DYE REMOVAL EFFICACY OF ADSORBENT DERIVED FROM TANNERY LIMING SLUDGE

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ABSTRACT

In leather tanning, many types of synthetic dyestuffs are used to make the leather colourful, attractive, adaptable, and fashionable. Succeeding in leather dyeing, dyeing wastewater is discharged into the water body which makes the environment polluted, especially aquatic life. In Bangladesh, every day a huge amount of dyeing wastewater is discharged from the tannery into the environment. The dyeing wastewater increases the Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD), colour, and turbidity of the receiving water body. This study describes the efficacy of adsorbent derived from the hair-burning liming sludge for the removal of dye from tannery wastewater. The derived adsorbent was characterized using Energy-Dispersive X-ray (EDX) spectroscopy, Scanning Electron Microscopy (SEM), and Fourier Transform Infrared (FT-IR) spectroscopy. Before and after the adsorption process, physicochemical characteristics of the tannery dyeing wastewater were conducted. Results reveal that the adsorption process showed the efficacy of dyestuffs, BOD, COD, and turbidity removal achieved at 84.2%, 48.0%, 36.6%, and 76.2%, accordingly. The use of the derived adsorbent from the hair-burning liming sludge for dyestuff removal is a way of waste-to-waste treatment approach.

Keywords: *Tannery wastewater, adsorbent, liming sludge, environment.*

1. INTRODUCTION

Leather industries play a substantial national economic growth for many countries e.g. India, Pakistan, Ethiopia, Bangladesh, Brazil, China, etc. (Lofrano et al. 2013; Chowdhury et al. 2013; Haydar & Aziz 2009 & Wang et al. 2014). Processing of 1-ton raw hide or skin generates 850 kg of solid waste and 30–35 m^3 of wastewater (Yoseph et al. 2020). Dyeing is one of the most frequent operations in the leather industry needed to provide the final appearance of a product. Synthetic dyestuffs are a significant source of pollution not only in the leather, and textile sectors but also in the paper and plastic industries. Since sunlight cannot penetrate the wastewater with a heavy brown colour, photosynthesis is hampered, which is harmful to aquatic life (Durai & Rajasimmam, 2011). Therefore, dyestuffs are getting into the alimentary chain and reaching human beings (Heredia et al. 2009). Due to their resistance to sunlight and other oxidizing factors, certain dyes contain complex aromatic compounds that resist degradation in traditional wastewater treatment techniques, and microorganisms (Mishra & Tripathy, 1993). Therefore, before being discharged into the environment, the effluent must go through some treatment process.

The treatment of wastewater via adsorption is important for preventing environmental contamination. Researchers have developed activated carbon, adsorption, electrochemistry, ultrasonic technology, membrane technique, and reverse osmosis to remove colour from wastewater (Tan et al. 2008). At present, tertiary treatments like nanofiltration or ozonation are also conducted after the coagulation-flocculation or adsorption process to remove almost 100% of the dye from the wastewater (Tech et al. 2016). An immobilized fungi growth technique was used to remove the dye. It is reported that 45 types or more dyestuffs were removed using 22 types of fungi with versatile immobilized support separately (Couto et al. 2009). Therefore, when a technique is chosen, numerous factors, including the financial, and efficacy, should be taken into account.

Due to the presence of high amounts of sulfide, lime, dissolved proteins, fat, suspended particles, and dissolved solids; the wastewater produced through the liming process has a distinctive brown colour and odour (Tamersit & Bouhidel, 2020). In the liming process, more than 70% of the sludge is generated (Kanagaraj et al. 2020). Each year, about 4.08×10^4 tons of liming sludge are produced in Bangladesh and they are disposed of carelessly without any treatment (Paul et al. 2013). A variety of methods have been used to handle the sludge, such as landfilling (Srivastava & Chakma, 2022), incineration (Yang et al. 2020), and the production of fertilizer (Mirmohamadsadeghi et al. 2019). However, each of these methods either tends to produce secondary pollutants or has a significant cost of upkeep (Verma & Sharma, 2020).

During the leather manufacturing process, the use of Na_2S in liming generates one of the most toxic substances, which is the largest threat to the environment because sulfide-containing wastewater discharges poisonous H_2S gas into the atmosphere (Nazer et al. 2006). Furthermore, the liming process discharges wastewater with high levels of sulfide, chemical oxygen demand, biological oxygen demand, and total suspended and dissolved particles (Jian et al. 2011). Additionally, it produces enormous quantities of solid waste that include fleshing, hair, and lime sludge (Saravanabhavan et al. 2003). However, liming wastewater from tanneries comprises organic carbonaceous residue, and it has fewer harmful components (Khambhaty et al. 2017). Due to its poor solubility, lime does not present a significant environmental risk; on the contrary, its major limitation is the emission of a large amount of lime sludge (Saravanabhavan et al. 2003). Liming sludge can be recycled into value-added products for internal tannery wastewater treatment by drying or thermal activation. The majority of the sludge–based on activated carbon, however, contains hazardous chemicals that may be to blame for secondary contamination, depending on the wastewater (Jellali et al. 2021). Compared to other adsorbents such as algae, peat, chitin, sawdust, tree barks, clays, fly ash, and biomass, the adsorbent made from tannery liming sludge has the potential to solve two major issues: (i) it will show how to manage solid waste, and (ii) it will allow for the recycling of wastewater treatment waste. This study is based on the removal efficiency of recycled alum sludge for the adsorption of dye from dyeing wastewater of a tannery in dynamic conditions.

In this study, the dye adsorption capacity was investigated of the derived adsorbent from the tannery liming sludge. The derived adsorbent was characterized using Energy-Dispersive X-ray (EDX) spectroscopy, Scanning Electron Microscopy (SEM), and Fourier Transform Infrared (FT-IR) spectroscopy.

2. MATERIALS AND METHODS

2.1 Materials Collection

The liming wastewater was collected in plastic containers from a local tannery Superex Leather Ltd. in Khulna, Bangladesh. Through coagulation, the sludge was extracted from the liming wastewater. Aluminium sulfate ($Al_2(SO_4)_3$) was gradually added to the liming wastewater through continuous stirring and pH was adjusted at the range of 8.5-9.0 to avoid the formation of hydrogen sulphide (Dixit et al. 2015). The sludge was removed from the bottom of the drum after 24 hours of settling. The liming sludge was retained in a burlap cotton towel. The sludge was separated from excess water and then dried in the sun for four days before being put in an oven set to 105°C. The dried material was removed, ground, screened, and stored in an airtight container. The dyeing wastewater was collected into a plastic container from Superex Leather Ltd. in Khulna, Bangladesh.

2.2 Reagents

Commercial aluminium sulfate was used as a coagulant. The analytical grade reagents-sodium thiosulfate, sulfuric acid, alkaline azide, manganese sulfate, starch, potassium dichromate, sulfuric acid, ferrous ammonium sulfate, and ferroin indicator were collected from the local scientific and used for this study.

2.3 Experimental system for dye removal

The adsorbent was used to remove dye from the tannery wastewater. The pH of dye wastewater was 3.5 for raw dyeing wastewater. For dye adsorption, a fixed bed glass column with a 2 cm internal diameter and 10 cm length was created. The column was prepared with 10 g of adsorbent. Glass wool was layered on either side of the adsorbent to assist support the adsorbent (Mthombeni et al. 2018). Throughout the experiment, gravity flow was used to introduce the effluent into the column at a controlled flow rate of 0.4 mL/min. After a certain period, samples taken from the column outlet were analyzed for their dye removal.

2.4 Application process

The wastewater with pH 3.5 was treated with the adsorbent made from alum sludge in dynamic conditions at the flow rate of 0.4 mL/min. The first sample was collected after 60 minutes and then each sample was taken at a 300-minute time interval. A total of 22 samples were collected and filtered. The absorbance of the eluted sample was measured by a double-beam spectrophotometer (4802, UNICO, Germany). The physicochemical parameters pH, electrical conductivity (EC), turbidity, total dissolved solids (TDS), Dissolved Oxygen (DO), and Chemical Oxygen Demand (COD) of the eluted samples were monitored. The pH of raw dye wastewater and treated liquor was measured using a calibrated pH meter (UPH–314, UNILAB, USA). After calibrating the conductivity meter (CT-676, BOECO, Germany) with a standard solution, TDS, EC, and salinity were measured.

2.4 Characterization of absorbent

The pure adsorbent and dye-loaded adsorbent were investigated employing a Fourier Transform Infrared (FTIR) spectroscopy (Perkin Elmer, USA). The recorded FT–IR spectra were within 4000 to 400^{-1} cm wavelength for the adsorbent placed on KBr discs. The pure adsorbent and dye-loaded adsorbent were analyzed for metal content with an EDX (sigma HV, Carl Zeiss Microscopy Ltd.) by coating the sample with gold. The surface morphology of pure and dye-loaded adsorbents was evaluated employing SEM (JEOL JSM-6490, USA) operated at 3kV on the gold–coated sample. Surface images were obtained at 20kV with 50000X magnification.

3. RESULTS AND DISCUSSION

1. 3.1 Characteristics of liming wastewater and dyeing wastewater

Table 1 shows the physicochemical parameters of liming wastewater before and after coagulation. The physicochemical parameters- pH, TDS, EC, turbidity, BOD, and COD values are very high and exceed the discharged limit (ECR, 2023). It seems that after coagulation, all the physicochemical parameters significantly decreased but were still beyond the permissible range. Only the pH (8.7) falls within the discharged level 6-9 (ECR, 2023). The reduction of TDS, EC, turbidity, BOD, and COD was achieved at 55.63%, 51.23%, 41.94%, 77.98, and 72.98, respectively. Although still, the parameters are several times higher than the permissible level.

Table 1: Physiochemical parameters of liming wastewater

Parameters	Liming wastewater		(ECR, 2023)	Unit
	Before treatment	After treatment		
pH	11.5±1.6	8.7±0.3	6-9	-
TDS	21.86±1.1	9.7±1.4	2.1	g/L
EC	36.7±1.6	17.9±1.3	1.2	mS/cm
Turbidity	124±3.4	72±2.09	10	NTU
BOD	12.17±2.6	2.68±2.4	0.25	g/L
COD	17.81±1.6	4.82±1.7	0.4	g/L

Table 2 represents the dyeing wastewater characterization before and after treatment. Before treatment pH, TDS, EC, turbidity, BOD, and COD of the dyeing wastewater were 3.5, 8.76 g/L, 16.15 mS/cm, 98.23 NTU, 2.07 g/L, and 5.8 g/L, respectively. It is noticeable that all monitored physicochemical parameters are beyond the permissible levels. pH was very acidic 3.5 and below the discharged level (ECR, 2023). After treatment at optimized conditions, pH was 7.1 which is within the discharged level 6-9 (ECR, 2023). The reduction of TDS, turbidity, BOD, and COD of dyeing was obtained at 10.3%, 76.2%, 48.0%, and 36.6%, respectively. However, it can be concluded that the used adsorbent affects the wastewater treatment.

Table 2: Physiochemical parameters of dyeing wastewater

Parameters	Dyeing Wastewater		(ECR, 2023)	Unit
	Before treatment	After treatment		
pH	3.5±0.6	6.5±0.5	6-9	-
TDS	8.76±0.9	5.15±1.3	2.1	g/L
EC	16.15±1.6	14.9±1.4	1.2	mS/cm
Turbidity	98.23±0.9	23.56±0.7	10	NTU
BOD	2.07±2.4	1.06±0.78	0.25	g/L
COD	5.8±2.3	3.682±1.6	0.4	g/L

3.2 Effect of solution pH on dye removal

Figure 1 depicts the effect of solution pH on the dye removal from the dyeing wastewater concerning time. The raw dyeing wastewater pH was 3.5. It seems that by increasing the solution pH, the percentage of dye removal was gradually increased. At 3.8 pH, dye removal was 44.23% after 81h. Then gradually the removal percentage was increased with increasing the solution pH. It is clear from Figure 1 that up to pH 4.5 (67.98%) dye removal was very steep and linearly increased (R^2=0.915). At pH, 6.1, the removal efficiency of dye was 80.44%. Then, the dye removal percentage slowly increased. At pH 6.5, maximum dye removal was achieved 84.2%. Mim et al. (2023) achieved the dye removal of 98.89% from the tannery wastewater in a batchwise experiment at pH 7.4. However, the higher the solution pH, the higher the dye removal percentage was obtained.

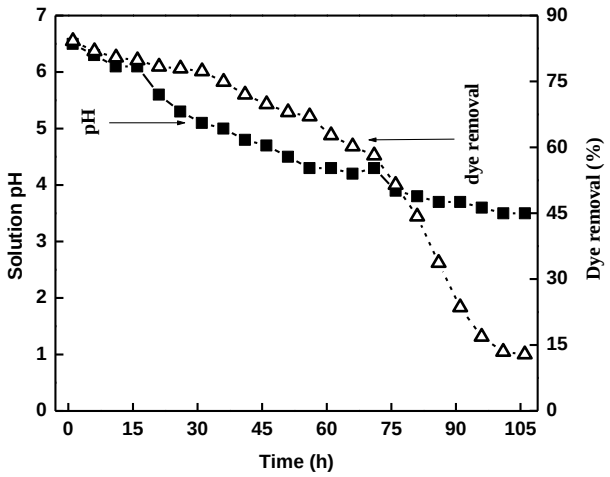


Figure 1: Effect of solution pH on dye removal with respect to time

3.3 Behaviour of TDS on dye removal

Figure 2 depicts the behaviour of TDS on dye removal with respect to time. The TDS of the raw dyeing wastewater was 8760 mg/L. In the column test, initially, the adsorbent surface is active to adsorb, therefore, the dye molecule, as well as dissolved particles, were adsorbed on the adsorbent resulting in removal efficiency being higher than the elapse of time. In the column experiment, for time, first 1 (one) h, TDS was 7730 mg/L where dye removal was 84.19%. At 11 h, TDS suddenly increased and the percentage of dye also decreased. The column test was conducted for 106 h, the adsorbent exhibited a decreased TDS at 5150 mg/L. Finally, a maximum TDS removal and dye removal was obtained at 41.2% and 12.85%, respectively. On the other hand, considering the dye removal (84.1%) after 1 h, TDS removal was only 10.3%.

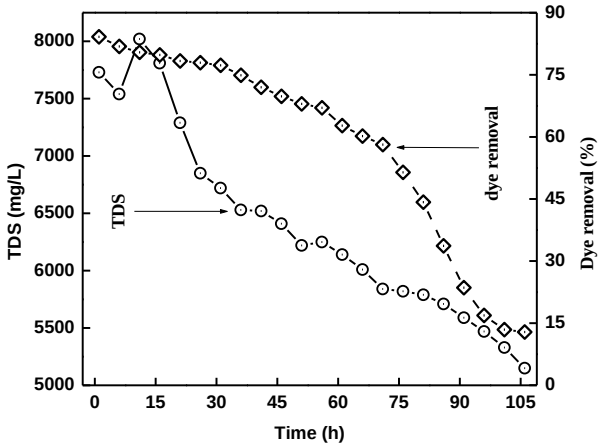


Figure 2: Behavior of TDS on dye removal with respect to time

3.4 EC analysis

Figure 3 depicts the behaviour of EC on dye removal with respect to time. The EC of raw dyeing wastewater was 16150 µS/cm. For the first 1 h, the wastewater dissolved the mineral from the adsorbent and released it to the eluate. Therefore, the EC of the wastewater was drastically increased and it was 32400 µS/cm, while dye removal was 84.19%. With time elapsed, EC was gradually decreased. At 21 h, EC was 13660 µS/cm. Hence, after 21 h, the adsorbent exhibited a maximum EC removal efficiency of 15.42% with dye removal of 78.35%. It might be the reason that some adsorbed minerals were released from adsorbent to the eluate. Nevertheless, the EC again slowly increased at 46 h and the final EC was observed at 17150 µS/cm. The column test was conducted for 106 h at that time the EC was 14920 µS/cm. Finally, the adsorbent exhibited a

maximum EC decreased to 7.62% with dye removal of 12.85%.

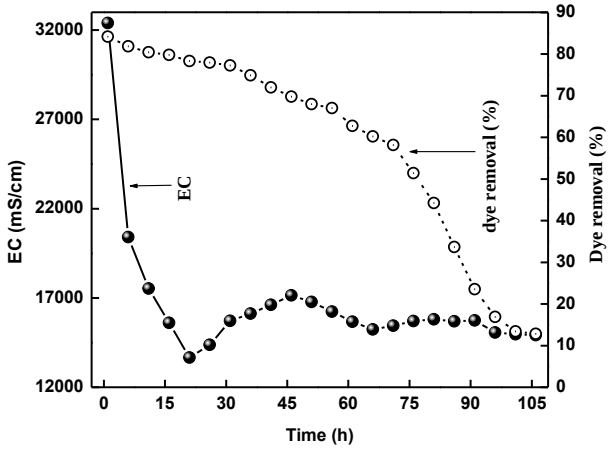


Figure 3: Behavior of EC on dye removal with respect to time

3.5 Turbidity analysis

Figure 4 depicts the variation of turbidity on the dye removal. The turbidity of the raw wastewater was 98.23 NTU, which was decreased to 39.80 NTU after 1 h. After 6 h, the turbidity was 23.10 NTU with 81.86% dye removal. This decreasing behavior was observed up to 15 h then turbidity gradually increased up to 89.40 NTU at 66 h. After 15 h, the turbidity was 10.40 NTU, which was almost 89.41% lower than the raw wastewater with 79.80% dye removal. After a certain time, responsible turbidity performer substances were released from the adsorbent as well as desorbed, therefore, turbidity was increased. After 106 h, the final turbidity was observed at 68.90 NTU which was lower than the initial turbidity.

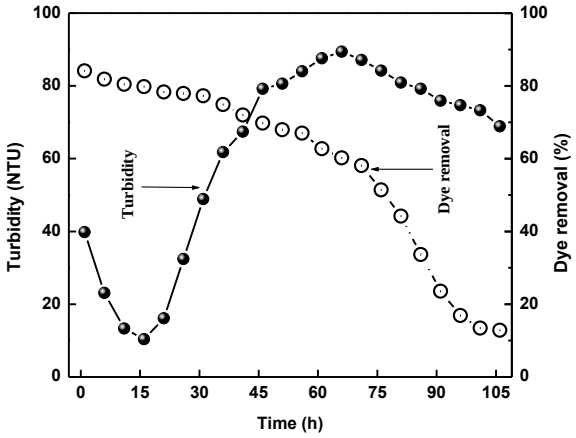
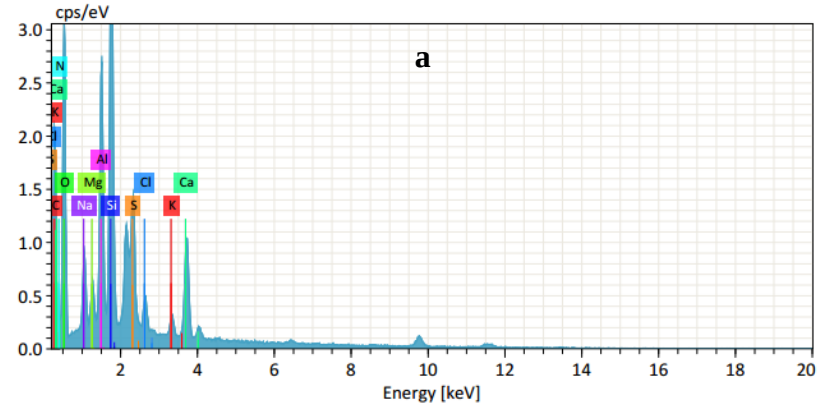


Figure 4: Variation of turbidity on dye removal with respect to time

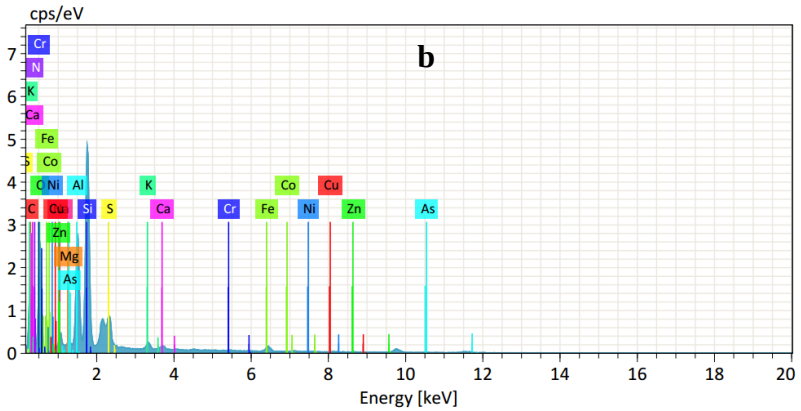
3.6 EDX analysis

Figure 5 demonstrates the EDX analysis of the prepared adsorbent before and after use. Figure 5(a) indicates that the adsorbent contains C, O, Al, Si, and Ca, possibly from organic elements and aggregate production during sludge preparation (Villalobos-Lara et al. 2021). The lime (calcium oxide, CaO) used in the liming process may be the source of the Ca. The adsorbent shows obvious changes in the peak after dye adsorption as depicted in Figure 5(b). The presence of Cr, Co, Ni, Cu, Zn, and As in the loaded adsorbent confirms the adsorption process successfully. The primary chromium sulfate used in chrome tanning processes could be one of the origins of S. (Villalobos-Lara

et al. 2021). It is evident from the shift in the Si, O, Na, and Al peaks that chemical components were involved in the adsorption process (Rambabu et al. 2020).



Spectrum	C	N	O	Na	Mg	Al	Si	S	Cl	K	Ca
1144	37.85	1.09	39.12	2.40	0.99	4.70	6.30	2.99	0.78	0.45	3.33



Spectrum	C	N	O	Na	Mg	Al	Si	S	K	Ca	Cr	Fe	Co	Ni	Cu	Zn	As
1145	41.54	0.00	35.97	1.26	0.24	6.14	10.21	2.56	0.58	0.36	0.04	0.86	0.00	0.02	0.05	0.08	0.10

Figure 5: EDX study of before adsorbent treatment

3.7 SEM Analysis

Figure 6 depicts the SEM images of pure adsorbent and dye-loaded adsorbent. The pure adsorbent is rougher and porous as shown in Figure 6(a). The dye-loaded adsorbent showed the accumulation of particles on the adsorbent surface as depicted in Figure 6(b).

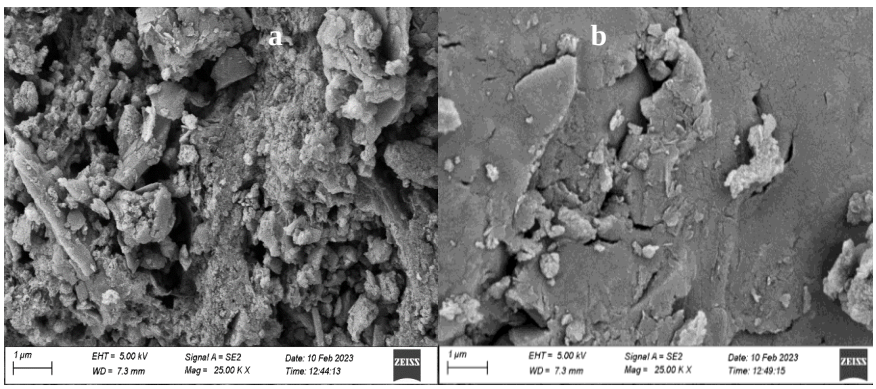


Figure 6: SEM images of pure and dye-loaded adsorbent

The pore size and uniformity of the dye-loaded adsorbent differed from the surface texture of the pure adsorbent. Comparison between the pure adsorbent and dye-loaded adsorbent, pure adsorbent, and dye–loaded adsorbent demonstrated that the adsorbent prepared from the liming sludge became less porous after the adsorption of dye. This indicated that the dye was successfully adsorbed by the adsorbent derived from the liming sludge.

3.8 FT-IR analysis

Figure 7 represents the FTIR spectrum for the pure and dye-loaded adsorbent. The pure adsorbent showed peaks at 3451 cm-1, 2931 cm-1, 2363 cm-1, 1656 cm-1, 1545 cm-1, 1033 cm-1, 783 cm-1, 693 cm-1 that represented the existence of –OH stretching, C–H stretching, O=C=O stretching, C=N stretching, N–O stretching, C–F stretching, C–H bending, C=C bending. However, the addition of new peaks was found after adsorbent activation at regions 1455 cm-1, 1151 cm-1, and 1033 cm-1 representing the existence of C–H bending, C–O stretching, and S=O stretching. The adsorption spectrum changes for the dye-loaded adsorbent. However, the addition of new peaks was found after adsorbent activation at regions 1455 cm^{-1}, 1151 cm^{-1}, and 1033 cm^{-1} representing the existence of C–H bending, C–O stretching, and S=O stretching.

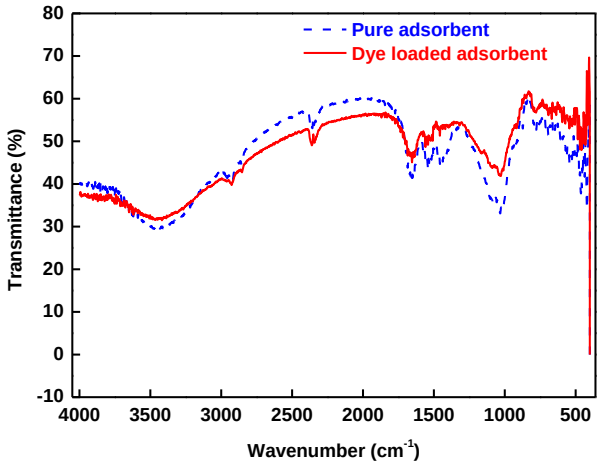


Figure 7: FT-IR spectrum of pure adsorbent and dye-loaded adsorbent

4. CONCLUSION

In this study, derived adsorbent from the tannery liming sludge was used to remove dye from tannery wastewater. FT-IR analysis reveals the shifting of functional groups before and after dye adsorption. EDX spectrum and SEM images suggest the dye adsorption. The SEM analysis also indicated that the adsorption process led to a change in the surface morphology of the adsorbents. The pure adsorbent was more porous than the dye-loaded adsorbent indicating that some dye elements accumulated on the surface adsorbent. The adsorbent exhibited a maximum dye removal efficiency of 84.19%. Also, the reduction of TDS, turbidity, BOD, and COD of dyeing was obtained at 10.3%, 76.2%, 48.0%, and 36.6%, respectively. This study not only recognizes the innovative “waste control by waste” strategy but also demonstrates how to effectively utilize the tannery liming sludge.

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