



Utilization of flower waste for the removal of chromium from tannery effluent

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Abstract: In this work we used flower waste biomass as a biosorbent to remove Cr from tannery effluent through column experiments. The sorption capacities of biosorbent (Fine, coarse and rough grades) were also evaluated by employing chemical pretreatments viz., sodium hydroxide, acetic acid, glutaraldehyde and hydrogen peroxide. The order of percentage removal of Cr using the above pretreatments was: 10% hydrogen peroxide < Raw powdered-FWB < 2% Glutaraldehyde < 10% Acetic acid < 0.1N sodium hydroxide. Among the different grades of biosorbents used, fine grade adsorbed more Cr (70 %) than that of coarse (64%) and rough (62 %) sorbents. The removal percentage of Cr from tannery was analyzed by using Atomic Absorption Spectroscopy, the functional groups which are responsible for adsorption was examined by Fourier Transform- Infrared Spectroscopy and the amorphous behaviour of FWB facilitating metal biosorption was indicated by the X-ray diffractogram. This study showed that pretreated flower waste biomass is a potential sorbent of Cr, which could be successfully used to reduce the Cr content in tannery effluent.

Keywords: AAS, Chromium, Flower waste biomass, Tannery

INTRODUCTION

The increase of industrial activities by human kind has intensified stress and several contamination problems to the environment, which has resulted in deteriorated ecosystems. Now a days, heavy metals are considered major environmental contaminants and one of the most toxic pollutants due to their persistence in the environment, mainly due to their no biodegradability, which results in potential problems for public health (Vargas *et al.*, 2012). Leather industry is one of the greatest contributors towards the economy of the nation as it is one of the oldest and most practiced manufacturing industries in India. The leather manufacturing process is divided into three sub-processes: preparatory stages, tanning and crusting. Tanning is the process of converting protein of the raw skin into a flexible and stable material and a large number of different tanning materials and methods have been employed. The most commonly used tanning material is chromium. Cr forms cross-links between the collagen fibres. As a result the skin gains a good mechanical resistance, an extraordinary dyeing suitability and a better moisture resistance in comparison with skins treated with vegetable substances (Dantas Neto *et al.*, 2004).

At present two methods of Cr recovery are practiced in tanning industry: direct recycling which involves filtration of the waste water followed by chemical precipitation of Cr as chromic hydroxide; other methods include electrochemical, ion exchange, membrane filtration and electro coagulation. The above processes are

not economically feasible, because of high costs and expertise required to implement and sustain the operation. A conventional pretreatment method followed by biological processes makes it uneconomical. Biosorption is a potential attractive technology for removal of heavy metal ions that utilizes low-cost biosorbents (Fourest and Roux, 1992). Adsorption at a solid solution interface is an important means for controlling the extent of pollution due to metallic species of industrial effluents. Most of the biosorbent materials contain functional groups (Carboxyl, hydroxyl and etc.) with proteins, polysaccharides and cellulose as major constituents. Metal uptake is believed to occur through a sorption process involving the functional groups mentioned above (Hasar, 2003). Modification of a biomass using different pretreatment methods such as heating, autoclaving, freezing, drying, boiling and chemical pretreatment showed enhancement or reduction in metal biosorption (Bhatti *et al.*, 2008) as well. Hence, this study has been under taken to explore the possibility of utilizing the flower waste biomass for adsorption of Cr from tannery effluent.

MATERIALS AND METHODS

Chemicals: All reagents used in this study were of analytical grade. Sodium hydroxide, glutaraldehyde, acetic acid, hydrogen peroxide, sulphuric acid, nitric acid, per-chloric acid were obtained from Merck while Chromium atomic absorption spectrometer standard solution (1,000 mg/L) was purchased from Fluka

chemicals. All glassware and polypropylene flasks used were overnight immersed in 10% v/v HNO₃ and rinsed several times with distilled deionised water (DDW).

Biomass collection and preparation: The Flower Waste Biomass (FWB) was obtained from locally available flower markets and was extensively washed with distilled water to remove particulate material from its surface, and oven dried at 60°C for 72 hrs. Dried biomass was ground well using Willey mill. The dried biomass was sieved through Octagon sieve (OCT-DIGITAL 4527-01) to obtain homogenous biomass with desired particle size [Rough (5 mm), Coarse (3 mm) and fine (2 mm)].

Tannery effluent: Effluent coming out from tanning process (chrome tan liquor) was collected from commercial tanning industry at Dindigul and stored at 4°C without pre-treatment.

Treatment details:

T₁- Raw powdered flower waste biomass (FWB)

T₂- Pretreated (FWB) with 0.1 N Sodium hydroxide

T₃- Pretreated (FWB) with 10% Acetic acid

T₄- Pretreated (FWB) with 2% Gluteraldehyde

T₅- Pretreated (FWB) with 10% Hydrogen peroxide

Pre-treatments: For chemical treatments, 5 g of raw powdered biomass of different grades separately soaked in 100 mL of respective reagent for 1 hr as reported by Jayabalakrishnan and Mahimaraja (2007). After pretreatments the biomass was extensively washed with DDW and filtered thoroughly. Finally the resulting biomass was oven dried at 60°C for 48 h to constant weight.

Column experiment: The column experiment was carried out by using locally fabricated glass column of 50 cm height and 5 cm internal diameter (Fig.1). The wire mesh (0.1 mm) and filter paper (Whatmann No.1) was placed at the bottom of each column. The head space of the column was closed using a rubber cork with a glass tube insert for air outlet. The bottom of the column has a closed end. The outlet was connected to a conical flask for the collection of treated sample. The tannery effluent from the reservoir was allowed to pass through the column upward at constant flow rate. The effluent flow rate was standardized to get maximum pollutant adsorption. The flow rate was maintained uniformly throughout the experiment. The treated effluent was collected in a conical flask.

Atomic absorption spectroscopy (AAS): The amount of total chromium in the effluent was determined by digesting the sample with triple acid mixture (Nitric, Sulphuric and Per-chloric acid in the ratio of 9:2:1) in a hot plate for about 110°C for 2 hrs. The concentration of Cr in the digests was measured by Atomic Absorption Spectrophotometer (AAS, Varian Spectra AA 200) using air- acetylene flame (USEPA, 1979). The adsorption of Cr from tannery effluent is calculated by the difference between initial and final concentration. The adsorption percentage is also calculated by using the formula,

$$\% \text{ sorption} = (C_i - C_f) / C_i \times 100$$

C_i (mg/L) and C_f (mg/L) are the initial and final metal concentrations.

Fourier transform- infrared spectroscopy (FT-IR): The potential of chromium binding sites as well as functional groups in the biomass were examined using an FTIR spectrometer (Shimadzu) equipped with highly sensitive pyroelectric detector (DLATGS). Translucent discs were prepared by pressing the samples with KBr (spectroscopic grade) where a sample/ KBr ratio ~ 1/100.

X-ray diffraction (XRD) analysis: X-ray diffractometer (BRUKER, axs, D8 Advance) was used to determine the crystallinities of the pristine, pretreated and chromium adsorbed FWB. The scanning scope and scanning speed were 10–90° and 0.02 min⁻¹, respectively, using Cu Kα radiation operated at 40 kV and 40 mA.

RESULTS AND DISCUSSION

Characterization of the tannery effluent and flower waste biomass: Tannery effluent and FWB were characterized and the results are given in table 1.

The tannery effluent has very low pH (3.5), high EC (33.6 dS/m) and TDS (79,800 mg/L) and also contains huge Cr content (2,947 mg/L) (Table 1). It shows that more amounts of soluble salts are present in the effluent. The characteristics of tannery effluent vary considerably depending upon the size of the tannery, chemicals used for a specific process, amount of water used and type of final product produced by the tannery.

Various physical and chemical treatments were applied to enhance the Cr adsorption by the FWB, and a series of experiments were carried out with the raw and treated flower waste biomass. The pH of chrome tannery effluent (treated) was increased due to the treatment with adsorbent. The pH of the influent (raw effluent) was 3.5 which upon treatment increased up to 74 % (T₂), 50 % (T₃), and 43 % (T₄). In all the experiments, the pH varied from 3.8 to 6.4 due to various

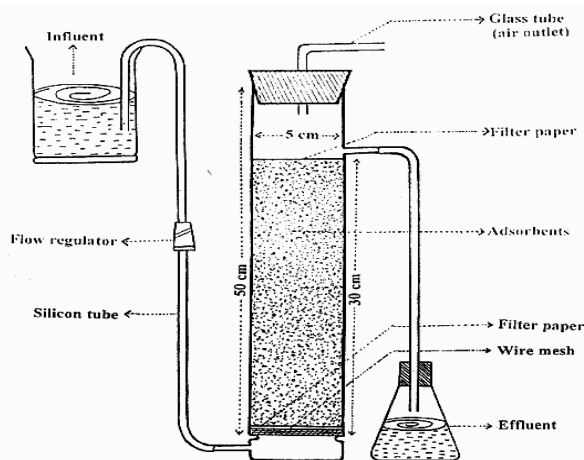


Fig.1. Schematic set-up of the glass column used in this study (Sumathi *et al.*, 2005 Analytical method for the determination of Cr).

Table 1. Physico-chemical properties of tannery effluent and flower waste biomass.

S.No.	Parameter	Tannery	Flower waste biomass
1.	Colour	Bluish green	Brown
2.	pH	3.5	7.18
3.	EC (dS/m)	33.6	1.62
4.	TDS (mg/L)	79,800	-
5.	TSS (mg/L)	570	-
6.	Total Cr (mg/L)	2,947	0.08

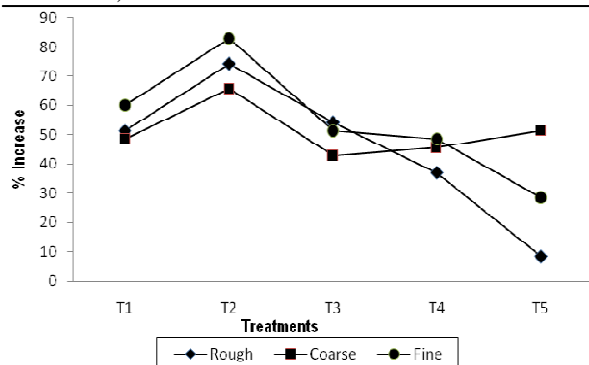


Fig. 2. Influence of flower waste biomass on pH of the tannery effluent.

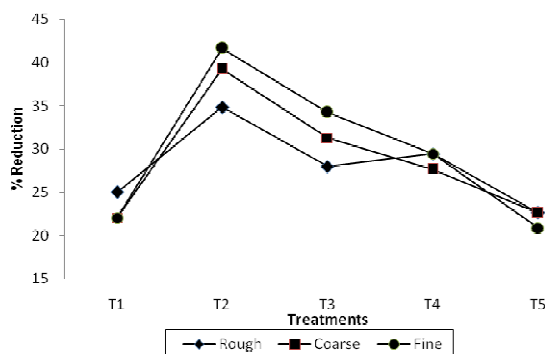


Fig 3. Influence of flower waste biomass on EC of the tannery effluent.

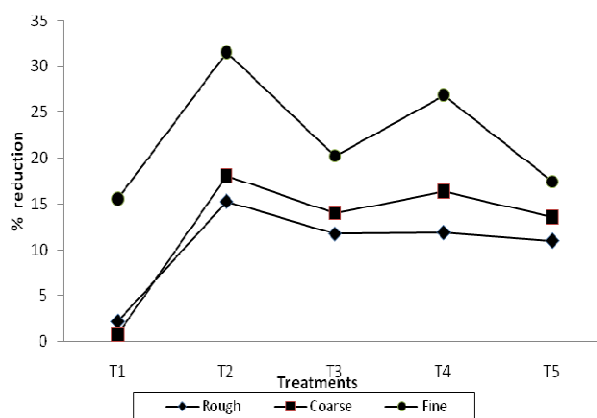


Fig. 4. Influence of flower waste biomass on TDS of the tannery effluent.

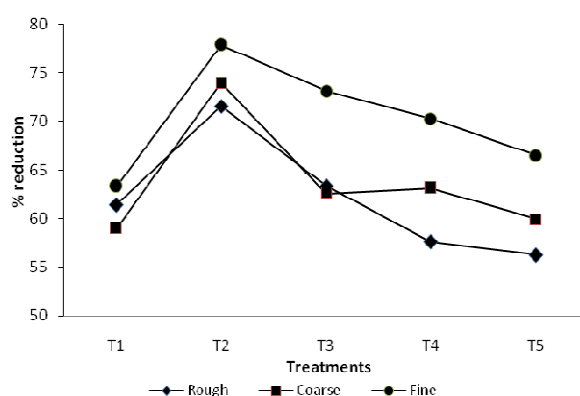


Fig. 5. Influence of flower waste biomass on chromium content of tannery effluent.

pretreatments. Among the five treatments and three grades of FWB, Pretreated- FWB with 0.1N Sodium hydroxide performed well to bring the effluent pH near neutral (Fig.2). The pH of the solution was found increased after adsorption from its original value also reported by Jayabalakrishnan and Mahimaraja (2007). The removal of metal ions from aqueous solution by adsorption depends on the pH of solution, surface charge of biosorbent, solution chemistry of metals, activity of functional group in the biomass, the degree of ionization and the species of adsorbate (Sag *et al.*, 2003). Due to high proton concentration at lower pH, metal ion biosorption decreases due to the positive charge density on metal binding sites, i.e. hydrogen ions compete effectively with metal ions for binding. The negative charge density on the cell surface of the biosorbent increased with increasing pH due to deprotonation. The metal ions then compete more effectively for available binding sites, which increased the biosorption and sorption decreased at lower pH values (Salim *et al.*, 1994).

Contradictorily the EC of the effluent gets reduced to the tune of 21 to 42 % (Fig.3). This is due to adsorption of salts on the surface of the biomass. Wide variations were observed in pH and EC between influent (raw effluent) and effluent (treated effluent) due to

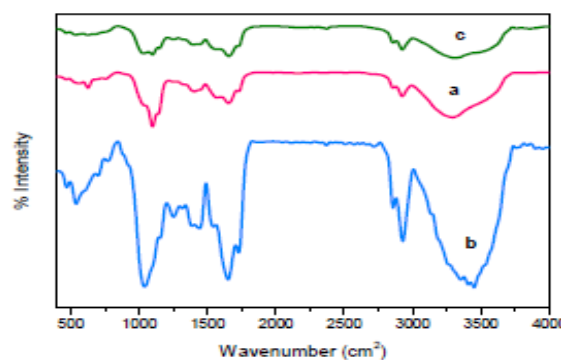


Fig. 6 FT-IR spectra of flower waste biomass (FWB) (a), Before (T2) (b) and after (T2+Cr) (c) Cr loading.

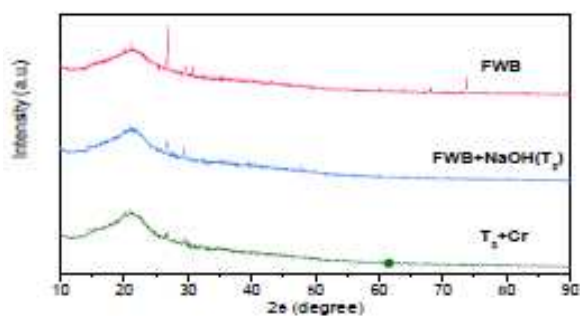


Fig. 7 X-ray diffractogram of flower waste biomass (FWB), Before (T₂) and after (T₂+Cr) Cr loading

treatment with adsorbents in columns.

Tannery effluent has higher amount of total dissolved solids (TDS) (79,800 mg/L), this might be, due to the presence of more amount of soluble salts. The chemical modification of the flower waste biomass substan-

tially reduced the TDS content (Fig.4). The reduction of TDS in industrial effluent by using herbal plant parts *viz.*, Vetiver root, Indian gooseberry bark, lemon peel and peanut husk were also found to reduce the dissolved solids effectively (Ebrahim, *et al.*, 2011). The percentage of chromium biosorption by flower waste biomass is varied from 60 to 75%. Among the treatments flower waste biomass pretreated with 0.1N NaOH (T₂) registered highest removal of 74.49% followed by 10% Acetic acid treatment (T₃) 66.37% (Fig.5). This might be due to the presence of more number of active sites on the biomass surface. Irrespective of the treatments, fine grade flower waste biomass adsorbed more number of chromium ranging from 63.32 to 77.91%. This is due to higher surface area to volume ratio and cation exchange capacity (CEC) of biomass.

In all treatments, removal of chromium varied due to

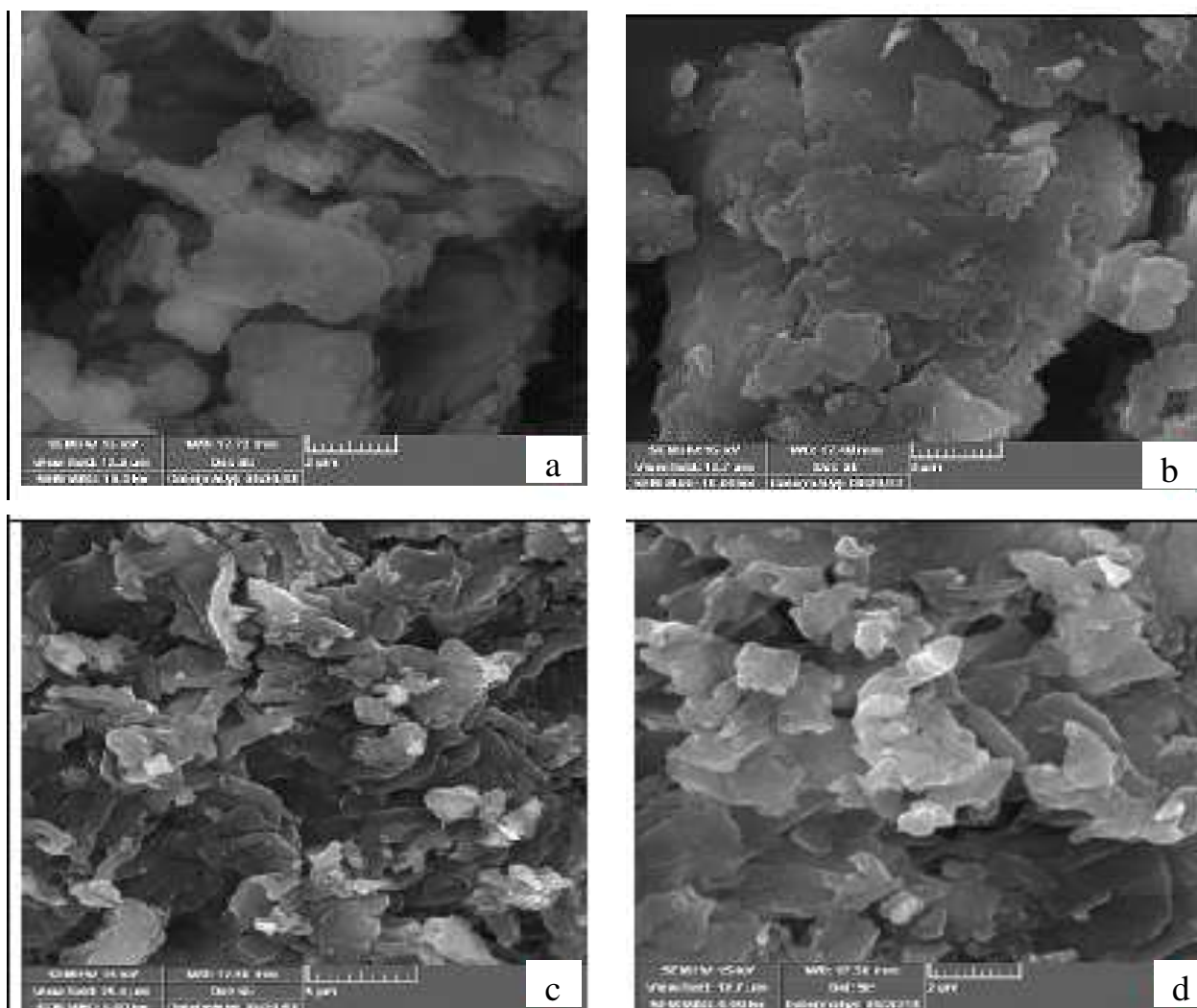


Fig. 8. SEM images of flower waste biomass (FWB) (a), FWB+ 0.1N NaOH (T₂) before Cr loading (b), after Cr loading (c) and high magnification of Cr loaded (d).

the surface and also the binding sites created by the chemical pre-treatments. From this column adsorption studies the highest adsorption of Chromium was achieved under pretreated- FWB with 0.1N Sodium hydroxide and the fine grade has the greater removal than the other forms of powdered biomass. Chemical modification of the biomass with NaOH enhanced the metal binding capacity. Metal ion binding was rapid, indicating that the metals were probably adsorbed to the cell walls of the plant tissues. As indicated previously, there have been very few reports on employing biosorbents for effluents; of which Tobin and Roux (1998) used *Mucor meihi* biomass to remove chromium from tanning industry effluents.

The percentage removal is in the increasing order of flower waste biomass pretreated with 10% Hydrogen peroxide < Raw powdered-FWB < 2% Gluteraldehyde < 10% Acetic acid < 0.1N Sodium hydroxide. Assessments revealed that the flower waste biomass exposed to alkaline supplements/salts exhibited significantly higher biosorption efficiency in comparison to untreated biomass. Whereas some of the pretreatments induced reduction in uptake efficiency and capacity of the adsorbents as compared to control (untreated biomass). It was previously observed that dried biomass generally acts as ion exchange resins and its biosorbing capacity depends on the available charges on the cell surface that bind to the biosorbent metal ions. Whereas, chemical treatments can be used to enhance the metal uptake capacity of the biomass, which led to removal, hiding or exposing chemical groups that binding or exchange with the adsorbed metal ions (Saleh *et al.*, 2009). In current research work, an increase in biosorption of Cr (VI) ions was noticed as a result of alkali pretreatments particularly NaOH. Similar enhancement in metal uptake capacity of the fungal biomass regarding alkali pretreatment was recorded by Yan and Viraraghavan (2000); El-Sayed and El-Morsey (2004) and Das *et al.* (2007). It could be due to modifications of the cell wall components by chemical pretreatments. The modification of biomass probably destroys autolytic enzymes that cause putrefaction of biomass and remove lipids and proteins that mask the reactive sites (Muraleedharan and Venkobachar, 1990). On the contrary, Kapoor and Viraraghavan (1998) reported 45% decline in adsorption of Ni(II) ions due to treatment with NaOH. They stated that deprotonation should theoretically reduce metal retention. Reduction in adsorption efficiency and capacity of adsorbent due to rest of treatment could be results of more affinity of active chemical groups to the cell wall components of the adsorbent. Hydrogen peroxide, gluteraldehyde and acetic acid pretreated FWB had slight improvement in Cr sorption. Gluteraldehyde treated FWB was better than hydrogen peroxide, due to cross linking structure with multi functional groups.

Presently, recorded reduction in sorption capacity of biosorbent for metal ions was evidenced due to acid pretreat-

ment compared to alkali. The assessments seemingly are in agreement with observations recorded in the case of *A. niger* (Kapoor and Viraraghavan, 1998) and *Mucor rouxii* (Yan and Viraraghavan, 2000) and *A. fumigatus* (Saleh *et al.*, 2009). It could possibly be explained in terms of H⁺ ions binding to the biomass after acid treatment being responsible for the reduction in adsorption of heavy metals. This indicated that the acids destroyed the absorbing groups and their positive ions (H⁺) may covalently bonded to the absorbing surfaces. Bux and Kasan (1994) have suggested that, higher the biomass electro negativity the greater would be the attraction and adsorption of heavy metal cations. Thus, the remaining H⁺ ions on the acid pretreated biomass may change the biomass electro negativity, resulting in a reduction in biosorption capacity. In contrast to that, Huang and Huang (1996) have been reported that acid pretreatment can strongly enhance the adsorption capacity of *Aspergillus oryzae* mycelia.

Comparison of the FT-IR spectra of native (Raw FWB), Pre-treated with 0.1N Sodium hydroxide (T₂) and tannery effluent treated biomass (T₂+Cr) revealed that, the peaks indicating the involvement of C=O, -NH and -OH groups (Fig. 6). The assignment of a specific stretching frequency for a functional group was unfeasible because the adsorption bands of a variety of functional groups are overlap and shift, depending on their molecular structure and environment. These Shifts in absorption positions may be caused due to different factors involved in that. These include: (1) the physical state (2) electronic and mass effects of adjacent substitutes (3) conjugation (4) intra-molecular and intermolecular hydrogen bonding and (5) ring strain (Mohan *et al.*, 2006). Although some inference can be made about the functional groups from IR spectra, the weak and broad band do not provide any authentic information about the nature of materials. The IR spectrum of FWB indicated weak and broad peaks in the region of 4000-400 cm⁻¹. Approximate FT-IR band assignment indicated the presence of carbonyl, carboxylic acids, and phenols. The 1800-1540 cm⁻¹ bands were associated with C=O stretching mode in carbonyls and carboxylic acids, while 1440-1000 cm⁻¹ band was assigned to the C-O stretching and O-H bending modes such as phenols and carboxylic acids. The aromatic stretching band assigned by around 2900-2980 cm⁻¹.

The band around 3270-3344 cm⁻¹ is the N-H stretching of aromatic compounds. The band at 1650 cm⁻¹ and 1592 cm⁻¹ corresponds to carbonyl stretching vibration of amide considered to be due to the combined effect of double bond stretching vibrations and NH deformation band for FWB, T₂ and Tannery Effluent treated with T₂ respectively. The intensities of these bands on FWB increases due to the pre-treatment with 0.1N Sodium hydroxide (T₂) and decreased with the loading of Cr from tannery effluent. In raw FWB the

peaks were obtained at 3298, 2916, 1652, 1439 and 1096 cm^{-1} for N-H stretching, C-H stretching, N-H bending (Primary amines), C-H bending (alkanes) and C=O stretching respectively. In pre-treated FWB (T_2), the intensity of the peaks enhanced and also some shifts occurred in their wave numbers due to increasing the numerous improvements in those functional groups present in the FWB. So, the peaks were obtained at the region of 3452, 2916, 1652 and 1107 cm^{-1} . This enhancement in functional groups might be increase the adsorption. After loading of Cr in the column process, the intensities of the peaks were very much reduced, because of the hydrogen ions which were present in the some of the functionalities are replaced by Cr with the presence of CrO_4^- ions. Spectra analysis after Cr biosorption showed that, there was a substantial decrease in the wave number of asymmetric stretching of the carboxylic C=O double bond from 1096 to 1042 cm^{-1} in normally. Not only for the raw FWB, had the pretreated (T_2) showed the decrease in the frequencies 1107 to 1042 cm^{-1} as well. This result indicates that, the carboxylic acid groups were likely responsible for binding Cr by biosorbent. The groups C-OH and C-O-C involved in Cr binding to some extent. Peaks in the region of lower wave numbers (under 800 cm^{-1}) appeared as a broad peak and this could be attributed to N containing bioligands. It is evidenced that from the previous results, the functional groups which can absorb metal ions are of the type OH, NH, COO^- and CO. Any changes in molecular and crystalline structure of the adsorbate due to biosorption would provide valuable information. In general, well-defined peaks are observed for the crystalline material, whereas amorphous materials show hallow peak. The XRD patterns of the FWB of before and after Cr loaded presented in fig. 7. The diffractogram of the FWB indicates the presence of a crystalline province of cellulose structure evidenced by the emergence of peaks at $2\theta \sim 21.07^\circ$, 26.85° , 30.72° , 68.10° and 73.61° . This clearly suggests that cellulose molecules are arranged in ordered lattices in which some functional groups were present in it, like O-H. In the mean time these peaks were disturbed due to the loading of Cr. The diffraction patterns of Cr loaded biomass clearly pointed out decrease in the degree of crystallinity ($2\theta \sim 20.85^\circ$, 26.65° and 29.31°) and some were not observed (68.10° and 73.61°) as well. This was due to chromium incorporation on the biomass has been reported. The decrease in crystallinity may be due to the removal of amorphous components as well as disruption of functional groups within and/or in between cellulose chains, which occur in Cr adsorption. Already we examined that, large numbers of potential binding sites *viz.*, carboxyl, hydroxyl, and amine are present on FWB surface from FT-IR studies.

Scanning electron microscopic (SEM) studies for Cr sorption: The assessment of morphological

changes as a result of chromium binding within/ surface of the pretreated FWB were done with SEM. It offers topographical and elemental information of the samples virtually. The SEM images provide modification of the samples at different stages *via.*, FWB (T_1), T_2 (before Cr loading) and $T_2+\text{Cr}$ (after Cr loading). The surface of the raw FWB was completely changed after the loading of Cr (Hana *et al.*, 2006). The surface morphology of the native FWB as shown in Fig.8a. It is evidenced that (fig. 8), the pretreatment enhances the surface functionalization as modification of its arrangement like sheets of multi layers (Yayuz *et al.*, 2006). The adsorption / sorption behavior increases due to its porous nature of after pre-treatment. The remarkable physical disintegration obtained due loading of Cr and that resulting in the emergence of protrusions and rough surface (Onwuka *et al.*, 2011). According to Fig. 8c, it revealed that, the flakes like morphology due to the loading of Cr on the pre-treated FWB (Reya *et al.*, 2013)

Conclusion

Column experiments were used to assess the effectiveness of biosorbent (flower waste biomass) in removing Cr from tannery effluent. The biosorbents pretreated with chemicals showed that among these, the pretreatment 0.1 N sodium hydroxide treatment adsorbed more Cr (75%) than other treatments. Among the biosorbents, irrespective of the pretreatments fine grade adsorbed more Cr (70%) than coarse (64%) and rough (62%). This study has, therefore, demonstrated that easily available low-cost biosorbents could be used to reduce chromium present in the tannery effluent.

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