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Chick-Pea Pod Husk: A Natural Green Waste Material and its Application for Removal of Hexavalent Chromium Oxyanion from Water

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Abstract

Green Waste Material Chick-Pea husk, an agricultural waste product utilized for withdrawal of hexavalent chromium oxyanion from water. Chick-Pea husk surface area was analyzed by BET surface area analyzer. The further characterization of Chick-Pea husk was achieved by FT-IR spectroscopy, SEM and EDX analysis. Various parameters were investigated. Hexavalent chromium oxyanions were analyzed by U.V-Vis spectrophotometer. Desorption of hexavalent chromium oxyanions was successfully made by sodium hydroxide. The results obtained through various parameters are employed to explore Langmuir, Freundlich, D-R isotherms, adsorption-kinetics and thermodynamics of adsorption. The objective of this research is to eliminate toxic Cr (VI) oxyanions from polluted water. The technique successfully utilized to withdrawal hexavalent chromium oxyanions from water utilizing Chick-Pea husk by applying Batch mode.

Keywords: Contaminant, Adsorption, Adsorbent, parameters, Characterization

Introduction

Oxoanions or oxyanions are poly atomic ions involving one or more oxygen atoms plus another metal or non-metal atom (Chen K, et al., 2024). Oxyanions commonly have the negative charges such as chromite, chromate, arsinite, arsenate, selinite, selenate, nitrate, phosphate (Fiol et al., 2008). These oxyanions of metals dispatched by various waste products such as industrial waste results the water pollution (Shah, et al., 2023). This contaminated water is used for various purposes. These toxic oxyanions results numerous diseases (Senthil and Santhil, 2021).

The WHO threshold limits for drinking water in micro gram per liter for Cr, As, Se, Hg, Cd are 50, 10, 40, 6, and 3 respectively (WHO, 2019). To clean water it is necessary to remove these toxic oxyanions from water (Hu, et al., 2024). Numerous modes are acquired for removal of toxic oxyanions from water (Akula and Philip, 2023).

So many green waste materials have been explored for removal of these oxyanions from water (Kainth, et al., 2024).

Currently, adsorption of oxyanions by green waste materials becomes apparent eco-friendly and contemptible technologies (Yuan and Lu, 2024).

Many green waste materials such as orange peel (Gao, et al., 2024), wood activated carbon (Wang, et al., 2023), pomelo peel (Tan, et al., 2024), avocado seed (Mobalane, et al., 2024), grape stalk and yohimbe bark (Fiol, et al., 2008), corn straw (Li, et al., 2024), nutmug fruit rind (Vaishna, et al., 2023), red brick waste (Mao, et al., 2024), fly ash waste (Elidrissi, et al., 2023), chitosan composite (Wang, et al., 2023) explored to withdraw Cr (VI) oxyanions from water.

Adsorption is a suitable way to remove these oxyanions from polluted water (Senthil and Santhil, 2021). These green waste materials consists ROH, RCOR, $RCONH_2$, groups (Peiravi-Rivash, 2023). These groups interact with Cr (VI) oxyanions which results in adsorption of oxyanions from water on their surface (Akula and Philip, 2023) changing the characteristics of adsorbing green waste.

Every year, large amounts of Chick-Pea pod husk is produced worldwide after the harvest of crop. This green waste is either used as food for cattle or thrown as waste. The utilization of this waste as adsorbent to remove chemical pollutants from drinking water can be a promising, cost-effective, and environment friendly technique. The objective of this work is, therefore, to utilize this green waste for removal of Cr (VI) oxyanions from polluted water. Further in this study, various parameters such as pH, shaking speed, shaking time, concentration by applying Batch mode and removed toxic chromium oxyanions from polluted water were optimized.

MATERIALS AND METHODS

Chick-Pea pod husk, a green waste material, has been selected to remove the contaminating agent i.e., oxyanions from water. The material was washed with deionized water, dried, powdered and characterized by U.V-Vis Spectrophotometer, FT-IR spectroscopy, scanning electron microscope analysis, and Brunauer-Emmett-Teller surface area analyzer afterward Batch-mode employed. The kinetics and isotherms were explored to achieve the mechanism of adsorption and the real applications of Chick-Pea pod husk.

Chemicals and Instrumentation

The chemicals and reagents used throughout experiments were pure and scientific or equivalent grade.

Chick-Pea pod husk, Deionized water, Filter papers, Conc. HCl, Conc. H_2SO_4, HNO_3, 2-Propanol, NaOH, $K_2Cr_2O_7$, CH_3COOH, CH_3COONa, 1,5-diphenylcarbazide, NH_4OH, NH_4Cl, Sodium EDTA, Methanol, were obtained from Sigma-Aldrich and Merck (Germany).

Characterization

Chick-Pea pod husk loaded with Cr (VI) oxyanions was characterized by FT-IR, SEM and energy dispersive X-ray spectroscopy.

Adsorption by Batch method

A weighed amount of Chick-Pea husk was equilibrated with the metal ion solution by variations in parameters like pH of solution, Chick-Pea husk dosage, Cr (VI) oxyanions volume, shaking speed, shaking time, concentration of Cr (VI) oxyanions that affect Cr (VI) oxyanions adsorption onto Chick-Pea husk.

Optimized Parameters

The parameters like pH, dosage of adsorbent, concentration, temperature, volume, agitation time, shaking speed were examined by complexing Cr (VI) oxyanions with 1,5-Diphenylcarbazide (D-P-C) using UV-Vis spectrophotometer Perkins Elmer (Uberlingen, Germany) for the analysis at 540 nm.

The FT-IR spectra of Chick-Pea husk was evaluated in the range of 4000 to 625 cm^{-1}. Thermo-electron Co. (Madison. USA) FT-IR model Nicolet-Avatar 330 in connection with Omnic-Software Version─7.0 with Zn-Se A.T.R cell used for recording the spectrum.

The surface morphology of Chick-pea husk was elaborated by Hitachi S-2300 SEM with 10 kV. The SEM was equipped with EDX, which elaborated the elemental composition of Chick-Pea husk and Cr (VI) oxyanions. The samples were carbon-coated using Edwards-Scancoat.

To maintain particular pH various buffer solutions, e.g. for pH 2, 0.1 M HCl / KCl; for pH 3 to pH 7, 0.1 M CH_3COOH / CH_3COONa and for pH 8 to pH 9, 0.1 M NH_4OH / NH_4Cl were used respectively.

RESULTS

FT-IR Spectrum of Chick-Pea Husk

Chick-Pea pod husk infrared spectrum was attained to explore the presence of functional groups in Chick-Pea pod husk. Peaks appeared were 3290.67, 2919.86, 1729.56, 1628.56, 1420.68, 1318.39, 1238.01 and 1030.58 cm^{-1} were O.H stretching, C.H stretching of

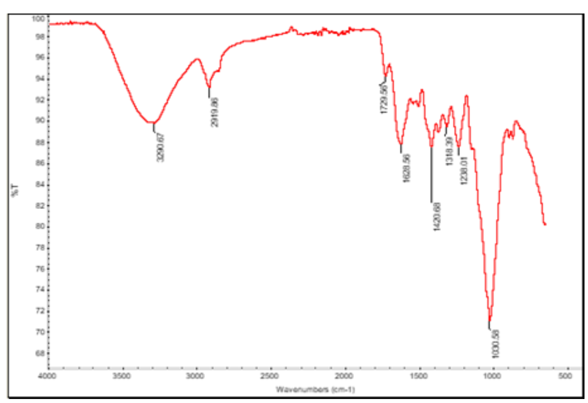


Figure 1. FT-IR spectra of Chick-Pea husk.

alkane, C.H and C=O stretch of CO.OH or esters, $CO.O^{-1}$ stretch, O.H bend, C-O stretch of RCOO.R/RO.R and N.H deformation of amines respectively were recorded in spectra of Chick-Pea husk as shown in Figure1.

SEM analysis of Chick-Pea husk

The surface morphology of Chick-Pea pod husk exhibits a micro-porosity at 1000x resolution while the images of Chick-Pea pod husk showed particle size of 10 micro-meters. SEM analysis showed the images of Chick-Pea pod husk before and after sorption of Chromium (VI) oxyanions. Figure 2 (A) before sorption and Figure 2 (B) after sorption of Chromium (VI) oxyanions.

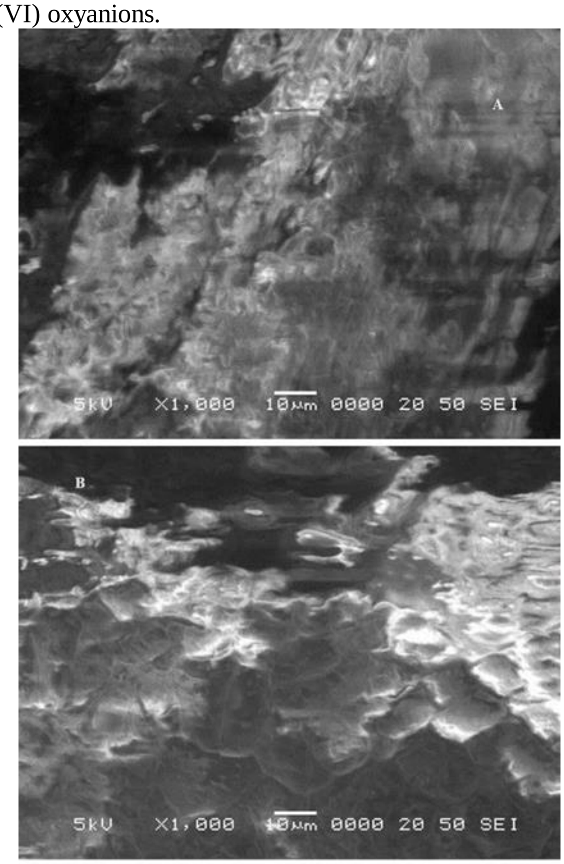


Figure 2. (A). SEM image of Chick-Pea husk before sorption of Cr (VI) oxyanions. (B) SEM image of Chick-Pea husk after sorption of Cr (VI) oxyanions.

EDX analysis of Chick-Pea Husk

Chick-Pea pod husk elemental composition was explored by E-D-X analysis. The E-D-X analysis of Chick-Pea pod husk as it is appeared in Figure 3 (A), elaborate the presence of different elements comprises maximum quantity of K. E-D-X spectrum of Cr (VI) oxyanions Chick-Pea pod husk, shown in Figure 3 (B) showed the sorption of Cr (VI) oxyanions onto the surface of the Chick-Pea pod husk.

Effect of pH

pH of Cr (VI) oxyanions ranges 1-9, while keeping all other parameters constant. The optimum adsorption was achieved at pH 2. The electrostatic attraction takes place between positively charged adsorbent surface and negatively charged Cr (VI) oxyanions which results maximum sorption at pH 2 (Figure 4).

Effect of Chick-Pea pod husk dosages

Chick-Pea husk dosage was 0.01-0.5g. The % adsorption increases upto 0.5 g because of surface area and accessibility enhanced. The amount of

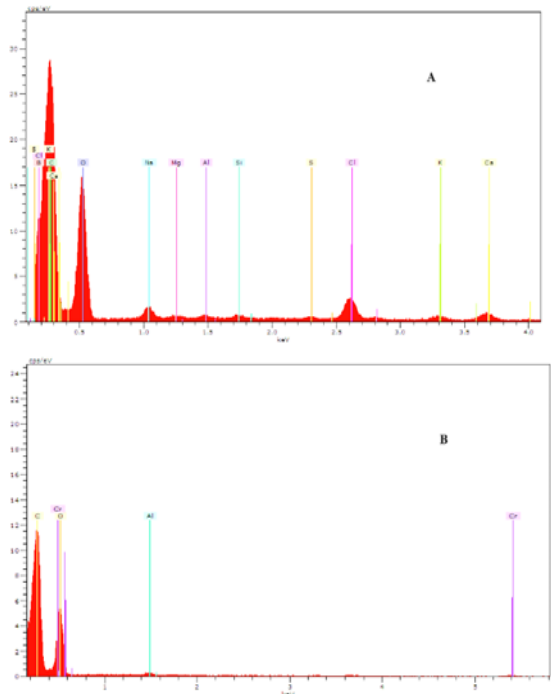


Figure 3. (A).Energy dispersive X-ray (EDX) spectra of Chick-Pea pod husk un-adsorbed. (B) Energy dispersive X-ray (EDX) spectra of Chick-Pea pod husk adsorbed with Cr (VI) oxyanions.

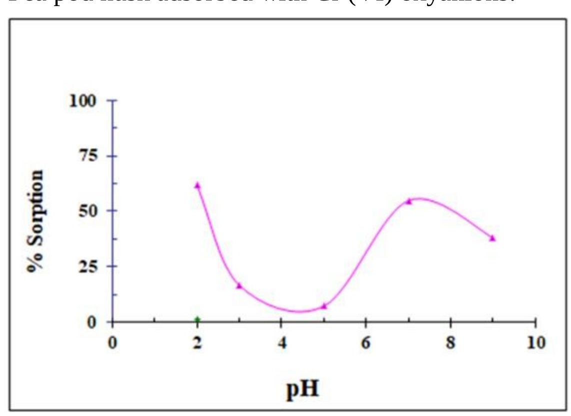


Figure 4. Effect of pH on Cr (VI) oxyanions sorption onto Chick-Pea pod husk

dosage is proportional to the chromium oxyanions solution (figure 5).

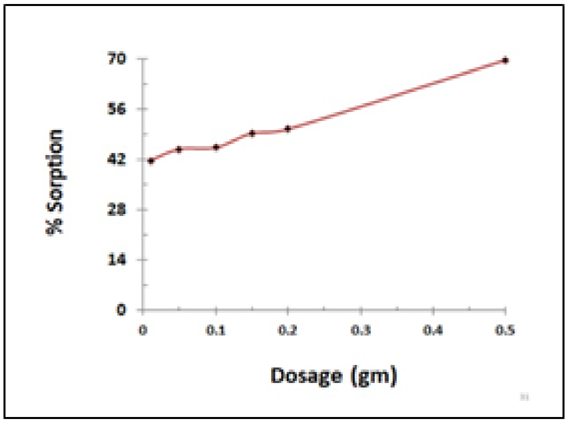


Figure 5. Effect of dosage on Cr (VI) oxyanions sorption onto Chick-Pea pod husk

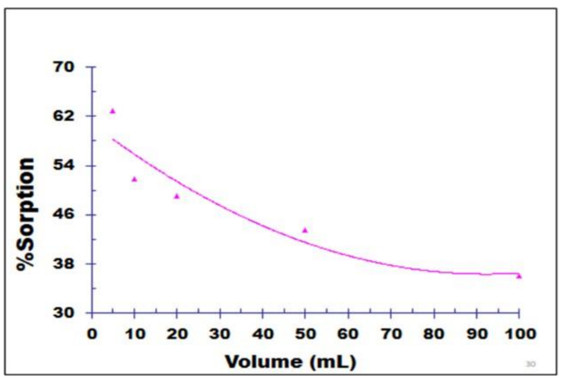


Figure 6. Effect of volume on Cr (VI) oxyanions sorption onto Chick-Pea pod husk

Effect of volume

Different oxyanions volumes 5 to 100 mL were taken. Highest uptake was observed at 5 mL (Figure 6).

KINETICS STUDIES OF ADSORPTION

Shaking time of oxyanions with Chick-Pea pod husk elaborate the % adsorption was rapid upto 30 min. After that no characteristic result was explored. Kinetics studies of adsorption were explored by equations:

Lagergren Equation

This identifies the adsorption-mechanism accumulated by kinetics model.

$\ln (Q_e - Q_t) = \ln q_e - kt$

Where q_e is the adsorbed-conc. of oxyanions by Chick-Pea Pod mol/g, Q_t is adsorbed-conc. at time (t) and (k) is Rate- constant. ($Q_e - Q_t$) VS t, elaborate the linearity accompanied (k) slope.

Figure 7.1 comprises a linearity with R^2 (Regression-Coefficient) of 0.998 and Plotted ($Q_e - Q_t$) VS t, display the linearity with k (slope) = 0.01 ± 0.000197 min^{-1}.

Morris Webber equation

$Q_t = R_d \sqrt{t}$

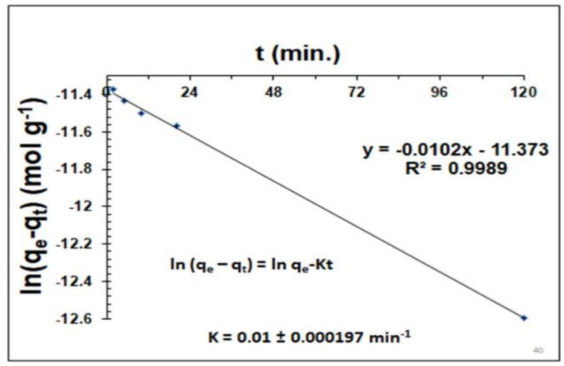
R^2 appeared 0.993 and the R_d was 1.47 ± 0.06 µ. mol. g^{-1}. $min^{-1/2}$ elaborated by slope of the plot Q_t VS $\sqrt{t}$ and Q_t was the conc. of oxyanions at $t.^{1/2}$ (time). Q_t VS $\sqrt{t}$ doesn't proceed from zero in Figure 7.2.

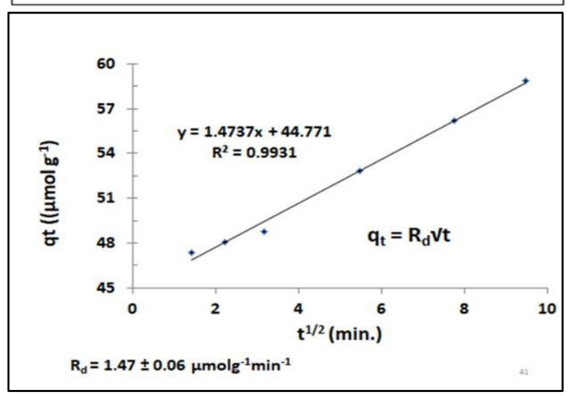
Reichenberg Equation

$Bt = -0.4977 \ln (1-F)$

$F_r = Q_t / Q_e$ & $\beta = \pi D_i / r^2$

Bt VS t slope is linear from 0-90 min. doesn't proceed





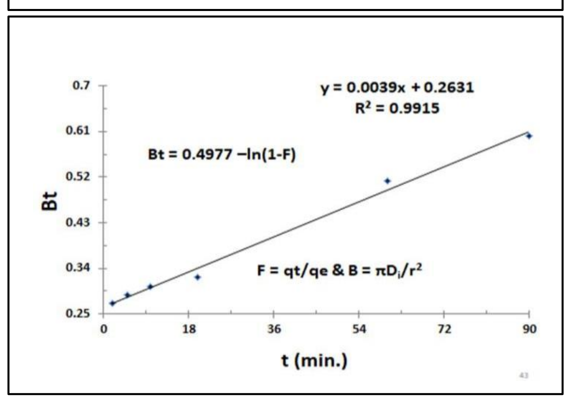


Figure 7.1 to 7.3. (Top) Lagergren plot for the sorption of Cr (VI) oxyanions, (middle) Morris- Webber plot for sorption of Cr (VI) oxyanions and (Bottom) Reichenberg plot for sorption of Cr (VI) oxyanions onto Chick-Pea pod husk

by zero indicates a thin-layer produced at adsorbent-plane. R^2 regression-coefficient appeared 0.991.

ADSORPTION ISOTHERMS

Adsorption-isotherms for oxyanions at different temperatures in the concentration of 0.5-1000 mg mol^{-1} were obtained. A decline in removal of Cr (VI) oxyanions with amplified temperature indicates exothermic reaction. Following isotherms were used:

Langmuir-Isotherm

Langmuir-isotherm evaluates the utmost adsorption-capacity through below eq:

$C_e/C_{ads} = 1 / Q.b + C_e / Q$

Plotting C_e / C_{ads} VS C_e evaluate the slope. A linear plot of Langmuir isotherm for Cr (VI) oxyanions appeared.

R_L elaborated by:

$R_L = 1 / (1+b.C_i)$

C_i = initial conc. of oxyanions

& b = Langmuir-constant.

R_L (dimensionless constant) appeared 0.99-0.37 for oxyanions elaborate suitable adsorption. Plot of C_e /C_{ads} VS C_e elaborate 1 / Q. Value of b & Q explored by slope and intercept. Q or K_L (Langmuir-adsorption capacity), was explored 6.5 ± 0.3 mmol g^{-1} and b (adsorption-equilibrium constant) explored 0.005674 ± 0.000118 Lg^{-1}.

Freundlich-Isotherm

Freundlich-isotherm plotted log C_{ads} VS log C_e appeared in Figure 8.2 and R^2 (Regression-Coefficient) explored 0.991 which elaborate that the experimental results suitability to Freundlich-isotherm. K_f (Freundlich-adsorption capacity) explored 0.48 ± 0.013 $mmolg^{-1}$ and 1/n (Intensity-adsorption) was explored 0.91 ± 0.03.

The Freundlich-isotherm accumulated by: Log C_{ads} = log K_f+ 1/n log C_e

Dubinin-Radushkevich

D-R isotherm plotted ln K_c VS ε (Polanyi-Potential) in Figure 8.3 and R^2 (Regression-Coefficient) was explored 0.99.

D-R elaborates the porosity and free-energy of adsorption.

The D-R accumulated by:

$\ln C_{ads} = \ln X_m – \beta \varepsilon^2$

C_{ads} is the quantity of oxyanions-adsorbed per unit mass, X_m and β are D-R constant.

ε accumulated:

$\varepsilon = R.T \ln (1 +1/C_e)$

C_e = equilibrium conc. while R = gas-const. and T = temp. (K). β = slope and ln X_m / K_{D-R} = 0.1959 ± 0.02954 mmol g^{-1} as intercept. E from slope accumulated can as:

$E = 1 /\sqrt{-2 \beta}$

E = 7.84 ± -0.009 kJ mol^{-1} gives the idea that adsorption-mechanism is physical adsorption (1-9 kJ mol^{-1}).

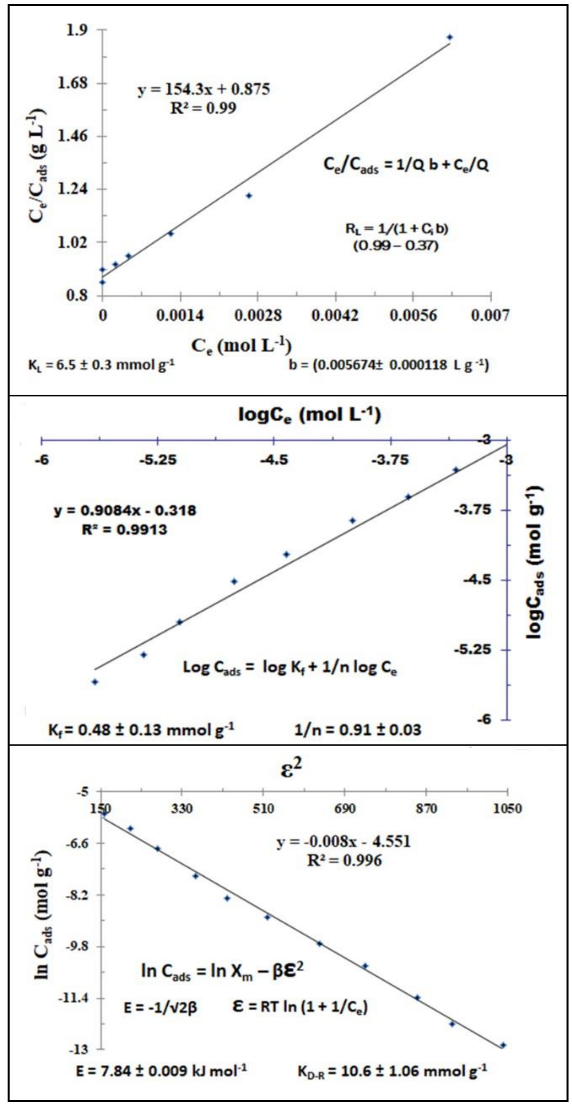


Figure 8.1to 8.3. Langmuir isotherm (Top), Freundlich isotherm (Middle) and D-R isotherm (Bottom) for sorption of Cr (VI) oxyanions onto Chick-Pea husk.

THERMODYNAMICS

Temperature effect on the Chick-Pea husk oxyanions-adsorption explored 10-50 °C. The slope accumulated by K_c VS 1/T was linear as shown in Figure 9.

Thermodynamics parameters ΔG_o (Gibb's free energy), ΔS_o (entropy) and ΔH_o (enthalpy). Van't-Hoff equations as below:

$\Delta G_o = -RT \ln K_c$

$\ln K_c = \Delta S_o / R - \Delta H_o / RT$

Slope and intercept of plots accumulated ΔG_o, ΔS_o and ΔH_o for oxyanions. The -ΔS elaborate the spontaneous

& randomness of oxyanions and -ΔH elaborate exothermic nature.

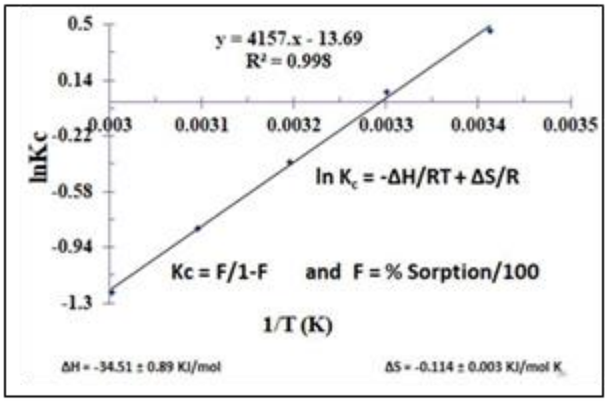


Figure 9. Effect of temperature on sorption of Cr ***(VI) Oxyanions onto Chick-Pea husk.***

Desorption

The NaOH of various concentration and volumes used for elution of Cr (VI) oxyanions. Maximum elution took place by 1M 5mL NaOH as shown in Table 1.

Table 1. The results of desorption of Cr (VI) oxyanions by NaOH

CONCENTRATION	VOL	%RECOVERY
0.5M	5mL	58.68
0.5M	10mL	36.11
1M	5mL	97.53

Applications of Developed method

The application of Chick-Pea pod husk was tested to remove the Cr (VI) oxyanions from samples of water collected from Badin, University of Sindh Jamshoro, Ghooni Matli, Matli channel & Matli-groundwater. The samples of water were filtered and digested with 0.1M HNO_3 at optimum conditions and experiment was carried out using Batch mode. Percent **r**emoval of Cr (VI) oxyanions by Batch-mode under optimum parameters from these water samples is shown in Table 2.

DISCUSSION

Removal of Cr (VI) oxyanions from contaminated water using Chick-Pea pod husk was studied. Cr (VI) oxyanions are poly atomic ions involving one or more oxygen atoms plus another metal or non-metal atom (Chen K, et al., 2024). Cr (VI) oxyanions commonly have the negative charges such as chromite, chromate (Fiol, et al., 2008). Drinking water is polluted by Cr (VI) oxyanions due through disposal of various industrial waste products (Shah, et al., 2023). Adsorption by applying Batch method removed Cr (VI) oxyanions from polluted water (Senthil and Santhil, 2021).

Chick-Pea pod husk worldwide cultivated as green waste material was utilized as adsorbent to remove these toxic Cr (VI) oxyanions from polluted water. These green waste materials consists various functional groups such as ROH, RCOR, $RCONH_2$ (Peiravi-Rivash, 2023). These groups interact with Cr (VI) oxyanions which results removal of these Cr (VI) oxyanions from water (Akula & Philip, 2023).

Various parameters optimized such as pH, shaking speed, shaking time, concentration by applying Batch mode and removed these toxic chromium oxyanions from environmental water (Wang, et al., 2023). The maximum uptake was attained at pH 2 due to the electrostatic attraction between positively charged adsorbent surface and negatively charged Cr (VI) oxyanions which results sorption more efficient at pH 2 while other pH ranges are not suitable for optimum adsorption due to basic medium. The adsorption increases from 0.01 to 0.5 g because of more surface area. Sorption is proportional to the surface area of adsorbent. Highest adsorption was observed at 5 mL. Cr (VI) oxyanions volume increased the amount of chromium ions which results the decreases in adsorption. Shaking time of Cr (VI) oxyanions with Chick-Pea pod husk elaborates the adsorption was rapid upto 30 min. After that no characteristic result was explored. Kinetics studies of adsorption were explored by Lagergren, Morris-Webber and Reichenberg equations. The adsorption data follows Langmuir, Freundlich and D-R isotherm. Adsorption-isotherms for Cr (VI) oxyanions at different temperatures in the concentration from 0.5 to 1000 mg mol^{-1} were obtained. The -ΔS elaborates the spontaneous & randomness of Cr (VI) oxyanions and +ΔS elaborates endothermic nature. A decline in removal of Cr (VI) oxyanions with amplified temperature indicates exothermic reaction and negative entropy elaborates the spontaneous nature of reaction. Various concentrations and volumes of sodium hydroxide were used for desorption of Cr (VI) oxyanions and maximum desorption takes place by 1M 5mL NaOH while higher concentrations and volumes decreased desorption because of increasing the basic medium which results less desorption (Yuan and Lu, 2024).

Table 2. Determination of Cr (VI) oxyanions from polluted water samples

S.No.	Sample collected from	Cr (VI) oxyanions found in% Removal (μg mL^{-1})
1	Badin channel	0.318±0.0006290
2	University of Sindh Jamshoro	0.454±0.0000280
3	Ghooni Matli	0.181±0.00094100
4	Matli Channel	0.181±0.00029100
5	Matli-Underground	0.090±0.00166100

CONCLUSION

The Chick-Pea pod husk is an effective adsorbent to remove Cr (VI) oxyanions from polluted water. The

Chick-Pea pod husk was characterized by FTIR, SEM-EDX, BET analysis. BET surface area analyzer was used and found to be $1.661 m^2 g^{-1}$. Different parameters were optimized using U.V Spectrophotometer, FT-IR, SEM, EDS, BET etc. FT-IR of Chick-Pea pod husk elaborates functional groups while SEM micro-porosity of Chick-Pea Pod & EDS analysis confirmed Cr (VI) oxyanions adsorbed by Chick-Pea pod husk from water samples. Kinetics and thermodynamics data obtained at the optimum conditions for Cr (VI) oxyanions adsorption by Chick-Pea pod husk follows Langmuir, Freundlich and D-R isotherm. Enthalpy was found to be -34.51 ± 0.89 kJ mol^{-1} and Entropy -0.114 ± -0.003kJ mol^{-1} expressed the order and a uniform layer formation. Energy of adsorption is 7.84 ± 0.009 kJ mol^{-1} indicates that adsorption mechanism is physical adsorption (1 - 9 kJ mol^{-1}). R_L ranges 0.37 - 0.99 for oxyanions elaborate favorable adsorption.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

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