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Performance of flocculation titanium salts for seawater reverse osmosis pretreatment

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ABSTRACT

This study evaluated the performance of titanium tetrachloride ($TiCl_4$) and titanium sulphate ($Ti(SO_4)_2$) as coagulants to remove organic matter and solids from actual seawater. The coagulant performances were evaluated at different doses in terms of turbidity, dissolved organic carbon (DOC), humics (UV_{254}), zeta potential and pH of the solution. The performance of Ti-salts were compared to ferric chloride ($FeCl_3$), a commonly used coagulant. The results showed that at pH of 8.0 (closely similar to seawater pH), $TiCl_4$ showed relatively better performance over $FeCl_3$ and $Ti(SO_4)_2$ for the same coagulant dose of 20 mg/L. $TiCl_4$ achieved a 70% DOC and UV_{254} removal. This was approximately two times higher than achieved by $FeCl_3$ and $Ti(SO_4)_2$. Based on a floc zeta potential evaluation, the difference in performance of the coagulants were attributed to the coagulation mechanism. The coagulation mechanisms of Ti-salts were mainly charge neutralization while $FeCl_3$was adsorption mechanism.

Keywords: Ferric chloride; Flocculation; Seawater; Titanium sulphate; Titanium tetrachloride

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1. Introduction

Water scarcity is a major problem in many parts of the world especially in arid regions [1]. Seawater desalination offers the potential of meeting fresh water demands. Among desalination technologies, seawater reverse osmosis (SWRO) is widely used at desalination plants for capabilities such as ease of operation and cost-effective drinking water production [2]. Nevertheless, a key challenge of SWRO is membrane fouling, specifically, organic, colloidal and bio-fouling [3]. This underlines the importance of pretreatment of seawater. Pretreatment can reduce membrane fouling and potentially extend the life span of SWRO membranes. Pretreatments are commonly grouped into two categories; conventional and non-conventional.

Conventional pretreatment includes acid addition, coagulation-flocculation, disinfection and media filtration [4–7]. Coagulation–flocculation is a well-established SWRO pretreatment that can remove particles, colloids and organics in water and wastewater treatment [4,6]. Coagulation applies chemicals to assist particulates in water to adhere together. Flocculation leads to the formation of larger particles that easily settle in water which can be removed.

Aluminium sulphate ($Al_2(SO_4)_3$), ferric chloride ($FeCl_3$), polyferric sulphate (PFS) and polyaluminum chloride (PACl) are some examples of commonly used coagulants for water and wastewater treatment [8]. Al-salts are suspected of being harmful to human life and living organism [9]. By comparison, $FeCl_3$ as an Fe-salt flocculant is more widely applied due to its better DOC removal efficiency with no significant toxicity [10–12]. However, $FeCl_3$ flocculation produces a large amount of sludge (the settled floc) that needs to be disposed [11].

The quest for new coagulants in water treatment to meet increasingly stringent guidelines has led to renewed interest in Ti-salt. The possibility of using Ti-salt as coagulant

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in water treatment was first investigated by Upton and Buswell [13]. Subsequently, a number of studies evaluated the performance of Ti-salts such as $TiCl_4$ and $Ti(SO_4)_2$ for wastewater treatment using synthetic solutions [14,15]. Similarly, Zhao et al. [16] used reservoir water to compare the coagulation effect of $TiCl_4$ with PACl. These studies highlighted the superior flocculation of Ti-salt and the additional advantage of being able to recover valuable TiO_2 from the sludge. Although these studies analysed the performance of Ti-salt, the application of Ti-salts in seawater has not been analysed on detail.

Presently, only a few studies have evaluated the suitability of applying Ti-salt flocculation for seawater pretreatment. For instance, Okur et al. [17] analysed the capacity to produce TiO_2 from sludge after Ti-salt flocculation of seawater. Jeong et al. [18] studied in detail organics removal by $TiCl_4$. In a recent study, Checkli et al. [19] evaluated the performance of Ti-salts for the removal of algal organic matter in seawater. These studies compared the results with that of $FeCl_3$ coagulant and highlighted the efficiency of Ti-salts to reduce low molecular weight (LWM) organics in seawater. LMW organics are associated with biofouling development in SWRO membranes [7,20]. Nevertheless, conditions to enhance the performance of Ti-salts flocculation of actual seawater in terms of coagulant mechanism, the influence of dose and pH has not been evaluated in detail.

In this study, the performance of Ti-salts ($Ti(SO_4)_2$ and $TiCl_4$) and $FeCl_3$ coagulants in flocculating seawater were investigated in terms of turbidity, organics removal (DOC andUV_{254} absorbance) and zeta potential at varying coagulant doses (1 to 30 mg/L) and pH (5–9).

2. Materials and methods

2.1 Feed solution

Seawater was collected from Cabarita, Sydney. The characteristics of seawater are shown in Table 1.

2.2. Coagulants

In this study, different doses of coagulants (1.0 to 30.0 mg/L) were used. $FeCl_3 \cdot 6H_2O$ stock solution (i.e. 10 g/L) was prepared by dissolving $FeCl_3$ powder in deionized (DI) water. $TiCl_4$and $Ti(SO_4)_2$ stock solution was prepared by adding concentrated solution drop by drop to frozen cubes of deionized water to obtain a final concentration of 1% w/w. All chemicals were reagent grade (>99% purity; Sigma Aldrich, Australia).

Table 1
Seawater characteristics

Specification	Value
UV_{254} (cm^{-1})	0.029–0.049
Turbidity (NTU)	6.1–6.7
DOC (mg/l)	1.9–2.9
pH	7.8–8.0
Zeta potential (mV)	–1.86
Total suspended solid (mg/L)	5.0–10.0

2.3. Jar-test

Flocculation was conducted using a programmable jar test apparatus (PB-900TM, Phipps and Bird, USA). For each jar test, beakers were filled with raw seawater (500 mL) and different coagulants (Ti salts and $FeCl_3$) at varying concentrations (1–30 mg/l) and pH (5–9). Here the concentration was expressed as mg of $FeCl_3$/L or $TiCl_4$/L. The seawater pH was adjusted with 0.1 N solution of HCl and NaOH prior to coagulant addition. The solution was subjected to rapid mixing (100 rpm) for 2 min followed by slow mixing (20 rpm) for 30 min. It was then stopped to allow aggregated flocs to settle for 30 min. After settling, the supernatant samples were drawn from around 5 cm below the water surface without disturbing the sediment of aggregated flocs in the sample to measure turbidity, UV_{254} absorbance and DOC, and zeta potential. The water samples were pre-filtered using a 0.45 µm membrane syringe filter before the UV_{254} and DOC analysis while residual turbidity and zeta potential were directly measured as discussed in Section 2.4.

2.4. Analytical methods

The zeta potential charge value of the samples were measured using a zetasizer (Malvern Instruments, UK). After 2 mins of stabilisation, the data was recorded automatically. Turbidity was measured using a 2100P turbidimeter (Hach, USA). The initial and final pH values of the solution for each experiment were measured using pH meter (Hach, USA).

The water samples were pre-filtered using 0.45 µm membrane syringe filter before measuring DOC and UV_{254} absorbance. DOC concentration was measured using liquid chromatography–organic carbon detector (LC-OCD) (LC-OCD model 8, DOC Labor, Germany) [21]. The UV_{254} absorbance was measured with a spectrophotometer (UV-1700 UV-VIS, Shimadzu, Japan) at 254 nm wavelength using a quartz cells.

Sample analysis were carried out in triplicates and the average results were reported in this study.

3. Result and discussion

3.1. Performance of $TiCl_4$, $Ti(SO_4)_2$ and $FeCl_3$ flocculation with seawater

3.1.1. Turbidity removal efficiency

The removal of turbidity from seawater (average pH = 8.0, turbidity = 6.67 NTU) increased as the coagulant dose was increased from 1 to 15 mg/L. The highest turbidity removal of 71–73% was achieved at a dose of 15 mg/L for all coagulated water (Fig. 1a). At higher doses (20–30 mg/L), a slight reduction in turbidity removal was observed. This was especially apparent for with $FeCl_3$ coagulant. A similar pattern of turbidity removal from seawater at higher $FeCl_3$ doses was observed in other studies [6,22]. This was because the addition of more $FeCl_3$ at higher concentrations generated ferric hydroxide and ferric oxide which caused an increase in turbidity after coagulation. Okur et al. [17] reported the presence of micro-suspended flocs with Ti-salts and $FeCl_3$ in seawater, causing increased turbidity at higher coagulant doses. They therefore applied a hybrid flocculation followed by media filtration.

3.1.2. DOC and UV_{254} removal efficiency

A pattern of higher removal efficiency of DOC (Fig. 1b) and UV_{254} (Fig. 1c) was observed as coagulant dosages were increased in seawater (average pH = 8.0, DOC = 2.0 mg/L). Better DOC and UV_{254} removal efficiency was achieved with the $TiCl_4$ coagulant than with $Ti(SO_4)_2$ and $FeCl_3$. A 68% and 71% removal of DOC and UV_{254} was obtained at 20 mg/L dose of $TiCl_4$ with a slightly lower removal (61–65%) at 30 mg/L. By comparison, only 34–40%removal of DOC and UV_{254} was achieved with a 30 mg/L dose of $FeCl_3$. Jeong et al. [18] and Checkli et al. [19] similarly reported the superior removal of DOC by $TiCl_4$ compared to $FeCl_3$. The difference in DOC removal efficiency achieved by the coagulants could be attributed to the coagulant mechanism. It is also worth highlighting that Checkli et al. (2017) reported significantly lower coagulant dose requirement (5 mg/L) in achieving 80% DOC removal in synthetic seawater containing high algal organic matter. This was associated with the presence of algal organic matter acting as polymer aid which decreased the coagulant requirement. The coagulant mechanism has not been widely reported with regards to flocculation in actual seawater. As such, a further evaluation on the charge (zeta potential) was carried out in Section 3.2. The changes in floc zeta potential are used to analyse the coagulation mechanism in terms of charge neutralization and sweep flocculation [19,23].

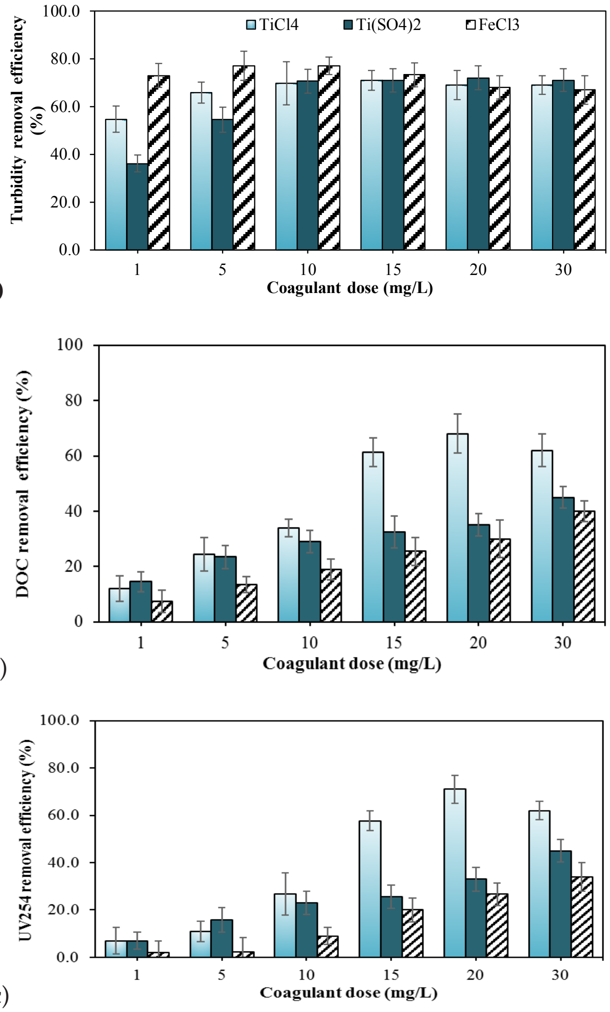

Fig. 1. Performance of $TiCl_4$, $Ti(SO_4)_2$ and $FeCl_3$ in terms of (a) turbidity, (b) UV_{254} and (c) DOC removal efficiencies at different coagulant doses (sea water solution: average pH = 8.0; turbidity = 6.67 NTU; UV_{254} = 0.030 cm^{-1} and DOC = 2.1 mg/L).

Overall, the results indicated the substantially higher removal of DOC with $TiCl_4$ at lower coagulant dose. By 15 mg/L $TiCl_4$ dose, more than 60% DOC removal was achieved from seawater. Comparatively at 30 mg/L dose, only 35–40% DOC removal was achieved by $Ti(SO_4)_2$ and $FeCl_3$. This is about twice the amount of $TiCl_4$ dose. The lower $TiCl_4$ coagulant dose required to achieve substantially high DOC removal from seawater will be an added advantage in terms of the lower sludge production potential in actual SWRO plants. This factor is highlighted in Section 3.4.

3.2. Influence of charge (zeta potential)

All three coagulants showed a similar pattern of negative values of zeta potential at lower doses followed by a shift towards positive values of zeta potential at high doses (Fig. 2). However, $TiCl_4$ showed a significant shift to positive values of zeta potential at doses higher than 15 mg/L (3.0 mV for $TiCl_4$ at 30 mg/L). By comparison, $FeCl_3$ and $Ti(SO_4)_2$ showed negative values of zeta potential with a slight shift from –1.56 to –1.65 mV at 1 mg/L doses to –0.52 to –1.34 mV at 30 mg/L.

As the dose of $TiCl_4$ coagulant was increased, the negative charge of colloidal particles decreased and an isoelectric point appeared at about 20 mg/L. In line with this, the highest DOC and UV_{254} removal efficien-

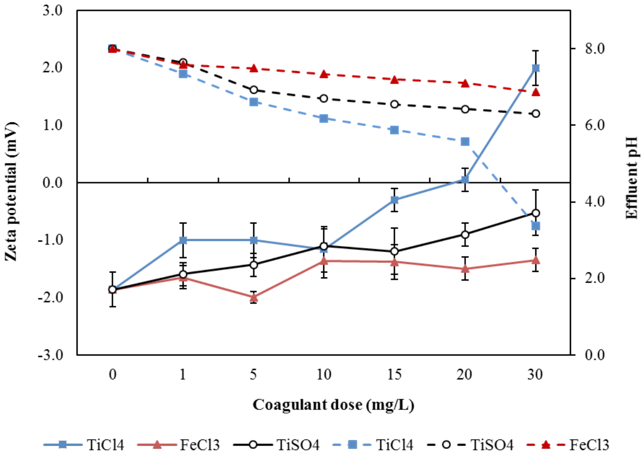

Fig. 2. $TiCl_4$, $Ti(SO_4)_2$ and $FeCl_3$ zeta potential (------) and effluent pH (------) at different coagulant doses (seawater feed solution: average pH = 7.84 ± 0.02; zeta potential = –1.86 mV).

cies (UV_{254} of 71% and DOC of 68%) was achieved at $TiCl_4$ dose of 20 mg/L. At this dose, the zeta potential was close to the isoelectric point. At even larger doses, a pattern of positive zeta charge became more dominant, which may have led to electrostatic repulsion between particles. This can possibly explain the slightly lower DOC and UV_{254} removal efficiencies at 30 mg/L (Fig. 1b and c). This may have also caused a charge repulsion among the particles because of similar charges resulting in a lower turbidity removal efficiency compared to $Ti(SO_4)_2$ and $FeCl_3$ (Fig. 1a). Based on the zeta potential results, charge neutralization appears to play a dominant role in the flocculation process using the $TiCl_4$ coagulant. Charge neutralization is a well-established coagulation mechanism [23,24]. Nevertheless, apart from charge neutralization, high removal efficiency can be associated to the change in coagulation mechanism from dominant charge neutralization to sweep flocculation. In this condition, coagulation is enhanced due to adsorption and enmeshment in metal hydroxide precipitates [19].

$Ti(SO_4)_2$ showed the same zeta potential pattern as $TiCl_4$, however with comparatively weaker charge value. This could be the reason for $Ti(SO_4)_2$displaying a lower DOC and UV_{254} removal compared to $TiCl_4$. Meanwhile, for $FeCl_3$, the low negative zeta potential values suggest that charge neutralization was rather weak. This may explain the lower DOC and UV_{254} removal efficiency of $FeCl_3$ (34–40%). In this regard, previous studies of $FeCl_3$ have highlighted that charge neutralization was not a dominant coagulation mechanism because its zeta potential remained negative. Rather precipitation entrapment as $Fe(OH)_3$ and adsorption mechanism play a more dominant role for humics removal by $FeCl_3$ at a pH range of 7–9 [23,25]. Therefore, the influence of initial solution pH on the different coagulant's removal mechanism is a key factor that requires evaluation. This is discussed in Section 3.3.

3.3. Influence of initial solution pH

3.3.1. Turbidity removal

The initial pH of the solution plays a significant role in influencing the turbidity removal pattern as shown in Fig. 1a. A pattern of higher turbidity removal was observed with increase of pH from 5 to 7. Beyond pH 7, a slight decrease of turbidity was observed, which was more apparent for $FeCl_3$. Overall, $FeCl_3$ coagulant displayed better turbidity removal compared to Ti-salts coagulants especially in acidic conditions. Generally, turbidity removal is associated with adsorption and physical entrapment of colloids, which appears to be the dominant coagulant mechanism of $FeCl_3$ as highlighted in Section 3.2. This could explain $FeCl_3$ better turbidity removal capacity compared to Ti-salts. At pH of more than 7, the decrease of turbidity removal with $FeCl_3$ might be attributed to the competition between hydroxyl ions and negatively charged colloidal particles.

3.3.2. DOC and UV_{254} removal

All coagulants showed a similar pattern of removal for DOC and UV_{254}. $TiCl_4$ achieved the highest removal efficiencies at pH of 8. Meanwhile $FeCl_3$ and $Ti(SO_4)_2$ achieved the highest removal efficiencies at pH < 6 with a gradual reduction at higher pH. The different pattern of DOC and UV_{254} removal for different coagulants shows that pH plays a significant role in the coagulant removal mechanisms. This could be related to coagulant hydrolysis as discussed next.

3.3.3. Zeta potential

The floc charge of $Ti(SO_4)_2$ and $FeCl_3$ showed a similar pattern, where the zeta potential values decreased from positive to negative with higher solution pH from 5 to 9 (Fig. 2b). Meanwhile $TiCl_4$ floc charge showed an opposite pattern with a change from negative to positive zeta values for pH change from 5 to 8. There was a decrease in the value of zeta potential when the pH increased from 8 to 9.

Generally, at different pH ranges, floc zeta potential changes correlate with hydrolysis variation of the coagulant. Studies have shown that the hydrolysis of coagulants such as Ti-salts and $FeCl_3$ are inhibited at pH 5 resulting in dominant positive monomer hydrolysates [26]. This enables these coagulants to neutralize negative charges of particles and organic matters, and destabilize colloids. Nevertheless, when the positive charges were not enough for full charge neutralization, flocs with negative charges were produced. This was especially apparent in the case of $TiCl_4$, where zeta potential remained significantly negative at pH 5–6. This could explain the reason for low DOC and UV_{254}removal at pH 5–6 for $TiCl_4$ (Fig. 2a). By comparison, at these pH ranges, $Ti(SO_4)_2$ and $FeCl_3$ displayed positive zeta potential, and both these coagulant achieved the highest DOC and UV_{254}removal at pH 5.

For $TiCl_4$, it is likely that when the pH was increased from 5 to 8, polymerichydrolysis with high positive charges and large surface area are formed [26,27]. The charge neutralization between negative colloids and the positive coagulant resulted in high DOC and UV_{254} removal (Fig. 2a) and produced flocs with positive charges. At pH higher than 8, the drop in zeta potential value suggests that bulk and rapid hydrolysis had likely occurred, producing coagulant species with low positive charges such as $Ti(OH)_4^-$. In this condition, complete neutralization was not achieved, producing the flocs with negative charges. As a result, DOC and UV_{254} removal in this pH range was lower. Similarly, for $TiSO_4$ and $FeCl_3$, at pH < 6, the reaction between the negatively charged pollutants and positively charged coagulants generated flocs with positive charges. Beyond pH 6, the drop in zeta potential value suggests that bulk and rapid hydrolysis had likely occurred, producing coagulant species with low positive charges such as Fe $(OH)_4^-$. The pH of seawater is in the range of 7.8–8.0 as shown in Table 1. At these pH ranges, a high DOC removal was achieved with $TiCl_4$ compared to $FeCl_3$ and $Ti(SO4)_2$. The results indicated the suitability of $TiCl_4$ for organic removal from seawater.

3.4. Feasibility of $TiCl_4$ as a seawater coagulant

An important aspect in establishing the feasibility of $TiCl_4$ as a coagulant is the level of residual Ti concentration in the treated effluent to ensure it does not result in any environmental health toxicity risk or does not contribute towards membrane fouling in the subsequent RO process. In this regard, studies have established the low level of residual Ti concentration in effluent [28,29]. For instance Wu et al. [28], reported on the low Ti concentration in the treated water. In another study, Lee et al. [29] used bioassay mortality and LC50 toxicity tools to evaluate the aquatic toxicity of $TiCl_4$ flocculation process (in wastewater, sewage effluent, and seawater) and TiO_2 recovered from the sludge. The results highlighted the low toxicity (low Ti residual) in both the supernatant and recovered TiO_2. These results indicate that the low residual Ti concentration is not likely to contribute towards RO membrane fouling.

The results of this study showed that at low doses of $FeCl_3$ (1–10 mg/L), good turbidity removal (around 80%) was achieved. Nevertheless, in terms of DOC removal, even at the highest tested dose of this study (30 mg/L), only 34% DOC removal was achieved with $FeCl_3$. Comparatively, at 15–20 mg/L $TiCl_4$, more than 65% DOC removal was achieved. Overall, the results highlighted the better removal efficiency of $TiCl_4$ to treat seawater at lower dose requirement. The lower dose requirement of $TiCl_4$ indicate its potentially lower sludge production compared to $FeCl_3$. Moreover, an added advantage of $TiCl_4$ application is the capacity to recover the sludge into TiO_2 [14,17]. Recovering TiO_2 from $TiCl_4$ sludge would be a practical approach to overcome sludge management issues arising from large-scale treatment plants and potentially economically viable. This is because TiO_2 has a wide range of industrial application in the field of cosmetics, paint, paper and solar cells [14].

Although the results does indicate the favourable performance of $TiCl_4$ for seawater coagulation, a thorough cost comparison must be carried out before the feasibility of applying $TiCl_4$ can be established.

4. Conclusions

This study evaluated the performance of $TiCl_4$, $Ti(SO_4)_2$ and $FeCl_3$ coagulants with seawater. The results showed that at a natural seawater solution pH of 8, $TiCl_4$ had advantages over $FeCl_3$ and $Ti(SO_4)_2$ at the same coagulant dose of 20 mg/L. $TiCl_4$ achieved higher removal of DOC and UV_{254}. Nevertheless, $FeCl_3$ and $Ti(SO_4)_2$ showed slightly better turbidity removal. At higher coagulant dose (30 mg/L), the turbidity removal of $TiCl_4$, was worse. The difference in the performance of the coagulants were associated with the coagulant mechanisms based on floc zeta potential evaluation. The coagulant mechanisms of Ti-salts were mainly charge neutralization while for $FeCl_3$was adsorption mechanism. A further study on the detailed floc characteristics such as the dynamic variation of the floc size and floc breakage would be useful to establish the suitability of $TiCl_4$as a coagulant for SWRO pretreatment.

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