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Research article

Harnessing potential of *Eucalyptus* derived biochar to improve physico-chemical characteristics of tannery effluent

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HIGHLIGHTS

- Biochar can be used as an effective mean for the purification of wastewater.
- *Eucalyptus* biochar significantly affects physico-chemical properties of wastewater through adsorption.

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Wastewater,
Adsorption,
Tannery effluent,
Treatment

ABSTRACT

Water pollution in Pakistan and all over the world is one of the major concerns, because it is badly affecting the living beings and reducing the chances of survival of life on the planet earth. Tannery industry with all its waste from leather products manufacturing and dyes wastes is one of the leading contributor to the water pollution, especially in developing countries like Pakistan where people are mostly unaware of the harmful drawback of their acts of today on their future life. To reduce this problem many methods and techniques have been used and are still being in process. These methods are quite expensive and cannot be used by informal industry holders. The dangerous waste materials from tannery wastewater must be treated before discharging into the environment. In the present study, tannery wastewater will be purified using *Eucalyptus* biochar (prepared at different temperatures and heating durations). The adsorption of pollutants on the surface of biochar and the extent of purification of wastewater was analyzed by comparing the difference in the values of physico-chemical parameters like: pH, TSS, TDS, COD, DO, turbidity, hardness, chlorides, calcium and magnesium using standard methods of APHA. A Multi-barrier Water Treatment System was used. The water treatment system efficiency and *Eucalyptus* biochar adsorption capacity for the wastewater pollutants was examined.

1. Introduction

In Pakistan the total quantity of wastewater annually produced is 4369 MCM, (million cubic meters). This includes 3060 MCM from municipal and 1309 MCM from industrial use. 1/3rd of this wastewater is discharged to the major rivers, which includes 1,438 MCM of municipal and 344 MCM of industrial effluents. Untreated industrial and municipal wastewater disposal has become one of the largest environmental problems in Pakistan (Arshad *et al.*, 2017; Tunio, 2013). Rivers, lakes and Fresh water reservoirs are major sources of ground and surface water, but these natural water resources are being polluted by industrial and domestic effluent (Kazi *et al*., 2009). In Pakistan, presently 596 tanneries exist in the formal sectors and equally large numbers of tanneries exist in the informal sectors (ETPI, 2001). Severe environmental degradation is caused by increase in the number of tanneries in Pakistan, because the effluents are continuously being released in nearby water reservoirs. Many environmental pollution problems can be caused by the industrial wastewater containing heavy metals (Gulnaz *et al*., 2005). About 80% of the untreated tannery wastewater is discharged directly onto open land or into water bodies (Awan, 2004). Treatment of heavy metals in wastewater by the use of low cost adsorbents has proven to be effective and simple (Verma, 2000). For thousands of years water has been treated by the use of charcoal (Kearns, 2012). In developing countries sand can be successfully used for the removal of heavy metals from waster containing low concentration of metals (Awan, 2003). Visekruna *et al.* (2011) studied the heavy metal adsorption capacities of various low cost adsorbents such as activated carbon, sand, sawdust, bark, Carbon anode dust, Zeolites. Biochar has been widely used for the enhancement of soil nutrients and plants growth, yet we need to examine its usefulness in wastewater purification as well (Regmi *et al*., 2010). High cation exchange rate and adsorption capacity make the biochar effective and efficient for sewage treatments and filtration plants (Schafford and Meiler, 2012). Most of the work reported for biochar is on targeted contaminant or either on synthetic waste however little work is done specifically on Physico-chemical parameters. Therefore present study is designed to check the potential of biochar derived from *Eucalyptus* for improving physico-chemical characteristics of wastewater.

2. Materials and Methods

2.1 Waste water Sampling

Wastewater sample used in the study was obtained from a tannery industry located in Daska near Sialkot. The sample was collected in a 2.5L container from the effluent site of the industry and brought to Rawalpindi the same day after collection. Sample was then preserved in refrigeration at 4˚C till analysis.

2.2 Wood Sampling

For biochar preparation *Eucalyptus* (*Eucalyptus urophylla*) wood was collected from a single tree from F-8 Islamabad. The selected wood was taken from small healthy branches and shredded down into smaller wood chips with a chip size smaller than 0.5 mm.

2.3 Biochar Preparation

Eucalyptus biochar was prepared by the process of pyrolysis. *Eucalyptus* wood chips were pyrolised temperature at 200˚C for two hours. Biochar was prepared at three different temperatures i.e. 300˚C, 450˚C and 600˚C for two hours respectively. After pyrolysis the biochar was taken out of the container and immediately placed in a bowl of water in order to stop any reaction with oxygen which might turn it to ash. Biochar was then dried for a day and crushed until it could pass a 2mm sieve.

2.4 Pre Treatment of the Wastewater Samples

Initially the wastewater samples were pretreated to remove the larger particles form the wastewater through sedimentation. No chemical was added to enhance the sedimentation process because it might have caused increase in chemical oxygen demand or other parameters. The sample was let to rest for a day and wastewater was separated from the settled particles using a pipette to carefully collect the wastewater from the container.

2.5 Waste Water Treatment Using Sand

Collected sand samples were washed several times with distilled water and allowed to dry for a day. 200ml jars were washed properly and air dried. 25 g of sand was measured carefully using digital balance. All three jars were filled with 25g of sand each. 100ml wastewater sample was measured using a measuring cylinder and added in each jar. The jars were sealed tightly and placed on an orbital shaker. The orbital shaker was set up at 250 rpm for 2hrs each day for a week. The samples were filtered and analyzed for parameters like pH, EC, TDS, Turbidity, Total hardness, Chlorides, COD, Calcium & Magnesium and Heavy metals (Pb, Cu, Cd).

2.6 Wastewater Treatment Using Biochar

Biochar prepared by the pyrolysis of *Eucalyptus* wood at three different temperatures i.e. 300˚C, 450˚C and 600˚C, was crushed and passed through a 2mm sieve. This biochar was then carefully weighed on a digital balance. 4.5 g of biochar of each temperature was used and 1.5 g of biochar was added into each jar. The jars were carefully washed, cleaned and air dried prior to use. 100ml of wastewater was added into each of the jars. There were a total of 9 jars. The jars were tightly sealed and placed on an orbital shaker. The orbital shaker was set up at 250rpm for 2hrs. The shaking was continued for a week. After a week the jars were taken off and wastewater was filtered using a filter paper, prior to analysis.

2.7 Physicochemical Analysis

Pre and post-treatment analysis of tannery industry wastewater samples was performed for following parameters: pH, Electrical conductivity (EC), Total Dissolved Solids (TDS), Turbidity, Total Hardness, Chemical Oxygen Demand (COD) and Chlorides. Table 1 shows detailed mean values of all the parameters under all treatments.

3. Results and Discussion

Figure 1 shows the results of pH changes between samples before and after the treatment with biochar and sand. It also shows the difference in the changes brought about by sand and biochar as well as biochar prepared at different temperatures (300 ˚C, 450 ˚C, 600 ˚C). The analysis was done after one week of treatment with sand and one week of treatment with biochar of samples. Original sample was analyzed after 24hours of the sample collection. It can be seen by the P value that the difference is significant ($P < 0.005$). This shows that both sand and biochar are effective in lowering down the pH of tannery wastewater from basic towards the neutral. The pH of the original sample was 9.64 proving to be very basic as expected from the tannery wastewater. After treatment with sand it was observed that the pH lowered to a significant level i.e 7.6, and became very close to neutral. In case of biochar, the biochar prepared at 300 ˚C lowered the pH up to 8.34, biochar prepared at 450 ˚C reduced the pH the most close to neutral among the three biochar i.e. up to 7.4, biochar prepared at 600 ˚C lowered the pH up to 8.28. From the results obtained by conducting careful analysis showed that pH was reduced from basic to very close to neutral, greater reduction was achieved by sand as compared to biochar. As pH plays a very important role in adsorption and it has been studied that the process of adsorption is highly dependent on pH, temperature and surface to volume ration of the adsorbents (Zhao *et al.,* 2008; Singh *et al*., 2009).

Trends in the change in Electro conductivity can be seen in the Fig. 2. Results from one way ANOVA shows that the P value is greater than 0.05 that proves that results are not significant.. EC trend shows that there was an increase in the EC compared to the original sample's EC in case of Biochar. In case of sand only 0.99% decrease in EC was observed i.e. from 1145 µS/cm to 1143µS/cm.

In case of biochar there was a linear increase in the value of EC (Hamer *et al.*, 2004; Nawrocki and Kasprzyk-Hordern, 2010).It can be observed from the graph that the values increased non-significantly and none of the biochar samples caused a decrease in EC. The values of EC after treatment with biochar were as follows: Biochar 300 ˚C 1192 µS/cm, biochar 450 ˚C 1201 µS/cm, biochar 600˚C 1244 µS/cm respectively.

Hence, biochar increases the EC of treated waste water, this EC increases with the increase in temperature at which biochar was prepared. However, as biochar increases the EC of waste water, similar impact of newly isolated bacterial strains was found of waste water treated with them in order to carry out the purification. This increment in EC is also correlated with the increase in water pH due to increased salt concentration (Mahmood *et al.,* 2013). Figure 3 shows the increasing trend of TDS after the treatment with biochar while the value remained constant in case of sand treatment. The results are shown to be non-significant as seen by the comparison using one way ANOVA and P value ($P > 0.05$). EC and TDS do not pose much effect on adsorption and therefore the increasing trend is not of much significance.

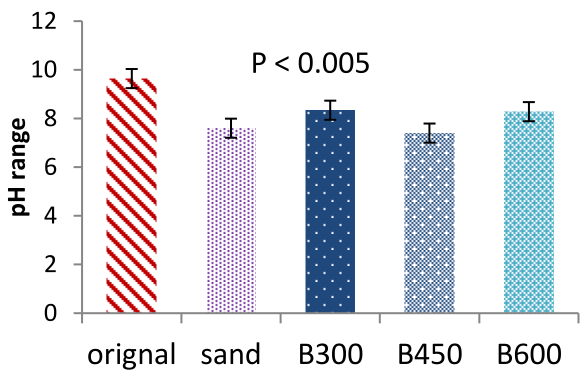

Figure 1: Trend showing the changes in pH before and after the treatment and difference in pH changes between sand and biochar samples as well as different biochar samples.

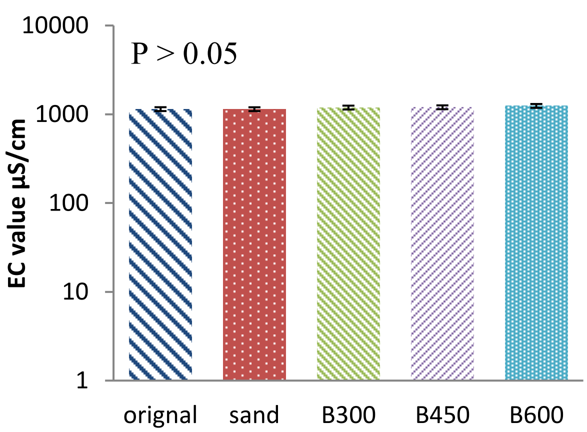

Figure 2: Comparison in means of original sample, sand treated wastewater samples and biochar treated wastewater samples, showing a trend in change of EC before and after the treatment.

Value of TDS for the original sample was 801 mg/L and it remained unchanged when wastewater sample was treated with sand for a week as seen from the results. In case of biochar treatment, the value of TDS showed a linear increase, it follows the following trend: biochar 300 ˚C gave the value 834 mg/L, biochar 450 ˚C value increased up to 839 mg/L and finally by treatment with biochar prepared at 600 ˚C the value obtained was 838 mg/L. 4%, 5.8% and 5.5% increase in the TDS value of the tannery wastewater was observed after treatment with biochar (Glass and Silverstein, 1999).

Decrease in pH mostly results in a high value of EC and TDS (WHO, 1997), and both these factors do not interfere much with the adsorption process, therefore their higher value is not of much concern (Qdais and Moussa, 2004).As seen in the results presented above, no significant changes were observed in both parameters and there was an increase in the value of both EC and TDS because both of these parameters are directly related to each other, increase in one result in the increase in the other as well. There increase followed a linear pattern, although in case of TDS there was no increase in the value after adsorption with sand.

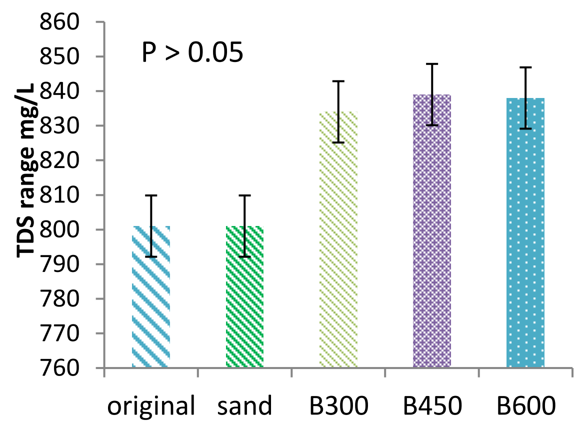

Figure 3: Mean value of TDS after the treatment with sand and wastewater compared using one way ANOVA (n=3).

A significant difference between the values of turbidity was observed before and after the treatment with the corresponding adsorbents. By the comparison of means using one way ANOVA it was found out that $P < 0.05$ that means that the adsorption was carried out in a successful way. As seen in the Fig. 4, turbidity of the original sample was 1000 FTU while sand caused a removal of 93.4% i.e. from 1000 FTU to 66 FTU of turbidity. Where as in case of biochar of various temperatures the decrease in turbidity was as follows: Biochar 300˚C decreased turbidity from 1000 to 336 FTU (66.4 %), biochar 450˚C decreased turbidity up to 241 FTU (75.9%), while biochar 600˚C caused a decrease up to 431 FTU (56.9%). Turbidity is a measure of suspended particles in the liquid and are the main cause of many diseases. Tannery wastewater contains a very high value of turbidity (Mohammed *et al*., 2004), as seen in the presented results. The original sample had a value of 1000 FTU or above and sand caused the maximum decrease in turbidity upto 93% and among the biochar, biochar prepared at 450˚C caused the maximum reduction in turbidity. Turbidity reduction by sand is a very important adsorption quality and it exceeds the adsorption capacity of biochar greatly (Eriksson *et al.,* 2002).

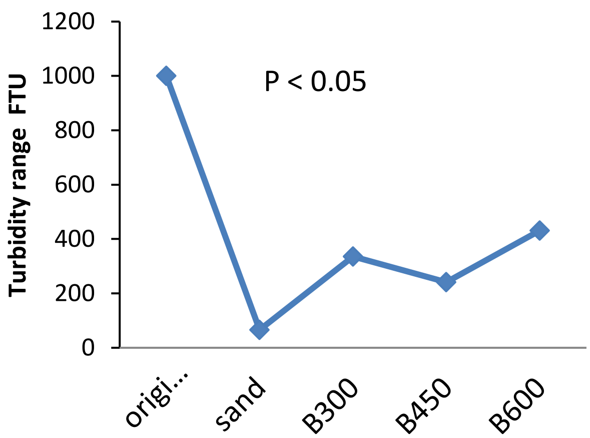

Figure 4.Changes in turbidity values before and after the treatment with the adsorbents.

Figure 5 shows the increasing and decreasing trend as well as the comparison of adsorption between sand and *Eucalyptus* biochar prepared at different temperatures. The COD value of the original sample was 6400 mg/L tannery wastewater usually has high COD. The removal of COD by the process of adsorption was analyzed using the closed reflux titrimetric method. It was found out that after adsorption by sand for a week the COD was removed up to 25.2% it got reduce to 5111.1 mg/L. By adsorption on biochar for a week, it was observed that the removal by biochar 300˚C was 61.2% and COD was reduced to 3970 mg/L, by biochar 450˚C maximum reduction in COD was observed i.e. 71.29% and value obtained was 1836.9 mg/L. Biochar prepared at 600˚C caused only 13.8% reduction and COD was 5511.06 mg/L.COD values showed a high degree of trend and comparing the adsorption of sand and biochar, biochar despite of its lesser amount proved to be a much better adsorbent of COD. Reduced COD supports the fact that biochar has better adsorption capacity compared to sand (Meriç *et al.,* 2004; Satyawali *et al.,* 2008). COD is a major factor when it comes to water quality. A lot of people have studied the COD reduction caused by adsorbents and it has been shown that biochar is an effective adsorbent (Xuejio, 2012). Only 25.2 % of COD was removed by sand while biochar removed COD up to 71.9% (Kuo, 1992).

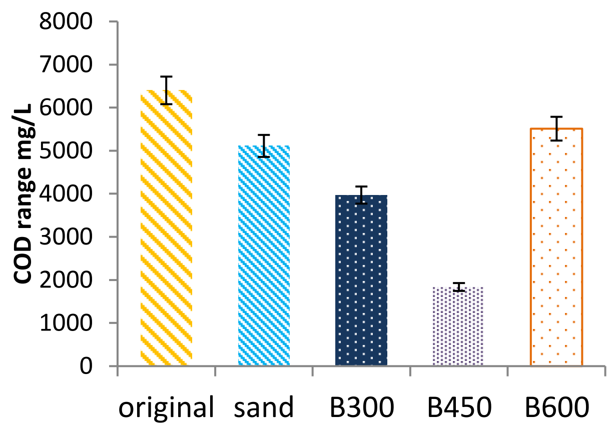

Figure 5. Trend showing difference in the COD value of the treated samples compared to the original sample.

Total Hardness trend has been shown in Fig. 6. Total hardness is the analysis in which total amount of calcium and magnesium is expressed as $CaCO_3$. Significant reduction in total hardness in tannery wastewater was observed after the treatment with sand and biochar because of the adsorption process. The value of total hardness analyzed in the original sample was 4500 mg/L. Sand samples did not cause significant decrease in the total hardness compared to biochar adsorbents. Sand only caused a reduction from 4500 mg/L to 4350 mg/L (3.3%). Reduction in total hardness after treatment with biochar adsorbents was up to 56.6% caused by biochar prepared at 450˚C the value obtained was 1950 mg/L. Biochar 300˚C reduced the total hardness to 2940 mg/L (34.6%) and 16.6% reduction was caused by biochar 600˚C, the value obtained was 3750 mg/L. Total hardness showed the same results and biochar was a more effective adsorbent compared to sand. The total hardness of the original sample was reduced up to 56.6% by biochar prepared at 450˚C compared to sand which only removed 3.3% of the total Ca and Mg ion concentration from the sample. In the case of COD, total hardness was also greatly reduced by biochar 450˚C. Same results were obtained in some other studies which used biochar to treat wastewater (Kadirvelu *et al.,* 2001; Shyamala *et al.,* 2008).

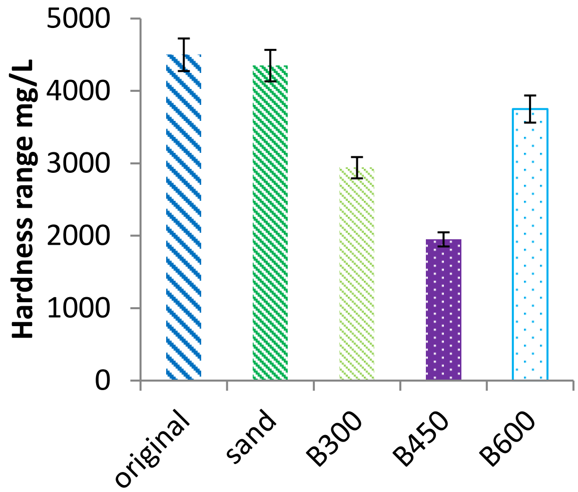

Figure 6. Trend showing reduction in total hardness before and after the adsorption process.

Argentometric titration method was used to analyze the amount of chlorides in the tannery wastewater sample before and after the adsorption process (Garg *et al.,* 2007). Very significant reduction was observed by biochar 450˚C as well as sand, whereas, biochar 300˚C and biochar 600˚C was not able to reduce a significant amount of chlorides from the wastewater. Value of chlorides obtained by analysis of the original sample was 1349.58 mg/L (53.7%). Biochar 450˚C reduced the chloride concentration up to 59.7% and the value obtained was 534.1 mg/L. Reduction caused by biochar prepared at 300˚C was 43.2% and the chloride concentration was observed to be 766.4 mg/L and in case of biochar prepared at 600˚C the chlorides were reduced to 783.06 mg/L (41.9%).Chlorides were significantly removed by both sand and biochar but in this study as well maximum reduction occurred by biochar 450˚C that was up to 59.7% and sand reduced chlorides up to 53.7%. Chlorides cause the water to become hard and hard water is not suitable for the reuse or disposal into the environment. Because sand was unable to reduce all important factors of wastewater pollution, so it cannot be considered as a good adsorbent as compared to biochar (Mullins *et al.*, 1990; Hossain *et al.,* 2009).

4. Conclusion

The results of our study clearly show the significance of the biochar for the waste water treatment. The E*ucalyptus* biochar significantly improved the physico-chemical parameters of the tannery effluent by altering the pH, EC, TDS of the waste water by the removal of pollutants through adsorption indicated by decrease in COD of the wastewater samples. At the same time the turbidity and hardness of water was reduced. However, the extent of the purification of waste water depends upon the temperature at which the biochar was prepared. Higher the temperature greater the purification as observed in our study. Hence, biochar can be used as an effective mean for the purification of waste water due to its high adsorption capacity.

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