

Research Article

Grewia spp. Biopolymer as Low-Cost Biosorbent for Hexavalent Chromium Removal

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In this study, *Grewia* spp. biopolymer was utilized as a biosorbent for elimination of hexavalent chromium from water. Fourier-transform infrared spectrometry (FTIR) and X-ray diffraction (XRD) were performed for characterization of the biosorbent. Experiments were conducted in a batch mode at room temperature ($25 \pm 2^\circ\text{C}$) and agitation speed of 100 rpm to determine the influence of biosorbent dose, contact time, Cr(VI) concentration, and initial solution pH. It was found that equilibrium was attained in 50 min. A pseudo-first-order model suited well than a pseudo-second-order model. Biosorption capacity of *Grewia* spp. biopolymer increased with increase in concentration and depended on the solution pH. Langmuir and Freundlich models described experimental data very well. These findings showed that *Grewia* spp. biopolymer can serve as a biosorbent for elimination of Cr(VI) from water.

1. Introduction

Water polluted with heavy metals has become a serious environmental problem [1]. Chromium is one of such heavy metals. Its presence in water originates from some anthropogenic source including industrial activities such as dyeing, electroplating, textile processing, alloying, and fertilizer production [2–4]. Chromium in aqueous solution exists in two oxidation states, trivalent state [Cr(III)] and hexavalent state [Cr(VI)]. The trivalent chromium is an essential trace element for metabolism in mammals (for living organisms), while the hexavalent is more dangerous because of its carcinogenic effects in mammals [5]. Therefore, elimination of Cr(VI) from water or at least its reduction below the maximum allowed limit is necessary. The technologies used for the treatment of waters contaminated by chromium are ion exchange, membrane separation, coagulation, adsorption, and solvent extraction [6]. However, these methods have technological and economical limitations [7, 8]. Therefore, cost-effective treatment for

chromium elimination is required especially in developing countries. Adsorption using biological materials is one of the recommended processes because it has many advantages including low cost, minimization of chemicals, and possession of great adsorption efficiency [9, 10]. Many biological materials (biosorbents) such as gum kondagogu [11] and brown algae *Pelvetia canaliculata* [12] have efficiently eliminated chromium from water. The efficiency of this process is attributed to the presence of some chemical groups like carboxyl and hydroxyl in the biosorbent. These groups are responsible for physical and chemical interactions of heavy metals with the biosorbent [13]. *Grewia* spp. is widespread in some northern regions of Cameroon. This plant is used by the population in the clarification of water wells and a local beer (bilibili). Biopolymer extracted from *Grewia* spp. contains chemical groups mentioned above that are involved in metal binding [14, 15]. Thus, it can be used for hexavalent chromium elimination from water.

The aim of this study is then to investigate the feasibility of *Grewia* spp. biopolymer as a biosorbent for elimination of

chromium (VI) by varying contact time, solution pH, biosorbent dose, and chromium concentration. Kinetics and isotherm studies were also performed.

2. Materials and Methods

2.1. Materials. *Grewia* spp. biopolymer barks were collected from Mokolo, a locality in the northern region of Cameroon, transported to the laboratory, and dried and stored at room temperature. Crude *Grewia* gum was obtained following the method of Somboonpanyakul et al. [16]. The dried and pulverized bark of *Grewia* spp. was dispersed in distilled water (1:80 w/v, pH 4) at 50°C for 4 hours. The fibrous material from the dispersed mucilage was removed by centrifugation at 5300 rpm for 20 min at 4°C. Thereafter, the crude mucilage was precipitated with 3 volumes of 95% absolute ethanol and freeze-dried.

All other reagents used in this study were of analytical grade. A synthetic solution of Cr(VI) was obtained by dissolving appropriate quantity of potassium dichromate powder (Riedel-de Haën) in distilled water.

2.2. Characterization of Biosorbent

2.2.1. Fourier-Transform Infrared Spectroscopy (FTIR). The identification of chemical groups on the surface of *Grewia* spp. biopolymer powder was performed by Fourier-transform infra-red spectrometer (Bruker Vertex 70). Approximately 1 mg of the sample was mixed with 25 mg KBr, and both were ground together in a ceramic mortar. Thereafter, a pellet was prepared using a 5-ton press and analyzed by an infrared spectrometer. The spectra were recorded in the 4000 cm⁻¹ to 400 cm⁻¹ wavenumber range with ten scans collected at 4 cm⁻¹ resolution.

2.2.2. Powder X-Ray Diffraction. X-ray diffraction was carried out on Desktop X-ray Diffractometer Rigaku, MiniFlex II. Samples were analyzed over an angular range of 2 theta (10°–90°).

2.2.3. Zeta Potential. Electrokinetic zeta potential (ZP) of *Grewia* spp. biopolymer was measured as a function of pH using dynamic light scattering (DLS) with the nanoparticle analyzer SZ-100 (Horiba Scientific).

2.3. Biosorption Experiments. The biosorption experiments were realized in a batch mode using 100 mL Erlenmeyer flasks fixed to a shaker, Polystest 20 (Bioblock Scientific). About 50 mL of chromium solution was poured into Erlenmeyer flasks, and the pH of the solution in each flask was adjusted to the desired value with either 0.1 M HCl or 0.1 M NaOH. A predetermined amount of biosorbent was then added to each flask, fixed on a shaker, and set on agitation at a speed of 100 rpm. After a given time, the shaker was stopped and the flasks removed. The suspensions were centrifuged at 5000g, and the supernatant was recovered. The supernatant absorbance was measured using

the spectrophotometer (Jenway 7310, UK) at 540 nm after complexation using the diphenylcarbazide solution as described by Clesceri et al. [17].

The amount of chromium sorbed at time “*t*” and the percentage of chromium elimination were calculated according to the following equations:

$$q_t = \frac{(C_i - C_t)}{m} V, \quad (1)$$

where C_i (mg/L) is the initial concentration of Cr(VI), C_t (mg/L) is the concentration of Cr(VI) at contact time t (min), V (L) is the volume Cr(VI) solution, and m (g) is the amount of the biosorbent added.

Elimination efficiency of Cr(VI) was determined by the following equation:

$$\%R = \frac{(C_i - C_e)}{C_i} 100, \quad (2)$$

where C_i and C_e are the initial and the equilibrium concentration (mg/L) of Cr(VI), respectively.

3. Results and Discussion

3.1. Characteristics of Biosorbent

3.1.1. X-Ray Analysis of *Grewia* spp. Biopolymer. XRD analysis was applied to detect the crystallinity of the *Grewia* spp. biopolymer. The XRD pattern showed zero peak characteristics; *Grewia* spp. biopolymer consists of amorphous structure (Figure 1). This suggests that chromium (IV) ion can easily penetrate into the surface of the biopolymer [18].

3.1.2. Functional Groups Determination by FTIR. The FTIR spectrum of *Grewia* spp. biopolymer before and after chromium biosorption is presented in Figure 2. In all spectrum sorption bands, free water was observed to be around 3300 cm⁻¹. The intensity of the peak engaged by free water molecules was found diminished little with the biosorption of Cr(VI) on the *Grewia* biopolymer. This suggests that, the biosorption may happen on -OH bending of water, the inner surface -OH stretching vibration. The important decrease of peak intensity was also observed for the bands of 1049, 1728, 1418, and 1595 cm⁻¹ after adsorption. All those changes can be attributed to the engagement of some chemical groups of *Grewia* such as CONH and COOH (uronic acid) during the biosorption.

3.1.3. Nature of Charge of *Grewia* spp. Biopolymer. The ZP is the potential of shear plane of the particle when it moves in liquid. ZP was measured to collect information on the stability and charge behavior of the polymer. The ZP of *Grewia* spp. biopolymer in the aqueous medium were recorded and found to be -30.45 ± 0.48 mV, -24.64 ± 0.49 mV, and -18.96 ± 0.66 mV at pH value of 9, 7, and 5, respectively (Figure 3). These results showed that, the *Grewia* spp. biopolymer is anionic in nature and can be attributed to the existence of uronic acids as previously showed FTIR spectral.

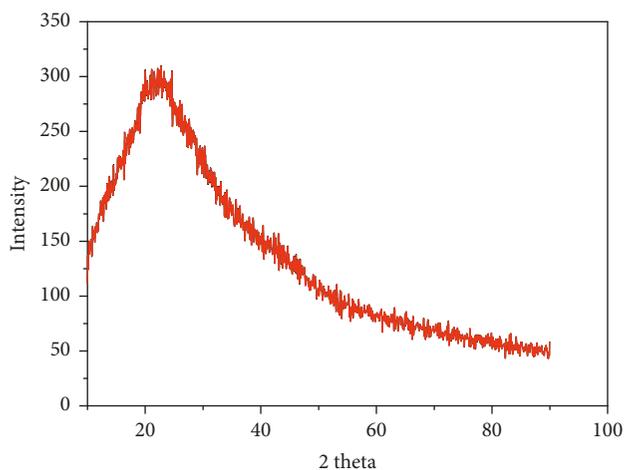


FIGURE 1: X-ray diffraction (XRD) pattern of *Grewia* spp. biopolymer.

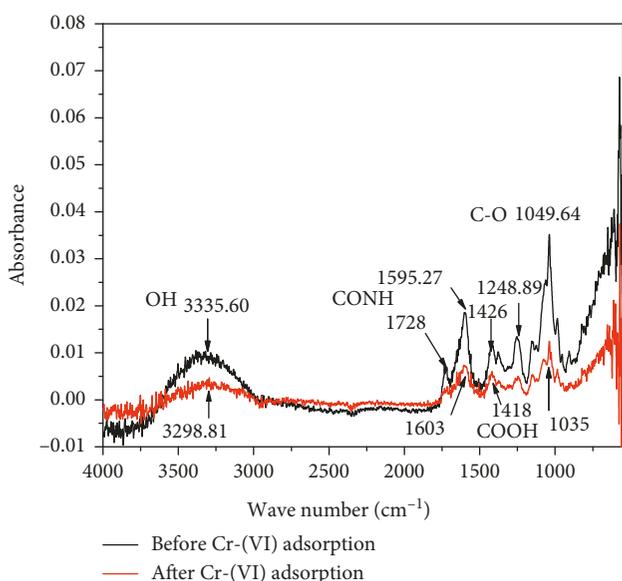


FIGURE 2: FTIR spectra of *Grewia* spp. biopolymer.

3.2. Effect of Biosorbent Mass. The effect of biosorbent mass on the biosorption of Cr(VI) was studied. The amount of the biosorbent used varied from 0.1 g to 1 g, while other parameters such as pH, agitation time, and agitation speed were kept constant. The biosorbent dose affects the extent of chromium as shown in Figure 4. The extent of chromium elimination varied from 47% to 98%, respectively, at 0.1 g/L and 0.8 g/L. The increase in chromium biosorption with increase of biopolymer dose may be due to the availability of higher surface area for chromium biosorption [19]. However, there was no noticeable variation in the extent of Cr(VI) adsorption above 0.8 g/L. This may be due to non-adsorbability of Cr(VI) ions as a result of sorbent-sorbate interaction [20]. Additionally, at high dose, the biosorbent also adhere to each other, consequently adsorption extent becomes constant, due to the fact that all the surface area is not available.

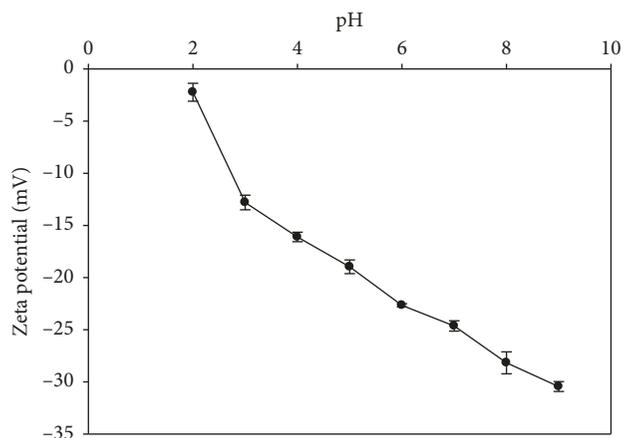


FIGURE 3: Zeta potential of *Grewia* spp. powder at different pH.

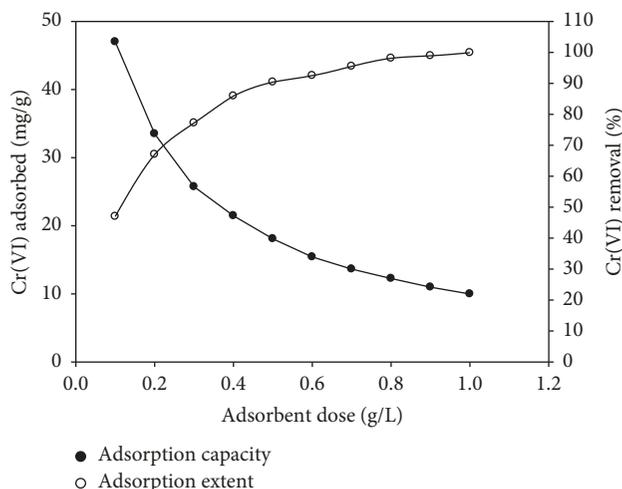


FIGURE 4: Effect of biosorbent mass on the biosorption of Cr(VI) ions onto biopolymer at pH 7 ± 0.05 , $T = 25 \pm 2^\circ\text{C}$, and initial concentration 10 mg/L.

When the mass is low, all biosorbent sites are occupied and the surface is saturated, resulting in a high value of the amount of chromium adsorbed.

3.3. Effect of Contact Time. The effect of contact time on Cr(VI) biosorption on *Grewia* spp. polymer was studied. It is observed that (Figure 5), as contact time increases, the amount of chromium biosorbed increases initially, afterwards it gradually approaches a constant value denoting attainment of equilibrium; this is independent of the pH studied. The rate of Cr(VI) biosorption is high in the initial 40 min, thereafter level off and approaches zero. These variations in the rate of biosorption may be due to the fact that, initially, all sorption sites were vacant and the solute concentration gradient was high. Afterwards, the chromium rate decreases due to decrease in sorption sites [20]. For other studies, 60 minutes was chosen as the equilibrium time.

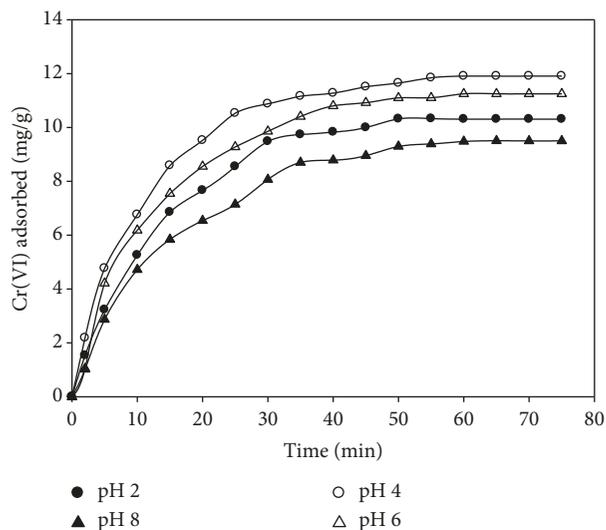


FIGURE 5: Effect of contact time at different pH. Biosorbent dose = 0.8 g/L; $T = 25 \pm 2^\circ\text{C}$; initial Cr(VI) concentration = 10 mg/L.

3.4. Effect of Chromium Initial Concentration. The experiments were carried out at different chromium concentrations (20, 40, 60, 80, and 100 mg/L) and a contact time of 60 min. The amount of Cr(VI) ion biosorbed on the biopolymer powder is presented in Figure 6. It can be observed from this figure that by increasing initial chromium (VI) concentration, biosorption capacity is also increased. This is due to the increase in the driving force of the concentration gradient [21]. Similar trend of Cr(VI) adsorption onto the biosorbent as function of initial concentration has also been reported by other studies [22–24].

3.5. Effect of pH. The process of biosorption depends on the pH of the solution as it affects the surface charge of biosorbent and the degree of ionization and specification of the adsorbate [25]. The pH of solution was varied from 2 to 8. From Figure 7, it was observed that the amount of Cr(VI) adsorbed increased with the increase of pH. The maximum Cr(VI) biosorbed was found around pH 4. The increase of the amount of chromium biosorbed with increasing pH can be attributed to the decline in competition between the proton and metal for the active sites, which results in a lower electrostatic repulsion between the surface and metal ions [1, 26]. As the pH increased, the protonation gradually weakened, and in alkaline solutions, the OH^- ions competes with Cr(VI) anionic species [27]. As consequence low amount of chromium adsorbed was observed at high pH values.

3.6. Biosorption Kinetics. In order to evaluate the kinetic mechanism that controls the biosorption process, the pseudo-first-order and pseudo-second-order models were applied to experimental data.

The pseudo-first-order model is given as follows [28]:

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t, \quad (3)$$

where q_t (mg/g) is the amount of chromium biosorbed at time t , k_1 (min^{-1}) is the rate constant of the first-order

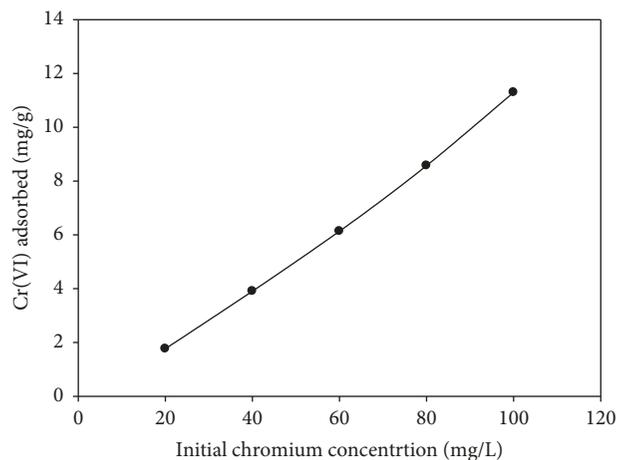


FIGURE 6: Effect of initial chromium concentration. pH 7 ± 0.05 ; $T = 25 \pm 2^\circ\text{C}$.

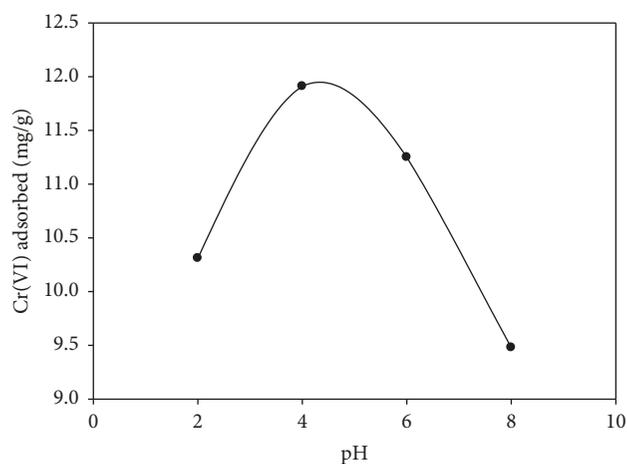


FIGURE 7: Effect of initial solution pH. Cr(VI) concentration = 10 mg/L; $T = 25 \pm 2^\circ\text{C}$.

reaction, and q_e the equilibrium sorption uptake derived from extrapolation of experimental data at time $t = \infty$. A plot of $\log(q_e - q_t)$ versus t is presented in Figure 8(a) q_e and k_1 are determined and presented in Table 1.

The pseudo-second-order kinetic model [28] is expressed as follows:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t. \quad (4)$$

The plot of t/q_t versus t is presented in Figure 8(b) and q_t and k_2 were determined (Table 1).

From Table 1, the values of correlation coefficients obtained for the pseudo-first-order and pseudo-second-order reactions were all above 0.90. However, the biosorption capacities calculated by pseudo-first-order model are close to those determined by experiments. That means, the pseudo-first-order model is appropriate to describe hexavalent chromium biosorption on *Grewia* spp. biopolymer, implying that sorption is of physical nature involving weak forces such as van der Waals bonding [29, 30]. The first-order adsorption rate decreases with increased pH, indicating that Cr(VI) biosorption is favorable at acidic pH.

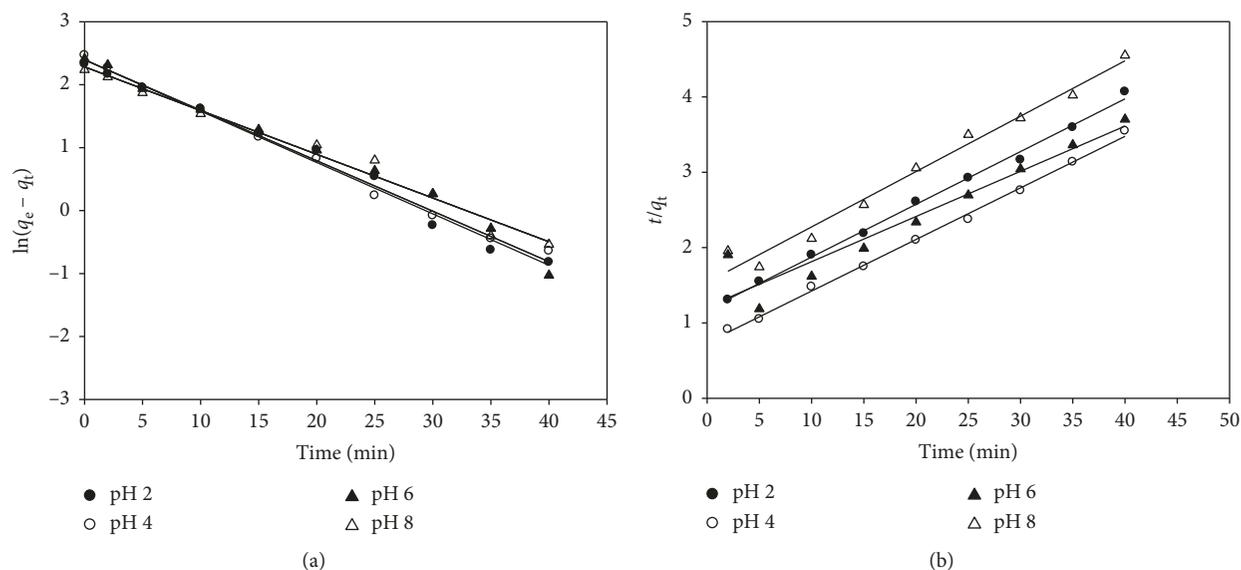


FIGURE 8: First-order (a) and second-order kinetics (b) modeling of Cr(VI) biosorption on *Grewia* spp. biopolymer. Biosorbent dose: 0.8 g/L, $T = 25 \pm 2^\circ\text{C}$.

TABLE 1: Kinetics parameters for Cr(VI) biosorption on *Grewia* spp. biopolymer.

| Initial pH of the solution | First-order model | | | | Second-order model | | |
|----------------------------|---------------------------|-----------------------------|---------------------------|--------|---|---------------------------|--------|
| | $q_{e,\text{exp}}$ (mg/g) | k_1 (min^{-1}) | $q_{e,\text{cal}}$ (mg/g) | R^2 | k_2 ($\text{g}\cdot\text{mg}^{-1}\cdot\text{min}^{-1}$) | $q_{e,\text{cal}}$ (mg/g) | R^2 |
| 2 | 10.268 | 0.1879 | 10.478 | 0.9875 | 0.0041 | 14.285 | 0.9963 |
| 4 | 11.803 | 0.1847 | 10.974 | 0.9941 | 0.0063 | 14.598 | 0.9973 |
| 6 | 11.159 | 0.1828 | 11.652 | 0.9836 | 0.0030 | 16.667 | 0.9107 |
| 8 | 9.374 | 0.1601 | 9.815 | 0.9813 | 0.0035 | 13.605 | 0.9796 |

3.7. Biosorption Isotherms. Isotherm studies were performed in a batch mode using concentrations of hexavalent chromium ranging from 6 to 19 mg/L at different solution pH. Two equilibrium models, namely, the Langmuir and the Freundlich were used to analyze the data. The linear form of the Langmuir isotherm model is given by the following:

$$\frac{C_e}{q_e} = \frac{C_e}{Q_m} + \frac{1}{Q_m b}, \quad (5)$$

where q_e and C_e are the equilibrium concentrations of chromium in the sorbent (mg/g) and liquid phase (mg/L) respectively. Q_m and b are the Langmuir constants which are related to the sorption capacity and energy of sorption, respectively. These values can be calculated from the intercept and slope of the linear plot, with C_e/q_e versus C_e .

The linear form of the Freundlich isotherm model can be expressed as follows:

$$\log q_e = \log k_F + \frac{1}{n} \log C_e, \quad (6)$$

where q_e and C_e are the equilibrium concentration of chromium in the biosorbent (mg/g) and liquid phase (mg/L), respectively. k_F and n are the Freundlich constants which are related to sorption capacity and intensity, respectively. These constants can be calculated from the slope and intercept of the linear plot, with $\log q_e$ versus $\log C_e$.

The isotherms constants calculated from the slope and intercept of Figure 9 for Langmuir and Figure 10 for Freundlich are presented in Table 2. According to regression coefficients, the experimental data fit well both Freundlich ($R^2 = 0.9871-0.9982$) and Langmuir models ($R^2 = 0.9029-0.9969$), suggesting that both monolayer and heterogeneous surface sorption affect the biosorption of Cr(VI) [31]. Similar mechanisms of Cr(VI) sorption onto biosorbent has also been reported by other studies [9]. The separation factor, an indicator of the reliability of the biosorption, is defined by the following equation:

$$R = \frac{1}{1 + bC_0}, \quad (7)$$

where C_0 is the initial Cr(VI) concentration and b is the Langmuir isotherm constant.

The value of R varied from 0.14 to 0.90. These values are less than 1, indicating the favorable biosorption of Cr(VI) on *Grewia* spp. biopolymer [31]. Moreover, the “ n ” values lie between 1 and 10, which shows that biosorption is beneficial [25].

4. Conclusion

Biosorption of hexavalent chromium onto *Grewia* spp. biopolymer has been studied in detail. *Grewia* spp. biopolymer is negatively charged and is able to remove 98% of

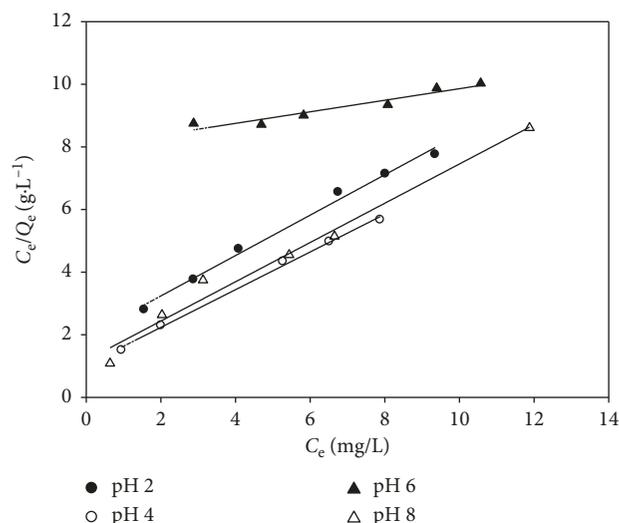


FIGURE 9: Langmuir plots for Cr(VI) biosorption by *Grewia* spp. biopolymer. Contact time 60 min, $T = 25 \pm 2^\circ\text{C}$.

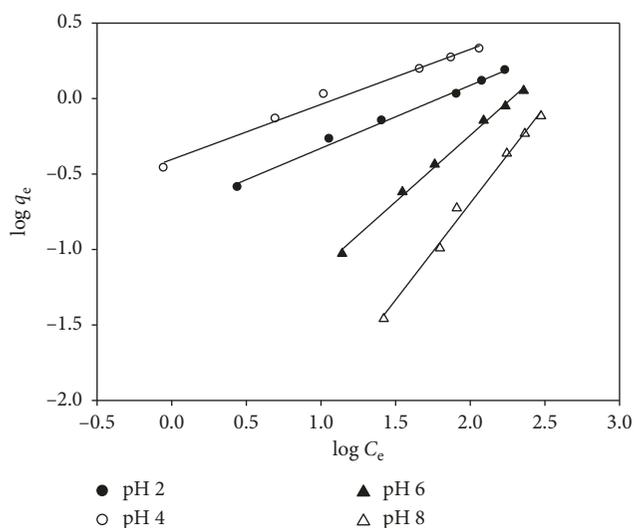


FIGURE 10: Freundlich plots for Cr(VI) biosorption by *Grewia* spp. biopolymer. Contact time 60 min; $T = 25 \pm 2^\circ\text{C}$.

TABLE 2: Langmuir and Freundlich isotherms parameters for Cr(VI) biosorption on *Grewia* spp. biopolymer.

| Isotherm model | pH 2 | pH 4 | pH 6 | pH 8 |
|-------------------|--------|--------|--------|--------|
| <i>Langmuir</i> | | | | |
| Q_{\max} | 1.555 | 1.661 | 5.258 | 1.613 |
| b | 0.331 | 0.584 | 0.240 | 0.52 |
| R^2 | 0.9919 | 0.9969 | 0.9356 | 0.9029 |
| <i>Freundlich</i> | | | | |
| k_F | 1.516 | 1.441 | 2.409 | 3.649 |
| n | 2.402 | 2.738 | 1.137 | 0.984 |
| R^2 | 0.9936 | 0.9871 | 0.9967 | 0.9982 |

hexavalent chromium. Equilibrium was attained within 50 min, regardless of the pH studied. The amount of chromium biosorbed was affected by the pH of the solution. This amount increases with the increase of biosorbent dose

and initial chromium concentration. Kinetics studies revealed that the pseudo-first-order model described well the Cr(VI) biosorption, which suggests that the process is of physical nature. Both Langmuir and Freundlich isotherms fit the biosorption data adequately. Being an inexpensive and abundantly available material, *Grewia* spp. biopolymer is a potential biosorbent for the elimination of Cr(VI) from aqueous solutions.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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Supplementary Materials

These data were used to plot curves shown in figures. (*Supplementary Materials*)

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