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ADSORPTION OF CHROMIUM FROM TANNERY WASTEWATER ON ADSORBENT EQUIPPED FROM *Argemone Mexicana*

Md. Abul Hashem*, Sadia Mim*, Md. Zillur Rahaman Shaikh* and Tanzila Parvin Ame *

**Department of Leather Engineering, Khulna University of Engineering & Technology (KUET), Khulna-923, BANGLADESH*

ABSTRACT

In this study, Argemone mexicana adsorbent has been introduced for the adsorption of chromium from tannery wastewater. The studies on the adsorption of chromium were conducted by varying adsorbent doses, stirring time and settling time. The surface charge of the Argemone mexicana adsorbent was obtained from pH_{PZC} analysis which was 8.9. Adsorption experimentations were performed through batch mode to check the effect of Argemone mexicana adsorbent on the uptake of chromium. The maximum removal efficiency was obtained at 99.80% applying 0.4 g of Argemone mexicana adsorbent into 25 mL of tannery wastewater at 20 min. The kinetic and isotherm of adsorption of chromium ion followed Pseudo 2nd order kinetic and Freundlich isotherm model. The value of pH in the treated effluent obtained was 6.4 which was within the discharge level. This investigation might be supportive to develop an environmentally friendly process for adsorption of chromium from tannery wastewater.

Keywords: *Argemone Mexicana*, Adsorbent, Chromium, Efficiency, Tannery.

INTRODUCTION

The leather industry has been considered one of the most prospective industries in Bangladesh due to contributing a significant role in the national economy. The availability of raw materials with lower manufacturing expenses has accelerated this prosperity to meet the requirement of about 10% of the total leather market worldwide (Moktadir et al. 2021). The raw animal skins are usually transformed into final leather through a series of integral steps e.g., pre-tanning (beamhouse), tanning and finishing. Tanning is one of the most important operational stages in leather manufacturing to stabilize the collagen fibers in the leather (Nazer et al. 2006). About 80-90% of the leather industries apply chrome tanning to obtain better hydrothermal stability in the final leather (Covington, 2009). However, only 60-70% of chromium (Cr) is usually uptaken by the pelt during the traditional chrome tanning process and about 30-40% of the Cr keeps remaining in the waste tanning liquor (Kanagaraj et al. 2020).

The large quantities of Cr in the tannery effluent may cause serious pollution in the soil and water (Ma et al. 2014). About 3×10^8 m^3 of high Cr-containing waste liquor is daily discharged by a medium-sized tannery (Kanagaraj et al. 2015). Both trivalent chromium, Cr (III) and hexavalent chromium, Cr (VI) are harmful (Agarwal et al. 2006). The Cr (III) released from tanneries may also convert into Cr (VI) through oxidation (Apte et al. 2005). This scenario is mainly responsible to gain negative comments for the leather industry. Cr (VI) is highly mobile and acutely toxic, mutagenic and carcinogenic to living organisms (Dotaniya et al. 2014). Exposure to Cr (VI) may also lead to nasal septum perforation, allergic and irritant dermatitis, skin ulcers, etc. (Saha et al. 2011). Therefore, the remaining Cr in the tannery effluent must be removed before disposal.

Several physicochemical techniques e.g., coagulation (Song et al. 2004), flocculation (Garrote et al. 1995), ozonation (Houshyar et al. 2012), reverse osmosis (Hintermeyer et al. 2008), ion exchange (Petruzzelli et al. 1995), and adsorption (Hashem et al. 2021) have already been introduced to remove Cr from tannery wastewater. The investment and set-up costs are lower in the adsorption process compared to others (Kulkarni et al. 2015). Moreover, adsorption is sludge-free and does not require any supplementary treatment to remove Cr from tannery wastewater. Research on cost-effective adsorbents should be carried out to remove Cr from tannery wastewater. Many researchers have already employed different low-cost adsorbents e.g., waste newspaper (Dehghani et al. 2016), agricultural waste (Dubey et al. 2007), grape waste (Chand et al. 2009), sugar industrial waste (Fahim et al. 2006), spent tea leaves (Mia et al. 2018) and saw-dust (Gupta and Babu, 2009), etc. for removal of Cr. Most of these adsorbents possess lower adsorption capacity and generate more Cr-loaded adsorbent after treatment posing disposal problems (Agarwal et al. 2006). Therefore, exploration of cost-effective and higher adsorption capacities adsorbent is needed to remove Cr from tannery wastewater.

Argemone mexicana (*A. Mexicana)* is an erect spiny annual or biennial herb and is available all over the world on both rivers and roadsides (Brahmachari et al. 2013). Thistle-like leaves are not attached to the stem by petioles in this herb. The grayish-white veins on the leaves stand out against the blue-green leaf surface (Shaukat et al. 2002). In this investigation, both leaves and stems of *A. mexicana* were used to prepare adsorbent through thermal treatment. This herb has no potential use in our country which can be employed to prepare a low-cost adsorbent. Adsorbent preparation from the stem and leaves of *A. mexicana* is simple, easy and possible without any chemical treatment. Moreover, the source of *A. mexicana* is also available in our country.

This study aims to introduce *A. mexicana* adsorbent to remove Cr from chrome tanning wastewater. The Cr removal efficiency employing *A. mexicana* adsorbent was determined in terms of adsorbent dose, stirring and settling time. The Langmuir and Freundlich isotherm models are applied to fit the data obtained in this study. Pseudo 1st and 2nd order kinetic models are also applied to evaluate the mechanism of adsorption.

MATERIALS AND METHODS

Wastewater collection

Chrome tanning wastewater was collected in a high-density polyethylene (HDPE) bottle from SAF Leather Ltd., Khulna, Bangladesh. The bottle was washed with nitric acid before pouring the waste chrome liquor into it following laboratory methods. Before experimentation, the wastewater was filtered with Whatman No. 1 filter paper to eliminate unusual suspended solids. The obtained liquor after filtration was employed for the experiment.

Adsorbent preparation

The stem and leaves of *A. mexicana* were collected from the bank of the river Bhairab, Khulna, Bangladesh. The stem and leaves were split into little segments and dried in the sun. Then, the dried samples were burnt at 600ºC in a furnace for 3 h and ground by using laboratory grinder. Finally, by screening on 80-mesh, the requisite size of the adsorbent was attained. Figure 1(a) and Figure 1(b) represent the stem and leaves of *A. mexicana* and prepared *A. mexicana* adsorbent, respectively.

Figure 1 Stem and leaves of *A. mexicana* (a) and equipped adsorbent of from *A. mexicana* (b)

Chemicals

All analytical grade reagents/chemicals e.g., concentrated nitric acid, HNO_3 (Merck KGaA, Germany), hydrochloric acid, HCl (Merck KGaA, Germany), sodium hydroxide, NaOH (Merck, India) applied in this investigation were procured from City scientific store, Khulna, Bangladesh.

Batch adsorption experiment

The batch adsorption experiment was performed by applying a fixed mass of *A. mexicana* adsorbent at 25±2ºC. Different doses of *A. mexicana* adsorbent (0.1, 0.2, 0.3, 0.4, 0.5, and 0.6 g) were applied into a series of six beakers containing 25 mL of Cr containing wastewater. Then, the adsorbent mixed wastewater was stirred on a magnetic stirrer for a predefined time. After stirring, the wastewater was allowed to settle for a time; Finally, the mixture was filtered through Whatman No. 1 filter paper. Similarly, experimentations were performed by applying 0.4 g of *A. mexicana* adsorbent into 25 mL of Cr-containing wastewater with varying stirring times (5, 10, 15, 20, 25, and 30 min) and settling times (15, 30, 45, 60, 75, and 90 min). Cr removal efficiency (%) was calculated from the Cr content before and after treatment.

Batch kinetic adsorption experiments were performed by applying 0.4 g of *A. mexicana* adsorbent to 25 mL of Cr-containing wastewater maintaining a period (0–90 min). Pseudo 1st and 2nd order kinetic models were applied to analyze the adsorption mechanism. Again, adsorption isotherm studies were carried out by applying 0.4 g of *A. mexicana* adsorbent to 25 mL of Cr-containing wastewater for 20 min at 25±2ºC. Then, Freundlich and Langmuir isotherm models were applied to fit the data obtained in this investigation. Cr adsorption capacity (mg/g) was calculated from the initial and final Cr concentration, the mass of the adsorbent, and the volume of the wastewater. Origin 8 was employed to analyze data.

Adsorption kinetic and isotherm

For examining the adsorption kinetics, pseudo 1st and 2nd order equations were succeeded by Lagergren (1898) and YS and Mckay (1999), respectively. The mathematical statement for the Pseudo 1st and 2nd order model can be presented using the below expression (1) and (2), respectively.

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (1)$$

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

Where 'k_1' is the adsorption rate constant for the Pseudo 1st order model (min^{-1}), 'k_2' is the rate constant for the Pseudo 2nd order model ($g\ mg^{-1}\ min^{-1}$), 'q_t' is the Cr adsorption capacity (mg/g) at a time 't' (min), and 'q_e' is the Cr adsorption capacity (mg/g) at equilibrium condition.

To examine the adsorption isotherm, Langmuir and Freundlich isotherms were applied. The mathematical statement for Langmuir and Freundlich isotherm model can be presented using the below expression:

$$\frac{C_e}{q_e} = \frac{C_e}{q_{max}} + \frac{1}{K_L q_{max}} \quad (3)$$

$$\log q_e = \frac{1}{\log K_F} + \frac{1}{n}\log C_e \quad (2)$$

Where, 'K_L' and 'K_F' is the Langmuir and Freundlich isotherm constant (L/mg), respectively, 'C_e' is the equilibrium concentration of the solution (mg/L), 'q_{max}' (mg/g) is the monolayer adsorption capacity and 'n' is the intensity parameter of Freundlich adsorption isotherm.

pH_{PZC} analysis of adsorbent

The surface charge, pH_{PZC} of the *A. mexicana* adsorbent was performed following the pH drift method (Jawad et al. 2019). The pH was adjusted between 3 and 12 in ten beakers containing 25 mL of Cr-containing wastewater using 0.1 M NaOH and 0.1 M HCl. About 1.25 g of *A. mexicana* adsorbent was added to each beaker and left for continuous stirring for 24 h at 25±2ºC. The value of pH was determined after 20 min of settling. The value of ∆pH was calculated from the difference between the initial and final pH. The pHpzc was determined by plotting initial pH (pH_i) and ∆pH where ∆pH=0.

Wastewater characterization

Before and after treatment, Cr-containing wastewater was characterized in terms of pH, Cr content, total dissolved solids (TDS), total suspended solids (TSS), and electrical conductivity (EC). The pH of the raw Cr containing tannery wastewater and treated effluent was measured using a pH meter (BT 675, BOECO, Germany). Cr content was measured in the wastewater before and after treatment using Atomic Adsorption Spectrophotometer (Spectr240FS AA, Agilent, USA). The TSS was measured following the standard APHA method (APHA-2540-D, 2012). The TDS and EC were measured using a conductivity meter (CT-676, BOECO, Germany).

RESULTS AND DISCUSSION

Effect of adsorbent dose

Figure 2 depicts the effect of *A. mexicana* adsorbent dose for the Cr-removal. The highest Cr removal efficiency was calculated at 99.80% by applying *A. mexicana* adsorbent 0.4 g into 25 mL of Cr-containing wastewater. Figure 2 represents the effect of *A. mexicana* adsorbent dose on Cr removal efficiency. The Cr removal efficiency with 0.4, 0.5 and 0.6 g of *A. mexicana* adsorbent obtained 99.80%, 99.82% and 99.83%, respectively. The Cr removal efficiency slightly varies with 0.5 and 0.6 g doses which can be negotiated. After considering the dose of *A. mexicana* adsorbent in terms of Cr removal efficiency, 0.4 g was preferred to 0.5 and 0.6 g.

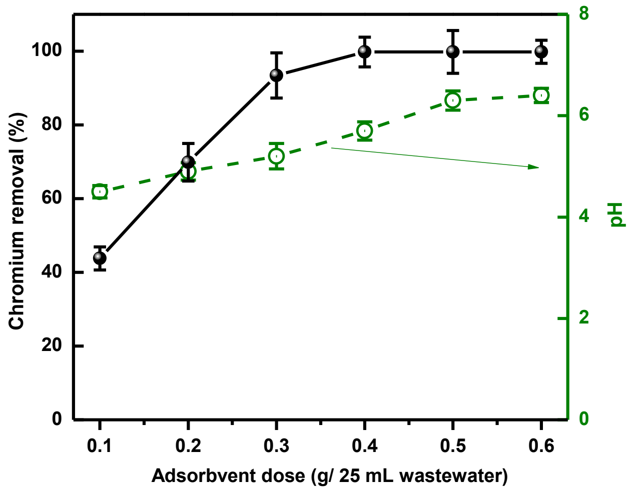

Figure 2 Changes in Cr removal (%) with *A. mexicana* adsorbent dose

The effect of *A. mexicana* adsorbent dose on the relative pH of the effluent was also monitored (Figure 2). The measured relative pH was 4.5, 4.9, 5.2, 5.7, 6.3, and 6.4 for adsorbent doses of 0.1, 0.2, 0.3, 0.4, 0.5, and 0.6 g, respectively. Here, the value of pH was also noticed to be increased with increasing adsorbent dose. As the value of pH obtained for 0.4, 0.5, and 0.6 g dose varies slightly, 0.4 g was considered optimum dose.

Effect of stirring time

Figure 3 represents the effect of stirring time on Cr removal efficiency. The highest Cr removal efficiency was calculated at 99.83% by applying 0.4 g of *A. mexicana* adsorbent into 25 mL of Cr-containing wastewater maintaining a stirring time of 20 min.

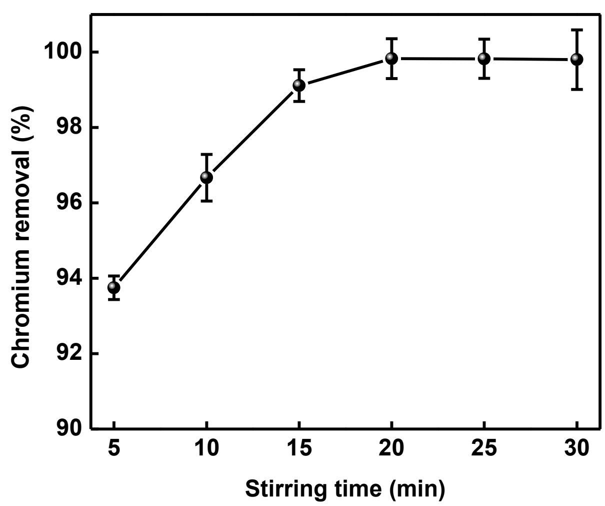

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Figure 3 Changes in Cr removal (%) with stirring time

From Figure 3, the stirring time differs from 5 to 20 min and the Cr removal efficiency was found to increase. The Cr removal efficiency maintaining 20, 25 and 30 min of stirring time obtained 99.83%, 99.82% and 99.80%, respectively. The Cr removal efficiency was found to decrease after 20 min of stirring time as a result of desorption. After considering the stirring time in terms of Cr removal efficiency, 20 min is considered the optimum stirring time during adsorption.

Effect of settling time

The effect of settling time for removal of Cr from tannery wastewater using *A. mexicana* adsorbent has been analyzed. The highest Cr removal efficiency was calculated at 99.82% by applying 0.4 g of *A. mexicana* adsorbent into 25 mL of tannery wastewater maintaining a settling time of 75 min. Figure 4 depicts the effect of settling time on Cr removal efficiency.

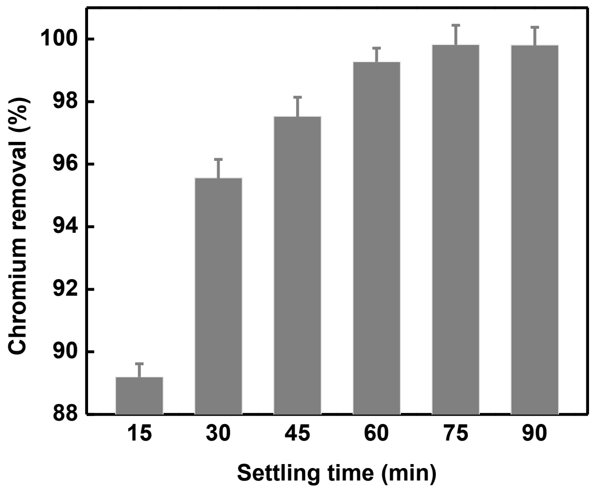

Figure 4 Changes in Cr removal (%) with settling time

From Figure 4, it can be seen that settling time varies from 15 to 75 min and the Cr removal (%) was also found to increase. The Cr removal efficiency maintaining 75 and 90 min of settling time obtained 99.82%, and 99.81%, respectively. The Cr removal efficiency slightly varies with 75 and 90 min of settling time which can be negotiated. After considering the settling time in terms of Cr removal efficiency, 75 min is considered as optimum settling time during adsorption.

Kinetic of adsorption

Pseudo 1st order kinetic

The values obtained from log (q_e - q_t) and t was plotted for Pseudo 1st order kinetic to evaluate the coefficients k_1, and R^2. The values k_1 and q_e were calculated from the slope obtained from the Pseudo 1st order kinetic (Figure 5a). The values of k_1, q_e and R^2 were obtained at 0.00033 min^{-1}, 40.271 mg/g and 0.5182, respectively (Table 1).

Pseudo 2nd order kinetic

The values of t/q_e and t were plotted for Pseudo 2nd order kinetic to evaluate the coefficients of k_2, and R^2. The value k_2 was calculated from the intercept obtained from plotting data for t/q_e and t (shown in Figure 5b). The values of k_2, q_e and R^2 obtained 0.00204 g mg^{-1} min^{-1}, 227.273 mg/g, and 0.9969, respectively (Table 1). The value of R^2 is 0.9969~1 which indicates that the adsorption mechanism can better be fitted to the Pseudo 2nd order kinetic model.

Table 1 Adsorption kinetic and isotherm study

Adsorption kinetic				Adsorption isotherm			
Pseudo 1st order		Pseudo 2nd order		Langmuir isotherm		Freundlich isotherm	
q_e (mg/g)	40.271	q_e (mg/g)	227.273	q_{max} (mg/g)	209.644	n	67.024
k_1 (min^{-1})	0.00033	k_2 (g mg^{-1} min^{-1})	0.00204	k_L (L mg^{-1})	3.9421	k_F (Lg^{-1})	224.889
R^2	0.5182	R^2	0.9969	R^2	0.5276	R^2	0.8633

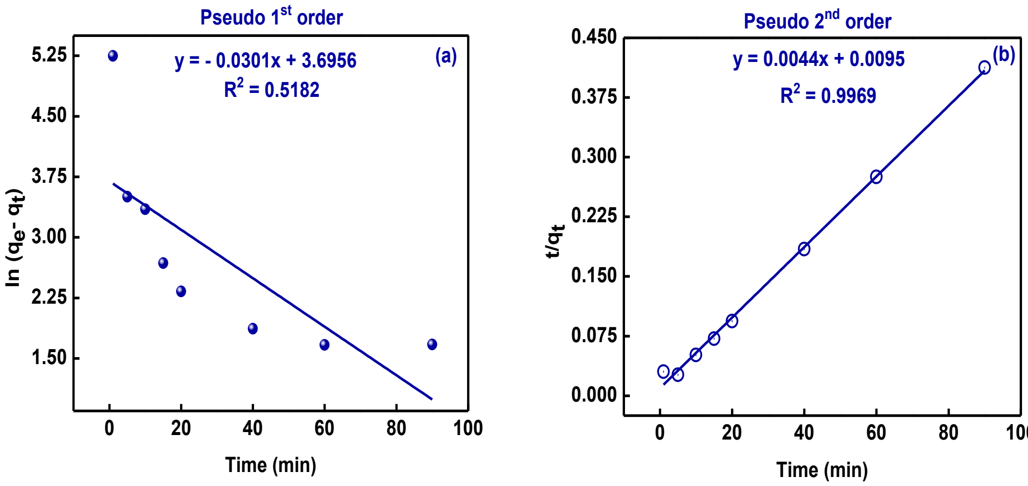

Figure 5 Pseudo 1st (a) and 2nd (b) order kinetic plotted for adsorption of Cr onto *A. mexicana* adsorbent (dose 0.4 g/25 mL)

Adsorption isotherm

Langmuir isotherm

The values of C_e/q_e and C_e were plotted in the case of the Langmuir isotherm model (shown in Figure 6a). The value of k_L was calculated from the slope and intercept obtained from the plot. The calculated values of k_L and R^2 were obtained at 3.9421 L mg^{-1}, and 0.5276, respectively (Table 1).

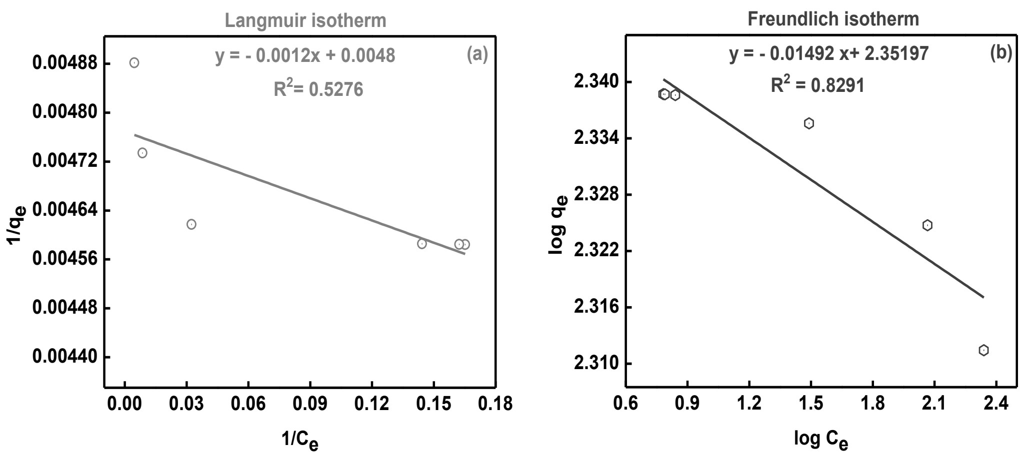

Figure 6 Langmuir (a) and Freundlich (b) isotherm model plotted for adsorption of Cr onto *A. mexicana* adsorbent (dose 0.4 g/25 mL)

Freundlich isotherm

The values of log q_e and log C_e was plotted for the Freundlich isotherm model (shown in Figure 6b). The k_F and n values were calculated from the intercept obtained from plotting data for log q_e against log C_e (shown in Figure 6b). The values of Freundlich constants k_F and n were 224.889 L g^{-1} and 67.024, respectively (Table 1). The value of R^2 for the Freundlich model was 0.8291. The values of R^2 for the Langmuir and Freundlich isotherm models were 0.5276 and 0.8633, respectively. Hence, the Freundlich isotherm model has better fitness for explaining the Cr adsorption mechanism.

pH_{PZC} analysis

The value of pH_{PZC} on the surface of *A. mexicana* adsorbent was determined 8.9 (shown in Figure 7). The value of pH_{PZC} is dependent on both chemical and electronic characteristics of the functional groups present on the surface of the adsorbent (Jiao et al. 2017). Here, the value of pH_{PZC} indicates that the surface of *A. mexicana* adsorbent was positively charged during experimentation.

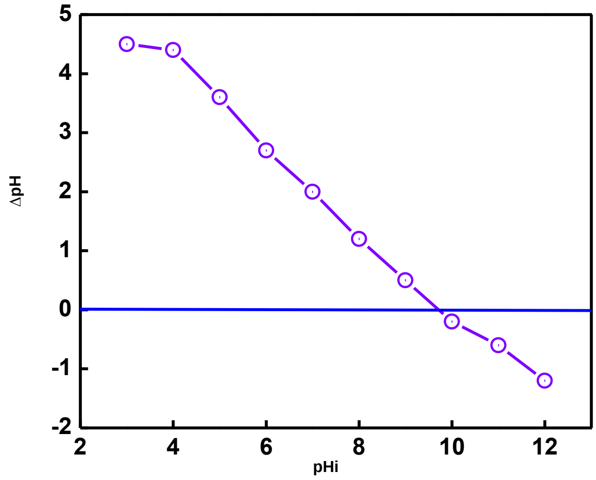

Figure 7 pH_{PZC} analysis of *A. mexicana* adsorbent

Process efficiency

Wastewater characterization was carried out before and after treatment by testing different physicochemical parameters e.g., pH, Cr content, TDS, TSS, and EC. Results obtained after adsorption employing *A. mexicana* adsorbent are represented in Table 2.

Table 2 Wastewater characterization

Parameters	Unit	Raw wastewater	Treated effluent
Cr	mg/L	3496.128	6.052
pH	-	3.9	6.4
TDS	mg/L	9870	12520
TSS	mg/L	6570	430
EC	mS/cm	23.32	29.12

From Table 2, it can be seen that the value of pH in the treated wastewater was within the discharged level after the adsorption process (ECR, 1997). About 99.80% of Cr removal was able to be achieved from this investigation. The Cr content in the treated effluent was 6.05 mg/L whereas in the raw wastewater it was around 3496.13 mg/L. the TSS was also found to be decreased after the treatment. However, other parameters, e.g., TDS, and EC were found to be increased slightly due to the incorporation of minerals from *A. mexicana* adsorbent. Thus, *A. mexicana* adsorbent can be employed to remove Cr from tannery wastewater.

CONCLUSION

Argemone mexicana adsorbent was applied to remove chromium from tannery wastewater. This investigation reveals that adsorbent prepared from the stem and leaves of *Argemone mexicana* had chromium removal efficiency (99.80%). This study also points out that adsorption could be an economical method to reduce pollution load from the chrome tanning wastewater by applying low-cost adsorbent. Preparation of *Argemone mexicana* adsorbent is comparatively easier and cost-effective. Different parameters e.g., adsorbent dose, stirring time and settling time are also determined to find out the suitable adsorption conditions. The pH_{PZC} analysis also revealed the suitability of *Argemone mexicana* adsorbent as an effective adsorbent. From adsorption kinetic and isotherm analysis, Pseudo 2nd order kinetic and Freundlich isotherm have better fitness for explaining Cr adsorption mechanism on *Argemone mexicana* adsorbent. Therefore, *Argemone mexicana* adsorbent could be easily employed in a tannery for the removal of chromium from waste chrome liquor to obtain a greener environment in a tannery.

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