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ORIGINAL ARTICLE

Adsorption of chromium (Cr) from tannery wastewater using low-cost spent tea leaves adsorbent

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Abstract

Leather tanning is consuming a large quantity of water and discharging a large volume of wastewater. This wastewater contains the high value of COD, BOD, TSS, TDS and heavy metals. In this study, spent tea leaves, a valueless waste produced during the manufacturing of tea beverage, were assessed for their potential to remove chromium (Cr) from tannery wastewater. Cr removal was studied by the batch process with varying adsorbent dose, contact time and pH of the solution to finding optimum conditions. The experiment results showed that maximum removal of Cr by spent tea leaves was 95.42% at 14 g/L of adsorbent dose and pH 10. The maximum adsorption capacity of Cr on tea waste was found 10.64 mg/g.

Keywords Tanning · Chromium (Cr) · Spent tea leaves · Adsorption · Batch process

Introduction

Tanning is a process of converting putrescible outer coverings of animals to non-putrescible leathers with definite physical, chemical and biological properties so that they can be used in our daily life and industries (Dutta 1999). In Bangladesh, the leather industry is well established and ranked fourth in terms of earning foreign exchange. This sector includes 220 tanneries, 3500 SMEs and 110 large firms of leather products controlling more than 90% of the export market (PKF 2013). The first tannery of Bangladesh established at Narayanganj by R.P. Shaha in the1940s. Later on, it was shifted to Hazaribagh area in Dhaka city. During the Pakistan period, in 1965 there were 30 tanneries in the then East Pakistan now Bangladesh (Mamun et al. 2016). The main tanning processes in Bangladesh are chrome tanning and vegetable tanning. But most of the tanneries produce chrome-tanned leather and a huge quantity of chromium is discharged with the effluents. In chrome tanning, the quantity of chromium needed for a complete tannage is about 02% or more of the weight of the hide and the fixation of the chromium is about 60–70%. Thus, about 0.5 per 1000 kg of hides tanned may be in the spent tanning solutions (Thorstensen 1999). Today, 80–90% of leathers in the world are tanned by chrome tanning (https://www.silvateam.com/en/products-and-services/leather-tanning-solutions/ecotan-tanning-processes/hybrid-chrome-tanning.html). During processing, a lot of untreated effluents discharge to the nearby water bodies which degrade the environment seriously and increase the human health risks. Chromium used in leather tanning is a heavy metal. The term ‘heavy metal’ is collectively applied to a group of metals (and metal like elements) with the density greater than 5gm/cm^{3}and atomic number above 20 (Gakwisiri et al. 2012). The concentration of heavy metals namely chromium, lead, nickel, mercury and cadmium in the aquatic environments is known to cause physiological disorders in organisms and phytotoxicity.

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Among the toxic heavy metals, chromium, in its hexavalent form, it is known to cause wide-ranging human health effects including mutagenic and carcinogenic risks (Park and Jung 2001). Chromium is used in leather tanning, dye and pigment manufacturing, the making of wood preservatives and so on. The untreated effluent from leather processing contains chromium higher than the permissible limit of 02 mg/L for tannery (DoE 1997). Chromium is an essential element involved in the normal metabolism of carbohydrate and lipid in humans. Furthermore, chromium is present in all plants, but it is unknown whether chromium is an important element for plants (Dabanovic 2016). For the general population, chromium is particularly important as a trivalent chromium, while the other forms of chromium are toxic and without function in the body (Hussain 2006). High level of Cr content was found in the surface water of Hazaribagh tanning area (939.81 mg/L) (Nur-E-Alam et al. 2017).

Tea (*Camellia sinensis*) is a very popular beverage in Bangladesh, and tea consumption of the year 2010–2014 was 57.63, 58.50, 61.19, 64.00 and 67.17 m.kg, respectively (Board xxxx). The East India Company first took the initiative to establish tea industry in Chittagong in 1840. Now the country has about 172 commercial tea estates which contribute about 3% global tea production and this industry employs more than 4 million people (Nasir et al. 2011). Once the beverage has been brewed, tea leaves discarded as waste which can be used as adsorbent for treatment of wastewater. Now tea waste generated from café, teashops or factories are discarded into environment without any treatment (Nandal et al. 2014). Some researchers have worked on different low-cost available adsorbent (Nandal et al. 2014; Dhanasekaran and Satya 2016; Ozdes et al. 2009; Wang et al. 2006; Aikpokpodion et al. 2010) for the removal of heavy metals from wastewater such as saw dust, rice husk, bagasse, tea waste. Waste mud from copper mine can be used as adsorbent for the removal of Pb (II) ions from aqueous solutions (Ozdes et al. 2009). As tea leaves consist of some functional groups associated cellulose, hemicelluloses, lignin, condensed tannins and structural proteins, these functional groups are responsible for metal uptake (Nandal et al. 2014; Wang et al. 2006; Aikpokpodion et al. 2010). Gundogdu et al. (2013) produced activated carbon from tea waste using $ZnCl_2$ as activating agent and used to remove phenol from aqueous solutions (Gundogdu et al. 2012), H_3PO_4 (Yagmur et al. 2008) and H_2SO_4 (Duran et al. 2011) can also be used as activating agent. Wastewater can be treated in various conventional treatment methods, and adsorption process is considered as effective and economical method (Lakdawala and Lakdawala 2013).

Adsorption is a sludge-free process, and it has a low investment to set up, which gives the cost-effective advantage compared to the other treatment methods (Dabanovic et al. 2016). Low-cost alternative adsorbents can be classified in two ways either (1) on basis of their availability, i.e., woo and (2) depending on their nature, i.e., inorganic or organic material (Grassi et al. 2012). This study dealt with the results of the batch process for its simplicity (Bhavsar and Patel (2014)) to establish the adsorption capacity of the spent tea leaves as the adsorbent for removal of Cr from tannery wastewater.

Materials and methods

Sample collection

Tannery effluents were collected from a tannery outlet and from a drain near the Institute of Leather Engineering and Technology (ILET), Hazaribagh, Dhaka, during the time from November 2016 to January 2017. Pre-washed plastic bottles were used for sample collection. Sampling locations:

Sample S1: Drain near ILET main gate
Sample S2: A tannery outlet
Sample S3: Drain backside of ILET

Tea waste is a cheap material which is easily available in our country. Adsorbent sample (tea waste) was collected from some local tea stalls near Leather Research Institute, Savar (Fig. 1a). So its utilization in wastewater treatment particularly in tannery wastewater would be convenient.

Preparation of the adsorbent

Waste black tea leaves were used for the treatment as adsorbent. Surface impurities were removed from tea wastes by washing with boiling water. Color was also removed by repeating washing. The tea leaves were then ovendried for 6–8 h at 105 °C. Prepared adsorbent is shown in Fig. 1b.

FTIR analysis has been used as a useful tool to identify the presence of certain functional groups of the

a b

Fig. 1 **a** Tea waste generated from a roadside tea stall and **b** prepared adsorbent bioadsorbent (Gakwisiri et al. 2012). The FTIR spectrum of STL is shown in Fig. 2 within the range of 500–4000 cm^{-1} wavenumber. The surface contains various functional groups such as peak around 3311.3 cm^{-1} in the spectrum indicates the free O–H group on the surface of the adsorbent and confirms the presence of alcohols and polyphenols in cellulose and lignin. Peaks 2919 and 2851.4 cm^{-1} assign the –CH stretching mode from the aliphatic. The spectrum around 1622.8 cm^{-1} indicates the aromatic C=C. The band appeared at 1032.1–1151.6 cm^{-1} can be due to C–O stretching in alcohols (Nur-E-Alam et al. 2017).

The TGA profile shows (Fig. 3) typical weight loss pattern for the adsorbent, and complete degradation of adsorbent starts around 300 °C and by 550 °C the degradation is complete. In the range between 40–90 °C, the weight loss is purely due to removal of moisture.

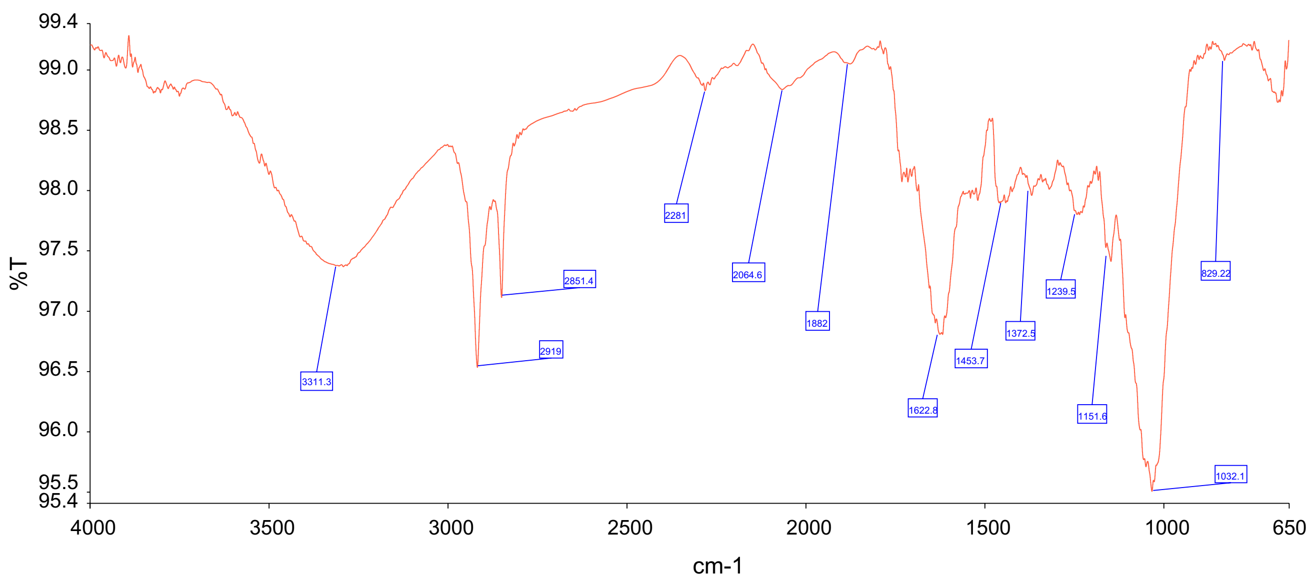

Fig. 2 FTIR spectra of spent tea leaves (STL)

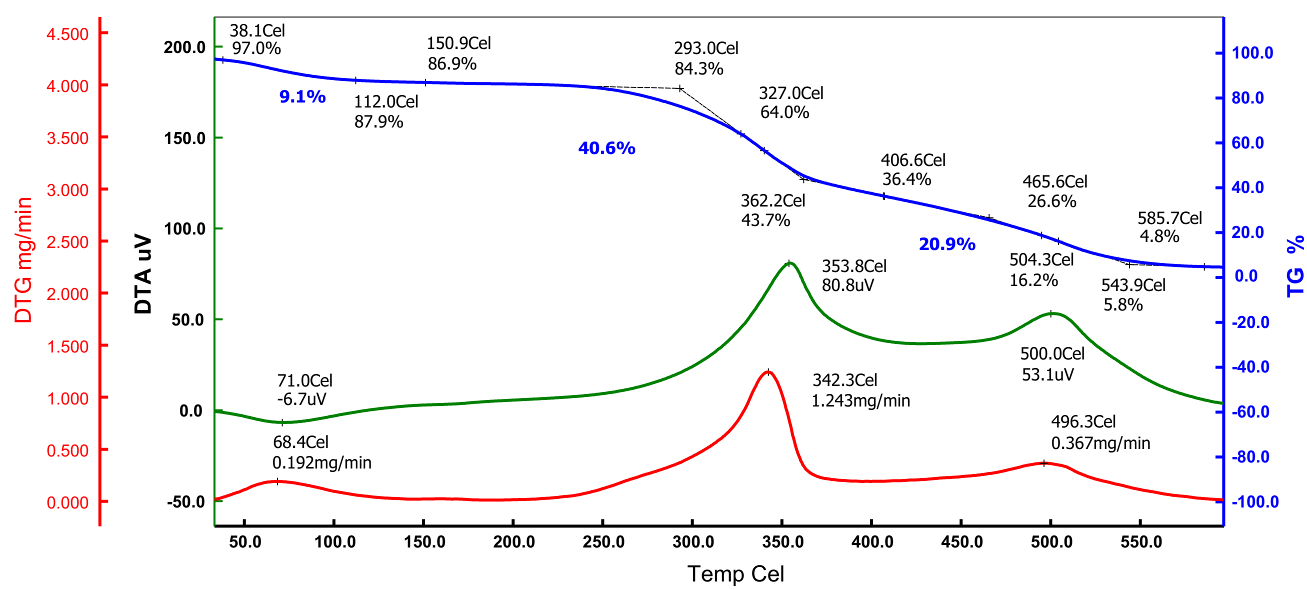

Fig. 3 TGA of spent tea leave

Experimental procedure

The study was performed in a batch process in a series of beakers equipped with stirrers by stirring the tannery effluent. The batch technique was selected for its simplicity (Bhavsar and Patel (2014)). At the end of predetermined time, the suspension will be filtered and the remaining concentration of metal ions in the aqueous phase will be measured by atomic absorption spectrophotometer (AAS). A known volume of sample effluent (250 mL) was conducted with varying adsorbent dose (03–20 g/L), contact time (30–80 min) and pH (4–10) at room temperature. The pH of the adsorptive solution was NaOH/HCl solution. Finally, Langmuir and Freundlich isotherms were used to analyze the experimental data to determine the best model that characterizes the adsorption mechanism.

Glassware and apparatus used

All glasswares used were of Borosil/Ranken. Major working instruments were automatic stirrer, digital weight balance, atomic absorption spectrophotometer, etc.

Results and discussion

The tannery effluent samples were characterized by the parameters of pH, BOD, COD and Cr (Table 1).

Effect of adsorbent dose

The effect of adsorbent dose on the adsorption process can be carried out by preparing the adsorbent–adsorbate solution with the different amount of adsorbents added to fix initial Cr concentration and shaken together for 60 min. The percentage of Cr removal is seen to increase with adsorbent dose up to 14 g/L for the samples (S1, S2 and S3). From Fig. 4, it is shown that the highest% of Cr removal was found about 91.79, 78.60 and 85.71 at the dose of 14 g/L for Samples S1, S2 and S3, respectively. It is apparent that the percentage removals of metals increase rapidly with increase in the dose of the adsorbents due to the greater availability of the exchangeable sites or surface area (Thakur and Parmar 2013).

Table 1 Characteristics of sample

Parameter	Sample S1	Sample S2	Sample S3
pH	6.5	8.2	6.2
BOD mg/L	1700	12,600	2100
COD mg/L	2490	21,060	3200
Cr mg/L	10.35	616.770	15.40

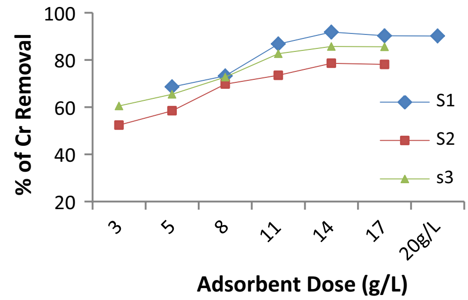

Fig. 4 Effect of adsorbent dose on Cr removal

Effect of contact time

The effect of contact time on adsorption of Cr can be carried out by preparing the adsorbent–adsorbate solution with fixed adsorbent dose (14 g/L) and initial Cr concentrations for different time intervals (30–180 min) and shaken together. The highest% of Cr removals were found in times 60, 150 and 60 min for Sample S1, Sample S2 and Sample S3 which were about 91.79, 85.4 and 88.64%, respectively (Fig. 5). The contact time required to attain equilibrium is dependent of the initial concentrations of the pollutants. The percentage of removal increases with time until equilibrium is attained for sample of same concentration.

Effect of pH

The effect of pH on the Cr reduction from wastewater is shown in Fig. 6. This study was conducted at a constant adsorbent dose of 14 g/Land different agitation period of 60, 150 and 60 min for Sample S1, Sample S2 and Sample S3, respectively. The highest% of Cr reductions were found at pH 10 for Samples S1, S2 and S3 which were about 95.42, 83.57 and 88.38%, respectively. With increasing pH, the percentage of Cr removal increases because of de-protonation of binding sites which makes different functional groups available for metal binding and vice versa (Dhabab 2011).

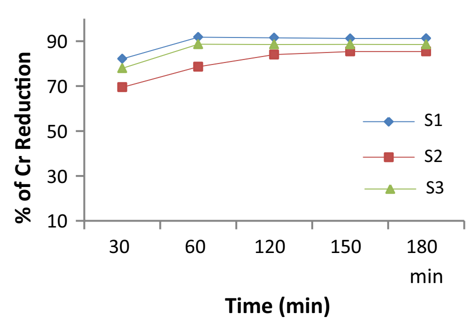

Fig. 5 Effect of contact time on % removal of Cr by tea waste adsorbent

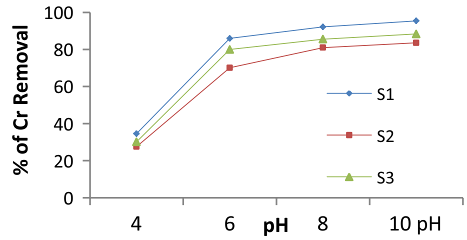

Fig. 6 Effect of pH on % removal of Cr by tea waste adsorbent

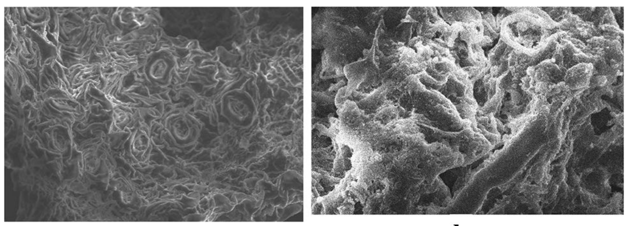

Fig. 7 SEM images of spent tea leaves before (**a**) and after treatment (**b**)

Figure 7 shows the scanning electron microscope (SEM) image of STL before and after treatment. SEM image was used to examine the surface morphologies. Gaps, slits or channels can be seen as well as get the useful information about the pore structure by SEM image (Gundogdu et al. 2013). The surface of STL was found smooth and with uniform microporous structure (Fig. 7a). After adsorption, the Cr molecules cover the most of the available pores present in spent leaves adsorbent causing the surface to become saturated as shown in Fig. 7b.

Adsorption isotherms

Adsorption isotherm is an equilibrium plot of the solid phase (q_e) versus liquid phase concentration (C_e) at fixed temperature. Freundlich and Langmuir's isotherms are the simplest known relationships which describe the adsorption equation. Freundlich and Langmuir adsorption isotherm parameters are shown in Table 2.

Freundlich model with linear plotted log q_e versus log C_e shown in the following equation;

$$\log q_e = \log K_f + 1/n \log C_e$$

where K_f is, roughly, an indicator of the adsorption capacity (mg/g), C_e is the equilibrium concentration (mg/L) and $1/n$ is the adsorption intensity. A linear form of the Freundlich expression will yield the constants K_f and $1/n$. Freundlich isotherm model assumes a non-ideal adsorption on heterogeneous surfaces in a multilayer coverage which suggests that stronger binding sites are occupied first, followed by weaker binding sites. In other words, as the degree of site occupation increases, the binding strength decreases (Jamhour et al. 2016).

Langmuir model with linear plotted $1/q_e$ versus $1/C_e$ *is* shown in the following equation:

$$\frac{1}{q_e} = \frac{1}{q_{\max}} + \frac{1}{q_{\max} K_L C_e}$$

where q_e is the equilibrium adsorbate concentration in solution; q_{max} is the maximum adsorption capacity (mg/g) which is determined from the slope; C_e is the equilibrium concentration (mg/L); and K_L is Langmuir constant related to of the binding sites and determined from the intercept(L/mg). The Langmuir isotherm model is valid for monolayer adsorption onto the surface containing a finite number of identical sorption sites, and according to this model, adsorbed molecules cannot move across the surface or interact with each other (Jamhour et al. 2016; Kord et al. 2013).

Freundlich adsorption isotherm

From the Freundlich isotherm model as shown in Fig. 8, constants obtained are as follows: adsorption capacity, K_f is −1.01 and adsorption intensity, 1/n is 0.99. The regression coefficient is 0.935.

Langmuir adsorption isotherm

The Langmuir equation was used to describe the data derived from the adsorption of Cr from the wastewater as shown in Fig. 9. The constants obtained are as follows: Langmuir

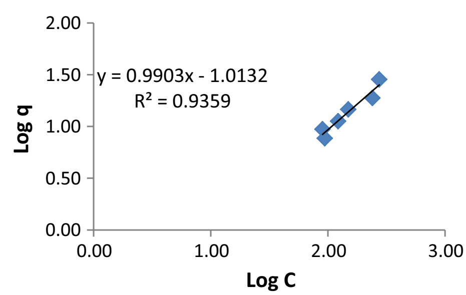

Fig. 8 Freundlich isotherm of spent tea waste

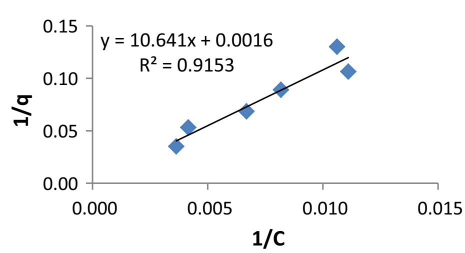

Fig. 9 Langmuir adsorption isotherm of spent tea waste

Table 2 Freundlich and Langmuir adsorption isotherm parameters

No.	Ads. dose, m, (g/L)	Eq. conc. Ceq (mg/L)	Rev. $x = C_0$–Ceq (mg/l)	q_e = V(x/m), (mg/g)	Rev. %	Log Ceq	Log q_e	1/Ceq	1/q_e
01	0	616.77	–	–	–	–	–	–	–
02	3	274.61	342.16	28.51	55.48	2.44	1.46	0.004	0.04
03	5	240.2	376.57	18.83	61.06	2.38	1.27	0.004	0.05
04	8	149.49	467.28	14.60	75.76	2.17	1.16	0.007	0.07
05	11	122.22	494.55	11.24	80.18	2.09	1.05	0.008	0.09
06	14	90.05	526.72	9.41	85.40	1.95	0.97	0.011	0.11
07	17	94.2	522.57	7.68	84.73	1.97	0.89	0.011	0.13

constant K_L is 0.002 and maximum adsorption capacity is 10.64. The regression coefficient is 0.915.

The effect of isotherm shape is discussed from the direction of predicting whether adsorption system is "favorable" or "unfavorable". Hall et al. (1966) proposed a dimensionless separation factor or equilibrium parameter, R_L, as an essential feature of the Langmuir isotherm to predict if an adsorption system is "favorable" or "unfavorable", which is defined as (Dhanakumar et al. 2007):

$$R_L = 1/(1 + bC_0)$$

where C_0= reference fluid-phase concentration of adsorbate (mg/L) (initial concentration).b = Langmuir constant, (intercept, L/mg).

The value of R_L indicates the shape of the isotherm accordingly as shown in Table 3. For a single adsorption system, C_0 is usually the highest fluid-phase concentration encountered.

The value of separation factor (R_L) for the present study is 0.448 indicating that the shape of the isotherm is favorable.

Table 3 Characteristics of adsorption Langmuir isotherm

Separation factor, RL	Characteristics of adsorption Langmuir isotherm
RL > 1	Unfavorable
RL = 1	Linear
0 < RL < 1	Favorable
RL = 0	Irreversible

Table 4 Adsorption isotherm constants and coefficient of determination

Langmuir isotherm constants			Freundlich isotherm constants		
q_{max} (mg/g)	K_L (L/mg).	R^2	K_f (mg/g)	1/n	R^2
10.64	0.002	0.915	− 1.01	0.99	0.935

From Table 4, the correlation coefficient (R^2) of Freundlich (0.935) is slightly higher than that of Langmuir adsorption isotherm (0.915).

Conclusion

Discharged heavy metals with wastewater cause many environmental and health effects. The solution would be preventing discharge of heavy metal directly into the water bodies. The conventional process of removing heavy metals has many disadvantages like high investment and operational cost, not suitable for small-scale industries. Adsorption is one the important process for removal of heavy metals from wastewater. Spent tea leaves are a cheap and available material discarded as waste material from teashops to hotels in the environment without any treatment can be converted into valuable product as adsorbent for chromium (Cr) removal from tannery wastewater. Experimental results showed that maximum removal of Cr was at optimum conditions of pH 10 and adsorbent dose of 14 g/L. Contact time is dependent on the initial effluent concentration. For Samples S1 and S3, contact time was 60 and 150 min for Sample S2. The percentage of removal increased with time until equilibrium was attained for sample of same concentration. Experimental data were justified by Langmuir and Freundlich adsorption isotherm which were 0.915 and 0.935, respectively. The maximum adsorption capacity of Cr on tea waste was found 10.64 mg/g. Based on this study, tea waste can be considered as low cost, locally and freely abundantly available, eco-friendly and efficient bioadsorbent for removal of Cr from tannery wastewater.

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Author contributions MNA and MASM carried out a major part of the literature review and drafted the manuscript. FA carried out literature review for selected sections. MNA conceived the study. MMR supervised the research project and helped to finalize the manuscript. All authors read and approved the final manuscript.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

Ethical approval Research and manuscript are original and unpublished. All authors read and approved the final manuscript.

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