UTILIZATION OF TANNERY LIMING SLUDGE IN ACID DYESTUFF REMOVAL FROM TANNERY WASTEWATER

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ABSTRACT

Dyeing is one of the most important operations in the leather manufacturing process. Aquatic life is harmed by a large volume of wastewater that is released from the tannery following the dyeing process. Reducing the pollution load at the tannery has become extremely problematic due to the removal of acid dyestuffs from the actual dyeing wastewater. In recent times, there has been a growing interest in using tannery solid waste as a byproduct to filter out contaminants in tannery effluent. In this work, acid dyestuff has been removed from actual tannery effluent using tannery liming sludge. Thermal alteration of liming sludge was used to create an affordable adsorbent. Both before and after treatment, the adsorbent was characterized using Fourier Transform Infrared (FTIR) Spectroscopy, Energy Dispersive Spectrometer (EDS), and Scanning Electron Microscope (SEM). The parameters of adsorbent dosage, contact time, and settling time were optimized to enhance the removal of dyestuffs from tannery effluent. To eliminate dyestuff and other contaminants from the effluent, adsorption was performed without adjusting the initial pH. The pH of the treated effluent was closer to 7.0 after adsorption. The findings of the adsorbent showed that 54.83% of the dyestuff was removed at 0.2 g/50 mL of adsorbent dose for a 20-minute stirring as well as settling time. The considerable interconnectedness between the molecules of the adsorbent and dyestuff was determined via the Temkin isotherm model and pseudo-second-order (PSO) kinetics. Additionally, the procedure decreased the dyeing wastewater's chemical oxygen demand (COD) by 58.63% and its biochemical oxygen demand (BOD) by 72.41%. This work provides a cost-effective valorization route for tannery liming sludge and a practical strategy for reducing dyestuff pollution in tannery effluents.

Keywords:. *Adsorbent, Dyeing wastewater, Environment, Pollution, Solid waste.*

1. INTRODUCTION

Large quantities of solid and liquid waste are produced during leather processing, and these wastes are typically disposed of in the water bodies or dumped on land. Liming, often referred to as unhairing and liming, is one of the leather-making procedures that uses a significant amount of lime and sodium sulfide to break down the wool and hair (Hashem et al., 2017). Destructed wool, hair, epidermis, interfibrillary proteins, etc., are thus produced as solid waste. These solid wastes must be limited in order to prevent biodegradation and limit the potential management scopes (Delolme et al., 1998; Mim et al., 2018). These pollutants combine with liming water to form hazardous liming sludge that also contains a significant amount of insoluble lime. Furthermore, this phenomenon frequently results in deadly accidents because it lowers pH, which releases large amounts of hydrogen sulfide (Hashem et al., 2018). Furthermore, the breakdown of hair or wool increases wastewater parameters such as total dissolved solids (TDS), total suspended solids (TSS), chemical oxygen demand (COD), and biochemical oxygen demand (BOD), etc. (Andrioli et al., 2015; Dettmer et al., 2013). The amount of pollution produced by liming wastewater can be reduced to a certain level using liming sludge as a byproduct.

Many chemicals are used in the wet or pre-finishing stage of leather processing to achieve all the intended reactions. To provide the required quality of leather and physico-mechanical characteristics, the wet finishing process entails tanning, re-tanning, and coloring (Piccin et al., 2016). It is estimated that 12000–16,000 mL of wastewater is released for processing each kg of raw animal skins, liable for 30–50% of the tannery's inclusive environmental damage (Rajamani, 1998). Preventing proper passage of sunlight, causing acute and chronic toxicities, and creating challenges for biological treatment, dyestuffs in wastewater lower the oxygen content in aquatic bodies (Piccin et al., 2009). Most dyestuffs are aromatic chemicals that are carcinogenic, mutagenic, and non-biodegradable for aquatic life and human health, according to Vikrant et al. (2018). To lower the contamination load before releasing wastewater into the environment, dyestuffs must be removed from the actual tannery effluent.

Researchers have previously examined methods for photocatalytic degradation (Govindan et al., 2019), coagulation (Mella et al., 2017), and advanced oxidation (Sivagami et al., 2018) to remove dyestuffs from wastewater. However, their utilization in wastewater treatment is constrained by the intricate chemistry customization and the elevated costs associated with the specific application. To reduce the pollutant load, it is essential to eliminate dyestuffs from tannery effluent by developing cost-effective and environmentally sustainable technologies. Adsorption has become a favored technique for pollutant removal from wastewater because to its efficacy, cost-effectiveness, reusability, and suitability for large-scale implementation. Adsorption has recently been studied to remove dyestuff from synthetic effluent using various adsorbents (Awad et al., 2019; Mella et al., 2015). Synthetic dyeing solutions and actual tannery dyeing effluents possess distinct characteristics. Consequently, research is required to identify cost-effective and accessible adsorbents for the removal of dyestuffs from actual tannery effluent. This study employs thermal activation of tannery liming sludge to produce a cost-effective adsorbent. Pollutants from dyeing effluents in the tannery industry can be eradicated by utilizing hazardous liming sludge as an adsorbent. This inquiry will generate opportunities for the management of both liquid and solid tannery waste reduction.

This work aims to remove dyestuff from actual tannery wastewater by introducing an affordable adsorbent from tannery liming sludge. The goal of the experiment is to reduce the amount of tannery liming sludge by producing adsorbents that may remove dyestuff from wastewater. The Energy dispersive spectrometer (EDS), scanning electron microscope (SEM), and Fourier transform infrared (FTIR) spectroscopy were utilized to evaluate the resulting adsorbent both before and after treatment. Demonstrating the interaction between the adsorbent and dyestuff, the adsorption behavior was examined and contrasted using linear equations of the pseudo-first-order (PFO), pseudo-second-order (PSO) kinetics, Langmuir, Freundlich, and Temkin isotherm models. Overall, the investigation was performed to determine whether thermally activated tannery liming sludge meets the criteria of a scalable adsorbent for real tannery effluent treatment, potentially enabling waste valorization and pollution reduction.

2. METHODOLOGY

2.1 Materials

2.1.1 Wastewater Collection

The high-density polyethylene (HDPE) bottle was used to capture dyeing and liming waste from the local tannery in Jashore, Bangladesh. To prevent any contamination, clean high-density polyethylene (HDPE) bottles were used. An acid Green dyestuff (C.I. 44025, CAS 12768-78-4) was found in the wastewater collected from the tannery. Unusual suspended particles were removed from the dyeing wastewater using a filter before the research.

2.1.2 Reagents

The following reagents were used in this investigation: nitric acid (Merck, Germany), commercial aluminum sulfate, ferrous ammonium sulfate (Merck, India), magnesium sulfate (Merck, India), calcium chloride (Merck, India), sulfuric acid (Merck, India), ferric chloride (Merck, India), ferroin indicator, potassium dichromate (Merck, India), and phosphate buffer. All of these were procured from Khulna, Bangladesh.

2.2 Methods

2.2.1 Preparation of Adsorbent

Aluminium sulphate was used to treat the collected liming wastewater, causing the fines to clump into a bigger floc and making it easier to separate the liming sludge from effluent. The formed liming sludge was collected after six hours of settling. After that, the collected sludge was gradually sun-dried and oven-dried. The dried liming sludge was then pulverized after being burned for three hours at 600℃ in a muffle furnace.

2.2.2 Adsorption Experiment

A fixed amount (0.20 g) of derived adsorbent was combined with a fixed amount (50 mL) of dyeing effluent in the dyestuff adsorption experiment. Adsorption was carried out to eliminate more contaminants from the effluent without adjusting the initial pH (3.8). On a magnetic stirrer set to 500 rpm, the adsorbent-mixed wastewater was agitated for a predetermined time (20 min). After a specified amount of time (20 min) for settling, filtration was done. The impact of adsorbent dose, settling time, and stirring time were tested in batch experiments. To optimize the dose, adsorbent doses of 0.05, 0.10, 0.15, 0.20, and 0.25 g were considered for each batch (50 mL), and the settling and stirring times were unchanged. A preset period of 5, 10, 15, 20, 25, and 30 minutes was established to optimize stirring time, while other variables, including the ideal adsorbent dosage of 0.2 g, were maintained constant. A predetermined time interval of 5, 10, 15, 20, 25, and 30 minutes was established to optimize settling time, while other parameters, including the optimal adsorbent dosage (0.20 g) and stirring duration (20 min), remained constant. The absorbance obtained from the UV-Vis spectrophotometer (UNICO SQ2800, Princeton, USA) was used to calculate the concentration of dye present in raw effluent and post-treatment following adsorption. The following mathematical formula (i) was used to determine the percentage of dyestuff removal (R).

$$R\ (\%) = \frac{C_i - C_f}{C_i} \times 100 \quad (1)$$

Where "C_i" and "C_f" stand for the dyestuff concentration in mg L^{-1} for dyeing effluent prior to and following the adsorption process, respectively. Adsorption kinetic tests were conducted by adding 50 mL of raw dyeing wastewater to the ideal dose (0.20 g) of prepared adsorbent from liming sludge and continuing stirring for 5, 10, 15, 20, 25, and 30 min, respectively. The acquired data was then analyzed using the PFO and PSO equations. After adding the ideal amount (0.20 g) of adsorbent to the 50 mL of raw tannery dye wastewater and stirring for 20 min, the adsorption isotherms were investigated. Then, Origin8 software was used to examine the Langmuir, Freundlich, and Temkin isotherm models.

2.2.3 Adsorbent and Wastewater Characterization

The adsorbent characteristics were investigated using FTIR, EDS, and SEM analysis. The structural characteristics of the surface of the adsorbent was characterized via SEM (JSM-6490LA, JEOL, USA), EDS for elemental analysis, and FTIR, (Spectrum 100, Perkin Elmer, USA) for functional group study. The dyeing wastewater was assessed both before and after treatment using additional characteristics: pH, TSS, TDS, EC, COD, and BOD.

3. RESULTS AND DISCUSSION

3.1 CHARACTERIZATION OF ADSORBENT

A sophisticated technology known as SEM was utilized to examine the surface morphology of the adsorbent. The SEM image illustrates the morphology and pore architecture of the adsorbent, as depicted in Fig. 1(a). The SEM image was captured at a magnification of 10,000 times the adsorbent's dimensions to facilitate a detailed examination of the surface morphology. The liming sludge underwent thermal activation at 600ºC in a furnace, yielding a rough and porous surface for the adsorbent, as illustrated in Fig. 1(a). The adsorption of the dyestuff was significantly aided by the porosities on the adsorbent's surface. The mass and atom fraction of the element included in the adsorbent are displayed in Table 1. Fig. 1(b) displays the EDS spectra of the adsorbent, which ensures the presence of C, O, Na, Ca, Al, K, Cl, and S. Table 1 shows that the mass of carbon was approximately 11.49% and the atomic percentage was 17.29%. According to Sivaranjanee and Kumar (2021), the high percentage of Carbon makes the adsorbent suitable for adsorbing pollutants, e.g., dyestuffs and heavy metals. The other elements, such as O, Na, Al, Ca, S, Cl, and K, have relative mass percentages of 56.77%, 1.95%, 15.04%, 10.72%, 2.30%, 1.64%, and 0.09%. When liming sludge was collected, aluminum may originate from the aluminum sulfate; however, throughout the liming process, the adsorbent may contain sodium and calcium.

Table 1. Elemental composition of the adsorbent

Element	KeV	Mass (%)	Atom (%)
C	0.277	11.49	17.29
O	0.525	56.77	64.11
Na	1.041	1.95	1.53
Al	1.486	15.04	10.07
S	2.307	2.30	1.29
Cl	2.621	1.64	0.84
K	3.312	0.09	0.04
Ca	3.690	10.72	4.83

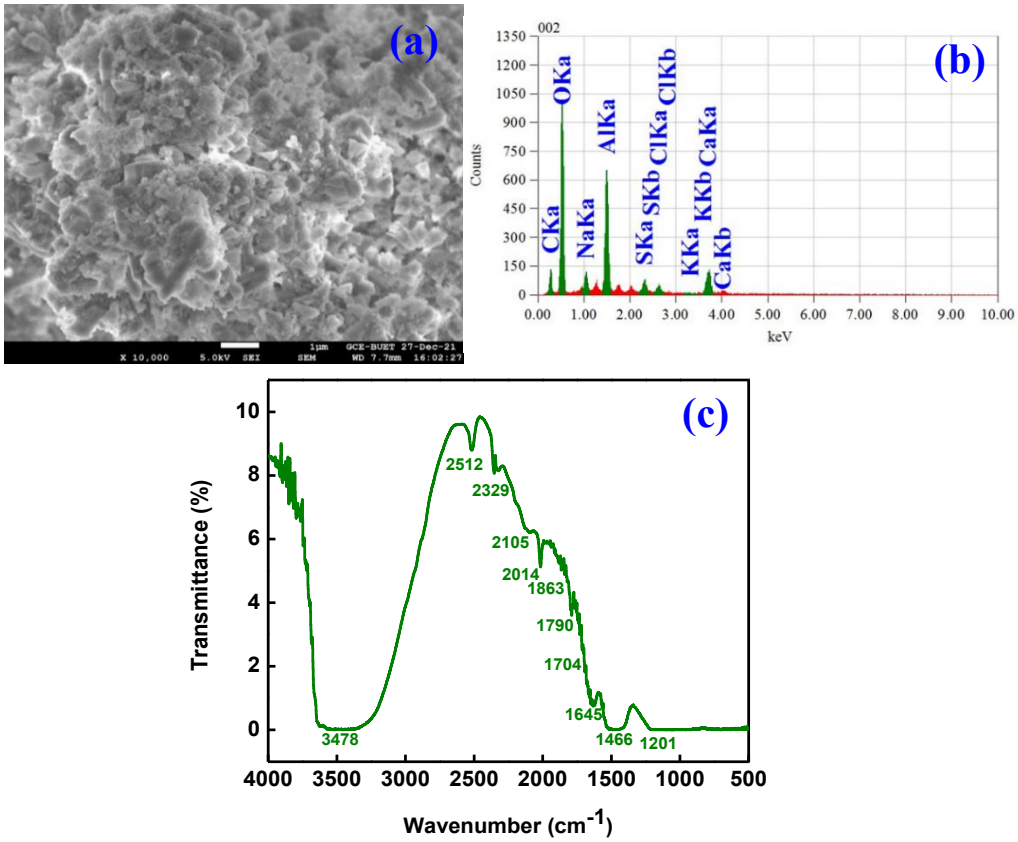

Figure 1: (a) SEM, (b) EDS, and (c) FTIR analyses of the adsorbent from liming sludge

The functional groups present within the adsorbent in the 4000-400 cm^{-1} range were identified by FTIR analysis (FTIR Spectrum 100, Perkin Elmer), displayed in Fig. 1(c). The presence of alcohol, ether, ester, and carboxylic acids is indicated by a broad band at 1201 cm^{-1} in the adsorbent's infrared spectrum, which represents the presence of C–N and C–O functional groups. The N=O functional group, which represents the nitro ($R–NO_2$) compounds present in the adsorbent, is indicated by the peak present at 1466 cm^{-1}. The stretching vibration of the C=N functional group could be the cause of a band observed at 1645 cm^{-1}. Possible stretching of the C=O functional groups of carboxylic acids is represented by a band at 1704 cm^{-1} in the adsorbent (Hienerwadel et al., 1997). The peaks observed at 2105 cm^{-1} and 2512 cm^{-1} denote alkyne-representing C-C group and the carboxylic acid-representing O-H group, respectively. N-H and O-H functional groups, which stand for primary and secondary amine, amide, and alcohol, phenol, respectively, are seen in the peak that is centered at 3478 cm^{-1} (Nagaraj et al. 2018). The prepared adsorbent from liming sludge has a good adsorption ability due to having sufficient functional groups, as shown in Fig. 1(c). According to Liu et al. (2010), the more functional groups facilitate simpler access of present dyes to the available sites of absorbent throughout the dyestuff's adsorption process.

3.2 Effect of Investigating Parameters

One of the most crucial factors of an adsorbent is how a given quantity of adsorbent can treat wastewater (Bulut and Aydın, 2006). The influence of adsorbent dose helps determine the potentiality of dyestuff from an economic standpoint and gives information on the amount of dyestuff that can be

adsorbed with the least amount of adsorbent (Yagub et al., 2014). Fig. 2 illustrates the results of an analysis conducted at ambient temperature (>25℃) for determining the possible impact of the adsorbent dosage on removing the dyestuff from tannery dyeing effluent. The dyestuff removal efficiency (%) increases as the adsorbent dosage rises because of additional sorption sites present on the adsorbent's surface as its amount increases (Kannan and Sundaram, 2001). Fig. 5 shows that the dyestuff removal efficiency rose from 20.03% to 54.83% when the adsorbent dose was raised from 0.05 g to 0.20 g. When 50 mL of tannery raw dyeing effluent was treated with an adsorbent dosage of 0.20 g, a maximum dyestuff elimination efficiency of 54.83% was achieved. The dyestuff removal efficiency was 54.83% and 54.90%, respectively, using 0.20 g and 0.25 g. It is possible to overlook the tiny deviation in the dyestuff removal efficiency in this instance while using an adsorbent dosage of 0.25 g. The ideal adsorbent dose was determined to be 0.20 g after taking into account the percentage amount of dye removal in relation to the adsorbent dosage. Using adsorbent doses of 0.05, 0.1, 0.15, 0.20, and 0.25 g, the pH values were measured at 4.4, 5.3, 6.2, 6.9, and 7.2, respectively. In this instance, when the adsorbent dose grew, so did the pH value. According to ECR (2023), the pH of the treated effluent was within the discharge level at 6.9 and 7.2, respectively, after applying an adsorbent dose of 0.20 g and 0.25 g.

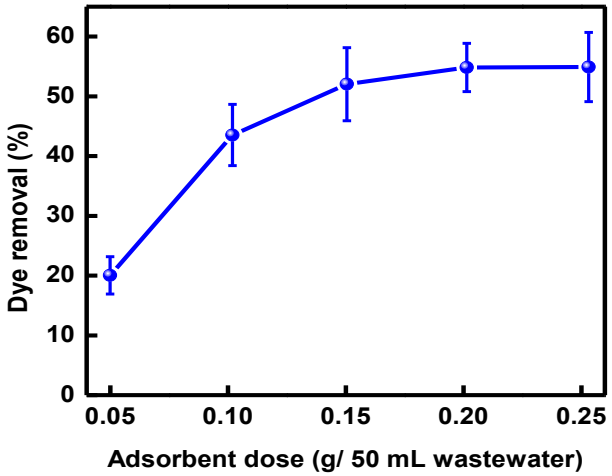

Figure 2: Effect of adsorbent dose on dyestuff removal efficiency

The results of the examination of the effects of contact and settling time on the efficacy of dyestuff removal are displayed in Fig. 3(a) and Fig. 3(b). It is evident from the figures that the dyestuff removal effectiveness improved within a definite time (5-20 min) as both contact and settling time increased. However, it was shown that the dyestuff removal effectiveness decreased after 20 min of both contact as well as settling time (Fig. 3(a) and Fig. 3(b)). After 20 min of contact and a settling period, a dominant desorption rate the adsorption rate may be the cause of the decline in dyestuff removal percentage. When all other factors, e.g., adsorbent dose, are the same, the removal effectiveness is impacted by variable contact and settling time. Maintaining both contact and settling duration for 20 min yielded maximum dyestuff removal efficiencies of 54.44% and 54.29%, respectively. The adsorption process may not always be the best method for eliminating dyestuffs from tannery effluent due to excessive contact time and settling time. So, 20 min was determined to be the ideal duration for this experiment after taking into account the dyestuff removal (%) in relation to contact and settling time.

3.3 Adsorption kinetics

PFO and PSO kinetic analyses were performed to evaluate the adsorbent's adsorption capabilities. Plotting the values for $\ln(q_e-q_t)$ and t allowed for the evaluation of the determination coefficient, or R^2, for PFO kinetic (Fig. 4(a). The R^2 value that was achieved was 0.3063. To assess the determination

coefficient, or R^2, for the PSO kinetic, values acquired for t/q_e and t were shown (Fig. 4(b)). The adsorption mechanism can be better fitted and represented by the PSO equation, as indicated by the derived R^2 value of 0.9985~1

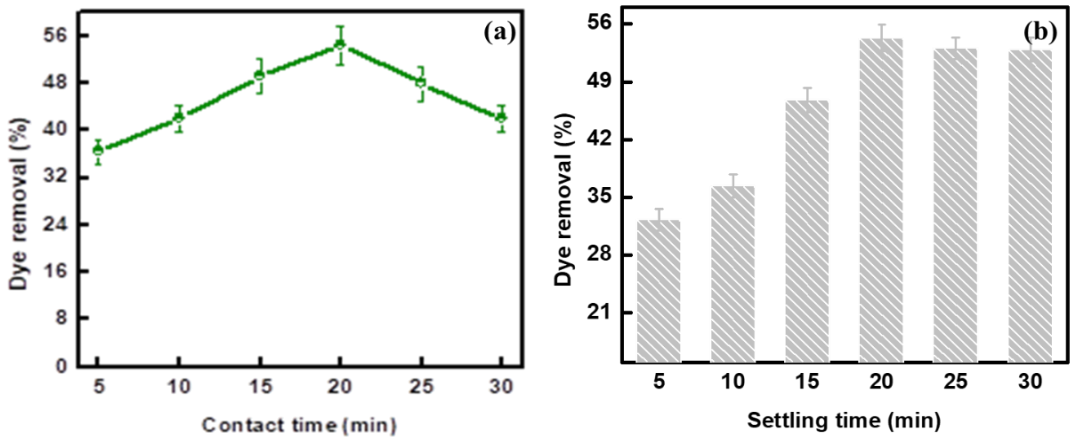

Figure 3: Effect of (a) contact time and (b) settling time on dyestuff removal efficiency

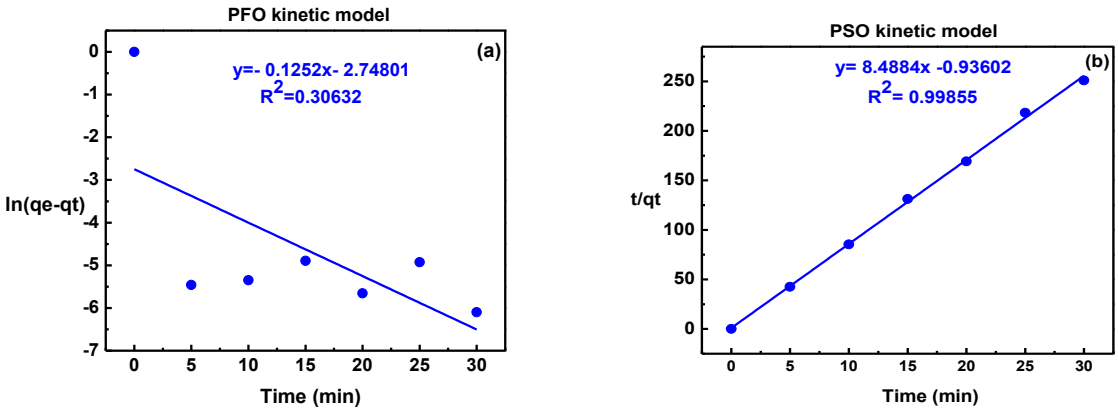

Figure 4: (a) PFO and (b) PSO kinetics plotted for dyestuff adsorption on liming sludge adsorbent

3.4 Adsorption Isotherm

Three isotherm models, including the Langmuir, the Freundlich, and the Temkin models, were used to examine the equilibrium data, to determine the potential adsorption process. The values obtained for $1/q_e$ and $1/C_e$ were shown in the Langmuir isotherm model (Fig. 5(a)). According to the Langmuir plot, the determination coefficient, or R^2, was 0.98315. The values obtained for log q_e and log C_e were shown for the Freundlich isotherm model (Fig. 5(b)). Log q_e vs log C_e showed a good fit to the equilibrium data from Fig. 5(b). For the Freundlich model, the determination coefficient, or R^2, had a value of 0.98725. The value obtained for q_e and ln Ce was shown for the Temkin isotherm model (Fig. 5(c)). According to Fig. 5(c), the Temkin model's determination coefficient, or R^2, was 0.9999. Compared to the Freundlich and Langmuir isotherm models, the Temkin isotherm model yielded a higher R^2 value. Therefore, in this instance, the Temkin isotherm model is considered suitable for explaining the adsorption mechanism. Once more, the graph's value for B was -0.23034 J mol^{-1}. An endothermic process took place when this constant value, which has been connected to the heat of adsorption, was negative.

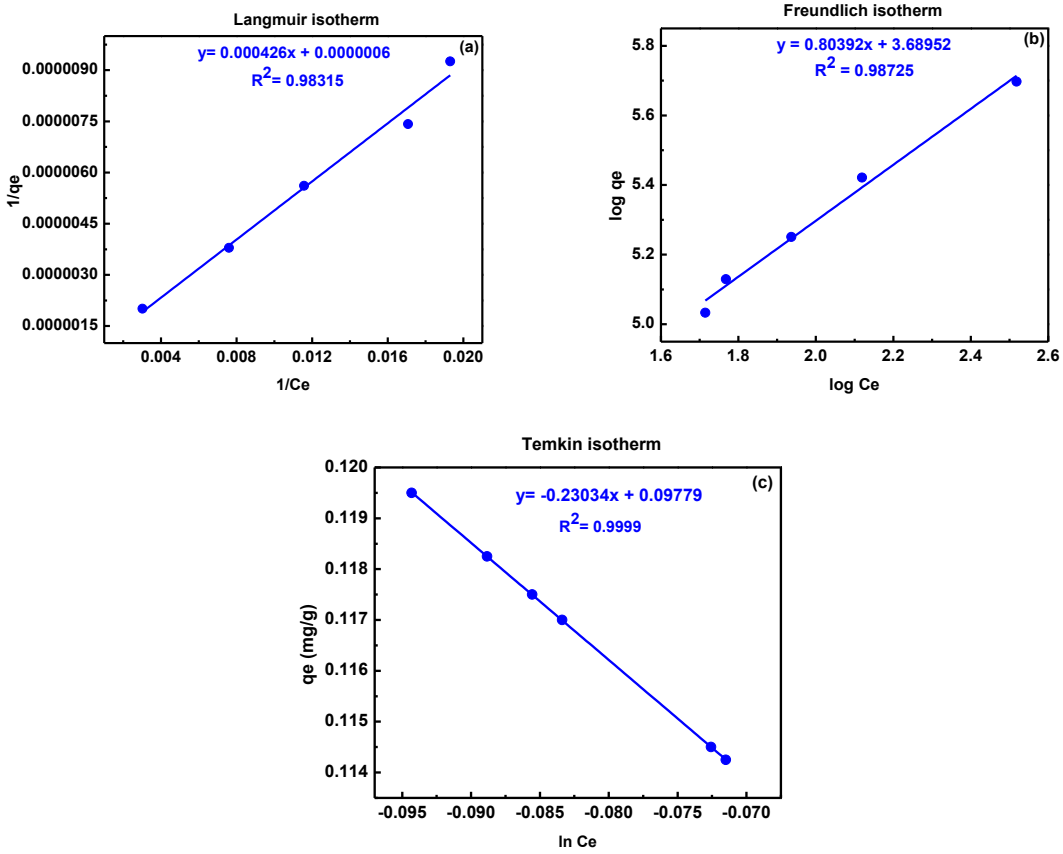

Figure 5: (a) Langmuir, (b) Freundlich, and (c) Temkin isotherm models plotted for the adsorption of dyestuff on liming sludge adsorbent

3.5 Efficiency of the Adsorption Process

Several physico-chemical factors, like pH, TSS, TDS, BOD, COD, and EC, were tested in order to characterize wastewater both before and after treatment. The findings of all these experiments are shown in Table 2. The pH values before and after treatment were 3.8 and 7.0, respectively, as shown in Table 2. Therefore, following the adsorption process, the pH value was within the discharged level. The raw tannery dyeing wastewater had BOD and COD levels of 2610.0 mg L^{-1} and 3836.8 mg L^{-1}, respectively. The BOD and COD levels after adsorption were 720.0 mg L^{-1} and 1587.2 mg L^{-1}, respectively. Thus, this treatment can reduce BOD by roughly 72.41% and COD by 58.63% while removing dyestuff by 54.83%. Before and after treatment, the EC values were 2.65 mS cm^{-1} and 3.65 mS cm^{-1}, respectively. The addition of certain minerals and other contaminants from the collected liming sludge may cause the treated wastewater's EC value to rise.

Table 2. Physico-chemical properties of raw and treated wastewater

Sl.	Parameters	Raw wastewater	Treated effluent	ECR (2023)
1.	pH	3.8±0.1	6.9±0.2	6-9
2.	TDS (mg L^{-1})	1176.0 ±3.0	1493.0±4.0	2100
3.	TSS (mg L^{-1})	233.33±4.0	1293.33± 3.0	100
4.	BOD (mg L^{-1})	2610.0±14	720.0±2.0	150
5.	COD (mg L^{-1})	3836.8±2.2	1587.2±3.4	200
6.	EC (mS cm^{-1})	2.95±0.2	3.65±0.2	1.20

3.6 REGENERATION AND REUSABILITY

The disposal of wasted adsorbents may cause hazards and result in secondary pollution if treated as garbage. Initially, adsorption was conducted under uniform conditions (starting pH: 3.8, adsorbent dosage: 0.20 g/50 mL, contact duration: 20 min, settling duration: 20 min) to get the exhausted adsorbent for the regeneration experiment. Following Mim et al. (2024), a regeneration experiment for liming sludge adsorbent was conducted using 1.5 g of adsorbent in 100 mL of 0.1 M sodium hydroxide (NaOH) solution. Following desorption, the adsorbent was deionized in water for 30 minutes at 25±1°C using an orbital shaker. After three rounds of washing with deionized water, the spent adsorbent powder was removed from the suspension, dried for three hours at 105°C in an oven, and then used for dyestuff adsorption. Four consecutive cycles of this regeneration experiments were conducted. To determine the regeneration efficiency (Rg), the amount of dyestuff adsorbed following different cycles was measured and documented. The regeneration efficiency is determined using the subsequent mathematical expression (2):

$$\text{Regeneration efficiency: } R_g\ (\%) = \frac{M_{De}}{M_{Ad}} \times 100 \quad (2)$$

Where M_{De} and M_{Ad} are the amount of dyestuff adsorption capacity of the liming sludge adsorbent in mg g^{-1} before and after regeneration, respectively.

Table 3. Regeneration efficiency of liming sludge adsorbent for the adsorption of dyestuff

Cycle	Cycle I	Cycle II	Cycle III
Regeneration efficiency, R_g (%)	96.12 ± 0.34	94.18 ± 0.83	91.15 ± 0.47

Table 3 indicates liming sludge adsorbent regeneration efficiency (R_g) for Acid Green V dyestuff from tannery dyeing wastewater. R_g values in each cycle were above 90%, indicating that adsorbent regeneration may be cost-effective. Table 3 shows that R_g decreased from 96.12% to 94.18% following the second cycle. In cycle three, removal was reduced to 91.15%, indicating its potency. Rg fell from 96.12 to 91.15% during the third cycle (Table 3), possibly due to surface active site degradation during adsorption-desorption. Thus, tannery liming sludge adsorbent can be recycled and utilized to remove dyestuffs for numerous cycles in tannery dyeing wastewater treatment.

3.7 COMPARISON STUDY

Table 4 compares the dyestuff removal efficiency using tannery liming sludge adsorbent with other adsorbents. Tannery dyeing wastewater is typically more complex and harmful since it mixes multiple unreacted dyes with synthetic, resin, dye leveler, etc. (Piccin et al., 2016).

Table 4. Comparison of dye removal efficiency using tannery liming sludge adsorbent with other adsorbents

Adsorbent and wastewater type	Type of dyes	Removal efficiency	References
H_3PO_4-activated cattle hair adsorbent in synthetic solutions	Acid Brown 414	71.06%	Mella et al., 2019
H_3PO_4-activated cattle hair adsorbent in synthetic solutions	Acid Orange 142	73.05%	Mella et al., 2019
Cr-tanned shaving waste adsorbent in tannery dyeing wastewater	Acid Red 357	87.37%	Gomes et al., 2016
Cr-tanned leather waste adsorbent in tannery dyeing wastewater	Acid Red 357	58%	Piccin et al., 2016
Tannery liming sludge adsorbent in tannery dyeing wastewater	Acid Green V	54.83	This study

The study conducted by Mella et al. (2019) found that a cattle hair adsorbent activated with phosphoric acid (H_3PO_4) was able to remove 71.06% of Acid Brown 414 and 73.05% of Acid Orange

142 from the synthetic dyeing effluent. The percentage of Acid Red 357 dye removed from tannery dyeing effluent by non-activated Cr-tanned shaving waste is 87.37%, while the percentage removed by leather waste adsorbent is 58% (Gomes et al. 2016). This study removed 54.83% of dyes (Acid Green V), 58.63% COD, and 72.41% BOD from tannery dyeing effluent. These findings suggested that tannery liming sludge adsorbent can treat complex tannery dyeing wastewater.

4. CONCLUSIONS

According to this study, the adsorption process was able to remove 54.83% of the dyestuff from tannery effluent while reducing BOD by roughly 72.41% and COD by 58.63%. According to this study, the adsorption process was able to remove 54.83% of the dyestuff from tannery effluent while reducing BOD by roughly 72.41% and COD by 58.63%. Additionally, the pH of the treated wastewater was obtained closer to 7.0. This study reveals that a cost-effective way to lower pollution load from the tannery effluent could be to use an adsorbent made from tannery liming sludge. Liming sludge adsorbent preparation was somewhat simpler and less expensive. It possesses excellent surface properties, atomic and elemental compositions and functional groups to adsorb acid dyestuffs. Liming sludge can therefore be employed as an effective adsorbent in wastewater treatment as well as tannery solid waste management. With a little further research and development, the adsorption technique might be readily used in industry to eliminate acid dyestuff from the tannery dyeing effluent.

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AI DECLARATION STATEMENT

The authors confirm that no AI tools were used in the preparation or completion of this assessment. This submission is entirely their own work.

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