



## RESEARCH ARTICLE

# Adsorptive remediation of hexavalent chromium using agro-waste rice husk: Optimization of process parameters and functional groups characterization using FTIR analysis

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## Abstract

Hexavalent chromium [Cr (VI)] is considered as a toxic heavy metal in water bodies, posing global environmental and health risks. It has various detrimental impacts on the environment and human beings. The remediation of such toxicants from polluted sites has become essential for the good health of our ecosystem. Adsorption is a sustainable and eco-friendly solution for the efficient removal of such toxic heavy metals. In this study, the potential of an agro-waste material like rice husk (adsorbent) was investigated for remediation of the Cr(VI), and various parameters affecting the adsorption process were optimized to obtain the optimal conditions for effective remediation. By utilizing rice husk, the maximum 96.5% of Cr(VI) remediation efficiency was observed at an initial Cr(VI) level of 100 mg/L, pH 2, temperature 40°C with an adsorbent dosage of 26 g/L. The fourier transform infrared (FTIR) spectroscopy of rice husk with and without Cr (VI) indicated the functional group involvement in the metal adsorption. This study highlights the potential of agricultural waste, rice husk as an effective adsorbent material that may possibly employed in the remediation of Cr (VI) at a larger level.

**Keywords:** Adsorption, Rice husk, Hexavalent chromium, Agro-waste, FTIR, Aquatic pollution.

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## Introduction

Heavy metal like chromium is utilized as raw materials for various industries such as electroplating, metal processing, metallurgy and tanning industries (Gan *et al.*, 2019). Chromium is present mainly in two oxidation forms, Cr(VI) and Cr (III). While, Cr (III) is less hazardous and functions as an essential trace element, and Cr (VI), is more toxic with more solubility in water as compared to Cr (III). Researchers reported that Cr (VI) is more toxic compared to Cr (III) (Garg *et al.*, 2012; Levankumar *et al.*, 2009; Monnot *et al.*, 2014). Also, Cr(VI) is recognized as a carcinogenic and mutagenic chemical (Garg *et al.*, 2012; Nishioka, 1975; Pellerin and Booker, 2000; Venitt and Levy, 1974). Several physical and chemical strategies have been implemented to manage the pollution of Cr (VI) from industrial discharge and wastewater (Aman *et al.*, 2008; Dave *et al.*, 2012; Garg *et al.*, 2012). Nevertheless, these traditional methods are costly and unsustainable due to high capital investment and requirement of energy (Elmolla *et al.*, 2016; Chakraborty *et al.*, 2020). Therefore, due to the simplicity, effectiveness, cost-efficiency and flexibility of the adsorption method, it is becoming a more prevalent remediation approach (Bibi *et al.*, 2018; Satyam *et al.*, 2024).

Different types of waste biomass can be applied for effective Cr (VI) remediation through the adsorption process (Tripathi *et al.*, 2024). Due to the easy and plentiful availability of different agricultural vegetables and crops, a range of agro-waste biomass, such as vegetables and fruits, can be utilized for adsorption purposes (Lu *et al.*, 2021; Zaidi *et al.*, 2023). Various agricultural waste biomass (as adsorbents) for Cr (VI) remediation such as rice husk (Khalil *et al.*, 2020, 2021), mango kernel (Gning *et al.*, 2024), mosambi peel (Mondal *et al.*, 2019), maize cob (Kant *et al.*, 2024), *Moringa stenopetala* seed (MSSP) and banana peel (Badessa *et al.*, 2020; Hernandez and Romero, 2020), raw spent coffee waste (Loulidi *et al.*, 2021), *Heinsia crinite* seed coat (Dawodu *et al.*, 2020).

Although, there are multiple researches about the effective removal of Cr (VI) by utilizing rice husk or any agro-waste, yet, more studies are needed to understand the optimization of various parameters required for the efficient removal of Cr (VI) from contaminated water. More in-depth understanding and research is required for the application of this approach in practical settings. This study was focused on the Cr (VI) remediation by utilizing a sustainable agricultural waste material biomass (rice husk as an adsorbent), and optimization of different process factors like pH, dosage of adsorbent, temperature, initial metal level to understand the effect of various factors on the removal of Cr (VI) by rice husk. Finally, the FTIR analysis of rice husk was conducted for the characterization of possible functional groups involvement in the adsorption process.

## Materials and Methods

### Chemicals

The desired pH was maintained by the addition of concentrated HCl (0.1N) and NaOH (0.1N). The DPC (1,5-diphenyl carbazide) reagent was prepared for the analysis of Cr (VI).

### Adsorbent

The rice husk was used as an adsorbent for the experiment and was collected and washed with distilled water to remove unwanted particles. Further, it was sun-dried for 2 days and oven-dried at 50°C for 3 days. The dried rice husk was grounded thoroughly in an electric grinder and sieved to obtain powdered rice husk utilized as adsorbent the study. The biomass was prepared following the protocol of Khalil *et al.* (2021) with some modifications.

### Optimization of Parameters Affecting Cr (VI)

#### Adsorption

The factors affecting the adsorption process were optimized. The effect of temperature was investigated by varying it from 25 to 50°C to obtain the optimal temperature that is suitable for efficient Cr (VI) adsorption. Different adsorbent doses 14, 16, 18, 20, 22, 24, 26 and 28 g/L were employed to know

the optimum biomass required for an effective remediation process. The pH was varied from 1 to 5 for knowing the optimal pH value for metal adsorption, and the effect of different initial Cr (VI) concentrations (100–350 mg/L) was also studied. The solution was filtered and their absorbance was measured at 540 nm spectrophotometrically via DPC analysis. The determination of percent Cr (VI) remediation via the adsorption process was calculated by using the formula:

$$\% \text{ Cr (VI) remediation} = \frac{C_o - C_e}{C_o} \times 100$$

where,  $C_o$  = initial Cr (VI) concentration  
 $C_e$  = equilibrium Cr (VI) concentration

### Fourier Transform Infra red (FTIR) analysis

The characterization of adsorbent material, rice husk, was done by FTIR analysis to know the functional groups involved in the adsorption of Cr (VI). FTIR analysis of the sample was performed at the CytoGene Research & Development, Lucknow, India

### Analysis of Cr (VI) removal

The analysis of Cr (VI) removal by adsorbent was done by DPC method using (1, 5- Diphenyl Carbazide). DPC reagent is utilized for the spectrophotometric estimation of Cr (VI) ions in the solution after the adsorption experiment. Cr (VI) in an acidic solution of  $K_2Cr_2O_7$  gives red violet color when combined with DPC. The intensity of the color is directly proportional to the concentration of Cr (VI) present in the solution. For the preparation of DPC reagent for Cr (VI) estimation the standard protocol was followed (US EPA; APHA 1998).

The biomass was added in the flask containing the chromium solution for the adsorption of Cr (VI). The flask was kept in orbital shaker (for 2 hours at 140 rpm). The adsorbent was filtered out after being settled at bottom. The filtrate was analysed by DPC method and the Cr (VI) concentration was measured by spectrophotometer at 540 nm wavelength (Tripathi *et al.*, 2011a).

### Statistical Analysis

All the experiments in this study were carried out in triplicate, and the Microsoft Excel<sub>7</sub> was used for the statistical calculations.

## Results and Discussion

The results of this study demonstrated the efficacy of rice husk for the effective remediation of Cr (VI). Multiple factors such as pH, initial Cr (VI) concentration, temperature and adsorbent dosage influencing the process of adsorption were evaluated and optimized for environmental sustainability. The findings showed that rice husk exhibits significant adsorption capacity, with optimal hexavalent chromium removal potential under optimal conditions. The functional groups were characterized, of adsorbent material, using FTIR

spectroscopy. These results support the potential utilization of rice husk as a cost-effective and green sustainable adsorbent for the effective remediation of Cr (VI).

### Factors Influencing the Hexavalent Chromium Adsorption

#### Effect of temperature

The efficiency of Cr (VI) removal by adsorbents can be considerably affected by the alteration in temperature. The percentage Cr (VI) removal was studied with the change in temperature ranging from 25 to 50°C. As the temperature was increased above 25°C, the Cr (VI) removal efficiency of the adsorbent was also reported to be increased. The % Cr (VI) removal increased from 94.5 to 96% as the temperature was increased from 25 to 40°C, whereas the adsorbent efficiency decreases after the elevation of temperature above 40°C to 94.8% (Figure 1). The efficiency of adsorbents can be considerably altered by the alteration in temperature (Giri *et al.*, 2021). The increase in the Cr (VI) may be due to the enhanced attraction of metal ions for the binding locations at the surface of adsorbent (Aliabadi *et al.*, 2012; Pandey *et al.*, 2014). In our study, there is lesser changes in adsorption efficiency with alternation in temperature. In another study, Mondal *et al.* (2019) investigated the effect of temperature on adsorption of hexavalent chromium by mosambi peel adsorbent and obtained similar results on the alteration of temperature. Higher temperature assists in the formation of more new sites for the sorption, on the surface of adsorbents, resulting in the increase in removal rate of Cr (VI) (Das *et al.*, 2000; Guo *et al.*, 2002). Giri *et al.*, (2021), also reported the similar trend in the % Cr (VI) removal with the change in temperature utilizing pomegranate peel as adsorbent for hexavalent chromium remediation.

#### Effect of pH

pH is one of the important parameters in the efficient remediation process. In this study, rice husk as an adsorbent was utilized with different pH values during Cr (VI) adsorption. At pH 2, the higher Cr (VI) remediation was observed, indicating it as the optimum pH for the effective

hexavalent chromium removal (Figure 2). Further increase in pH up to 5, the %Cr (VI) adsorption was decreased from 85 to 50% indicating the inverse relation between pH and % removal by using the rice husk. The maximum percentage hexavalent chromium removal was observed at pH 2. The pH is important for effective adsorption of Cr (VI) (Kumar *et al.*, 2008; Tripathi *et al.* 2011b). The ionic form of chromium are dichromate ( $\text{Cr}_2\text{O}_7^{2-}$ ), chromate ( $\text{CrO}_4^{2-}$ ), etc. (Jobby *et al.*, 2018). The protonation of the adsorbent's surface assists in the electrostatic attraction between Cr ions and adsorbent surface (Sanchez-Hachair and Hofmann, 2018; Garg *et al.*, 2012). Similarly, Yadav *et al.*, (2022), observed that the with increase in pH, decreased adsorption of Cr (VI) by rice husk. Mondal *et al.*, (2019), also reported the similar trend and outcome by utilizing mosambi peel dust for removal of Cr (VI).

#### Effect of initial Cr (VI) concentration

The initial Cr (VI) level is also an essential factor for the efficient adsorption. The effect of initial Cr (VI) concentration (100–500 mg/L) was studied, while keeping other parameters such as pH, adsorbent dose and temperature constant. The %Cr (VI) removal was found to be decreased from 96.5 to 55%, respectively with increasing level of metal concentration. The effect of initial Cr (VI) concentration is an essential parameter which assists in the adsorption of Cr (VI) ions on the surface of adsorbent (Ghorbani *et al.*, 2016). Similar results were obtained by Khalfaoui *et al.*, (2024), by utilizing orange peel as biosorbent and obtaining 97% Cr (VI) removal at 50 mg/L initial Cr (VI) concentration. In another study, Giri *et al.*, (2021) investigated the effect of initial Cr (VI) concentration on adsorption by varying the concentration from 20–100 mg/L and observed maximum efficiency of 96.1% at 20 mg/L concentration, while at 100 mg/L, the Cr (VI) removal efficiency was lesser.

#### Effect of adsorbent dosage

The results showed that the Cr (VI) removal efficiency was optimum with 26 mg/L adsorbent dosage, further no significant effect was observed with increase in adsorbent dose (Figure 3). The optimum adsorbent dose obtained for the efficient Cr (VI) adsorption was 26 mg/L. The increase in

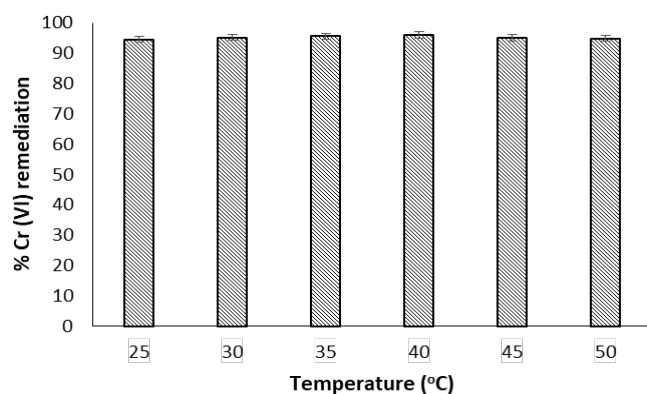


Figure 1: Effect of temperature on %Cr (VI) remediation by rice husk

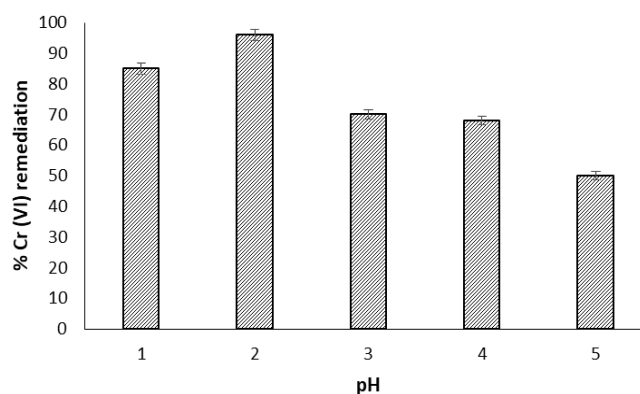


Figure 2: Effect of pH on %Cr (VI) remediation by rice husk

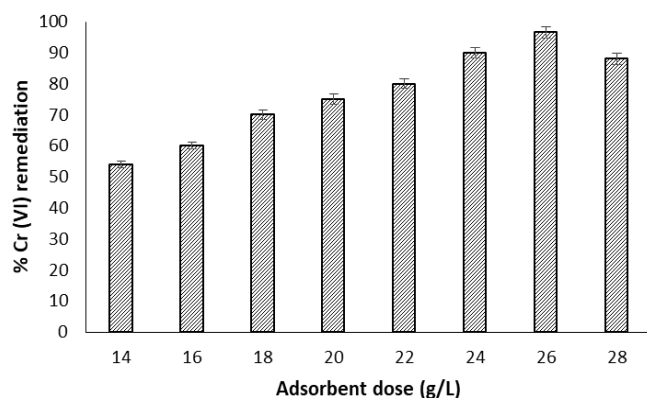


Figure 3: Effect of adsorbent dose on %Cr (VI) remediation

the Cr (VI) removal might be observed due to increase in the surface area and the sites of exchange as the adsorbent dose is increased. However, with increase in the adsorbent dose, the %Cr (VI) removal is decreased due to overcrowding of adsorbent particles, therefore, decreasing the binding sites of Cr (VI) (Sahmoune *et al.*, 2011). Other researchers, Gning *et al.*, (2024), reported that the optimal 45 mg adsorbent for the 97% Cr (VI) removal by utilizing mango kernel powder for the remediation of hexavalent chromium. Similarly, Giri *et al.*, (2021), also observed the adsorbent dose of 500 mg as optimal dose for the 100% Cr (VI) removal in 30 minutes by employing pomegranate peel as adsorbent. In another study, Yadav *et al.* (2022), reported %Cr (VI) removal using rice husk, and observed that their removal rate of hexavalent chromium was increased with increase in the biomass dosage from 4–10 g/L, and achieved equilibrium at 10 g/L, and observed 10 g/L as the most effective adsorbent dose.

#### FTIR Analysis of Adsorbent

The functional groups characterization on adsorbent surface was done by FTIR analysis. The unloaded rice husk and loaded rice husk after Cr (VI) adsorption from the chromium solution was studied. Functional groups present on the surface of rice husk, that are involved in the Cr (VI) adsorption was analysed by FTIR spectra, spanning 400 to 4000  $\text{cm}^{-1}$  wavenumber. Various peaks at different wavelength were observed. Some peaks were obtained at certain wavelength, some bands were present at certain wavelength representing the specific functional groups involvement in the adsorption process. FTIR analysis of without and with Cr (VI) rice husk was conducted and FTIR spectra was obtained signifying the various functional groups, present on the surface of adsorbent (Figure 4). In the spectral analysis, the peak was obtained at 3330  $\text{cm}^{-1}$  of rice husk without Cr (VI), and the peak was shifted to 3339  $\text{cm}^{-1}$  in the rice husk spectra with Cr (VI). Other peak was obtained at 1842  $\text{cm}^{-1}$  was also found to be shifted near 1800  $\text{cm}^{-1}$  in the spectra of rice husk with Cr (VI). The peaks at 1502  $\text{cm}^{-1}$ , 1337  $\text{cm}^{-1}$  and 977.8  $\text{cm}^{-1}$  observed in the FT-IR spectra

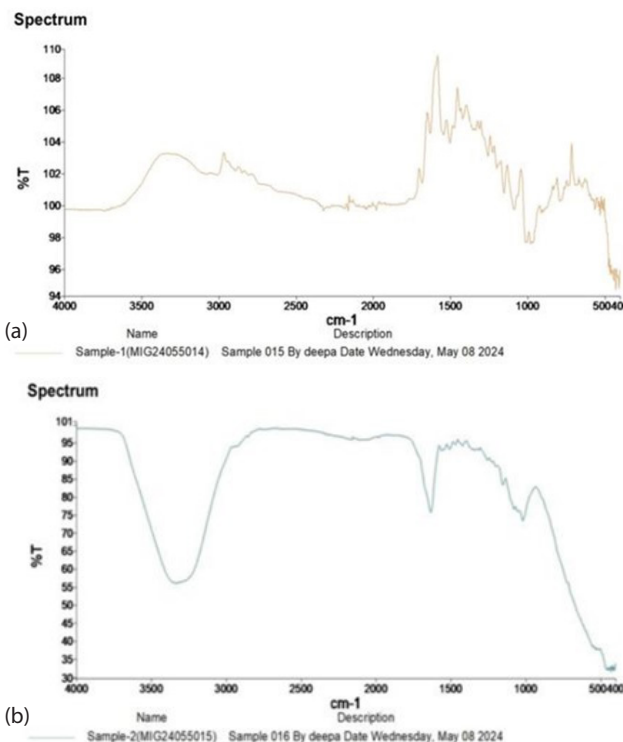


Figure 4: FTIR spectra of adsorbent material, (a) without Cr (VI), and (b) with Cr (VI)

of unloaded rice husk was observed to be shifted to 1508, 1338 and 1018  $\text{cm}^{-1}$ , respectively, indicating the interaction of various functional groups present on the surface of rice husk with hexavalent chromium ions.

In the spectra, the peaks were obtained at 3354  $\text{cm}^{-1}$  of unloaded rice husk and at 3377  $\text{cm}^{-1}$  of Cr (VI) loaded rice husk, indicating the hydroxyl functional group (-OH stretching vibrations). These peaks might be associated with the macromolecules found in the rice husk particularly lignin and cellulose. The shift in the peak from 3354  $\text{cm}^{-1}$  (unloaded rice husk) to 3377  $\text{cm}^{-1}$  (hexavalent chromium loaded rice husk) demonstrate the interaction between the rice husk %Cr (VI), indicating that the adsorption of Cr (VI) affects -OH functional group present on the surface of rice husk (Nadeem *et al.*, 2015; Garg *et al.*, 2007). The Cr (VI) removal was studied, by adsorption on modified groundnut hull (Owalude and Tella, 2016). The results revealed the possible role of -OH and C-H, while others like -NH<sub>2</sub> and COO- with low peaks may also to be involved in the adsorption process. The similar trend was obtained in other studies, with the utilization of adsorbent almond shell and apricot shell (Khazaei *et al.*, 2011), rice husk (Khalil *et al.*, 2021), coconut husk (Verma *et al.*, 2021).

Various researchers investigated the adsorption capacity of waste biomass in the Cr (VI) remediation. In a study, Mancilla *et al.*, (2022), utilized *Sambucus nigra* leaves which is an agro- forestry and industrial residue, and observed impressive Cr (VI) removal. Similarly, Diaf *et al.*, (2023), utilized pomegranate peel as adsorbent for the removal of

hexavalent chromium and reported 100% Cr (VI) removal efficiency at pH 1 and 50°C. In another study, the adsorption was examined by Hernandez and Romero, (2020), utilizing banana peel as adsorbent for the removal of Cr (VI) and observed > 92% of Cr (VI) removal. In another study, Channa *et al.*, (2022), employed orange peel as adsorbent for the remediation hexavalent chromium and achieved efficient removal of Cr (VI) in the batch adsorption studies.

## Conclusion

Rice husk was efficiently utilized for the remediation of Cr (VI). The study showed the maximum Cr (VI) removal was 96.5% by employing rice husk as an effective adsorbent under optimal conditions. Additionally, the FTIR analysis showed the possible involvement of specific functional groups in the Cr (VI) adsorption from the aqueous solution. Since, rice husk has been proved to be an efficient adsorbent for the effective removal of hexavalent chromium and are easily available, cost-effective and waste material biomass. Therefore, rice husk might be utilized as a sustainable and viable adsorbent for the effective remediation of Cr (VI) and aid in the overall health of ecosystem.

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