

Equilibrium isotherm studies for the sorption of hexavalent chromium (VI) onto groundnut shell

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Abstract: Groundnut shells, a low-cost agricultural waste material were used without any physical or chemical modification as an alternative and cheap adsorbent for the removal of Cr (VI) ion from aqueous solution. The aqueous solutions containing different concentrations of Cr (VI) were treated with fixed doses of groundnut shells powder for an equilibrium time of 90 min. The determination of Cr (VI) concentration in the sample solutions was carried out using UV-Vis spectrophotometer by the diphenylcarbazide indicator method. The equilibrium isotherm studies showed that the groundnut shell has a significant capacity for the adsorption of Cr (VI) from aqueous solutions. For the various adsorption isotherm models tested, Tempkin isotherm model showed a better representation of the equilibrium data with a good correlation coefficient (R^2) greater than 0.99. The FT-IR results gave an indication of the possible functional groups located on the surface of the groundnut shell. These groups influence the biosorption of Cr (VI) and are likely the mechanism of adsorption, which largely depended on the functional groups present such as alcohols, amines, and carboxylic acids.

Keywords: Adsorption, adsorbent, groundnut shell, isotherm, chromium

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I. Introduction

The presence of toxic contaminants in aqueous solution from the discharge of untreated metal containing effluents into water bodies have become a very serious environmental concern. The presence of heavy metals in the environment through human activities in recent times has increased due to industrialization, urbanization, automobile emissions and mining activities [1]. It is important to reduce pollutants to their permissible limits especially toxic metals in the aquatic environment. The exposure of heavy metals can be detrimental to human health and other living organisms [2]. Heavy metals are present in the soil, natural water and air in various forms and may become contaminants in food and drinking water [3]. Heavy metals are non-biodegradable and they tend to accumulate in the tissues of living species leading to many health disorders and several ailments such as accumulative poisoning, cancer, nervous system damage and consequently death [4]. Among the toxic heavy metals, chromium in its hexavalent form is known to cause wide-ranging human health effect including mutagenic and carcinogenic risks [5]. Chromium is globally used in electroplating, leather tanning, metal finishing, and chromate preparation and is mostly present in high concentration in the aqueous waste released directly into the environment without any proper pretreatment [6]. The determination of trace amounts of chromium in soils and other naturally occurring materials is of considerable interest due to the contrasting biological effects of its two common oxidation states, chromium (III) and chromium (VI) and the growing interest in environmental problems. Cr (VI) exists in aqueous solution as oxyanions such as chromate (CrO_4^{2-}) and dichromate ($\text{Cr}_2\text{O}_7^{2-}$, HCrO_4^- and HCr_2O_7^-) forms and its ingestion may cause dental caries, neuromuscular disorders, hepatic and renal dysfunction, and blood cancer [7]. Chromium (III) is an essential nutrient for maintaining normal physiological function whereas chromium (VI) is toxic and carcinogenic [8]. While an increase in chromium concentration in soils makes them infertile depending on the chromium oxidation state, the application of chromium salts into soils activate some biochemical processes [9]. The tolerance limit for Cr (VI) for discharge into inland surface waters is 0.1 mg/L and in potable water is 0.05 mg/L, however, its concentration in industrial wastewater ranges from 0.5-270 mg/L [8].

The contamination of potable water by toxic heavy metals has led to the development of various technologies for water purification. The conventional methods for removing heavy metals include evaporation, ion exchange, reverse osmosis, chemical precipitation and adsorption technique, which proved to be very effective [10]. Activated carbons have been recognized as the most used adsorbents for Cr (VI) mitigation because of their high surface area and high porous character [11, 12]. However, due to the high cost of activated

carbon, there is a need to find and use low-cost adsorbents for Cr (VI) removal from cheap and readily available natural materials, which can be used economically on a large scale. Several studies have reported on the potential of agricultural by-products as good substrates for the removal of heavy metals such as orange peel [13], sawdust [14] and maize leaf [15]. This process involved using waste to treat waste and become even more efficient because these agricultural wastes are readily available and often suffer waste disposal problems. Hence, they are available at little or no cost, since they are waste products. This makes the process of treating wastewaters or effluents with agro-based adsorbents more cost-effective than the use of conventional adsorbents like activated carbon [2]. The efficiencies of various non-conventional adsorbents towards adsorbate removal ranged between 50 and 90 % depending on the particle size of the adsorbent, the characteristics, and concentration of the adsorbate [16].

The purpose of the adsorption isotherms is to relate the adsorbate concentration in the bulk and the adsorbed amount at the interface [17]. It also helps to estimate the economic feasibility of an adsorbent for specific commercial applications [18].

This current study focuses on assessing the removal efficiency of chromium (VI) from aqueous solution using groundnut shell, an agricultural waste material readily available in the Northern part of Ghana. One-parameter, two-parameter and three-parameter adsorption isotherm models were used to investigate the adsorption process.

II. Material And Methods

2.1 Preparation of Adsorbent

The groundnut shells as an adsorbent were prepared using procedures described by [19] in an earlier study. The adsorbent was sieved through 250 μm pore spaces and used without any modification.

2.2 Preparation of Adsorbate

All chemicals used for the study were of analytical reagent grade and de-ionized water was used throughout the experiment. A stock solution of chromium (VI) solution (1000 mg/L) was prepared by dissolving 2.829 g of potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) in 200 mL of de-ionized water and consequently diluted to the 1000 mL mark of the volumetric flask using de-ionized water. The stock solution was further diluted as needed. The diphenylcarbazide indicator method which is a more sensitive method was used for the determination of hexavalent chromium (VI) where Cr (VI) forms a pink colored complex with 1,5-diphenylcarbazide in acidic medium and was analyzed spectrophotometrically [18].

2.3 Characterisation of the adsorbent

Spectroscopic analysis was performed on the groundnut shell using Fourier Transform Infrared Spectrometer (Spectrum Two 94133 series) to determine its functional groups. The characterization of the groundnut shell was done before and after it had been used in the removal of Cr (VI) ions from the aqueous solution.

2.4 Equilibrium isotherm studies

The equilibrium adsorption isotherm studies were carried out by contacting 1.0 g/L of groundnut shell (maximum dose) with initial Cr (VI) concentration of 15, 25, 50, 75, and 100 mg/L at room temperature (25 $^{\circ}\text{C}$). The pH of the aqueous solutions was maintained at pH 8.0 using 0.1 M NaOH or 0.1 M HCl. The adsorbate-adsorbent solutions were stirred at a constant speed of 120 rpm using a magnetic stirrer for equilibrium time of 90 min. The mixtures were filtered and the residual metal ions in the filtrate were analyzed using Carry 60 UV-Vis spectrophotometer.

2.5 Determination of adsorption capacity and removal efficiency

The adsorption capacity (q_e) and removal efficiency (RE) were evaluated using equations (1-2).

Adsorption capacity (q_e):

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

Removal efficiency (RE):

$$RE = \frac{(C_0 - C_e)}{C_0} \times 100 \quad (2)$$

where q_e is the equilibrium mass of the adsorbed substance per unit mass of adsorbent (mg/g), C_0 is the initial metal ion concentration (mg/L), C_e is the equilibrium metal ion concentration (mg/L), V is the volume of solution (mL) and m is the mass of the adsorbent (g).

III. Results and Discussion

3.1 Characterization of groundnut shell

The FT-IR spectra of groundnut shell before and after it was used in the adsorption of Cr (VI) ions from the aqueous solution are shown in Figs. 1 and 2 respectively. The distinct peaks in Fig. 1 were found at 3322.39, 2921.43, 1727.51, 1628.90, 1246.67 and 1024.22 cm^{-1} which corresponded to -O-H, -N-H, -C-H, -C=O, -C=C and -C-O stretching vibrations respectively. It was observed that the distinct peaks shifted to 3323.02, 2914.01, 1729, 1625.21, 1248.94 and 1028.02 cm^{-1} respectively after using the sample to adsorb Cr (VI) as shown in Fig. 2. The peaks at 3322.39, 2921.43, 1727.51, 1246.67 and 1024.22 cm^{-1} shifted to higher wavenumbers while peaks at 2921.43 and 1628.90 cm^{-1} shifted to lower wavenumbers. From Fig. 1, the C \equiv C stretching vibration appeared at peaks 2323.12, 2168.90, 2103.93 and 2050.79 cm^{-1} respectively. However, these peaks showed the same signals after the sample was used to adsorb Cr (VI) ions [Fig. 2].

In a study carried out by [20] indicated that agricultural waste materials have functional groups such as carbonyl, acetamido, phenolic, amido, carboxyl, amino, alcohols, esters, and sulphhydryl and these groups possess an affinity for complexation with metals. In this current study, the presence of hydroxyl group, coupled with the carbonyl group in the FT-IR analysis (Fig. 1 and 2) suggested the presence of carboxylic acid groups in the groundnut shell [21]. The most important adsorption sites in agricultural waste materials that are responsible for binding heavy metals are the hydroxyl (OH), amine (NH), carbonyl (C=O) and carboxylic (COOH) groups as indicated by [22] in their study. From Fig. 1, all these important functional groups were present on the surface of the groundnut shell indicating the potential of the groundnut shell to bind Cr (VI) ions from the aqueous solution. Fig. 1 showed that many functional groups shifted to different frequency levels after adsorption, indicating the possible involvement of those groups for the uptake of Cr (VI) ions [23]. Similarly, [24] indicated that shifting of bands usually confirmed the adsorption of metal ions on the surface of the adsorbent. The binding mechanism involved electron pair sharing between surface functional groups of the adsorbent and metal ions.

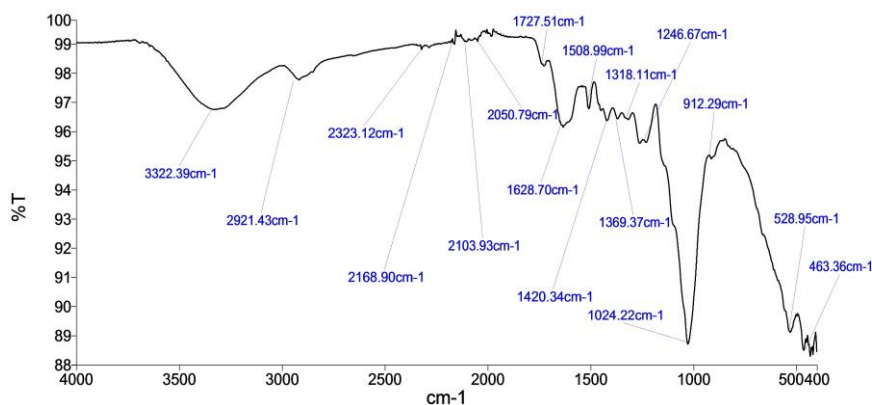


Figure 1: FT-IR spectra of groundnut shell without treatment with Cr (VI) ion

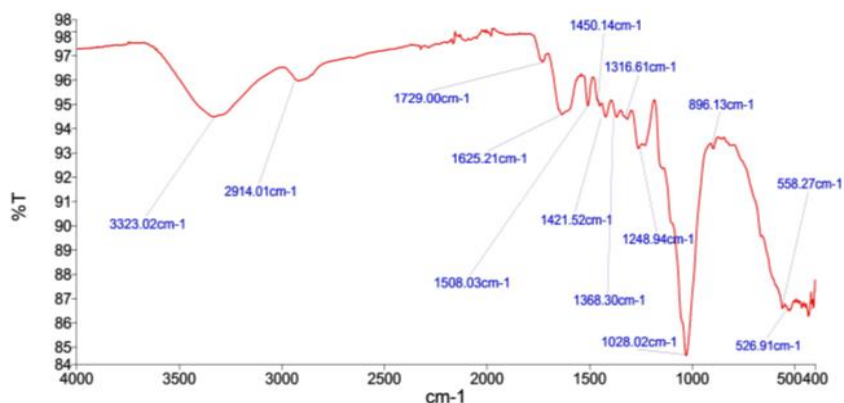
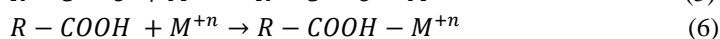
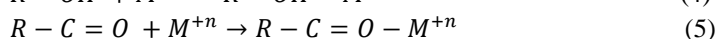
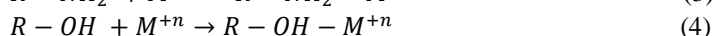
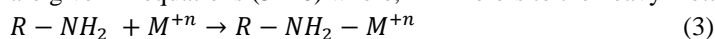


Figure 2: FT-IR spectra of groundnut shell after treatment with Cr (VI) ion

The possible mechanism of the reactions between the surface functional groups of the adsorbent and metal ions are given in equations (3 – 6) where, M^{+n} refers to the heavy metal ion (Cr) and the respective charge.



After the adsorption of Cr (VI) [Fig. 2] ions, the FT-IR spectra of the adsorbent nearly coincided with that of the original spectra of the raw groundnut shell (Fig. 1). Therefore, the basic structure and properties of the groundnut shell remained relatively stable during the process of the metal ions adsorption that suggested that it might be applicable as an effective renewable adsorbent.

3.2 Equilibrium adsorption isotherm studies

Adsorption isotherms simplify the relationship between the concentration of the adsorbate and the degree of surface coverage and are informative in providing details on the mechanism of adsorption. They further show how ions can be distributed between the liquid and solid phases at various equilibrium concentrations. The efficiency of groundnut shell to interact with Cr (VI) ion in the aqueous solution was determined by applying the experimental data to linearized equations of one-parameter (Henry), two-parameter (Langmuir, Freundlich, Dubinin-Radushkevich, Tempkin, Harkin-Jura and Elovich) and three- parameters (Redlich-Peterson and Jossens) adsorption isotherm models [19]. The calculated values of the various isotherm model constants and their correlation coefficients (R^2) for the adsorption of Cr (VI) ion onto groundnut shell are summarized in Table 1. The analysis of the one-parameter isotherm (Table 1) showed that the equilibrium data fitted the Henry isotherm model with a correlation coefficient (R^2) of 0.8959. From the assumption of Henry isotherm, the level of fitness suggested that the adsorption of Cr (VI) occurred at relatively low concentrations such that all metal ions were separated from their nearest neighbors [25]. The high values of K_{HE} (0.051 L/g) confirmed the fitness of the Henry isotherm model to the experimental data.

From the two-parameter isotherm models analyzed, the correlation coefficient (R^2) of 0.9905 meant that the Langmuir model fitted the equilibrium data well. This could indicate monolayer adsorption by the groundnut shell surface which could contain a finite number of identical sites [26, 27]. From Table 1, the R_L value was between 0 and 1 and this indicated the favorability of the isotherm model. The monolayer adsorption capacity (q_m) value was found to be 37.92 mg/g showing that the groundnut shell had high adsorption capacity. The value of K_L (0.083 L/mg) was also high implying high surface energy during the adsorption process as well as an effective collision between Cr (VI) ions and the groundnut shell. Freundlich model also fitted the data but the level of fitness was lesser than that of the Langmuir model as indicated by the correlation coefficient, R^2 (0.9535). The value of n was found to be 1.840 indicating that the groundnut shell had a heterogeneous surface since this value obeyed the heterogeneity condition, that is $1 < n < 10$. In addition, the value of $\frac{1}{n}$ was found to be below unity which showed a chemisorption process [28]. The value of K_F was 0.433 mg/g implying that there was low uptake of the Cr (VI) ion onto the groundnut shell surface. The correlation coefficient of 0.9301 showed that the Dubinin-Radushkevich isotherm fitted the equilibrium data. This showed that Cr (VI) uptake occurred onto both homogeneous and heterogeneous surfaces of the ground shell. The calculated value of Dubinin-Radushkevich constant (K_{DR}) was $2.0 \times 10^{-6} \text{ mol}^2/\text{kJ}^2$ and the mean sorption energy was 0.50 kJ/mol which indicated a physisorption process [29]. Among the two-parameter models, Tempkin adsorption isotherm model showed the best representation of the experimental data with the highest correlation coefficient of 0.9944. Tempkin isotherm assumed that the fall in the heat of sorption is linear rather than logarithmic [30]. The intensity of sorption (A) and the heat of adsorption (b) confirmed that the data fitted this model and indicated that the adsorption process was due to chemisorption. Harkin-Jura isotherm fitted less to the data with a low correlation coefficient of 0.7222 indicating that there was less possibility of multilayer adsorption of the metal ion on the surface of the adsorbent [31]. This indicated that Cr (VI) ions could be best adsorbed on a specific monolayer of the groundnut shell that is homogeneous in nature as Langmuir isotherm suggested. The Harkin-Jura constants, A and B were also found to be low indicating the non-conformity of the experimental data. The adsorption of Cr (VI) followed Elovich isotherm model with good a correlation coefficient of 0.9132 indicating a chemisorption process [32]. The high values of q_m and K_e showed that the experimental data fitted the model well. From the two-parameter isotherm data analyzed, it was found that Freundlich, Tempkin and Elovich models described the removal of Cr (VI) from the aqueous solution by the adsorbent to be due to chemisorption process.

For the three-parameter adsorption isotherm models investigated, the experimental data fitted well to the Redlich-Peterson isotherm model than that of Jossens model with correlation coefficients of 0.9353 and 0.9132 respectively. The conformity of the experimental data to the Redlich-Peterson model indicated that the mechanism of adsorption was a mixed type and did not necessarily follow ideal monolayer adsorption [33]. This

is because the Redlich-Peterson isotherm is a combination of the Langmuir and Freundlich isotherms. While Langmuir assumed homogeneous nature of the adsorbent, Freundlich isotherm assumed heterogeneity of the sorbent surface. These assumptions are in agreement with the Dubinin-Radushkevich isotherm, which suggested that the adsorption of Cr (VI) occurred on both homogeneous and heterogeneous surfaces of the groundnut shell. The relatively high values of β and A further confirmed this fitness. The fitness of the data to Jossens isotherm showed that the groundnut shell had a heterogeneous surface with respect to the interactions it had with Cr (VI) ion [34]. The Freundlich model also confirmed that the mechanism of the adsorption of Cr (VI) occurred on the heterogeneous surface of the groundnut shell. This was further confirmed by the high values of Jossens constants; H and F.

The analysis of the various adsorption isotherm models showed that the equilibrium data fitted well into nine models with increasing order of correlation coefficient (R^2) as follows; Harkin-Jura ($R^2 = 0.7222$), Henry ($R^2 = 0.8954$), Elovich and Jossens ($R^2 = 0.9132$), Dubinin-Radushkevich ($R^2 = 0.9301$), Redlich-Peterson ($R^2 = 0.9353$), Freundlich ($R^2 = 0.9535$), Langmuir ($R^2 = 0.9905$) and Tempkin ($R^2 = 0.9944$).

According to previous research studies, Langmuir and Freundlich models have the best fitness quality with experimental data in the case of two parameter isotherm models. Regarding three parameter isotherm models, Redlich-Peterson and Sips models provide the best agreement with experimental data [35]. This is in agreement with the findings of this present study. Among all the two parameter isotherm models, Langmuir and Tempkin were the best-fitted models with $R^2 > 0.99$ while Redlich-Peterson model was the well fitted three parameter isotherm model with $R^2 > 0.93$.

Comparatively, the Tempkin and Langmuir models showed the best representation of the equilibrium data indicating chemisorption and monolayer adsorption respectively [25, 26]. From literature, other studies also reported similar findings. Tempkin isotherm model was applied by [33, 34] and their study confirmed that the adsorption of cadmium ion onto nano zero-valent iron particles followed a chemisorption process. Similarly, [38] used fungal biomass for the removal of Cr (VI) from the industrial effluent. Among the equilibrium models tested, the Langmuir isotherm model was found to be the best fit for the obtained experimental data, thus suggesting a monolayer biosorption process.

In this present study, the entire adsorption process showed that the surface of the groundnut shell was more homogeneous than heterogeneous but with monolayer adsorption being the most superior.

Table 1: Calculated values of the various isotherm models constants and their correlation coefficients (R^2) for the adsorption of Cr (VI) ion onto groundnut shell

Henry		Langmuir				Freundlich				
K_{HE} L/g	R^2	q_m mg/g	K_L L/mg	R_L	R^2	K_F mg/g	n	$\frac{1}{n}$	R^2	
0.057	0.8954	37.92	0.083	0.243	0.9905	0.433	1.840	0.543	0.9535	
Dubinin- Radushkevich (R-P)				Tempkin				Harkin-Jura		
K_{DR} mol^2/kJ^2	q_s mg/g	R^2	E J/mol	A L/mg	b J/mol	B	R^2	A	B	R^2
2.0×10^{-6}	2.428	0.9301	0.500	1.028	3.139	0.833	0.9944	0.567	1.484	0.7222
Elovich			Redlich-Peterson			Jossens				
K_e L/g	q_m mg/g	R^2	A	β	R^2	H	F	R^2		
0.212	1.852	0.9132	0.433	0.457	0.9353	2.550	0.540	0.9132		

IV. Conclusion

This study concluded that groundnut shells could be used as an alternative adsorbent for chromium (VI) removal from aqueous solution. From the Langmuir model, the monolayer adsorption capacity (q_m) was found to be 37.92 mg/g indicating high adsorption capacity of the adsorbent. The application of the experimental data to the adsorption isotherm models showed that the Tempkin and Langmuir isotherm models (two-parameter models) fitted very well with correlation coefficients (R^2) > 0.99 . The Fourier transform infrared analysis confirmed the presence of amino ($-NH$), carbonyl ($-C=O$) and hydroxyl ($-OH$) functional groups on the surface of the adsorbent and their possible involvement for the removal of Cr (VI) ions from the aqueous media.

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