ORIGINAL ARTICLE



Adaptability of Teff husk activated carbon for removal of hexavalent chromium from tannery wastewater at optimized process condition

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Received: 29 March 2020 / Accepted: 22 June 2020 / Published online: 1 August 2020 © The Author(s) 2020

Abstract

Tanneries are the major contributors to chromium pollution of the environment. Exposure to chromium (VI) can cause cancer, brain damage, central nervous dysfunction, blood composition damage; damage of lungs, kidneys, liver, and other vital organs. Although various studies have been done on the removal of Cr (VI) from synthetic wastewater, there is a lack of study in the adaptability of these methods in the treatment of real wastewater and expanding to the industrial application level. Therefore, the present study was aimed to examine the effectiveness of Teff husk activated carbon for the removal of Cr (VI) from tannery wastewater at optimized process conditions. The laboratory-based study was conducted to determine the Cr (VI) removal efficiency of Teff husk activated carbon from tannery wastewater. Data were entered and analyzed using SPSS version 20 statistical software. A paired sample *t* test was also used to test the presence of a significant difference in the mean concentration of Cr (VI) before and after treatment of the wastewater with Teff husk activated carbon. Then *p* value < 0.05 was considered as a cutoff point for the presence of a mean significant difference. The removal efficiency was found to be 89.5%. There was a significant difference in the mean concentration of Cr (VI) before and after treatment with Teff husk activated carbon was an efficient with Teff husk activated carbon was an efficient adsorbent for removal of hexavalent chromium from real wastewater. Column adsorption needs to be studied in the future.

Keywords Tannery · Wastewater · Chromium (VI) · Teff husk activated carbon

Introduction

Human activities that use water produce wastewater. Industrial wastes are generated from different processes and the amount and toxicity of waste released vary with its specific industrial processes (Belay 2010). According to the fourth World Water Development Report, only 20% worldwide, 70% in high-income countries, and 8% in low-income countries of the wastewater generated get proper treatment before discharge to the environment (Sato et al. 2013). The potential of adverse impacts of industrial pollutants on water resources, human health, and the environment depends on the toxicity, mobility, and loading of the pollutants (Connor et al. 2017).

Tanneries are the major contributors to chromium pollution of the environment. Most tanneries in the world (about 90%) use chromium salts to produce leather through the chrome tanning method, because these salts provide better leather flexibility, better water resistance, and a high shrinkage temperature. About 30% of initial chromium is not fixed by skins and remains in tanning liquor, which will be discharged as wastewater (Gebrekidan et al. 2009). The conventional chrome tanning may lead to 1500-3000 parts per million (ppm) of chromium in wastewater, whereas the recent chrome tanning method may also lead to the release of wastewater containing 500-1000 ppm of chromium (Dargo and Ayalew 2014). This is beyond the WHO tolerance limit for Cr (VI) to be discharged into inland surface water (0.1 ppm) (Kebede and Gashaw 2017). For instance, in India, about 2000-3000 tone of chromium discharges into the environment annually from tannery industries (Belay 2010).

In Ethiopia, there are more than 30 tannery industries in operation (Birhanie et al. 2017). They are the main source of chromium pollution of the environment (Birhanu 2014) and



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about 200-300 tone of chromium is estimated to be released into the environment annually from tannery industries (Amabye 2015). Most of the Ethiopian tanneries, including Bahir Dar tannery, use chrome tanning and discharges their wastewater to the nearby water bodies without adequate treatment. This may lead to the accumulation of chromium which has a significant adverse impact on aquatic flora and fauna, and public health. This can cause cancer, brain damage, central nervous dysfunction, blood composition damage; damage of lungs, kidneys, liver, and other vital organs (Abdel-Raouf and Abdul-Raheim 2017). In Ethiopia, the impact of water pollution from leather processing industries is already felt (Amabye 2015). The solution to this problem is highly significant to protect the aquatic ecosystem and human health ultimately. In recent years, the need for safe and economical methods for the removal of Cr (VI) from contaminated water has demanded research. Due to its inexpensiveness and efficient removal of metal ions, adsorption was identified as the best-suited method to be used in low-income countries such as Ethiopia for the removal of Cr (VI) from drinking and wastewater (Adane et al. 2020; Zhang et al. 2015; Desta 2013). Adsorption using activated carbons prepared from low-cost agricultural wastes such as sugarcane bagasse (Yogeshwaran and Priya 2017), Teff straw (Tadesse et al. 2015; Desta 2013), Rice husk (Mullick et al. 2018; Tripathi and Ranjan 2015; Hegazi 2013; Singh and Singh 2012), coffee husk (Berihun 2017; Teshome 2015), sawdust (Bulut and TEZ 2007), coconut shell (Devi et al. 2012), corncob (Tesfaye 2016), neem bark (Tripathi and Ranjan 2015) has been investigated for the elimination of Cr (VI) from wastewater. But most of them remain on trial with synthetic wastewater which doesn't contain competitive cations and matrices and less fitted to scale up to the industry level, and their adaptability to real industrial wastewater was not investigated. Teff (Eragrostis Teff) is a widely cultivated cereal crop mainly produced in Ethiopia and its husk is abundant, locally available, and low-cost agro-waste which can be used to prepare activated carbon for removal of Cr (VI) from contaminated wastewater (Adane et al. 2020; Tadesse et al. 2015; Desta 2013). From our previous study, Teff husk activated carbon (THAC) is effective for the removal of hexavalent chromium from synthetic wastewater, and the process parameters were optimized (Adane et al. 2020). Therefore this study was aimed to examine the effectiveness of THAC for removal of Cr (VI) from real industrial (tannery) wastewater with the presence of matrices at optimized process conditions. This is the best fit to scale up the technique to be applied at the industry level for the treatment of wastewater.

Methods and materials

Study area, period, and design

A laboratory-based experimental study was conducted at the University of Gondar from March to June 2018 Gondar, Ethiopia. The tannery wastewater was taken from Bahir Dar Tannery S.C., Bahir Dar, Ethiopia. In the city, two large tannery factories discharge their effluent to the Nile River without proper treatment and there is common chromium pollution of water bodies that is why the study has been conducted in this study area.

Preparation of THAC

Teff husk activated carbon was prepared from raw Teff husk (common agro-waste) using the chemical activation method by mixing with 1:3 w/w % of H_2SO_4 . The detailed procedure used in the preparation of THAC is described in the article we published previously (Adane et al. 2020).

Sample size determination and sampling procedures

The sample size was determined by using an environmental sample size determination formula proposed by Manly Bryan (2008), by using the equation:

$$n = \frac{4\sigma^2}{\delta^2}$$

where n = Number of samples, $\sigma =$ standard deviation, $\delta =$ acceptable level of error.

Few samples were collected for pretest and the standard deviation (σ) becomes 0.09 and taking an acceptable level of error (δ) 3.3%, the total sample size to be taken was calculated and becomes 30. Therefore, to determine the adaptability of the technique to industrial wastewater, 3 grab samples at a different time of a day at morning, midday and afternoon (at 3:00, 6:30 and 10:00 h) for 10 days in a month was taken from Bahir Dar Tannery effluent at the point of discharge of the wastewater.

N = 3 samples/day $\times 10$ days = 30 samples.

Characterization of tannery wastewater

The samples of tannery wastewater were taken from Bahir Dar Tannery Share Company and characterized for its hexavalent chromium content, pH, and temperature using the standard protocol to water and wastewater examination (APHA 2005). Hexavalent chromium was measured using



a spectrophotometer; pH was also measured using digital pH meter after calibration with buffer solutions having a pH of 4.00, 7.00, and 10.00. The temperature was also measured using a digital thermometer. Both pH and temperature were determined onsite.

Application THAC to the treatment of industrial wastewater

The adaptability of the technique enlarged with the Teff husk activated carbon for chromium (VI) removal was undertaken with some actual effluent samples. Chrome tanning effluent was collected from Bahir Dar Tannery at the discharge point.

A sampling of wastewater

1000 ml of wastewater composite sample was taken from Bahir Dar tannery effluent using polyethylene bottle which is cleaned with $10\% \text{ v/v} \text{ HNO}_3$ and rinsed with distilled water.

Adsorption from wastewater

The wastewater was treated with THAC at optimum conditions (pH=1.92, adsorbent dose=20.22 g/l, and contact time=2.07 h) with agitation speed of 200 rpm at 25 °C (Adane et al. 2020). Then the samples were filtered using Whatman No.1 filter paper. Then the filtrate was digested for further analysis.

Acid digestion of wastewater

The digestion of wastewater for both treated with THAC and untreated samples was carried out by transferring a measured volume (50 mL) of well-mixed acid preserved water sample to a flask. Then 5 mL of concentrated HNO₃ and a few boiling chips were added into the flask. The mixture was boiled and evaporated on a hot plate to the lowest volume possible (10 to 20 mL). Heating and adding concentrated HNO₃ have been continued as necessary until digestion becomes complete as shown by a light color clear solution. The sample doesn't allow to dry during digestion. Then the flasks were washed down with water and filtered. Then the filtrate was transferred into a 50-mL volumetric flask and diluted to the mark and mixed thoroughly. A portion of this solution was taken for the required metal determinations (Berihun 2017).

Oxidation of Cr (III) to Cr (VI)

Since all forms of chromium in the effluent will be oxidized to Cr (VI) with different factors to cause environmental damage, determination of total chromium as hexavalent chromium is important. Therefore, Cr (III) was oxidized to Cr (VI) using a procedure reported elsewhere (Berihun 2017), by treating the portion of digested filtrate with strong oxidizing agents (H_2SO_4 and $KMnO_4$). Then total chromium to be discharged to the environment was determined as hexavalent chromium using UV–Vis spectrophotometer as follows.

Analysis using UV–Vis spectrophotometer

For analysis of hexavalent chromium in wastewater before and after treatment with THAC was analyzed using a UV-Vis spectrophotometer by carbazide method (Gebrekidan et al. 2009; Chasteen 1993). So the concentration of the residual Cr (VI) ion in the solution has been determined by microprocessor double beam UV-Vis spectrophotometer (PerkinElmer Spectrum65) at 540 nm. A 25% w/v solution of diphenyl carbazide was prepared in 100 ml acetone. Five milliliters of each of the sample solutions, containing various concentrations of Cr (VI) (0, 2, 4, 6, 8, 10, and 12 ppm) was pipetted out into 50-ml standard volumetric flasks. To this 2 ml of 3MH₂SO₄ was added following by 2 ml of diphenyl carbazide and the total volume was made up to 50 ml using distilled water. The solutions were allowed to stand 10 min before measurement. Chromium concentration estimated by the intensity of the color complex formed was measured using a microprocessor double beam UV-Vis spectrophotometer. The absorbance was measured against a reagent blank at a wavelength of 540 nm (Emirie 2015; Alfa-Sika et al. 2010). All samples were carried out in triplicate under the same conditions and the average results were taken. The percentage removal of Cr (VI) was calculated using the formula (Magoling and Macalalad 2017):

Removal efficiency(%) = $\frac{(C_o - C_t)}{C_o} \times 100$

where C_o and C_t are the Cr (VI) concentrations in ppm initially and at a given time *t*, respectively.

Data processing and statistical analysis

Data were entered and analyzed using SPSS version 20 statistical software. For most variables, data were presented by frequencies and percentages. Descriptive analysis was done to present the mean and standard deviation of hexavalent chromium before and after treatment of the wastewater with THAC at optimized process conditions. A paired *t* test was used to test the presence of a significant difference in mean hexavalent chromium concentration before and after treatment that shows the significant removal of Cr (VI) upon treatment with THAC. Then *p* value < 0.05 was considered as a cutoff point for the presence of a mean significant difference. Then the removal efficiency of THAC was determined by using the mean concentration of Cr (VI) before and after treatment.



Data quality control

The chemical reagents in the study were standardized. The UV–Vis spectrophotometer was calibrated with seven standard solutions of hexavalent chromium (0, 2, 4, 6, 8, 10, and 12 ppm). Then the calibration curve was drawn (absorbance vs concentration of Cr (VI)) and the analysis of samples was performed when the correlation coefficient is greater than 0.99 (99%) ("Appendix"). Reproducibility was also assured by triplicate each analysis and measurements. The recovery percent was also used as quality controls to evaluate the credibility of the method of digestion and measuring instruments.

Results and discussion

Characterization of tannery wastewater and THAC

From our previous study, THAC was already prepared, characterized, and optimized its application for the efficient removal of hexavalent chromium from synthetic wastewater. The process parameters including pH, initial concentration of Cr (VI), adsorbent dose, and contact time were optimized using response surface methodology. The hexavalent chromium removal efficiency of THAC from synthetic wastewater was determined in the previous study (Adane et al. 2020). But in the most adaptable form, its application in real industrial wastewater treatment was in question. The mean pH of the effluent was 4.0 ± 0.5 and the mean temperature was also found to be 20.2 ± 2.3 °C. The mean concentration of chromium (VI) in the effluent was found to be 19.996 ppm (Table 1). It was comparable with chromium (VI) concentration in tannery effluents in Bangladesh (Roy et al. 2018;

 Table 1
 Laboratory analysis results of actual wastewater samples taken from Bahir Dar Tannery SC, Ethiopia, 2019

Parameters	Number of samples	Concentration $(mean \pm SD)$
Cr (VI) (ppm)	30	19.996±0.088
pН	30	4.0 ± 0.5
Temperature (°C)	30	20.2 ± 2.3

SD standard deviation

Kabir et al. 2017). It was also lower than hexavalent chromium concentration in effluents of Batu and Modjo tanneries (Hassen and Woldeamanuale 2017). It was much higher than the WHO recommended level of chromium (VI) to be discharged to the environment, which is 0.1 ppm (Kebede and Gashaw 2017). This high discharge of hexavalent chromium is highly toxic to living organisms even at low concentration causing a carcinogenic effect (Dargo and Ayalew 2014). This is also indicated to the need for additional treatment to minimize toxic hexavalent chromium in the effluent be for discharged to the environment.

Application of THAC onto real industrial wastewater

Upon the treatment of effluent with THAC at optimized process conditions, the mean concentration of hexavalent chromium was decreased from 19.996 ppm to 2.097 ppm (Table 2 and Fig. 1). This is also found to be comparable with the national permitted limit of chromium concentration (2 ppm) for the tannery wastewater to be discharged directly to the environment. The THAC was better in its chromium removal efficiency as compared to the already existing treatment method. Therefore, it is advisable and better to use Teff husk activated carbon to treat the discharge effluents to reduce over 89.5% of toxic chromium and to protect the human health, aquatic ecosystem, and natural environment as a whole.

The paired t test also revealed that there was a significant difference in the mean concentration of hexavalent chromium before and after treatment with THAC with t = 956.899 and p value < 0.001 (Table 3). This implies the THAC was an efficient adsorbent for the removal of hexavalent chromium from real wastewaters and reduced the concentration at a significant level. The removal efficiency was found to be 89.5%. This was consistent with other studies done using Biomass-based activated carbons as an adsorbent such as dried water hyacinth adsorbent (Kumar and Roy 2013), Gooseberry seed bio-sorbent (Aravind et al. 2016) and coffee husk activated carbon (Berihun 2017). It was found to have better efficiency than the other adsorbents such as Teff straw (Desta 2013). It was also less effective than other adsorbents such as Rice Husk activated carbon (Mullick et al. 2018), Prosopis Juliflora Plant activated carbon (Emirie 2015) and Bamboo activated carbon (Dula et al. 2014). These differences in removal efficiency may be due to the variation in

Table 2 Descriptive Statistics on the application of THAC as an adsorbent for removal of Cr (VI) and total Cr in real industrial wastewater with pH 2, adsorbent dose of 2 g/100 ml of the solution, contact time 2 h, 200 rpm, and 25 $^{\circ}$ C

Conc. of Cr (VI) (ppm)	Ν	Minimum	Maximum	Mean	Std. deviation
Before treatment with THAC	30	19.784	20.201	19.996	0.088
After treatment with THAC	30	1.952	2.259	2.097	0.051



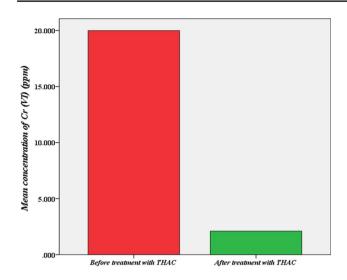


Fig. 1 concentration of hexavalent chromium in tannery effluent before and after treatment with THAC

the functional groups contained by adsorbents and the variation in the processes used for the preparation of adsorbents. The removal efficiency of THAC was also lower than its efficiency to remove hexavalent chromium from synthetic wastewater (Adane et al. 2020). This may be attributed to the presence of competitive ions and other matrices that lowers the removal efficiency of an adsorbent.

The practical implication of using THAC for removal of Cr (VI)

From the result of the current study, the application of Teff husk activated carbon on real industrial wastewater can remove above 89.5% of hexavalent chromium. This depicts that it can capable of reducing the diseases and environmental damages caused by chromium pollution with a significant amount, and can be used as a preventive measure to protect

Table 3 Paired sample t-test result on application of THAC as an adsorbent for removal of Cr (VI) in real industrial wastewater with pH 1.92, adsorbent dose of 2.02 g/100 ml of the solution, contact time 2.07 hrs, 200 rpm, and 25 °C

Table 4 Recovery tests forthe optimized procedure ofwastewater sample

human health and health of the environment. The industries should also develop a habit to use THAC for the removal of toxic chromium from industrial wastewater for the protection of human health and the environment.

Recovery percent as quality control

The recovery percent was used as quality controls to evaluate the effectiveness of the method of digestion and measuring instruments. The recovery percent was found to be about 95.35% (Table 4). This implies that the method of digestion and measuring instruments were able to retain 95.35% of hexavalent chromium that exist in the sample; only 4.65% may be missed and lost during digestion. Therefore result from this experiment is confirmed to be accurate, and the data generated were credible.

Limitation of the study

In this study the adsorption was conducted in batch adsorption manner due to the short period of the study, so the adsorption is not conducted in the column adsorption process which is better fit to design in industries. Since the sample is taken from one industry, it might not show the nature of tannery wastewater in Ethiopia.

Conclusion and recommendation

In conclusion, Teff husk activated carbon can be used as an effective, locally available, low cost, and environmentally friendly adsorbent for the removal of chromium (VI) from contaminated water. Since real industrial wastewater doesn't contain a single heavy metal, the competitive effect of other heavy metals on the Cr (VI) removal efficiency of Teff husk activated carbon should be studied. Once used up, the Teff husk activated carbon, the regeneration mechanism also needs further study.

Paired differences					t	df	Sig. (2-tailed)	
Mean	Std. deviation	Std. error mean	or mean 95% CI of difference		ce			
			Lo	wer	Upper	_		
17.899	0.102	0.019	17	.861	17.937	956.899	29	.000
Sample		Conc. in unspike sample (ppm)	ed	Amor	unt l (ppm)	Conc. in spike sample (ppm)		Mean % recovery
Untreated	d effluent reated with THAC	19.996 ± 0.086 2.097 ± 0.048		2.0 2.0		22.06 ± 0.088 4.005 ± 0.04		95.35



Acknowledgements The authors are pleased to acknowledge the University of Gondar for its unreserved contribution to the success of this study.

Author contributions TA: prepare the tools, conduct experimental analysis, analyze and interpret the data, and write up the manuscript. AD: Advice throughout the process. All authors read and approved the final manuscript.

Funding This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

Compliance with ethical standards

Ethical approval Ethical approval was obtained from the Institutional Review Board of Institute of Public Health, University of Gondar. Chrome tanning effluent samples were also collected from Bahir Dar tannery after getting appropriate permissions and oral consent from the responsible authorities.

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

Consent for publication All authors read and approved the manuscript.

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Appendix

See Table 5 and Fig. 2.

Table 5	Calibration	curve	for	Cr	(VI)	measurement	using	UV–VIS
spectrop	ohotometer							

Conc. of standards (mg/L)	Mean absorbance (%)			
0	0			
2	0.001			
4	0.001667			
6	0.003			
8	0.004			
10	0.005			
12	0.006			



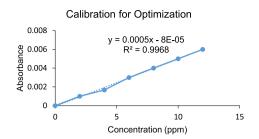


Fig. 2 Calibration curve for Cr (VI) measurement

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