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Water Quality and Wastewater Treatment Methods from the Process of Dyeing Cloth with Indigo Dye in Northeastern Thailand

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Abstract

The process of producing indigo dyed cloth involves a step of washing the dyed cloth, which requires a large amount of water and generates wastewater that cannot be reused and may have negative impacts on the environment. This research study collected 31 samples of wastewater from the process of dyeing cloth with indigo dye from production sources in Sakon Nakhon, Nakhon Phanom, and Mukdahan provinces to analyze the water quality and study wastewater treatment methods at laboratory scale using filtration and chemical methods, which employed aluminum sulfate, lime, and chlorine as coagulants. From the study, it was found that the collected samples of wastewater from the process of dyeing cloth with indigo dye had water quality that exceeded the standards set by the Department of Pollution Control. The average color value was 1,040.86 ± 744.83 PCU, the average COD was 711.59 ± 695.07 mg/L, the average total suspended solid was 1,495.87 ± 1,583.17 mg/L, and the average of total settable solids was 20.52 ± 25.85 mg/L. Therefore, the wastewater should be treated before being discharged into the public water system. Regarding the treatment of wastewater from the dyeing process, it was found that the most effective method is chemical treatment using lime and chlorine, which can reduce color in the wastewater by 98.73 %, COD by 91.67 %, total suspended solids by 97.09 %, and total settable solids by 96.47 %. The 2nd-best method is the use of aluminum sulfate, followed by filtration. All in all, both methods of treating the dyed fabric wastewater can improve the water quality to meet the standard before being used for beneficial purposes or discharged into public water sources, and the appropriate method of treatment can be selected for each area to minimize environmental impact.

Keywords: Indigo dyeing wastewater, Total suspended solids, Water quality, Coagulants, COD

Introduction

Indigofera tinctoria, also known as indigo, is a local plant in the northeastern region of Thailand that has been used to produce indigo-dyed fabrics. It has been promoted as a family industry and developed with support from both government and private organizations to make indigo-dyed fabric production an industry. In the provinces of Sakon Nakhon, Nakhon Phanom, and Mukdahan, indigo has been widely grown and used to produce indigo-dyed fabrics. The process of dyeing fabric with indigo involves 3 main steps. Firstly, indigo is extracted from the plant by soaking its leaves in water. During this process, the enzyme β-glucosidase in the chloroplast of the cells, mesophyll in the leaves, and bacteria, break down indican, a colorless and water-insoluble substance found in the leaves of the plant, into indoxyl and glucose, both of which are also colorless and water-soluble [1-3]. Indoxyl can easily be oxidized to become indigo in the presence of oxygen in the air. Indigo is a stable pigment that is small and blue in color. Next, the indigo pigment is made into indigo particles by mixing it with quicklime (CaO) and stirring it to expose it to oxygen in the air, which causes the blue particles to attach to the lime and settle down. The mixture is left to settle overnight, and the top liquid is then poured off. Finally, the indigo particles are collected and used for the next dyeing process. The 2nd step is the preparation of an indigo vat using a fermentation process. Indigo pigment is immersed in potassium hydroxide, which contains Alkalibacterium bacteria and reduced (the oxygen is removed). This transforms the indigo into leuco-indigotin (also known as indigo white). The dye solution changes color from blue to a dark yellow. This whole process takes approximately 15 days to prepare. The final step is to dye the fabric by immersing it in the dye bath that is ready to dye (indigo white). Then, the fabric is twisted and exposed to the air, causing the indigo white that was dissolved in the dye bath and attached to the fabric to undergo oxidation and convert back to indigo blue, which penetrates the fabric fibers and creates a natural indigo-dyed fabric [3]. In the process of dyeing natural indigo fabric, washing the dyed fabric involves using a large amount of water. The water used to rinse the dyed fabric is of poor quality and cannot be reused by the manufacturer. The wastewater containing indigo dye has high levels of chemical oxygen demand (COD), suspended solids, and dissolved solids, as well as a high color content [4]. The untreated water is usually discharged into the ground or municipal drainage systems, which could have long-term effects on public water sources, soil quality in nearby areas, or neighboring communities. The presence of color substances in public water sources can prevent sunlight, consequently reducing photosynthetic activity and the amount of dissolved oxygen, causing severe environmental problems and affecting the survival of living things [5]. Wastewater containing color, nutrients, and organic materials can be harmful to the environment if it is not treated before being discharged. These substances can pollute waterways, leading to eutrophication, algal blooms, and other problems [6]. Thus, it is important to treat wastewater to remove these substances before they are released into the environment. The treatment of indigo dyeing effluent is a complex issue, as there are multiple methods available with varying advantages and disadvantages. Some of the previously examined methods, such as the use of pure bacteria [3,7] or bacterial cellulose [8], have been shown to be effective in removing indigo dye color. However, these methods can be difficult and impractical to maintain on a large scale in actual field conditions. Membrane filtration technology aided by physicochemical pretreatment techniques has been identified as one of the most promising for the reclamation of textile effluents [9], a significant disadvantage of membrane technology is the decrease in membrane flux caused by membrane contamination. The use of advanced oxidation processes (AOP) is a widely recognized wastewater remediation technique due to its high efficiency in removing organic matter, odor, and color [10], but this treatment technique is expensive in terms of operational and maintenance costs. Hence, physico-chemical processes appear to be suitable remediation methods for indigo dye wastewater. Therefore, studying the quality of the water used to rinse indigo-dyed fabric and the process of treating the water to make it suitable for reuse or meet wastewater standards can help reduce water consumption costs, minimize environmental impacts, and provide a path towards producing environmentally friendly indigo-dyed fabric. This research aims to collect samples of wastewater from indigo dyeing sources to analyze the water quality. The wastewater treatment methods will then be evaluated in the laboratory to develop a standard method for treating indigo dye wastewater that meets the standards set by the Department of Pollution Control. This will help reduce the cost of water usage, minimize environmental impacts, and provide a sustainable way to produce environmentally friendly natural dyed fabrics.

Materials and methods

Study area

The study area for this research was the indigo-dyed fabric production in Sakon Nakhon, Nakhon Phanom, and Mukdahan provinces, northeastern Thailand. Thirty-one stations were established in the study area (Sakon Nakhon, Nakhon Phanom, and Mukdahan; n = 24, 4 and 3 entrepreneurs, respectively) (**Figure 1**). The topography of the study area is mostly flat, with some hills in the north and east.

Characterization of indigo dyeing wastewater samples

During June to July 2020, a total of 31 samples of wastewater from indigo-dyed fabric were collected from medium-sized production sources (with more than 15 dyeing vats). The physical and chemical characteristics of indigo-dyed wastewater samples were investigated. In situ measurements of temperature, dissolved oxygen, pH, and electrical conductivity (EC) were conducted using a multiprobe meter (YSI 556 MPS, USA), and salinity was measured using a refractometer (RHS-10ATC, China). Water samples were collected and analyzed in the laboratory for color using a colorimeter (Hanna-HI 727, Romania), biochemical oxygen demand (BOD) by direct method, chemical oxygen demand (COD) by close-reflux method, total solid, total suspended solid (TSS), total dissolved solid (TDS) by gravimetric analysis [11], total settable solids by imhoff cone method, total nitrogen (TN) by persulfate oxidation method, total phosphorus (TP) by persulfate oxidation method [12].

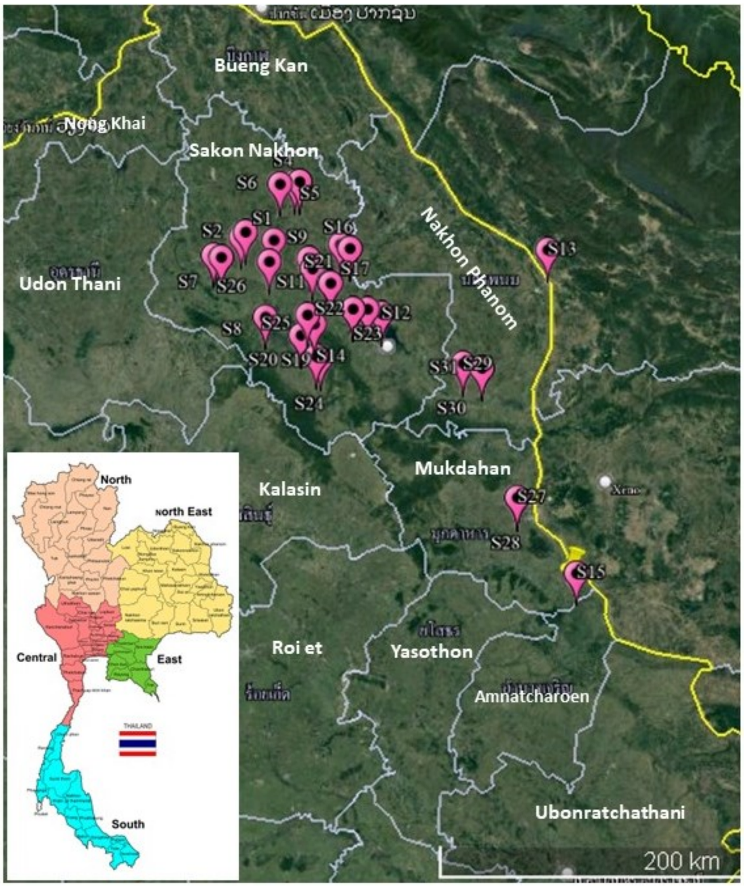

Figure 1 Map of study area.

Laboratory scale Indigo dyeing wastewater treatment

Treating wastewater from the indigo dyeing process by using a filtration method

Wastewater was treated using a filtration method that utilized filter materials consisting of glass wool, finely ground charcoal, and fine sand to remove impurities and dyes from the water. For filtering, a 5L cylindrical plastic tank (diameter 16 cm, height 30 cm) with a hole drilled in the bottom to allow water to escape was used. The tank's bottom layer was filled with 3 cm of glass wool. The center layer was filled with fine sand 10 cm thick and ground charcoal 10 cm thick. The top layer was filled with 7 cm of glass wool. The filter tank filtered water at a rate of 0.15 L/min (**Figure 2**). Water quality analysis was conducted before and after the treatment process.

Treating wastewater from the indigo dyeing process by using a chemical treatment method

Wastewater from the indigo dyeing process was treated using aluminum sulfate as a coagulant to remove dyes and impurities from the water through the coagulation system. Aluminum sulfate ($Al_2(SO_4)_3.18H_2O$) was added at a rate of 1 g/L of wastewater. The mixture was left to settle overnight, and the water was then filtered through a filter bag with a mesh size of 100 - 200 microns. After that, the filtered water was aerated for 30 min (**Figure 2**). Water quality analysis was conducted before and after the treatment process.

Dyed indigo wastewater was treated using lime ($Ca(OH)_2$) and chlorine by mixing lime at a concentration of 1 g/L of water and stirring for 30 min. Then, bleaching powder ($CaOCl_2$) was added at a concentration of 0.5 g/L of water and stirred for another 30 min. The mixture was allowed to settle overnight, and then the water was filtered through a 100 - 200 micron filter bag. After that, the treated water was aerated for 3 - 4 days until the remaining chlorine was completely dissolved (**Figure 2**). Water quality analysis was conducted before and after the treatment process.

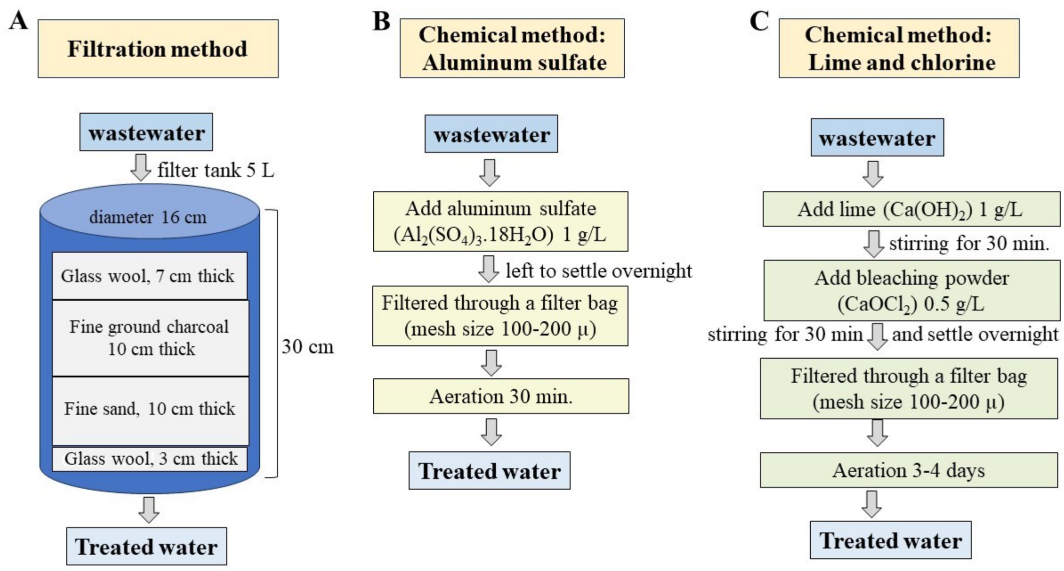

Figure 2 Diagram of the process of indigo dyeing wastewater treatment (A) filtration method, (B) chemical treatment method using aluminum sulfate, and (C) chemical treatment method using lime and bleaching powder).

Statistical analysis

Data analysis was conducted using the mean and standard deviation of the data obtained from the experiment. The mean differences were compared using a T-test with a 95 and 99 % confidence level.

Results and discussion

Characterization of indigo dyeing wastewater samples

From collecting and analyzing samples of indigo dyeing wastewater, totaling 31 samples from dyeing factories located in Sakon Nakhon, Nakhon Phanom, and Mukdahan provinces, it was found that pH, temperature, dissolved oxygen, EC, BOD, TDS, TN, and TP were within the standard limits of the Department of Pollution Control. The average pH was 8.86 ± 1.27, the average temperature was 29.86 ± 2.13 °C, the average dissolved oxygen was 2.25 ± 1.63 mg/L, the average EC was 1,716.12 ± 1,175.04 µS/cm, the average BOD was 1.84 ± 1.46 mg/L, the average TDS was 2,868.7 ± 1,812.4 mg/L, the average TN was 15.63 ± 17.37 mg-N/L, and the average TP was 1.72 ± 1.93 mg-P/L (**Table 1**). As for the water quality that exceeded the standard limits set by the Department of Pollution Control, it included color, COD, TSS, and total settable solids. The average color was 1,040.86 ± 744.83 PCU, the average COD was 711.59 ± 695.07 mg/L, the average TSS was 1,495.87 ± 1,583.17 mg/L, and the average total settable solids was 20.52 ± 25.85 mg/L (**Table 1** and **Figure 3**). The wide range of water quality values is due to the different dyeing and washing processes used by indigo dyeing operators. Wastewater with a quality exceeding the standard must be treated before being discharged to reduce environmental impacts. Abdallah and Halim [13] reported that textile industry wastewater had a COD value ranging from 993 to 1,606 mg/L and an average of 1,047 mg/L, which exceeded acceptable standards. Therefore, wastewater treatment should be performed before discharging it to the public. Senasri *et al*. [14] found that indigo dyeing wastewater with a concentration of 0.137 µg/mL had toxicity that caused 50 % mortality in white tilapia within 96 h. This is partly due to the high amount of suspended matter in the wastewater, which could obstruct fish gills and affect the transportation of oxygen to different parts of the body. This can ultimately lead to the death of the fish.

Table 1 Comparing water quality of the indigo dyeing wastewater (Mean±SD) with the water quality standards set by the Department of Pollution Control (n = 31).

Parameter	Water quality of the indigo dyeing wastewater (Mean ± SD)	Water quality standards set by the Department of Pollution Control	Types of water quality standards
pH	8.86 ± 1.27	5.5 - 9.0	Wastewater quality standards set for factories and industrial estates
Temperature (°C)	29.86 ± 2.13	≤ 40 °C	
Dissolved oxygen (mg/L)	2.25±1.63	2.0 mg/L	Wastewater quality standards discharged to irrigation canals and waterways connected to irrigation canals in irrigation project areas
EC (µS/cm)	1,716.12 ± 1,175.04	2,000 µS/cm	
Salinity (ppt)	2.40 ± 2.88	-	
Color (Platinum-cobalt unit; PCU)	1,040.86 ± 744.83	50 PCU	Wastewater quality standards discharged to groundwater
BOD (mg/L)	1.84 ± 1.46	≤ 20 mg/L	Wastewater quality standards set for factories and industrial estates
COD (mg/L)	711.59 ± 695.07	≤ 120 mg/L	
Total solid (mg/L)	3,772.48 ± 2,248.47	-	
Total suspended solid (TSS) (mg/L)	1,495.87 ± 1,583.17	≤ 50 mg/L	Wastewater quality standards set for factories and industrial estates
Total dissolved solid (TDS) (mg/L)	2,868.7 ± 1,812.4	≤ 3,000 mg/L	
Total settable Solids (mg/L)	20.52 ± 25.85	≤ 0.5 mg/L	Wastewater quality standards set for some building types and sizes
Total nitrogen (TN) (mg-N/L)	15.63 ± 17.37	≤ 20 mg-N/L	Wastewater quality standards set for local community water treatment system
Total Phosphorus (TP) (mg-P/L)	1.72 ± 1.93	≤2 mg-P/L	

Source: Thai water quality standards, Department of Pollution Control [15].

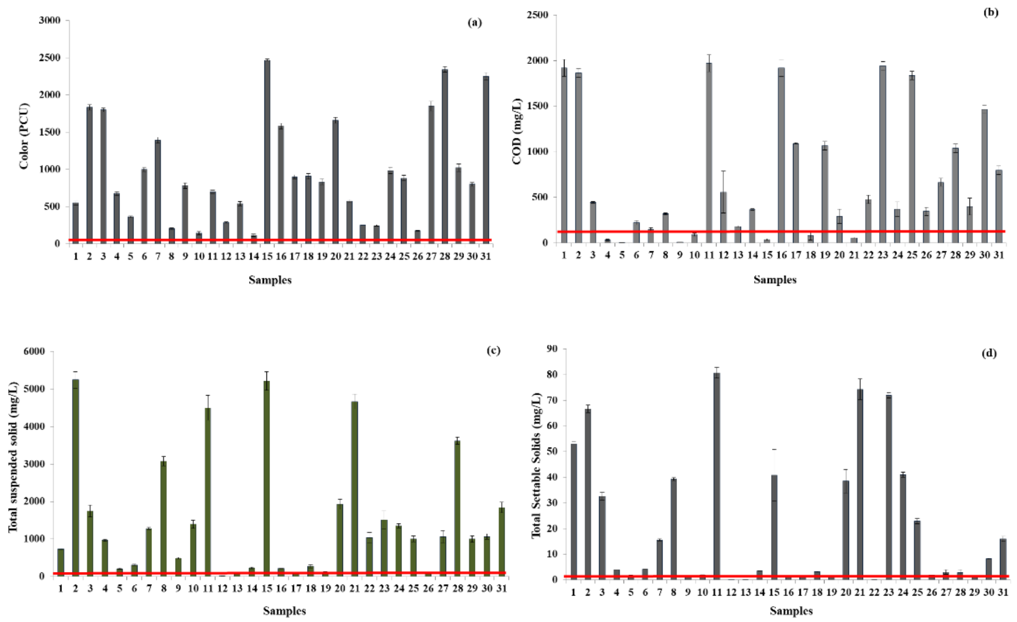

Figure 3 Water quality of indigo dyeing wastewater from 31 samples exceeded the wastewater quality standards. (a) color (PCU) (b) COD (mg/L) (c) total suspended solid (mg/L), and (d) total settable solids (mg/L).

Laboratory scale Indigo dyeing wastewater treatment

The analysis of the wastewater properties from the dyeing processes found that total settable solids, total suspended solids, and COD levels exceeded the wastewater quality standards, as well as the dark color of the wastewater. Two treatment methods, filtration and chemical treatments, were tested to treat the wastewater.

Treating wastewater from the indigo dyeing process by using a filtration method

The method of treating dyeing wastewater by filtration involves settling to remove total settable solids and total suspended solids, then passing the clear water through a filter made of finely ground charcoal, fine sand and glass fiber cloth to remove remaining solids and color from the water. The treated water is then aerated to adjust its condition before being used to water plants or released into public water sources. This method is effective in removing solids from the water, but some color remains in the water. Analysis of the treated water showed a 76.24 % reduction in total settable solids, a 45.99 % reduction in total solids, a 92.72 % reduction in total suspended solids, a 39.96 % reduction in total dissolved solids, a 20.84 % reduction in COD, and a 50.42 % reduction in color, according to **Table 2**. These results are consistent with the study conducted by Dogdu and Yalcuk [16] on the treatment of synthetic dye wastewater using a wetland system that is environmentally friendly. The system used sand, zeolite, and gravel as filter media (control: R1), planted *Canna indica* in the filter media (R2), and planted *Typha angustifolia* in the filter media (R3) for 91 days. The study found that the average COD removal efficiency was 61.95 ± 5.61 % in the R1 experimental group, 45.80 ± 19.65 % in the R2 group, and 39.15 ± 7.23 % in the R3 group. The percentage of color removal in R1, R2, and R3 was 97.10 ± 1.97 %, 90.18 ± 5.31 %, and 90.15 ± 3.55 %, respectively.

Table 2 Characterization of water quality parameters of indigo dyeing wastewater before and after treating using filtration method (n = 9).

Parameters	**Average**		**% Removal**
	Before treating	**After treating**	
Total settable solids (mg/L)	4.25	1.01	76.24
Total solid (mg/L)	1,831	989	45.99
Total suspended solid (TSS) (mg/L)	412	30	92.72
Total dissolved solid (TDS) (mg/L)	1,344	807	39.96
Color (PCU)	393.3	195	50.42
Total nitrogen (TN) (mg-N/L)	7.769	6.341	18.38
Total phosphorus (TP) (mg-P/L)	1.164	0.231	80.15
COD (mg/L)	640	506.6	20.84
BOD (mg/L)	3.37	3.31	1.78
pH	10.05	9.5	5.47
EC (μS/cm)	1,692	1,077	36.35
Dissolved oxygen (mg/L)	3.48	3.7	–6.32
Salinity (ppt)	4	4	0.00

Treating wastewater from the indigo dyeing process by using a chemical treatment method

The method for treating wastewater from indigo dyeing using aluminum sulfate as a coagulant aims to remove color and impurities from the water through a coagulation process. To do this, chemicals such as aluminum sulfate ($Al_2(SO_4)_3.18H_2O$) was added to the wastewater at a ratio of 1 g/L of water, and left to settle for 1 night. The water is then filtered through a 100 - 200 micron filter bag before passing through an aerator to improve its condition before it is used for watering plants or discharged into public water sources. This method produces clear water with some slight discoloration. When analyzing the properties of the water samples that have undergone the treatment process using aluminum sulfate and filtration through a filter bag, it was found that total settable solids reduced by 91.76 %, total solids reduced by 29.55

%, total suspended solids reduced by 95.15 %, total dissolved solids reduced by 18.75 %, COD reduced by 90.10 %, and color reduced by 36.87 % (**Table 3**). The dissolved oxygen level increases because the final stage of water treatment is aeration, which adds oxygen to the water. Besides, aluminum sulfate, being a sulfate salt, can increase the salinity of water. Sulfate compounds consist of sulfate ions (SO_4^{2-}) and additional cations, such as sodium, potassium, or calcium. These sulfate ions then contribute to the total salinity of the water. According to Unlu *et al.* [17], who used coagulation and membrane-based filtration processes to treat wastewater from denim production for reuse. Coagulation with aluminum sulfate and ferric chloride was effective in removing color. Aluminum sulfate at a concentration of 1,000 mg/L was found to be more effective in removing color than ferric chloride, with a 96 % removal rate. This is consistent with the work of Manu [18], who found that alum can reduce the color in indigo dye wastewater by 99 %. Microfiltration (MF) and nanofiltration (NF) were also effective in removing color and COD, with removal rates of 99 and 97 %, respectively.

Table 3 Characterization of water quality parameters of indigo dyeing wastewater before and after treating using aluminum sulfate (n = 9).

Parameters	**Average**		**% Removal**
	Before treating	**After treating**	
Total settable solids (mg/L)	4.25	0.35	91.76
Total solid (mg/L)	1,831	1,290	29.55
Total suspended solid (TSS) (mg/L)	412	20	95.15
Total dissolved solid (TDS) (mg/L)	1,344	1,092	18.75
Color (PCU)	393.3	248.3	36.87
Total nitrogen (TN) (mg-N/L)	7.769	2.035	73.81
Total phosphorus (TP) (mg-P/L)	1.164	0	100
COD (mg/L)	640	63.33	90.10
BOD (mg/L)	3.37	1.28	62.02
pH	10.05	9.09	9.55
EC (μS/cm)	1,692	1,456	13.95
Dissolved oxygen (mg/L)	3.48	4.59	–31.90
Salinity (ppt)	4	10	–150.0

The method of treating indigo dyeing wastewater using lime and chlorine involves adding lime to the wastewater at a concentration of 1 g/L of water and aerating it for 30 min before adding bleaching power ($CaOCl_2$) at a concentration of 0.5 g/L of water and aerating it again for 30 min. The mixture was left to settle for 1 night, and the water was filtered through a 100 - 200 micron filter bag. The filtered water was left for 3 - 4 days until the chlorine had completely dissipated. This method produces clear and colorless water without any suspended solids or impurities. The treated water showed a reduction of 96.47 % in total settable solids, 70.56 % in total solids, 97.09 % in total suspended solids, 20.98 % in total dissolved solids, and 91.67 % in COD, and a reduction of 98.73 % in color (**Table 4**). This method is consistent with the study by Manu [18], who used lime to treat wastewater from denim factories, achieving a 97 % reduction in color and a 66 % reduction in COD with lime at a concentration of 2.5 g/L, and a 99 % reduction in color and a 95 % reduction in COD with lime at a concentration of 1 g/L. In addition, the combination of chemical coagulation using 300 mg/L of ferric chloride and 87.5 mg/L of lime was effective in reducing COD by 57 %, BOD by 64 %, and TSS by 14.5 % [13].

Table 4 Characterization of water quality parameters of indigo dyeing wastewater before and after treating using lime and chlorine (n = 9).

Parameters	Average		% Removal
	Before treating	After treating	
Total settable solids (mg/L)	4.25	0.15	96.47
Total solid (mg/L)	1,831	539	70.56
Total suspended solid (TSS) (mg/L)	412	12	97.09
Total dissolved solid (TDS) (mg/L)	1,344	1,062	20.98
Color (PCU)	393.3	5	98.73
Total nitrogen (TN) (mg-N/L)	7.769	2.604	66.48
Total phosphorus (TP) (mg-P/L)	1.164	0.207	82.22
COD (mg/L)	640	53.33	91.67
BOD (mg/L)	3.37	0.97	71.22
pH	10.05	9.06	9.55
EC (µS/cm)	1,692	1,379	18.50
Dissolved oxygen (mg/L)	3.48	3.88	–11.49
Salinity (ppt)	4	4	0

This research investigates indigo dye wastewater treatment in laboratory settings. After treating the wastewater, the study found that the use of lime and chlorine is the most effective method to reduce total settable solids, total solids, total suspended solids, COD, and color in the water. The 2^{nd}-most effective method is using aluminum sulfate, followed by filtration. Regardless of the methods used, the treatment of wastewater from indigo dyeing processes can still meet wastewater quality standards before being utilized or discharged into public water sources. The appropriate treatment methods should be chosen according to the specific location.

Conclusions

The survey and analysis of the quality of wastewater from indigo dyeing processes revealed that color, COD, total suspended solids, and total settable solids exceeded the wastewater standard. Therefore, it is necessary to treat the wastewater before discharging it into public water sources. Based on this study, the best treatment methods for indigo-dyed wastewater are lime and chlorine, which can remove up to 98.73 % of color, 91.67 % of COD, 97.09 % of total suspended solids, and 96.47 % of total settable solids. The 2^{nd} effective method is using aluminum sulfate, which can remove up to 36.87 % of color, 90.10 % of COD, 95.15 % of total suspended solids and 91.76 % of total settable solids. The final effective method is filtration, which can remove up to 50.42 % of color, 20.84 % of COD, 92.72 % of total suspended solids, and 76.24 % of total settable solids. However, it is important to treat the wastewater before discharge every time to minimize the impact on the environment.

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