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Recovered poly manganese chloride ($PMnCl_2$) from industrial waste sludge to decolorize and detoxify textile dyes and comparison with alum

Keywords: coagulant recovery, characterization, decolorization and detoxification, poly manganese chloride, textile dyes

Introduction

Agriculture, forestry, fishing, hydropower generation, industry, and other innovative endeavors all depend heavily on water as a resource. Most industrial effluents are treated, and their properties are determined by the manufacturing methods and raw material types used (Tyagi et al., 2013).

To create, enhance, and put into practice suitable techniques for treatment to eliminate pollutants, a lot of research has been encouraged due to the production of massive output polluted wastewater (Ahmed et al., 2020). To remove color and other organic pollutants wastewater requires a post-treatment process (Hossain et al., 2019). Ion exchange, filtration, chemical precipitation, and the application

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of membrane technology are all well-established traditional methods for wastewater treatment (Vasudevan et al., 2010; Sher et al., 2013). These include the production of sludge and heavy metal residues that may be harmful to the ecology, as well as the end-of-life nanomaterials and enhanced color of wastewater (Zhao et al., 2014; Tetteh & Rathilal, 2020). High wastewater treatment system costs and other technological problems are caused by the issues listed above (Holkar et al., 2016).

Effectively reduce the organic burden before moving on to other methods of treatment. It is now important to pre-treat industrial wastewater utilizing coagulation and flocculation techniques (Saifuddin & Dinara, 2011). A crucial process that involves the addition of coagulants, coagulation destabilizes and neutralizes suspended particles, causing them to form big flocs or aggregates. Because of ion adsorption and surface group ionization, negatively charged suspended particles typically clump together when combined with positively charged coagulants. Filtration, flotation, or sedimentation processes are used to get rid of these aggregates (Sánchez-Martin et al., 2012; Sahu & Chaudhari, 2013; Balls, 2014; Bodlund, 2020). Since methods for managing industrial discharges containing toxic compounds are costly and a significant burden for most businesses in underdeveloped nations, these discharges represent major issues for rural communities worldwide (Hung & Kaya, 2020).

Due to the fast expansion of industrialization sectors, wastewater treatment plants are likely depleted and unable to provide granular discharge regulations. Industrial discharge of untreated wastewater into water bodies has an impact on aquatic diversity and human health (Jamali & Moradnia, 2018). Chemical phenolic pollutants are widely used, as demonstrated by materials such as reactive dyes, which are frequently used as synthetic dyes in apparel, paper, and leather sectors. These harmful pollutants are a serious concern to aquatic ecosystems because they seriously damage aquatic life and have a negative impact on human health as well as the larger ecological system (Mohammed & M-Ridha, 2024).

Industrial dyes are utilized all over the world today, and a lot of research is being done to create new synthetic processes that will enhance their application properties. However, textile dyes can provide serious wastewater issues for the dyeing industry because of their high solubility. Interest in decolorization techniques has grown as a result of growing ecological consciousness and public concern, as well as more stringent laws governing wastewater discharge in recent years. Analyzing and cleaning up industrial wastewater effluents contaminated with dyes are crucial procedures. Current cleaning methods are either expensive or harmful to human health because some colors are known to be particularly harmful to the environment, cancer, and mutagenicity (Mohammed & M-Ridha, 2025).

Coagulation has been the sole economically viable technique for color removal over the years. This approach still has a lot of benefits today and is still utilized both alone and in conjunction with other approaches. It was discovered that this combination treatment was highly successful. It was demonstrated that effluent had been completely decolored (Ghaly et al., 2014).

The purpose of this study has two purposes: utilizing environmentally polluting industrial sludge to recover manganese metal and transform it from an environmental burden into an effective coagulant applicable in the treatment of industrial dyes (1); and comparing the synthetic coagulant with a traditional coagulant (alum) and selecting the most efficient coagulant in the process of removing organic textile dyes (2).

Material and methods

The study was carried out in two experimental steps. In the first step, manganese chloride recovery is conducted to produce poly manganese chloride ($PMnCl_2$). Hydrochloric acid (HCl) and sodium hydroxide (NaOH) solutions from Sigma were used. A jar test is first conducted to determine the optimal recovered coagulant based on its effectiveness in pollutant removal. In the second step, to evaluate the effectiveness of recovered coagulants against traditional alum coagulants, an additional test is carried out. Figure 1 illustrates the whole procedure of purification of industrial water and recovery of $PMnCl_2$ of used in this study.

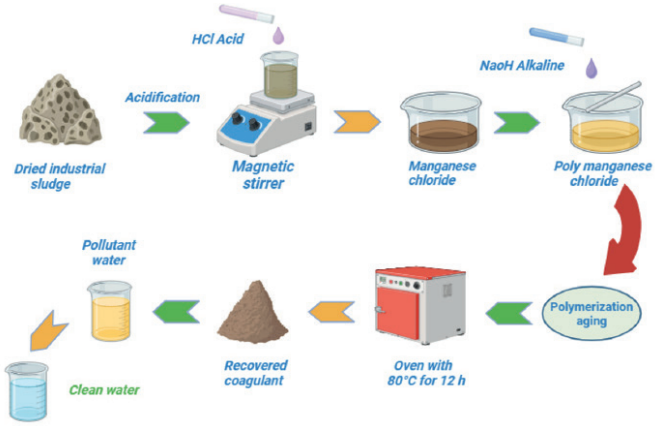

FIGURE 1. Study procedure

Source: own work.

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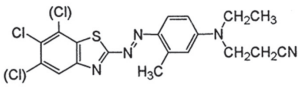
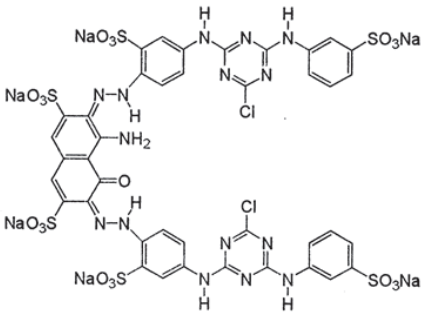
Textile dyes, namely reactive yellow (RY17) and direct blue (DB53), were used provided by an Iraqi textile factory made in Switzerland. According to the material safety data sheet for RY17 and DB53, these dyes have a negative consequences since they can irritate the respiratory system when inhaled. Furthermore, consumption may result in nausea, vomiting, diarrhea, and gastrointestinal irritation. To prepare a solution of 40 $mg \cdot l^{-1}$ of textile dyes, deionized water was used in laboratory experiments. Table 1 presents the characteristic details and structures of these pollutants.

The wastewater treatment plant in Al-Tajiat (a region in Baghdad, Iraq), which is part of the organized industrial area, supplied the industrial sludge. The sludge samples were dried for 48 h. The main characteristics of the industrial sludge sample were as follows: 300 $g \cdot l^{-1}$ of total solids (TS), 135 $g \cdot l^{-1}$ of total volatile solids (TVS), and 165 $g \cdot l^{-1}$ of fixed solids (FS) with 75.13 $g \cdot l^{-1}$ of chemical oxygen demand (COD).

All these characteristics were determined according to the procedure described in the standard methods (American Public Health Association [APHA], 2005), and the elemental composition is illustrated in Table 2.

TABLE 1. Dyes chemical properties

Pollutant	MWt [g·mol]	λ_{max} [nm]	Chemical structure
Reactive yellow 17	452.79	530	
Direct blue 53	1,418.94	545	

M_{wt} – weight-average molecular weight, λ_{max} – wavelength of maximum absorption.
Source: Safety data sheet.

TABLE 2. Elemental composition of the industrial sludge samples

Parameter	Unit	Value
Si concentration	$mg \cdot kg^{-1}$	13,600
Zn concentration	$mg \cdot kg^{-1}$	2,300
Cu concentration	$mg \cdot kg^{-1}$	1,100
Mn concentration	$mg \cdot kg^{-1}$	9,200
Fe concentration	$mg \cdot kg^{-1}$	7,850
Hg concentration	$mg \cdot kg^{-1}$	n.d.
Ni concentration	$mg \cdot kg^{-1}$	720
Cd, Cr concentration	$mg \cdot kg^{-1}$	b.d.
Pb concentration	$mg \cdot kg^{-1}$	680
Moisture	%	70
pH	–	6.6

n.d. – not determined, b.d. – below detection limit.

Source: own work.

Poly manganese chloride recovery

This step involves drying the industrial sludge then mixing the sample with hydrochloric acid at a pH of 2 in a glass beaker. For 30 min, the composite sample was agitated using a magnetic stirrer at different acidification stirring speeds. After combining, the composite sample is allowed to settle for 30 min. To create the $PMnCl_2$, NaOH is added progressively as a base material until the compound is homogeneous. After that, the polymerization process is carried out to improve the performance of the coagulant, then dried in an electric oven at 80°C for 12 h and ground. It is obtained within limits of 30 g of powder per 1 kg of sludge sample.

Factors effecting recovered poly manganese chloride

Solutions of HCl at varying concentrations are applied to the sludge. The factors that influencing on $MnCl_2$ coagulation are investigated such as:

– The acid concentration effect: at different concentrations 5%, 10%, 15%, 20%, 25%, 30%, and 35%; with parameters stirrer speed 100 rpm; pH of 2; reaction period 20 min; and temperature 40°C.
– The stirrer force effect: stirring at 100 rpm, 200 rpm, 300 rpm, 400 rpm, 500 rpm, and 600 rpm; 20 min reaction time; and a 30% acid concentration.

– The contact time effect: the chosen time intervals are 20 min, 40 min, 60 min, 80 min, 100 min, and 120 min, with a constant flow rate; 30% HCl concentration; 300 rpm stirring speed.
– The temperature effect: 40°C, 60°C, 80°C, and 100°C; 300 rpm of stirring; 100 min of reaction duration; and 30% acid concentration were applied.

Decolorization by recovered coagulant process

Using a jar test (JTL6) with four paddles, the recovered coagulation experiment was conducted with 500 ml of a solution containing 40 mg of dyes per 1 l was used for all tests, and each was assessed independently in beakers (Zhang et al., 2021). First, to obtain the required dosages of recovered coagulant, a dosage range of 10–60 $mg \cdot l^{-1}$ was examined. Equation 1 was used to determine the percentage of removal after samples were stirred with rapid mixing (150 rpm) for 2 min and slow mixing (30 rpm) for 15 min at moderate pH and room temperature, then the necessary coagulant dosage was added (Tetteh & Rathilal, 2021):

$$C_n = \frac{C_i - C_f}{C_i} 100\%, \tag{1}$$

where C_n is the removal efficiency (response parameter), C_i is the initial value of the contaminant, and C_f is the final value of the contaminant.

Characterization of recovered poly manganese chloride

By scanning electron microscopy (SEM), surface morphological analysis of $PMnCl_2$ and alum was conducted to define their elemental distributions and particle shapes. A scanning electron microscope, Nova Nano SEM, was employed to examine the morphological structure of the samples obtained. This was operated at an acceleration voltage under the scale of 20 μm with a landing energy capacity of 15 kV. Additionally, utilizing the Jasco FTIR 460 plus spectrometer, Fourier transform infrared (FTIR) in the 500–4,000 cm^{-1} range, the recovered poly manganese chloride and alum were characterized.

Decoloization by alum coagulant process

The traditional coagulation with alum was experimented by using a jar test (JTL6). All experiments were conducted using 500 ml of a solution containing dye at a concentration of 40 $mg \cdot l^{-1}$, tested individually in separate beakers. Alum

dosages ranging from 10 $mg \cdot l^{-1}$ to 60 $mg \cdot l^{-1}$ were examined under identical conditions to determine the optimal dosage. Dye removal efficiency was estimated using Equation 1.

The kinetics of agglomeration

Coagulation is driven by Brownian motion of the suspended particles coagulation is ejected (Nnaji et al., 2014). When colloidal particles destabilize and agglomerate to a diameter greater than 1 μm Brownian motion becomes less effective (Sun et al., 2019). Equation 2 describes the kinetics of the coagulation rate process (Kumar et al., 2016; Zahrim et al., 2017). Additionally, kinetics determine the floc formation rate and help terminate the critical period prior to floc destabilization. Because kinetic parameters (*n* and *k*) determine how quickly contaminant rates are removed from effluent, kinetic study is crucial. Kinetic parameters, a dependent variable (*C*), and an independent variable (*t*) make up the rate equation:

$$\frac{dC}{Dt} = -KC^n. \tag{2}$$

where *C* is the concentration of particles, *t* is the coagulation time, *k* is the *n*-th order coagulation rate constant, and *n* is the order of the coagulation process.

The particle concentration and time have an indirect relationship. The amount of pollutant concentration absorbed by the coagulant can be directly correlated with the rate of contaminant removal. Equation 3 yields the rate constant for a fast coagulation process (K_{RC}) by multiplying Smoluchowski's rate constant by the collision efficiency (*E*) (Daud et al., 2015):

$$K = EK_{RC}, \tag{3}$$

where K_{RC} is given by Equation 4:

$$K_{RC} = \frac{4KBT}{3\mu}, \tag{4}$$

where μ is the fluid viscosity.

By Equation 5, the Brownian diffusion coefficient (D_B) is given:

$$D_B = \frac{k_B T}{\beta}, \tag{5}$$

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$$\beta = 2k. \tag{6}$$

Equation 2 becomes Equation 7 when integrated for the first order reaction ($n = 1$):

$$\ln\left(\frac{C}{C_0}\right) = K_1 t, \tag{7}$$

where C_0 is the initial concentration, C is the final concentration, and k_1 is the first-order rate constant in $1 \cdot min^{-1}$.

A plot of $\ln\left(\frac{C}{C_0}\right)$ versus t will yield a straight line passing through the origin with a slope of K_1 using Equation 7 (Othman et al., 2011). However, if line does not pass through origin but intersects Y-axis at a different point, process is better represented by a second-order coagulation model ($n = 2$), in which case Equation 2 transforms into Equation 8:

$$\frac{dC}{dt} = -KC^2. \tag{8}$$

Then, Equation 3 yields Equation 4 after integration:

$$\frac{1}{C} = K_2 t + \frac{1}{C_0}, \tag{9}$$

where k_2 is the second-order rate constant expressed in $l \cdot mg^{-1} \cdot min^{-1}$.

Quality of water

Water quality parameters were applied to both untreated and treated solutions using $PMnCl_2$. Chemical oxygen demand (COD) meters were used to measure the total amount of oxygen required to chemically oxidize both organic and inorganic pollutants in water. For total organic carbon (TOC), 1.0 ml of 2N potassium dichromate ($K_2Cr_2O_7$), 1.6 ml of sulfuric acid (H_2SO_4), and 4.0 ml of pollutant samples were added to a digestion flask. The mixture was allowed to digest for 90 min at 110°C before being cooled to room temperature, and optical density at 590 nm was recorded. The toxicity of the untreated and treated water

was evaluated. The samples were examined in the Iranian Ministry of Environment laboratories. The means and standard errors of the means (mean ±*SE*) were calculated for each experiment, which was carried out in triplicate.

The findings and discussions

Factors influencing the efficiency of manganese chloride coagulation:

– The acid concentration effect: Figure 2 shows the relationship between the amount of HCl present and the amount of $MnCl_2$ generated. The results show that $MnCl_2$ generation increases as acid concentration increases up to a limit of 30% HCl content.
– The agitation forces effect: Figure 3 shows that the stirring velocity is a crucial factor in completing the reaction and achieving the best results. The results indicated that the maximum concentration of $MnCl_2$ was formed at a stirring velocity of 300 rpm.
– The contact time effect: Figure 4 illustrates the impact of contact time on the amount of $MnCl_2$ created. The results indicated that the synthesis of $MnCl_2$ increased with the length of contact between the reagents until the time limit; 100 min was the optimum time.
– The reaction temperature effect: Figure 5 illustrates that the production of $MnCl_2$ increases with temperature until a limit degree. The results showed the percentage of $MnCl_2$ produced rises with temperature. At 80°C, production conditions are ideal.

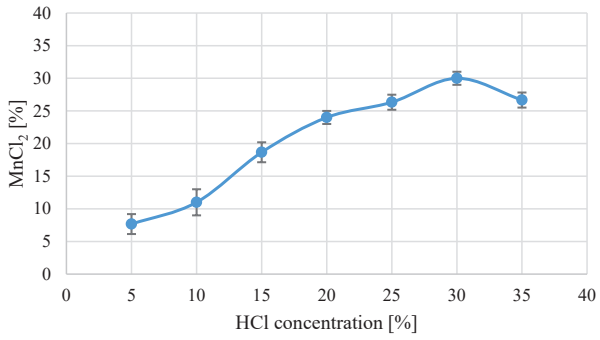

FIGURE 2. Effect of hydrochloric acid concentration on manganese chloride productivity
Source: own work.

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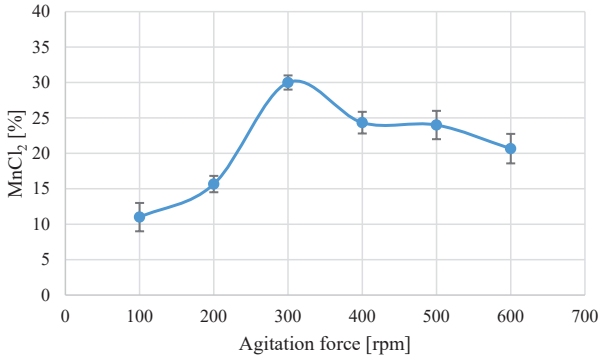

FIGURE 3. Effect of agitation forces on manganese chloride productivity
Source: own work.

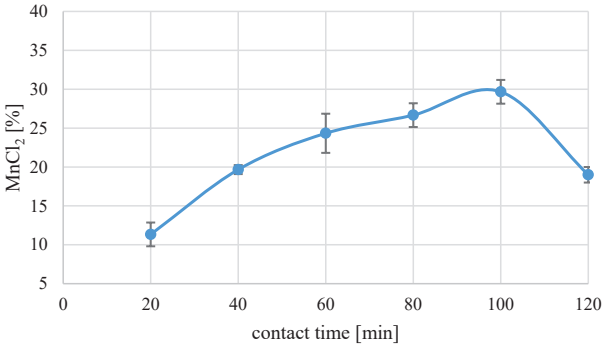

FIGURE 4. Effect of contact time on manganese chloride productivity
Source: own work.

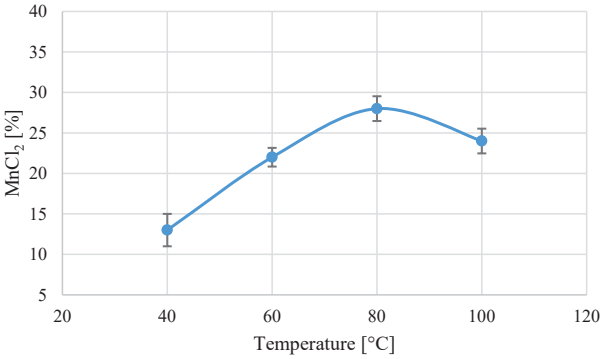

FIGURE 5. Effect of temperature on manganese chloride productivity
Source: own work.

Comparative decolorization between recovered and alum coagulant

Table 3 and Figure 6 illustrate the comparison of color removal efficiency for two coagulants at the variable dosages. The order of coagulant performance was as follows: recovered poly manganese chloride coagulant with an optimum dosage of 30 mg·l^{-1} achieved removal efficiencies of 90.33% and 86.11% for RY17 and DB53, respectively, while pure alum coagulant at the same dosage (30 mg·l^{-1}) achieved removal efficiencies of 85.42% and 80.34% for RY17 and DB53, respectively, which gives the recovered coagulant an advantage in removing the dyes over the alum coagulant.

Table 3. Summary of coagulant dosage and removal efficiency results

Coagulant	Dose of coagulants[mg·l^{-1}]	RY17 pollutant		Removal efficiency [%]	DB53 pollutant		Removal efficiency [%]
		initial turbidity [NTU]	final turbidity [NTU]		initial turbidity [NTU]	final turbidity [NTU]	
Recovered	30	24.93	2.41	90.33	26.82	3.71	86.11
Alum	30	24.93	3.63	85.42	26.82	5.27	80.34

Source: own work.

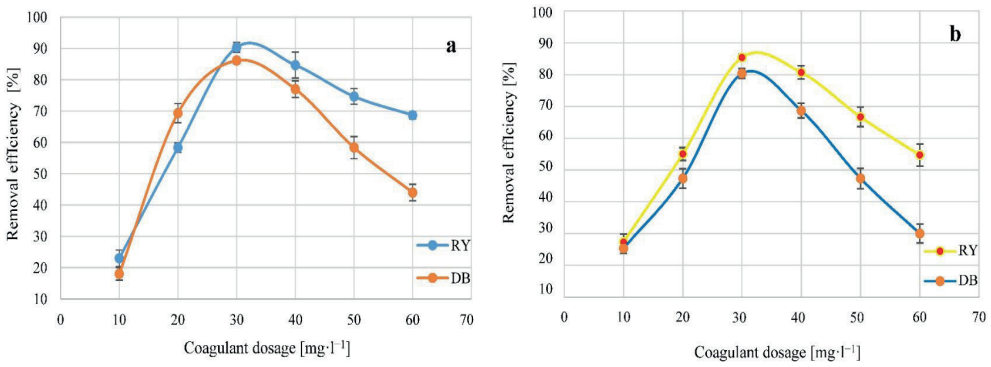

FIGURE 6. Effect of coagulant dosage on RY77 and DB53 removal efficiency by using poly manganese chloride (a) and alum (b)

Source: own work.

This supports findings from prior research showing that treatability performance increases with increasing coagulant dosage until agglomeration saturation is reached, at which point performance begins to stabilize or decline (Bressane et al., 2023). Because of the inverted net charge on suspended solids in wastewater, this resulted in a notable decrease in contaminant removal (Daud et al., 2015).

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Additionally, because an overdose would have prevented a polymeric chain reaction and allowed contaminants to find vacant places for adsorption bridging with a higher possibility of sweeping, it might have resulted in re-stabilization (Abreu et al., 2020). Furthermore, either too much or too little dosage might have a detrimental effect on the coagulation treatment procedure, increasing the expense of using chemicals (Kukić et al., 2018; Maurya & Daverey, 2018).

Scanning electron microscope test results

Figure 7 shows the SEM images of the coagulant grains at a scale of 20 μm with landing energy capacity of 15 kV. The crystal shape in the SEM micrograph indicates the poly manganese chloride in the sample. The manganese oxide could be the black spots, also explaining the ratios of manganese and chlorine which are compatible with the elemental analysis of poly manganese chloride. The agglomeration of the large flocs was facilitated by rough surfaces with vast heterogeneity, inconsistent form, and mesoporosity (Dos Santos et al., 2018). Recovered coagulant macromolecules showed highly selective, high-affinity manganese ions that improved precipitation. This indicates that morphological surfaces are strongly bonded to manganese ions, facilitating both adsorption and agglomeration. Furthermore, in the case of alum as a coagulant, the surface charge of colloidal particles in solution contributes to their destabilization, particularly when their settling process is slow, resulting in a state of apparent stability within the dispersion. A slight decrease in zeta potential combined with an increase in ionic strength can cause colloidal stability and destabilization (Tisti & Ghawi, 2020).

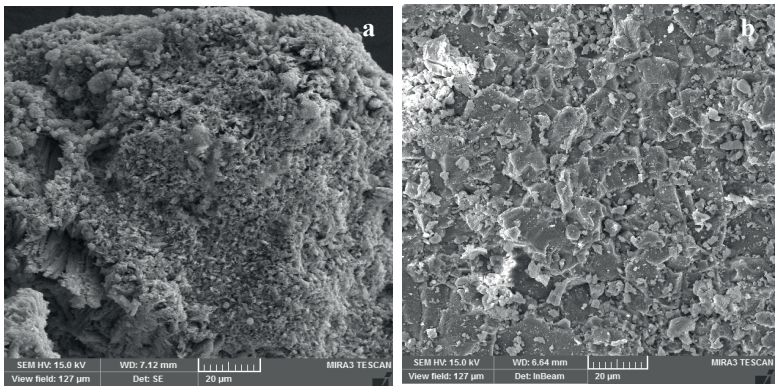

FIGURE 7. SEM images of poly manganese chloride (a) and alum (b)
Source: own work.

The energy dispersive spectrometer (EDS) was employed to analyze the alterations in support characteristics for poly manganese chloride. Figure 8a illustrates the energy dispersive spectrometer EDS techniques. The findings indicated that there was approximately 38.45 wt% of element Mn, followed by a 33.15 wt% of element O, and elements C, Si, Fe, Cl, and Zn at about 9.01 wt%, 11.93 wt%, 3.62 wt%, 2.16 wt%, and 1.68 wt%, respectively. In alum, the elements Al and O were about 26.20 wt% and 48.77 wt%, respectively, while C and S were about 9.17 wt%, and 15.86 wt%, respectively, as shown in Figure 8b.

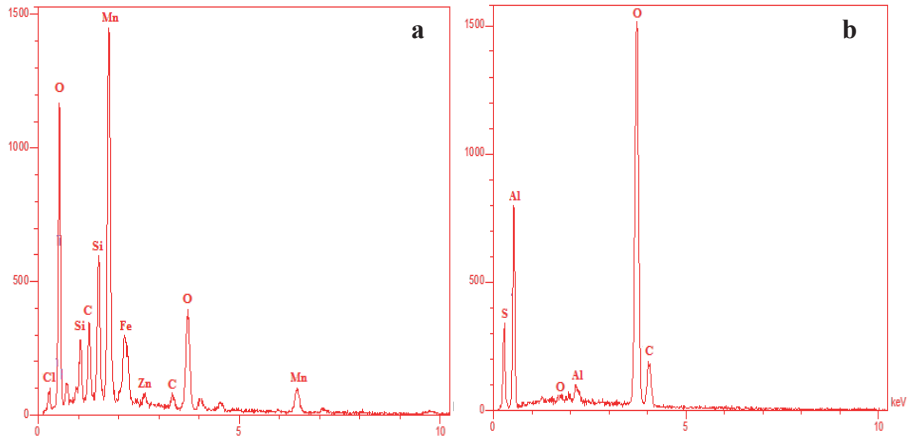

FIGURE 8. EDS analysis of poly manganese chloride (a) and alum (b)
Source: own work.

Fourier transform infrared test results

FTIR analysis of poly manganese chloride is shown in Figure 9a, where the hydroxyl vibration bands at 2,890 cm^{-1} are stretched. At 1,627 cm^{-1}, the absorption band is assigned to the OH bending vibration and the C–O stretching vibration. The asymmetric stretching vibration of Mn–OH–Mn and the Si–O bending and stretching vibrations are associated with the bands at 1,150 cm^{-1} and 1,210 cm^{-1}. Furthermore, three peaks for poly manganese chloride were identified at 1,270 cm^{-1}, 1,330 cm^{-1}, and 1,400 cm^{-1}; they were explained as bending vibrations of Mn–OH (Zhou et al., 2014).

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Alum's FTIR spectra, as shown in Figure 9b, showed a large peak at 3,442 cm^{-1} because of the presence of O–H groups in the material (Rong et al., 2013). A peak at 1,632 cm^{-1} indicates H–O–H stretching, which may have been caused by the hydroxyl group in the alum. Additionally, a peak at 531 cm^{-1} indicates Al–O stretching vibrations. Possible explanations for the other peaks at 1,060 cm^{-1} and 980 cm^{-1} include SO_4 stretching and the likely HOO matrix, respectively (Singh et al., 2012). The presence of OH and HOO groups may result from hydrogen bonds that occur within alum as it forms a variety of hydrates, the most prevalent of which are the octadecahydrate $Al_2(SO_4)_3 \cdot 18H_2O$ and the hexadecahydrate $Al_2(SO_4)_3 \cdot 16H_2O$.

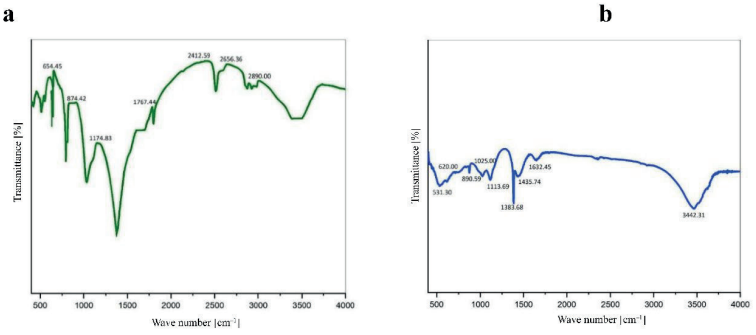

FIGURE 9. FTIR spectra of poly manganese chloride (a) and alum (b)
Source: own work.

Quality water evaluation of dye samples

COD and TOC were used to evaluate how well recovered poly manganese chloride reduced the organic pollutant in the tested colors. According to Figure 10, the COD values of RY17 and DB53 were calculated to be 788 $mg \cdot l^{-1}$ and 895 $mg \cdot l^{-1}$, respectively, before treatment. However, following poly manganese chloride treatment, the COD significantly decreased to 150 $mg \cdot l^{-1}$ and 210 $mg \cdot l^{-1}$, respectively. Observed percentage reductions in COD values were 80.96% for RY17, and 76.53% for DB53. The TOC values for RY17 and DB53 were estimated to be 680 $mg \cdot l^{-1}$ and 710 $mg \cdot l^{-1}$, respectively, before to treatment. However, following poly manganese chloride treatment, the TOC drastically decreased by 83.82% and 80.28%, respectively, as seen in Figure 11.

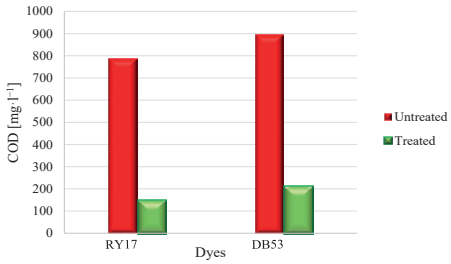

FIGURE 10. COD values of textile dyes before and after

Source: own work.

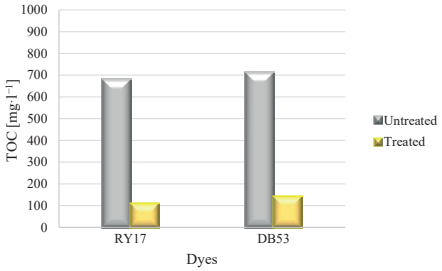

FIGURE 11. TOC values of textile dyes before and after

Source: own work.

Another study examined the use of sequential ozonation and an up-flow biological aerated filter method to remove color and COD from wastewater containing reactive dyes; the color and COD removal efficiencies were 97% and 90%, respectively (Lu et al., 2009). Another study used polyurethane foam (PUF) immobilized microbial consortia to reduce the COD and TOC levels of a carcinogenic azo dye, Congo red (CR), by 85% and 83%, respectively (Lade et al., 2015).

Conclusions

Among the available dye removal techniques, coagulation is extensively applied because of its simplicity and high efficacy. Recovered poly manganese chloride and alum are the coagulants applied to remove this pollutant. Manganese chloride can be recovered from industrial sludge to produce poly manganese chloride, which is an effective coagulant for treating textile dyes. The best recovery efficiency

in the recovered coagulant is achieved with a 30% HCl concentration, 300 rpm stirring speed, 100 min of reaction time, and 80°C reaction temperature according to the current analysis of lab experiments. The optimum dose for the recovered coagulant was 30 $mg \cdot l^{-1}$. The decolorization and reduction of the organic pollutant by poly manganese chloride are better than those by alum at the same dose, which shows the clear superiority of poly manganese chloride over alum in removal. In summary, our study has found an economical and effective way to create environmentally friendly wastewater treatment solutions. Additionally, money will be saved and industrial sludge, one of the most valuable resources given the global situation, will be safely reused by implementing this technique at the application level.

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Summary

Recovered poly manganese chloride ($PMnCl_2$) from industrial waste sludge to decolorize and detoxify textile dyes and comparison with alum. Th coagulation process for treating wastewater pollutants, due to its simplicity and safety, has received growing attention for a while. In this research, manganese chloride in industrial wastewater sludge can be recovered to produce poly manganese chloride as an effective and low-cost coagulant for the treatment of industrial pollutants. However, in recovering manganese chloride, there are some factors that affect efficiency, such as hydrochloric acid concentration, agitation force during acidification, contact time, and temperature. To describe the coagulant's morphological and elemental structure, scanning electron microscopy (coupled with energy dispersive spectroscopy) and Fourier transform infrared

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spectroscopy were used. The purpose of this research is to determine the ideal recovery coagulant conditions and assess this coagulant's efficacy in comparison to a conventional coagulant, alum, to treat textile dyes reactive yellow (RY17) and direct blue (DB53). In this paper, the results show that the optimum acidification concentration was 30% with a stirring speed of 300 rpm for 100 min at 80°C. Using a jar test, the optimum dose for the recovered coagulant was 30 $mg \cdot l^{-1}$. The decolorization of RY17 and DB53 was found to be 90.33% and 86.11%, respectively. The chemical oxygen demand and total organic carbon were reduced by 80.96% and 83.82%, respectively, for RY17, while for DB53 were reduced by 76.53% and 80.28%, respectively. At the same dose of alum, the decolorization of RY17 and DB53 was 85.42% and 80.34%, respectively. The decolorization performance illustrated that at the same dosage the recovered coagulant has slightly higher quality than the alum coagulant.