Removal of Metal Ions via Adsorption Technique using Low-Cost Adsorbent

Dattatraya Jirekar[1], Pramila Ghumare[2]

[1, 2]Department of Chemistry, Anandrao Dhonde Alias Babaji College, Kada. India
Email: *dattajirekar1[at]gmail.com*

Abstract: ***This study investigates the adsorption capacity of red gram seed husk (RGSH) as a potential bio-adsorbent for removing chromium, copper, and nickel metal ions from aqueous solutions. The adsorption behavior of these metal ions was investigated using batch experiments under varying conditions. The results showed that the adsorption of Cr, Cu, and Ni onto RGSH followed a pseudo-second-order kinetic model, suggesting chemical adsorption as the rate-controlling step. The Langmuir and Freundlich adsorption isotherm models fitted well for Cr and Ni metal ions, indicating surface heterogeneity. The adsorption capacity was pH-dependent, with optimum removal efficiencies observed at slightly acidic to neutral pH ranges. The presence of competing ions had a moderate influence on metal removal. The study concludes that RGSH is a promising and cost-effective bio-adsorbent for heavy metal remediation in wastewater treatment systems. Further investigations into regeneration and reusability are recommended to enhance its practical application potential.***

Keywords: Adsorption Capacity, Bio-adsorbent, Kinetic Model, Isotherm Model, Thermodynamic Parameters

1. Introduction

Heavy metal pollution, a global issue, is a significant concern due to its harmful effects on human health and the environment. Industries like mining, metallurgy, and manufacturing release toxic heavy metals into aquatic systems, contaminating water sources and potentially entering the food chain [1]. Conventional methods for heavy metal removal from industrial wastewater, like precipitation, coagulation, ion exchange, electrochemical treatment, membrane filtration, and chemical flocculation, are costly, generate sludge, and may not be environmentally friendly [2]. Conventional methods for heavy metal removal often have limitations like high cost, secondary pollutants, and low efficiency. The growing interest is in eco-friendly alternatives like bio-adsorbents derived from agricultural wastes like residues, plant materials, and fruit peels [3]. Seed husks, an agricultural by-product, have shown promising potential as a bio-adsorbent due to their abundant availability and effective metal ion adsorption ability [4]. Adsorption of heavy metal ions from water using natural and waste-derived materials, particularly agricultural waste, is promising due to their abundance, low cost, renewability, and biodegradability. A literature survey shows that so many low-cost alternative adsorbents have been proposed in recent decades, which include bamboo [5], oak wood biochar [6], pine wood, pig manure and cardboard [7], cedrelaodrorata seeds [8], green gram [9]; neem leaves [10], gram seed husk, [11], cucumber peel [12], palm kernel Wrap [13], Cajanuscajan, commonly known as red gram, is a widely cultivated legume crop in many tropical and subtropical regions.

Red gram seed husk's unique properties, including hydroxyl, carboxyl, and amine groups, make it an ideal candidate for metal ion removal from aqueous solutions through adsorption [14]. Numerous studies have explored the potential of bio-adsorbents, including agricultural waste-derived materials, for the removal of heavy metals [15, 16]. The adsorption capacity of Cr, Cu, and Ni ions in industrial effluents requires a comparative evaluation of red gram seed husk (RGSH) [17].

This study aims to analyze the adsorption capacity of red gram seed husk (RGSH) for removing Cr, Cu, and Ni from aqueous solutions. Batch experiments will be conducted under various conditions, and data will be analyzed using kinetic and isotherm models. The thermodynamic parameters will be evaluated to assess the process's feasibility and spontaneity. This study investigates the feasibility and practical applicability of red gram seed husk (RGSH) as a bio-adsorbent for heavy metal removal in wastewater treatment systems.

2. Materials and Methods

2.1 Preparation of adsorbent

Red Gram seed husk effectively removes heavy metal ions from aqueous solutions through a process that involves several steps to optimize its adsorption capacity. To initiate the process, collect fresh red gram seeds from a trustworthy source, ensuring they are free from contaminants and thoroughly cleaned to prevent interference with the adsorption process. After cleaning, seeds are separated from their husks and dried to reduce moisture content. Proper drying in a well-ventilated area or a low-temperature drying oven ensures better adsorption efficiency and prevents microorganism growth during storage. Dried husks can be reduced into smaller particles by grinding or cutting them to increase surface area and improve metal ion contact, ensuring uniform adsorption behavior.

2.2 Preparation of adsorbate

To prepare stock solutions, obtain A. R. grade chemicals from M/S S. D. Fine Chemicals Ltd, Mumbai, such as Potassium dichromate ($K_2Cr_2O_7$), Copper sulfate ($CuSO_4.5H_2O$), and Ammonium nickel sulphate [$NiSO_4(NH_4)_2.6H_2O$]. Accurately weigh each metal salt to achieve the desired concentration. To prepare a stock solution, accurately weigh each metal salt and determine the desired concentration based on the experimental design and heavy metal concentration range. Dissolve the weighed salt in deionized water and stir thoroughly. To prepare lower concentration working solutions, dilute stock solutions with deionized water and use volumetric flasks or graduated cylinders to measure the appropriate volumes of stock solution and deionized water. Adsorbate solution pH significantly impacts heavy metal adsorption behavior. Adjusting pH levels using acid or base solutions and using a pH meter allows accurate monitoring and control of solutions' adsorption effects. Store adsorbate solutions in clean, tightly sealed containers to prevent contamination and evaporation, and use them within a short period to avoid potential metal concentration changes. Adsorption experiments require specific concentrations and pH levels based on experimental goals, adsorbent properties, and initial heavy metal concentration in wastewater or aqueous solution. High purity metal salts are crucial to prevent contaminants from affecting adsorption experiments.

3. Batch Experiments

Batch experiments are widely used in adsorption studies to study the adsorption behavior of an adsorbent towards a specific adsorbate in a controlled laboratory setting, enabling researchers to determine the adsorption capacity, kinetics, and equilibrium characteristics. Batch adsorption experiments were conducted to study the kinetics, equilibrium, and thermodynamics of Cr, Cu, and Ni metal ions on red gram seed husk in 250 ml Erlenmeyer flasks. A RGSH bio-adsorbent was added to a flask, shaken for various times, and centrifuged for 10 minutes. The pH was adjusted with NaOH and HCl solutions, and the concentration of Cr, Cu, and Ni metal ions was determined using a UV-Vis spectrophotometer at different wavelengths. Competitive adsorption studies involve simultaneous introduction of multiple adsorbates into an adsorbent, resulting in different adsorption behaviors compared to single-component systems. The experiments simulate real-world wastewater scenarios, allowing evaluation of the adsorbent's selectivity for specific ions in wastewater containing multiple contaminants.

The concentration of adsorbed metal ion (mg/g) was determined using Vanderborght and Van Griekenm's formulas, and the kinetic adsorption parameters were also calculated.

$$q = \frac{V(Co-Ct)}{M} \quad (1)$$

Where q is the concentration of metal ion adsorbed from the solution (mg/g), Co is the concentration of RGSH before adsorption (mg/L) and Ct is the concentration of RGSH after adsorption. V is the volume of RGSH solutions (L) and M is the mass of RGSH
adsorbent (g).

The adsorption percentage of metal ion was calculated according to the following equation;

$$\text{Percentage adsorption} = \frac{(C_0 - C_e)}{C_0} * 100 \quad (2)$$

Where, C_0 and C_e are the initial and equilibrium concentrations respectively.

4. Results and Discussion

4.1 Effect of contact time

The contact time, the duration between the adsorbent and adsorbate, significantly influences the efficiency of the adsorption process, as illustrated in Fig. 1.

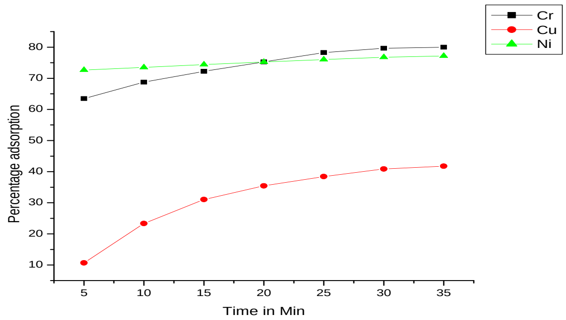

Fig.1: Effect of contact time of Cr, Cu and Ni metal ions on RGSH adsorbent.

The adsorption percentage of Cr, Cu, and Ni metal ions increased with longer contact time due to the larger surface area of the adsorbent.The study found that the maximum adsorption efficiency of Cr, Cu, and Ni metal ions was 79.98, 41.755, and 77.15% after 35 minutes.The study revealed a rapid adsorption process with an optimal contact time of 35 minutes for maximum metal ion removal, but slowed down after 35 minutes due to adsorbent surface saturation.In conclusion, it was found that the optimal contact time for the adsorption of Cr, Cu, and Ni metal ions on RGSH bio-adsorbent from an aqueous solution is 35 minutes, with the adsorption process slowing down after this time due to surface saturation.

4.2 Effect of adsorbent dose

The adsorption efficiency of Cr, Cu, and Ni metal ions from aqueous solutions using bio-adsorbents like RGSH is significantly influenced by the dosage of the adsorbent.The study investigated the adsorption of metal ions using RGSH in varying dosages, maintaining Cr, Cu, and Ni concentrations at 20 mg/L and pH 7.0 as shown in Fig. 2.

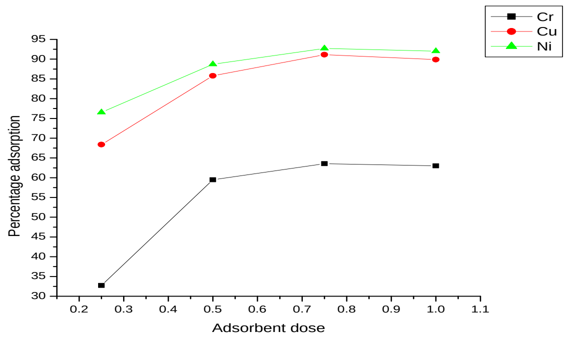

Fig. 2: Effect of adsorption dose on adsorption of Cr, Cu and Ni ions on RGSH adsorbent.

The adsorption capacity of RGSH for Cr, Cu, and Ni metal ions increases with increasing adsorbent dosage, but decreases beyond a certain point due to fewer available sites at lower doses. At higher doses, adsorption sites become

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saturated, and excess adsorbent may hinder the availability of these metal ions to active sites.

4.3 Effect of pH

The pH of a solution significantly impacts the adsorption of Cr, Cu, and Ni metal ions on RGSH adsorbent. The surface charge of the adsorbent and adsorbate affects electrostatic attraction, with the effect studied between 2 and 11, as illustrated in Fig. 3.

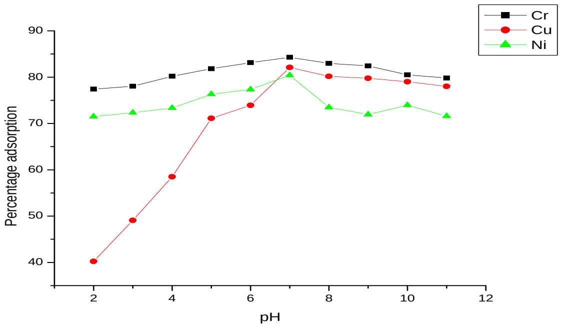

Fig. 3: Effect of pH on adsorption of Cr, Cu and Ni ions solution on RGSH adsorbent.

The study found that the adsorption percentage of Cr, Cu, and Ni metal ions significantly increased with an increase in pH from 6-7, consistent with previous findings.The adsorption capacity of RGSH for Cr, Cu, and Ni metal ions is pH-dependent, with maximum adsorption occurring within a specific pH range.

4.4 Effect of Salt

The study examined the impact of salt (KCl) on the adsorption of Cr, Cu, and Ni metal ions, with the results presented in Fig. 4.

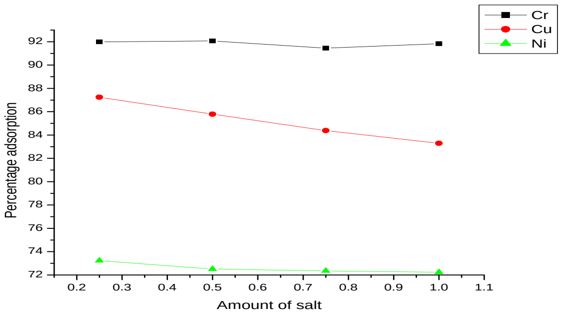

Fig. 4: Effect of salt on adsorption of Cr, Cu and Ni ions on RGSH adsorbent.

The adsorption of Cr, Cu, and Ni metal ions significantly decreased, with the maximum percentages of these ions being 91.83, 83.28, and 72.24, respectively.The negative impact of ionic strength on Cr, Cu, and Ni adsorption can be attributed to the competition between electrolyte anions and metal ions for surface sites [18, 20].K+ ions saturate material adsorptive surfaces, reducing metal ion uptake. Increased KCl concentration increases the thickness of the electrical double layer, inhibiting metal ion mobility and adsorption.

4.5 Effect of temperature

Temperature impacts the adsorption process by changing the surface properties of the adsorbent and the chemical nature of the adsorbate. The study examined the impact of temperature on various temperatures between 304.2 and 324.2 K, as depicted in Fig. 5.

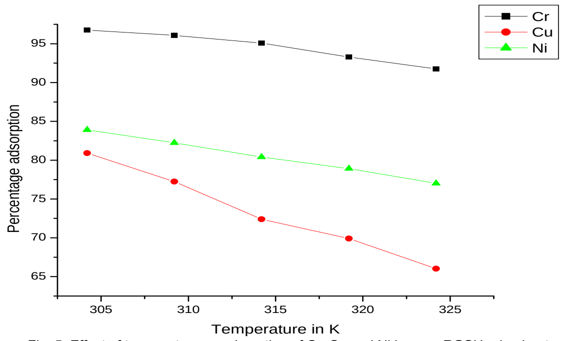

Fig. 5: Effect of temperature on adsorption of Cr, Cu and Ni ions on RGSH adsorbent.

The maximum adsorption capacity of Cr, Ni, and Cu metal ions on RGSH was found to be 91.75, 81.03, and 77.01 mg/g at 304.2 K.Graphical studies reveal a decrease in RGSH adsorption capacity with increasing temperature, indicating an exothermic and nonspontaneous process.

5. Thermodynamic Study

This study examined the thermodynamic impact of temperature on the equilibrium capacity of the RGSH for Cr, Cu, and Ni adsorbate, aiming to determine if the process is spontaneous.

The change in Gibb's free energy (ΔG°), enthalpy (ΔH°), and entropy (ΔS°) for the adsorption were calculated using the following equations:

$$\Delta G° = -RT \ln Kc \quad (3)$$

Where, R is gas constant and Kc is the equilibrium constant and T is the temperature in K.

According to Van't Hoff equation,

$$\log K_c = \frac{\Delta S°}{2.303\,R} - \frac{\Delta H°}{2.303\,RT} \quad (4)$$

The linear plots of ΔG° versus temperature, T, were evaluated, and the slope and intercept of the plots were used to determine ΔH° and ΔS°, as depicted in Table. 1.

Table 1: Thermodynamic parameters for the adsorption of Cr, Cu and Ni onto RGSH

Thermodynamic parameters of Cr, Cu and Ni onto RGSH									
Temp (K)	(−ΔG°) KJ/mole			(−ΔH°) KJ/mole			(−ΔS°) J/mole.k		
	Cr	Cu	Ni	Cr	Cu	Ni	Cr	Cu	Ni
306.2	-8.682	-4.168	-3.620						
311.2	-8.145	-3.940	-3.555						
316.2	-7.608	-3.713	-3.491	41.606	17.999	7.656	107.42	45.469	13.223
321.2	-7.071	-3.486	-3.426						
326.2	-6.534	-3.258	-3.362						

The negative value of ΔH° for RGSH indicates that adsorption is exothermic. The study found that physical adsorption was the predominant mechanism in the adsorption process for Cr, Cu, and Ni metal ions, with ΔG°values < -10 KJ/mole.Gibb's free energy indicates the spontaneity of the adsorption process, with a negative value indicating a more energetically favorable process.The negative value of ΔG° indicates favorable and spontaneous adsorption [21, 22].The negative values of ΔS° and ΔH°indicate decreased disorder and randomness at the solid solution interface with exothermic adsorption [23, 24].

6. Adsorption Isotherm

The linear form of Langmuir isotherm equation is

$$\frac{C_e}{q_e} = \left(\frac{1}{Q_0}\right) C_e + \frac{1}{bQ_0} \quad (5)$$

The value of ‘Q_0’, and ‘b’ are presented in Table 2 (a).

Table 2 (a): Langmuir parameter values of RGSH with Cr, Cu and Ni metal ions solution

Concentration of Cr, Cu and Ni metal ionssolution (ppm)	*Langmuir constants*											
	Q_0 (mg/gm.)			*b*10^{-3} (L/gm.)*			*R_L*			*R^2*		
	Cr	Cu	Ni	Cr	Cu	Ni	Cr	Cu	Ni	Cr	Cu	Ni
20	71.43	909.1	326.4	1.87	2.12	0.023	0.999	0.999	0.997	0.998	0.970	0.998

The Langmuir isotherm for Cr, Cu, and Ni metal ions has a ‘Q_0’ value of 71.43, 909.1, and 326.4 mg/g, respectively.The Langmuir isotherm is effective in explaining the adsorption of Cr and Ni metal ions on RGSH, as indicated by their R^2 values of 0.998, 0.970, and 0.998 mg/g respectively.The efficiency of RGSH, as indicated by the ‘Q_0’, was found to be lower due to the competition between Cr, Cu, and Ni metal ions.

The Freundlich isotherm was utilized to study the potential multilayer adsorption and non-linear energy distribution of desorption sites.

$$\log\frac{x}{m} = \frac{1}{n}\log C_e + \log K_f \quad (6)$$

The intercept value (K_f) and slope nTable 2 (b) were determined from the linear plots of Freundlich isotherm at 306.2.

Table 2 (b): Freundlich parameter values of RGSH with Cr, Cu and Ni metal ions solution

Concentration of Cr, Cu and Ni metal ionssolution (ppm)	***Freundlich constants***								
	n			***K_f (mg/gm.(L/gm.))$^{1/n}$***			***R^2***		
	Cr	Cu	Ni	Cr	Cu	Ni	Cr	Cu	Ni
20	1.013	1.111	1.243	14.897	13.621	17.9362	0.999	0.963	0.997

The Freundlich isotherm is effective in explaining the adsorption of Cr, Cu, and Ni metal ions on RGSH, with R^2 values of 0.999, 0.963, and 0.997 mg/g, respectively.The 1/n value of Cr, Cu, and Ni metal ions was found to be 0.987, 0.900, and 0.804 on RGSH. The Freundlich isotherm calculated K_f values for Cr, Cu, and Ni metal ions on RGSH, ranging from 14.897 to 17.936.The 1/n value, ranging from 0 to 1, indicates surface heterogeneity or adsorption intensity, which becomes more heterogeneous as its value approaches zero [25].The Langmuir adsorption isotherm, with a value below 1 indicating normal adsorption, and above 1 indicating cooperative adsorption, is a crucial factor in determining adsorption processes [26, 27].

7. Adsorption Kinetics

The study analyzed the adsorption of Cr, Cu, and Ni metal ions in RGSH using experimental data fitted to various kinetic models, including pseudo-first-order [28], pseudo second-order [29], and intra-particle diffusion [30].

The rate constant of adsorption is determined from pseudo first-order equation given by Lagergren, which is expressed as;

$$\log(q_e - q_t) = \log q_e - \frac{K_1}{2.303}t \quad (7)$$

whereq_e and $q_t$are the amounts of Cr, Cu and Ni metal ions adsorbed (mg/g) at equilibrium and at time t (min), respectively, and $K_1$is the rate constant adsorption (min^{-1}). Values of K_1 and $q_e$calculable from slope and the intercept of the plot of log $(q_e - q_t)$ versus t (not shown) at different concentrations are given in Table 3 (a).

Table 3 (a): Pseudo-First order kinetic parameter values of RGSH with Cr, Cu and Ni metal ions solution.

Concentration of Cr, Cu and Ni metal ionssolution (ppm)	*Pseudo-First order*								
	*K_1 (min^{-1})*10^{-3}*			*q_e (mg/gm)*			*R^2*		
	Cr	Cu	Ni	Cr	Cu	Ni	Cr	Cu	Ni
20	3.394	7.979	41.753	286.98	242.82	121.46	0.984	0.931	0.921

The pseudo second-order equation based on equilibrium adsorption can be expressed as;

$$\frac{t}{q_t} = \frac{1}{K_2\, q_e^2} + \frac{1}{q_e}t \quad (8)$$

whereK_2 (g/mg min) is the adsorption rate constant of pseudo second-order adsorption rate. The value of $q_e$and K_2 can be obtained from the slope and intercept of the plot of $\frac{t}{q_t}$ versus t respectively.

Table 3 (b): Pseudo-Second order kinetic parameter values of RGSH with Cr, Cu and Ni metal ions solution.

Concentration of Cr, Cu and Ni metal ionssolution (ppm)	*Pseudo-second order*								
	K_2 (gm/mg. min) 10^{-3}*			*q_e (mg/gm)*			*R^2*		
	Cr	Cu	Ni	Cr	Cu	Ni	Cr	Cu	Ni
20	0.695	0.133	0.556	312.50	708.48	154.46	0.995	0.999	0.999

The adsorption of Cr, Cu, and Ni metal ions on RGSH adsorbent was better represented by pseudo second order kinetics, with R^2values of 0.995, 0.999, and 0.999, respectively, was indicating that the adsorption system belongs to the second order kinetic model.

8. Conclusion

The study investigated the adsorption of metal ions like Cr, Cu, and Ni using low-cost bio-waste adsorbents, red gram seed husk and concluded with the following conclusions

1) The adsorption percentage of Cr, Cu, and Ni metal ions on RGSH increases with adsorbent dose, contact time, and temperature, but decreases with temperature and salt.
2) The study revealed that the pH of dyes significantly impacts the adsorption of Cr, Cu, and Ni metal ions.
3) The negative value of ΔG^0 confirms the reaction's feasibility and spontaneous nature of adsorption.The negative values of ΔS^0 and ΔH^0 indicate decreased disorder and randomness at the solid solution interface with exothermic adsorption.
4) The Langmuir and Freundlich adsorption isotherm model effectively fits the RGSH adsorbent for Cr and Ni metal ions more than Cu metal ions.
5) The discussion also explored the effectiveness of pseudo-first-order kinetic and pseudo-second-order kinetic models for the adsorption of Cr, Cu, and Ni metal ions on RGSH.The kinetic modeling study indicates that the experimental data aligns with a pseudo-second-order model, indicating a chemisorption process.
6) The separation factor, R_L, showed that Cr, Cu, and Ni metal ions showed favorable adsorption.

Since, red gram seed husk, a cheap, locally available, and renewable adsorbent, is abundant and readily available for the removal of Cr, Cu, and Ni metal ions from aqueous solutions.

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