (Krika et al. 2016). The pH_{zpc} value of the groundnut shell was found to be 5.5, which is less than the maximum pH (8.0) obtained for the adsorption of Cr(VI) ion.

Effect of adsorbent dose

Figure 4 shows that the adsorption of chromium(VI) increased rapidly when the adsorbent dose was increased from 0.5 to 2.0 g/L and remain constant after an

adsorbent dose of 2.0 g/L. The removal efficiency (Fig. 4a) of chromium(VI) was observed to increase from 56.6 to 87.6% when the dose was increased from 0.5 to 2.0 g/L. On the contrarily, the adsorption capacity decrease from 5.66–2.19 mg/g when the adsorbent dose was increased from 0.5–2.0 g/L as can be seen in Fig. 4b. The rapid increase in the removal efficiency of chromium(VI) as the amount of the adsorbent dose increases is due to the availability of more adsorption sites and greater availability of the surface area at a higher concentration of the adsorbent (Mohanty et al. 2005). The decrease in adsorption capacity (Fig. 4b) can be

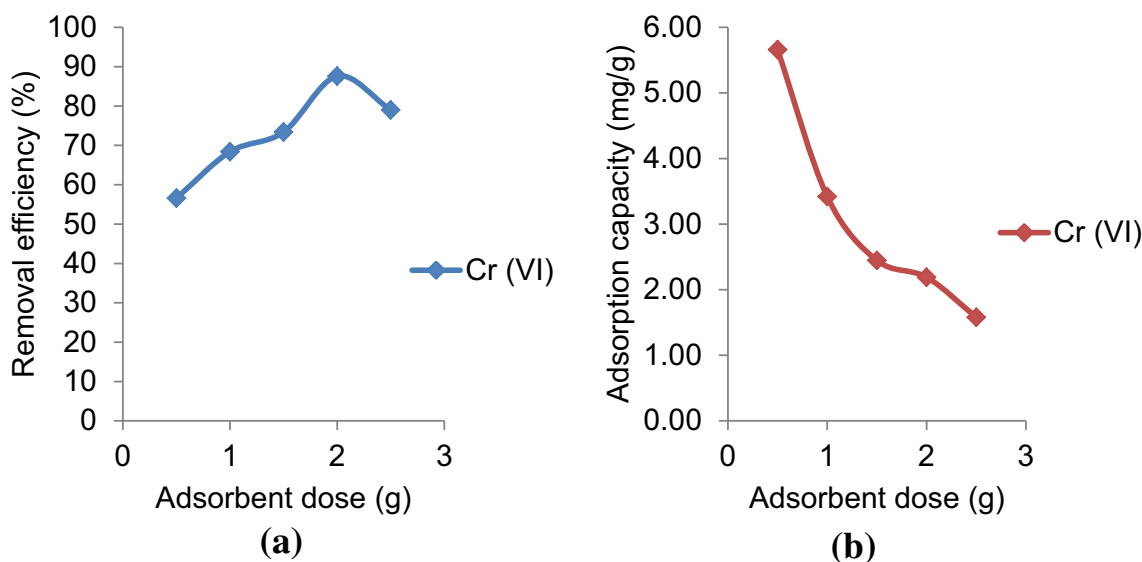


Fig. 4 Effect of adsorbent dose on the adsorption of Cr(VI) ion

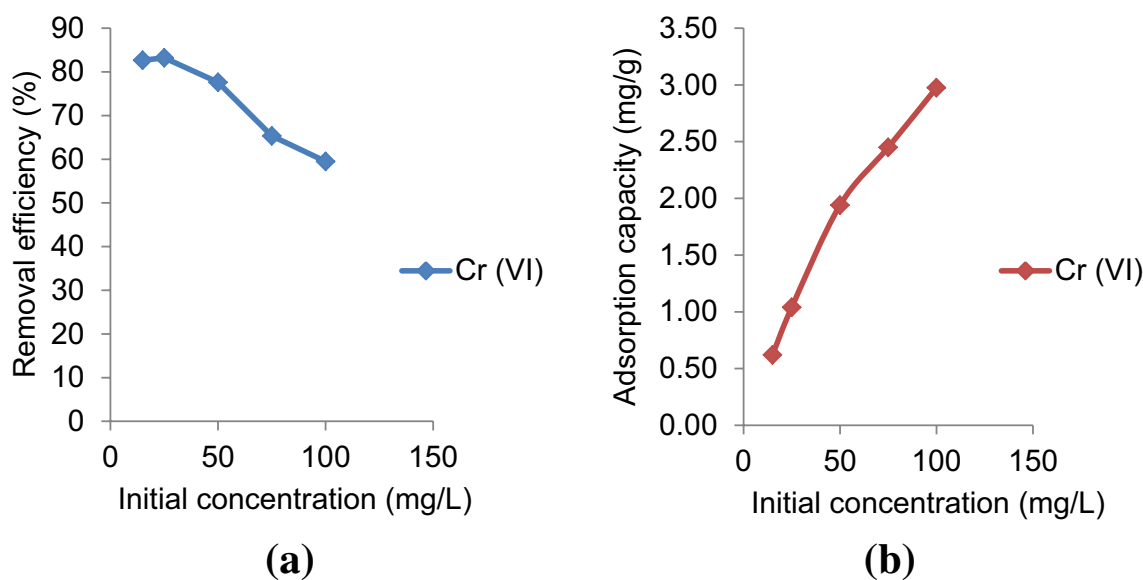


Fig. 5 Effect of initial concentration on the adsorption of Cr(VI) ion

attributed to the fact that some of the adsorption sites remain unsaturated during the adsorption process, whereas the number of available adsorption sites increases by an increase in adsorbent and this results in an increase in removal efficiency (Rengaraj et al. 2004).

Effect of initial metal ion concentration

The results presented in Fig. 5 showed that the removal efficiency (Fig. 5a) gradually decreased with increasing initial chromium(VI) ion concentration. The removal efficiency and adsorption capacity (Fig. 5b) at lower chromium(VI) concentration (15 mg/L) were found to be 82.7% and 0.62 mg/g, respectively, and at higher chromium(VI) concentration (100 mg/L), the removal efficiency and adsorption capacity were 59.5% and 2.98 mg/g, respectively. However, it was observed that the adsorption capacity of Cr(VI) ion increased with an increase in initial concentration, and thereafter, it reached a constant value indicating that no more Cr(VI) was further removed from solution as shown in Fig. 5.

The removal efficiency of Cr(VI) at higher concentration levels shows a decreasing trend due to the saturation of adsorption sites (Ahmet et al. 2004), whereas, at lower concentrations, all metal ions present in solution could interact with the adsorption sites and therefore higher removal efficiency. The increased in the adsorption capacity with increasing Cr(VI) ion concentration in the test solution is due to the high probability of collision between the metal ions and groundnut shell surface. Furthermore, higher concentration levels provided a better concentration gradient, which is an important driving force that helps

overcome the mass transfer resistance of the metal ion between the liquid and solid phases (Futalan et al. 2012).

Effect of temperature

From Fig. 6, the adsorption of Cr(VI) ion increased rapidly with an initial increase in temperature and later declined with a further increase in temperature. The removal efficiency (Fig. 6a) and adsorption capacity (Fig. 6b) of chromium(VI) increased from 43.2–76.8% to 0.61–0.96 mg/g, respectively, when the temperature was increased from 27.5 to 41.5 °C. Further increase in temperature beyond 41.5 °C resulted in a decline in removal efficiency and adsorption capacity. At the maximum temperature (41.5 °C), removal efficiency and adsorption capacity were found to be 76.8% and 0.96 mg/g, respectively. The removal efficiency and adsorption capacity for Cr(VI) ion increased with the increase in temperature indicating the process to be endothermic in nature (Ho 2006). This effect is characteristic of a chemical reaction or bond being involved in the adsorption process (Mohapatra et al. 2010; Aksu 2002). Alao et al. (2014), in their study on adsorption of Zn (II) ions on *Carica papaya* root powder, obtained similar results. They suggested that the enhancement of the adsorption capacity when the temperature is increased could be due to increased mobility and diffusion of ionic species.

Adsorption isotherm studies

The ability of groundnut shell to adsorb Cr(VI) ion from aqueous solutions was determined from the adsorption isotherms plot. The equilibrium data were applied to three two-parameter (Langmuir, Freundlich and Temkin) isotherm models, and the

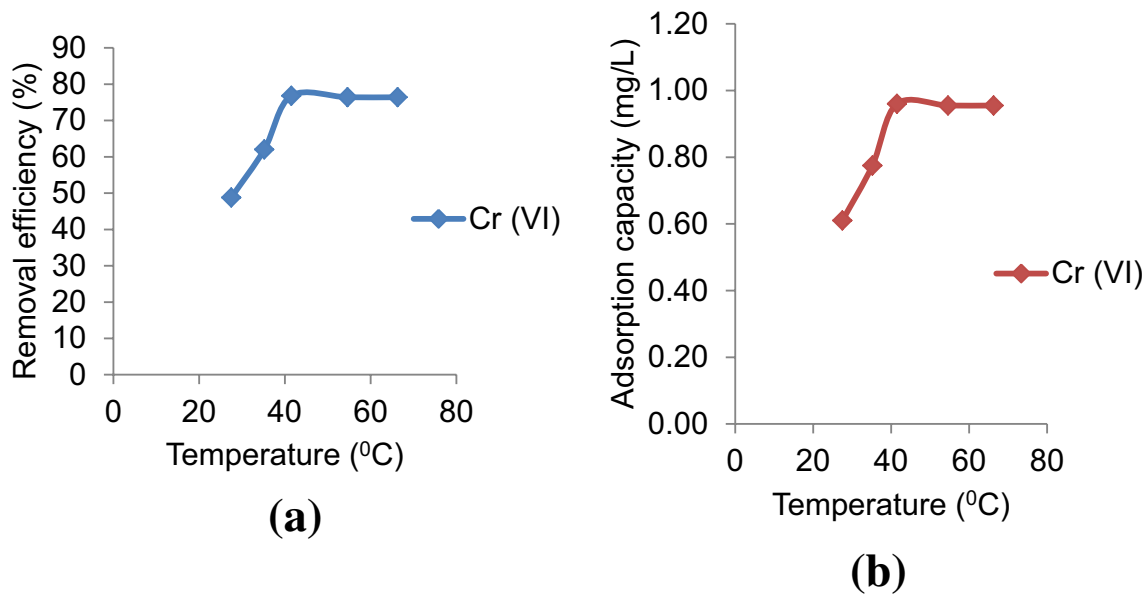


Fig. 6 Effect of temperature on the adsorption of Cr(VI) ion

results of their linear regressions were used to find the model with the best fit. The linear forms of Langmuir, Freundlich and Temkin isotherms are represented by Eqs. (4, 6, 8).

The Langmuir adsorption isotherm model is expressed as in Eq. (4):

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m} \tag{4}$$

where C_e is the concentration of chromium(VI) solution at equilibrium (mg P/L); q_e is the corresponding adsorption capacity (mg P/g); and q_m (mg P/g) and K_L (L/mg) are constants which are related to adsorption capacity and energy or net enthalpy of adsorption, respectively. The constants q_m and K_L are obtained by plotting $\frac{C_e}{q_e}$ versus C_e (Fig. 7) with a slope of $\frac{C_e}{q_m}$ and intercept of $\frac{1}{q_m K_L}$.

The conformity of the adsorption process to the Langmuir model is usually determined using Eq. (5). The value of R_L give an idea about the shape of isotherm (Chakravarty et al. 2010).

$$R_L = \frac{1}{1 + K_L C_0} \tag{5}$$

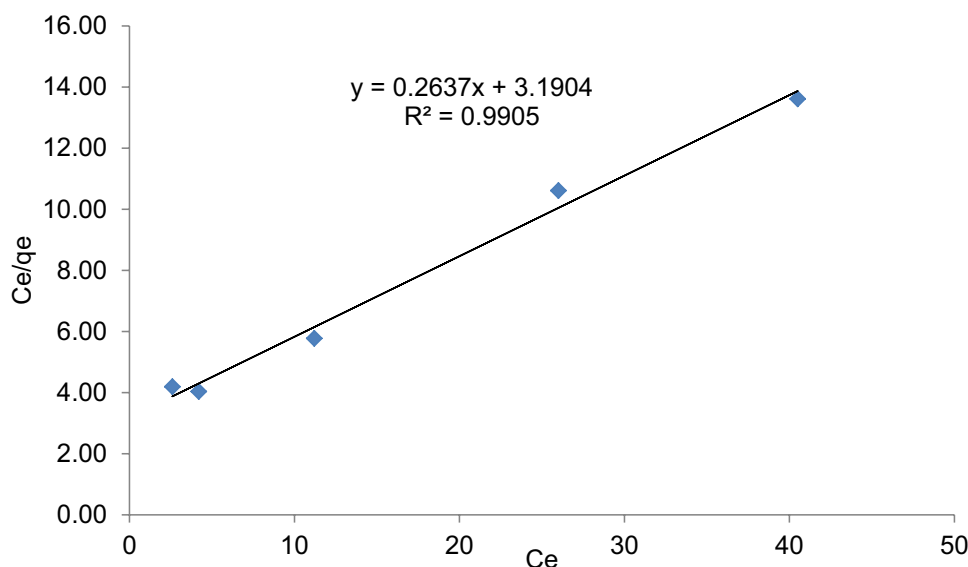
where R_L is the separation factor and K_L is the Langmuir constant (L/mg) which is related to the energy of adsorption through the Arrhenius equation. C_0 is the initial concentration of the metal ions. When $R_L > 1$, it indicates an unfavorable monolayer adsorption process, $R_L = 1$, linear, $0 < R_L < 1$, favorable and $R_L = 0$, irreversible.

From Fig. 7, it is evident that the Langmuir isotherm model describes the adsorption of Cr(VI) unto groundnut shell from aqueous solution as indicated by the high correlation coefficient (R^2) of 0.9905. The fitness of the Langmuir model to the adsorption process connotes that the metal ion molecules from bulk solution were adsorbed on specific monolayer that is homogeneous in nature (Oluwamukomi 2009; Kumar and Sivanesan 2007). As can also be seen from Table 1, the R_L value lies between 0 and 1 which confirms the adsorption processes to be favorable under the studied conditions. The monolayer adsorption capacity (q_m) value was found to be 3.792 mg/g indicating high adsorption capacity of groundnut shell. The value of K_L (0.083 L/mg) is relatively high which implies high surface energy in the process and consequently high bonding between the metal ion and the groundnut shell.

The Freundlich adsorption isotherm model is expressed as in Eq. (6):

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \tag{6}$$

where C_e is the concentration of solute in the bulk solution at equilibrium (mg/L); q_e is the corresponding adsorption capacity (mg/g); K_F is a constant indicative of the relative adsorption capacity of the adsorbent (mg/g) and n is a constant indicative of the intensity of adsorption. From the plot of $\log q_e$ versus $\log C_e$ (Fig. 8), the constants n can be obtained from the slope $\frac{1}{n}$ and K_F from the intercept $\log K_F$. The slope ranges between 0 and 1 is a measure of adsorption intensity or surface heterogeneity, becoming more heterogeneous as its value gets closer to zero, whereas, a value of $\frac{1}{n}$ below unity implies a chemisorption process, $\frac{1}{n}$ above one

Fig. 7 Langmuir adsorption isotherm plot of Cr(VI) ion**Table 1** Langmuir, Freundlich and Temkin isotherm constants for the adsorption of Cr(VI) ion onto groundnut shell

Model	Parameter	Value
Langmuir	q_m	3.792
	K_L	0.083
	R_L	0.243
	R^2	0.9905
Freundlich	K_F	0.433
	n	1.840
	$\frac{1}{n}$	0.54
	R^2	0.9535
Temkin	A	1.028
	b	3139.34
	B	0.833
	R^2	0.9944

is an indicative of cooperative adsorption (Haghseresht and Lu 1998; Brasquet et al. 1997).

The Freundlich adsorption isotherm also conforms to the sorption of Cr(VI) ion from aqueous solutions using groundnut shell as shown in Fig. 8.

However, the level of conformity is to a less extent compared to the Langmuir adsorption isotherm as indicated by a lower correlation coefficient value of 0.9535. From Table 1, the value of n (1.840) confirms that the groundnut shell have a heterogeneous surface since the value satisfy the heterogeneity condition, $1 < n < 10$. Also, a value of $\frac{1}{n}$ (0.54) below unity implies a chemisorption process (Haghseresht and Lu 1998; Brasquet et al. 1997). The values of K_F were found to be 0.433 mg/g which implies that there was low uptake of the metal ions unto the adsorbent surface.

The Temkin adsorption isotherm model is expressed as in Eq. (8):

$$q_e = \frac{RT}{b} \ln A + \frac{RT}{b} \ln C_e \quad (7)$$

Equation (7) can also be expressed as in equation (2.32)

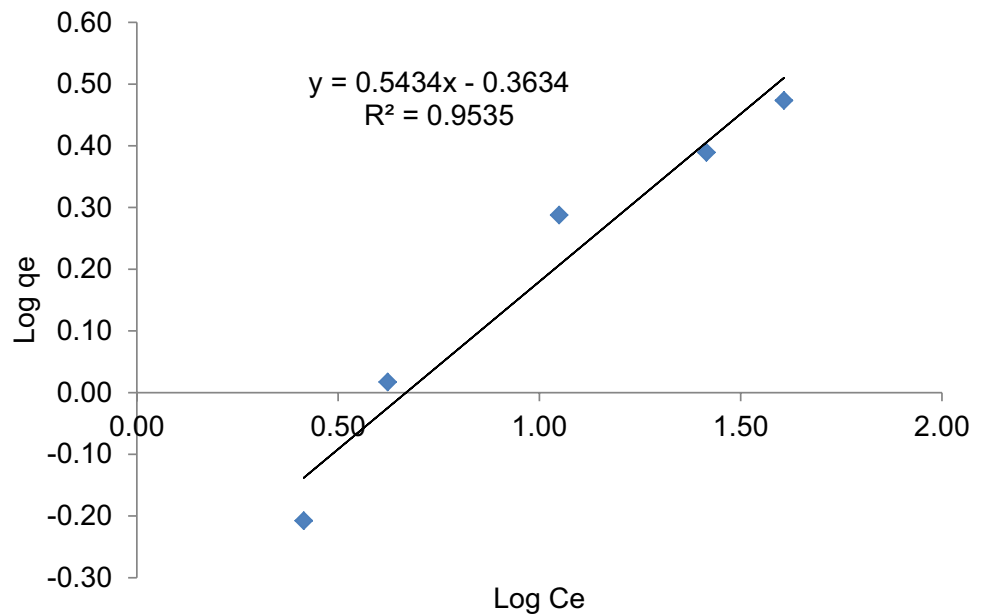
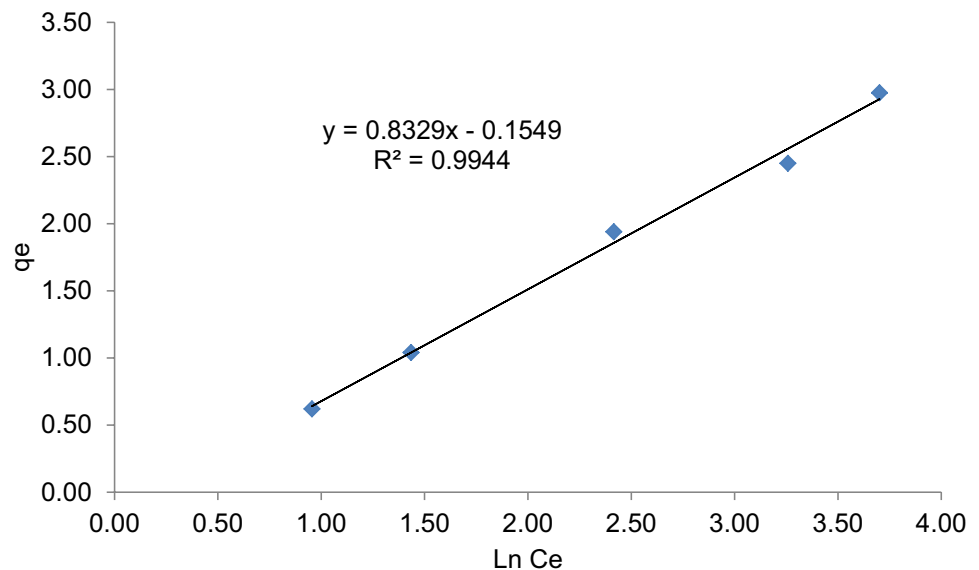
$$q_e = B \ln A + B \ln C_e \quad (8)$$

where

$$B = \frac{RT}{b} \quad (9)$$

R is gas constant (8.314 J/mol/K), T is temperature (K), C_e is the concentration of solute in the bulk solution at equilibrium (mg/L); q_e is the corresponding adsorption capacity (mg/g), b and A are Temkin's constants. By plotting q_e versus $\ln C_e$ (Fig. 9), the constant b can be obtained from the slope, $\frac{RT}{b}$ and A from the intercept, $\frac{RT}{b} \ln A$.

From Fig. 9, the correlation coefficient (R^2) for the Temkin isotherm is 0.9944 which is higher than the values obtained from the other isotherm models. Hence, the experimental data fit very well to this isotherm model and indicate that Cr(VI) adsorption occurs because of the heat of adsorption (ΔH°) of all molecules in the layer decreases linearly because of increased surface coverage (Aharoni and Ungarish 1977a, b). From Table 1, the intensity of sorption, A (1.03 L/mg) and the heat of sorption b (3139.34 J/mol) confirm further that the data best fit the Temkin isotherm. Hutson and Yang (1997) applied the Temkin isotherm model to confirm that the adsorption of cadmium ion onto non-zero-valent iron particles follows a chemisorption process.

Fig. 8 Freundlich adsorption isotherm plot of Cr(VI) ion**Fig. 9** Temkin adsorption isotherm plot of Cr(VI) ion

Conclusion

The groundnut shells investigated in this study exhibited high potential for the removal of chromium(VI) ion from aqueous solution even without physical or chemical modification. The adsorption of chromium(VI) ion was highly dependent on contact time, pH, adsorbent dose, initial metal ion concentration and temperature. The adsorption of Cr(VI) was found to be optimum at a contact time of 120 min; pH 8; adsorbent dose of 2.0 g/L; metal ion concentration of 25 mg/L; and temperature of 41.5 °C. For all the isotherm models tested, Temkin model was found to have the highest correlation coefficient (R^2) and hence the best fit.

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Compliance with ethical standards

Conflict of interest The authors report no conflict of interest.

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