Evaluation and comparison the performance of titanium and zirconium(IV) tetrachloride in textile wastewater treatment

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A R T I C L E   I N F O

Article history:
Received 5 January 2018
Received in revised form 16 March 2018
Accepted 23 March 2018
Available online 28 March 2018

Keywords:
Textile wastewater
Coagulation
Titanium tetrachloride
Zirconium tetrachloride

A B S T R A C T

Wastewater treatment is a key challenge in the textile industry. The current treatment methods for textile wastewater are insufficient or ineffective for complex dyes generated from the textile industry. This study evaluated the performances of two novel inorganic coagulants with high cationic charges, namely, titanium tetrachloride (TiCl4) and zirconium tetrachloride (ZrCl4). They were utilised to treat textile industry wastewater. Both coagulation processes were performed under the same experimental operational conditions. Turbidity, suspended solids (SS), colour, chemical oxygen demand (COD) and ammonia were measured to assess the efficiencies of the coagulants. Results indicated that ZrCl4 and TiCl4
exhibited high potentials for textile wastewater treatment. ZrCl4 presented high removal efficiency in COD and SS, whereas TiCl4 showed excellent removal in ammonia.

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Specifications Table

| Subject area            | Environmental Engineering |
|-------------------------|---------------------------|
| More specific subject area | Wastewater treatment    |
| Type of data            | Tables and figures        |
| How data was acquired   | All experiments were performed in 1000 mL glass beakers using jar test unit. ZrCl4 and TiCl4 were used as coagulants to treat textile wastewater samples. COD concentration, TSS, colour and ammonia were measured before and after each run, and the removal efficiencies were calculated. |
| Data format             | Analysed                  |
| Experimental factor     | Monitoring the removal efficiencies of turbidity, suspended solids, colour, COD, and ammonia from textile wastewater after each coagulation process. |
| Experimental features   | Treatment of textile wastewater using ZrCl4 and TiCl4 as a coagulation process, and compare the performance of both coagulants based on the maximum removal efficiencies for each parameter. |
| Data source location    | School of Civil Engineering, Engineering Campus, Universiti Sains Malaysia, 14300 NibongTebal, Penang, Malaysia |
| Data accessibility      | Data were presented in the article. |

Value of the data

- This article presents the data on the performances of two coagulants, ZrCl4 and TiCl4, in textile wastewater treatment
- The focus was on the comparison of the removal efficiencies of the parameters in textile wastewater, such as COD, TSS, colour and ammonia, under the effects of both coagulants.
- The dataset could also be used for reducing other parameters from other types of industrial wastewater, which is a challenging pollutant of natural water bodies.

1. Data

This study aimed to evaluate and compare the performances of titanium tetrachloride (TiCl4) and zirconium tetrachloride (ZrCl4) as coagulants in textile wastewater treatment. The performances of both coagulants were compared through the removal of such parameters as turbidity, suspended solids (SS), ammonia, chemical oxygen demand (COD) and colour. Table 1 presents the general characteristics of textile wastewater compared with Standard B of Environmental Quality (Sewage and Industrial Effluents) Regulation 2012 under the Environmental Quality Act 2012. Figs. 1–5 show the performances of ZrCl4 and TiCl4 in removing turbidity, SS, colour, ammonia and COD, respectively, under the effects of different coagulant dosages at natural wastewater pH. Figs. 5–10 present the effect of pH variation on the performances of the two coagulants (ZrCl4 and TiCl4) for turbidity, SS, colour, ammonia and COD removals. Table 2 summarise the operational conditions and performances of ZrCl4 and TiCl4 in parameter removal.
Table 1
Characteristics of textile wastewater compared with Standard B of Environmental Quality (Sewage and Industrial Effluents) Regulation 2012 under the Environmental Quality Act 2012.

| Parameter       | Textile wastewater | Standard B                  |
|-----------------|--------------------|-----------------------------|
|                 | Untreated¹         | Treated²                    |
| pH              | 11.8               | 7.9                         | 5.5 – 9.0 |
| COD (mg/L)      | 998                | 552                         | 250       |
| Turbidity (NTU) | 159                | 2.31                        | –         |
| Colour (Pt.Co)  | 1020               | 860                         | –         |
| Suspended Solid (mg/L) | 540               | 40                           | 100       |
| Ammonia (mg/L)  | 65                 | 8.2                         | –         |
| BOD₅ (mg/L)     | 80                 | 24                          | 40        |

Fig. 1. Actual (NTU) and percentage turbidity removals using ZrCl₄ and TiCl₄ as coagulants.

Fig. 2. Percentage SS removals by ZrCl₄ and TiCl₄ as coagulants and SS concentrations after treatments.
Fig. 3. Percentage removals of colour by ZrCl₄ and TiCl₄ coagulants and their residual (PtCo) after treatment.

Fig. 4. Percentage removal of ammonia for ZrCl₄ and TiCl₄ as coagulant in textile wastewater treatment.

Fig. 5. Percentage removals of COD by ZrCl₄ and TiCl₄ as coagulants in textile wastewater.
2. Experimental design, materials and methods

2.1. Sampling

A textile wastewater sample was collected from a textile factory in Prai Industrial Estate, Penang, was immediately transferred to the laboratory and was stored at 4 °C, in accordance with the Standard Methods for the Examination of Water and Wastewater [1]. The parameters measured for assessing the coagulants were pH, colour, turbidity, SS, COD, biochemical oxygen demand, ammonia, alkalinity and hardness.

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**Fig. 6.** Turbidity results of the ZrCl4 and TiCl4 treatments of textile wastewater over the 3–8 pH range.

**Fig. 7.** Removal of SS by ZrCl4 and TiCl4 as coagulants in textile wastewater treatment.
2.2. Preparation of stock solution

Stock solutions of both coagulants were prepared before each experiment. The TiCl4 stock solution was prepared by adding 1 mL of TiCl4 to 0.2 M hydrochloric acid. A 5000 mg/L stock solution of ZrCl4 was prepared by dissolving 5 g of ZrCl4 in 1000 mL of distilled water. These stock solutions were stored for approximately 14 h at room temperature to dissolve the metal salts [2].

2.3. Coagulation-flocculation

Jar testing was conducted under standardised conditions on wastewater to evaluate coagulant dosages and conditions required to achieve the optimum treatment process. A conventional jar test apparatus was used in experiments to evaluate the performances of ZrCl4 and TiCl4 in treating textile wastewater. An automated jar test apparatus, which consisted of six 1000 mL beakers and six-spindle steel paddles, was used. The jar testing was performed after leaving the textile samples at ambient...
temperature for 2 h. Stock solutions of ZrCl$_4$ and TiCl$_4$ coagulants were prepared before each experiment. The sample of textile wastewater was mixed with the coagulants before being poured into 500 mL beakers. After coagulant dosing into the wastewater samples, rapid mixing at 250 rpm for 2 min and slow mixing at 30 rpm for 20 min were performed. Flocs were allowed to settle for 30 min [2,3]. The treated wastewater samples were collected using a syringe from the supernatant surface for parameter measurements. In the jar test experiments, the TiCl$_4$ doses were varied from 100 mg/L to 800 mg/L at pH 3 to pH 8. The ZrCl$_4$ doses were varied from 300 mg/L to 800 mg/L at pH 4.5 to pH 7. The turbidity, colour, ammonia, COD and SS before and after treatment were measured. pH was measured on-site by using a portable digital pH/mV meter (WITEG, W-100, Germany) [4]. Colour measurements were reported as true colour (filtered using a 0.45 $\mu$m filter paper) at 455 nm using DR2800 HACH spectrophotometer in accordance with the Standard Methods for the Examination of Water and Wastewater [1] (Method No. 2120 C). The result was reported in platinum–cobalt (PtCo) which was the unit of colour being produced by 1 mg platinum/L in the form of chloroplatinate ion. Turbidity was determined using a DR/2100 turbidimeter. SS was measured using a DR2800 spectrophotometer in accordance with the HACH standard: Photometric Method 8006. Ammonia was determined using a DR2800 spectrophotometer in accordance with the Nessler method [5], adopted

Fig. 10. COD removal by ZrCl$_4$ and TiCl$_4$ as coagulants in textile wastewater treatment.

| Item               | ZrCl$_4$ | TiCl$_4$ |
|--------------------|----------|----------|
| Operating condition pH | 6        | 6        |
| Coagulant dose(mg/L)  | 500      | 300      |
| Rapid mixing time(min) | 1        | 1        |
| Rapid mixing speed(rpm) | 250     | 250      |
| Slow mixing time(min) | 20       | 20       |
| Slow mixing speed(rpm) | 30       | 30       |
| Settling time (min)   | 30       | 30       |

| Removal rate (%) | ZrCl$_4$ | TiCl$_4$ |
|------------------|----------|----------|
| Turbidity        | 97       | 97       |
| Colour           | 60       | 68       |
| Suspended solids | 99       | 98       |
| COD              | 36       | 28       |
| Ammonia          | 67       | 28       |

Table 2
Summary of the comparison of the performances of ZrCl$_4$ and TiCl$_4$ as coagulants.
from the Standard Methods for the Examination of Water and Wastewater 4500-NH₃ B and C. COD was determined in accordance with Method 5220D (closed reflux, colourimetric method) [5]. The removal efficiencies of turbidity, SS, colour, ammonia and COD were obtained using the following equation:

\[
\text{Removal(\%)} = \left( \frac{C_i - C_f}{C_i} \right) \times 100
\]

Where \(C_i\) and \(C_f\) are the initial and final concentrations of leachate, respectively.

Acknowledgment

This work is funded by the Ministry of Education Malaysia under FRGS grant scheme (Grant no. 203/PAWAM/6071280) for research associated with the Solid Waste Management Cluster, Engineering Campus, Universiti Sains Malaysia.

Transparency document. Supporting information

Supplementary data associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.dib.2018.03.113.

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