Desalination and Water Treatment
www.deswater.com
doi: 10.5004/dwt.2022.28152

250 (2022) 181–188
February

Coagulation and flocculation of dye wastewater by $FeCl_3$ and mucilage extracted from dragon fruit peel (*Hylocereus undatus*) in regard of side effects caused by the use of PACl and PAM

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Received 4 August 2021; Accepted 9 January 2022

ABSTRACT

Dye wastewater is the major and most polluted stream of wastewater generated from textile industry, which poses serious problems for the environment. Coagulation and flocculation have been the most practiced methods for dye wastewater treatment. However, the use of aluminum based coagulants and synthetic polymer are associated to human health; hence, iron based coagulants and plant-based polymers are alternatives of interest. This study determined the removal of turbidity and other pollutants from dye wastewater by a coagulation-flocculation process using $FeCl_3$ and mucilage extracted from the peel of dragon fruit *Hylocereus undatus* (DFPM) in regard to their alternative role as abovementioned. According to Jar-tests' results, optimal coagulation conditions of $FeCl_3$ were typically found in pH 4.0–6.0 and settling time of 30–60 min, whereas, $FeCl_3$ dose varied in range of 0.15–0.26 mM depending on types of dye wastewater. The addition of DFPM (0.5–50 mg/L) after $FeCl_3$ (0.04–0.74 mM) resulted in turbidity removal up to 97%, corresponding to 10%–20% increase compared to those obtained with $FeCl_3$ alone. DFPM showed a comparable effect to the comercial flocculant – polyacrylamide (PAM) and the potential to decrease 10%–50% the amount of $FeCl_3$ used to obtain the similar turbidity removal. In comparison to polyaluminum chloride (PACl), $FeCl_3$ showed better coagulation performance on investigated dye wastewater; While achieving similar performance in turbidity, TSS, color and COD removal when combined with DFPM.

Keywords: Dye wastewater; Coagulation/flocculation; Dragon fruit; *Hylocereus undatus*; Mucilage; Turbidity

1. Introduction

Dye wastewater is the most polluted stream in textile industry, one of the biggest water consumers in the world [1,2]. Dyeing process consumes more than 100 m^3/ton fabric material and discharges 80% of this volume as wastewater [2], including up to 50% of dye applied [2,3]. As a result, dye wastewater commonly has strong color, chemical oxygen demand (COD), low biodegradability [4], and is likely harmful to living organisms [1,5]. So far, coagulation and flocculation have been the most practiced methods in treatment of such kind of wastewater, especially in developing countries [1].

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Aluminum salts are the most used inorganic coagulants due to their good performance, large availability, easy handling and low cost [6]. The pre-hydrolyzed form of aluminum salt, polyaluminum chloride (PACl) is often reported more effective than the hydrolyzing form [1,7]. Nevertheless, residual aluminum is associated to Alzheimer's disease [5]. Iron salts (e.g., $FeCl_3$) have been used as alternatives for aluminum salts [5]; However, their concerns have not been fully investigated. Ferric coagulants could cause reddish brown staining of equipment or corrosion in condition of large dosage [8]. Hence, the reduction of its dose to reduce negative effect by mean of using flocculants is commonly considered. Nevertheless, some flocculants (e.g., PAM) are harmful to human and the environment [8,9].

The use of natural flocculants such as plant-based ones have been interested [5,10–14]. Different from chemical coagulants and flocculants, they are ecologically friendly and easily biodegradable [15]. These flocculants were reported to be as effective as synthetic flocculants such as polyacrylamide – PAM [10,14–16]. The textile effluent collected from a fabric dyeing mesh was optimally treated with 640 mg/L $FeCl_3$, 160 mg/L extract from *Opuntica ficus-indica* under pH 6 to remove 97.19% COD and 93.62% turbidity, respectively [5]. Also for this kind of effluent, de Souza et al. [12] found the optimal conditions of coagulation–flocculation as $FeCl_3$ 320 mg/L, *Cereus peruvianus* cactus mucilage 10 mg/L and pH 5; Corresponding to turbidity removal of about 86%, respectively. For an industrial laundry effluent, a maximum turbidity removal of about 97% was achieved at 88 mg/L Fe^{3+} (from $FeCl_3$) and okra mucilage dosage of 1.6 mg/L under pH 6.0 [11].

Mucilage extracted from dragon fruit peel (*Hylocereus undatus*) has been reported with coagulation activity on landfill leachate [17]; and flocculation activity on dye wastewater on dye wastewater [14] *Hylocereus undatus* (white flesh dragon fruit) is the most cosmopolitan cultivar of *Hylocereus* species, which are in the same family as cactus [17]. The cactus mucilage contains natural polymers such as polysaccharides (e.g., pectin) that facilitate the flocculation process. Galacturonic acid component of polysaccharides generally provide the predominant active sites at the polymeric chain for particle adsorption and encourage the coagulation process [18–20]. The dragon fruit peel contributes up to one-third of the fruit weight; Meanwhile, pectin was found with up to 20.14% of dry weight of the fruit [17,21]. In the research of Le et al. [14], dragon fruit peel mucilage was characterized with Fourier transform infrared (FTIR) spectra and particulate charge detector. It showed the typical characteristics of mucilage at low esterification degree, which benefit properties of water absorption, viscosity, and texture formation. The negative charge of mucilage was stable in a pH range from 6 to 9, implicating a stable working ability at different pHs.

In this study, the treatment efficiency of coagulant ($FeCl_3$) and flocculant (mucilage extracted from dragon fruit peel – DFPM) for different type of dye wastewater was assessed, especially in comparison to the case PACl and PAM were used instead [14]. The findings establish a basis for the choice of suitable coagulation–flocculation agents in real life practices.

2. Materials and methods

2.1. Materials

The DFPM and dye wastewater samples were the same kinds used in the study of Le et al. (2020) [14]:

- DFPM was extracted from *Hylocereus undatus* peels to serve as flocculant in the coagulation–flocculation of dye wastewater. The step-wise extraction procedure comprised (1) aqueous extraction with hot water (water: dried peel of 8:1 v/w, 60°C, 30 min.), (2) collection of mucilage in filtrate by filtration (8-fold muslin cloth), (3) precipitation of mucilage from filtrate by acetone (filtrate: acetone of 1:3 v/v), (4) isolation of mucilage by centrifugation, (5) washing by ethanol (96°, several times), and (6) oven drying (40°C).
- Three types of dye wastewater were taken from Huy Phat dyeing company (Duong Noi, Ha Dong, Hanoi, Vietnam) at collecting chamber of dye unit (~60°C), brought to the lab (~1 h) by 20 L cans and maintained at room temperature (27°C ± 2°C) after pre-treatment procedure with primary homogenization (60 L, pour and stir in a big tank), primary sedimentation (2 h), and secondary homogenization (transferred the settled wastewater to another tank and stirred) prior to the experiments. Their characteristics are clarified in Table 1.

$FeCl_3·6H_2O$ and PAM (A1110) was purchased from Longcin chemical (China) and KMR (UK).

Table 1
Characteristics of dye wastewater samples

Wastewater sample	Color	Dyes	pH	Turbidity (NTU)	TSS (mg/L)	Color index (Pt/Co)	COD (mg/L)
R	Red	Red GS 1.8% Red FB 0.4%	7.04	319	800	1,327	960
VB	Violet blue	Violet S3R 1.2% Blue 2BLN 0.6% Red SB 0.4%	6.72	403	670	1,027	1,536
NB	Navy blue	Navy HGL 2.6% Black EXSF 0.7% Red GS 0.3%	7.10	469	511	1,366	2,496

2.2. Methods

2.2.1. Optimal conditions for turbidity removal by coagulation–flocculation process using $FeCl_3$ and DFPM

2.2.1.1. Optimal coagulation conditions of $FeCl_3$ on dye wastewater

Settling time and pH: pH and settling time conditions were tested together according to Jar-test model (Le et al.) [14] under room temperature (27°C ± 2°C). Initial pH was varied between 4 and 9, while settling time was varied between 10 and 60 min (10 min interval). $FeCl_3$ was added to wastewater at fixed doses and quickly stirred at 200 rpm within 1 min, before slowing down to 30–40 rpm for 10 min. After settling time, the supernatant at 3 cm under the surface was collected to measure turbidity, using Hach 2100Q turbidity meter, in order to assess the turbidity removal in percentage of reduction. In order to justify the fixed doses of coagulant, test experiments were carried out with coagulation doses of large intervals (0, 5, 10, 50, 100, 150, 200, 300, 400, 500, etc.) and then smaller intervals for value ranges with considerable changes of turbidity removal. This prognosis was later utilized for investigating the effect of coagulant dose under optimal time and pH.

$FeCl_3$ dosage: Experiments were carried out similarly at optimal pH and settling time while $FeCl_3$ dosage was varied in the range of 0.04–0.74 mM (10–200 mg/L $FeCl_3·6H_2O$), respectively.

2.2.1.2. Optimal DFPM dose as flocculant in sequent coagulation–flocculation process using $FeCl_3$

To figure out optimal DFPM dosage, similar experiments were carried out with DFPM added right after 1 min flash mixing of $FeCl_3$ into the dye wastewater. DFPM dose was varied between 0 and 50 mg/L and the dose that resulted in highest turbidity removal was considered as optimal one. pH and settling time were fixed at optimal values while $FeCl_3$ was kept lower than the optimal dose. Similar experiments were carried out with PAM for comparison.

2.2.2. Pollutants removal by coagulation–flocculation process using $FeCl_3$ and DFPM

The optimal conditions were applied for the coagulation–flocculation process using $FeCl_3$ and DFPM in examination of pollutant removal. The treatment efficiency was assessed by comparing pollutant contents in the influent and effluent of the coagulation–flocculation process. All parameters, including TSS, COD, color index (Pt/Co) were measured according to APHA (2005) [22]. Similar experiments were carried out with PAM for comparison.

3. Results and discussion

3.1. Coagulation–flocculation conditions of $FeCl_3$ and DFPM for dye wastewater

3.1.1. Optimal settling time and pH

The effects of settling time and pH on coagulation efficiency of $FeCl_3$ for 3 types of dye wastewater are illustrated in Fig. 1. pH acid or slightly acidic (4–6) generally showed high and stable turbidity removal; while pH from neutral to alkaline value (7–9) showed much lower turbidity removal. Initial pHs of wastewater were around 7, hence pH 6 was found most suitable for the reason of chemical saving regarding pH adjustment.

In the range of 10–60 min, the longer the settling time, the higher the turbidity removal. Within 30 min of settling, turbidity removal increased steeply (up to 97%) and sludge volume as well (up to 97%). However, small changes in turbidity removal were observed when settling time was further increased to 60 min (mostly around 1%); while sludge volume wasn't changed. As such, settling time of 40 min was deemed reasonable for further experiments, taking in mind the three factors (turbidity removal, sludge volume and hydraulic retention time).

Compared to the results published by the same group (Le et al.) [14], $FeCl_3$ showed similar behavior as PACl in coagulation of dye wastewater (Fig. 1).

The pH of optimum coagulation falls in the 4.5–7.0 pH range, which is usually reported for Fe(III)-based coagulants [23–27]. When $FeCl_3$ are dissolved in water, the metal hydrates to form aqueous metal ions and then hydrolyzes to monomeric and polymeric ferric species including positively charged $FeOH^{2+}$, $Fe(OH)_2^+$, $Fe_2(OH)_2^{4+}$ and $Fe_3(OH)_4^{5+}$; neutral $Fe(OH)_3$; and negatively charged $Fe(OH)_4^-$ depending on pH and concentration of Fe^{3+} in the solution [24]. The higher the $FeCl_3$ concentration, the higher the isoelectric point of the solution is [24]. At pH not higher than 6.0, the ferric hydroxide precipitates and dissolved species are positively charged [27,28]. On the other hand, the charges of ferric hydroxide precipitates are close to zero at pH 7.8 [28]. The coagulation with $Fe(OH)_3$ has been elucidated by different mechanism including charge neutralization, precipitation, bridge-aggregation, adsorption and sweep-flocculation [8]. The positively charged hydrolysis species can be absorbed onto the surface of colloidal particles and destabilize the stable colloidal particles leading to charge neutralization. The precipitates (especially at high coagulation dose) can physically sweep the colloidal particles from the suspension, causing sweep-floc coagulation.

The similar pH of optimum coagulation by $Fe(OH)_3$ and PACl were observed in different studies [26,27]. Aluminum and ferric based coagulants, both have trivalent ion; Hence, similar overall stoichiometric reactions as well as coagulation mechanisms [29].

3.1.2. Optimal dosage of $FeCl_3$

The turbidity removal of $FeCl_3$ at different dosages under optimal pH and settling time are shown in Fig. 2 together with those of PACl obtained by Le et al. [14]. When $FeCl_3$ was increased to optimal value, the turbidity removals were increased up to about 97%, 91%, and 93% for R, VB, and NB dye wastewater, respectively. At higher $FeCl_3$ dosage, the turbidity removal dropped down quickly. The optimal $FeCl_3$ dosages were dependent on the types of dye wastewater and were 0.15, 0.26, and 0.22 mM Fe^{3+} (corresponding to 40, 70, 60 mg/L $FeCl_3·6H_2O$) for R, VB, and NB dye wastewater, respectively.

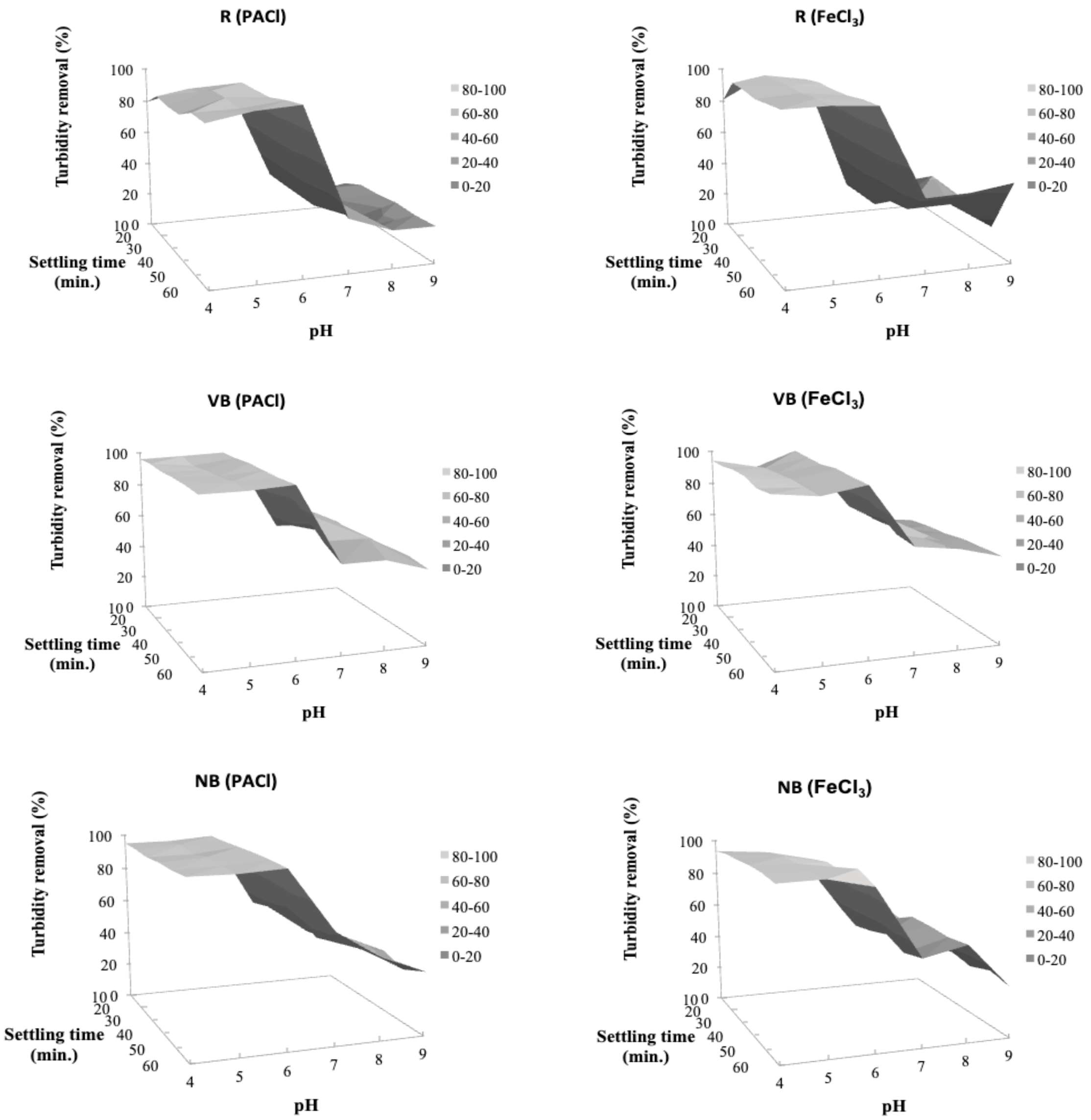

Fig. 1. The effect of pH and settling time on turbidity removal by $FeCl_3$ (this study) and PACl [14].

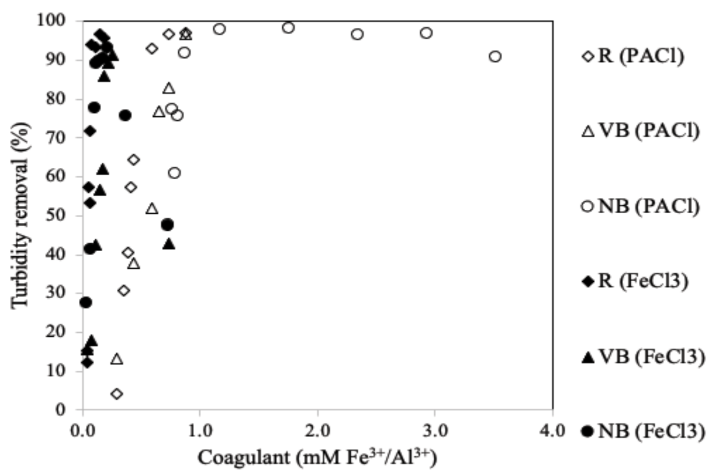

Fig. 2. Effect of $FeCl_3$ (this study) and PACl [14] dosage on turbidity removal.

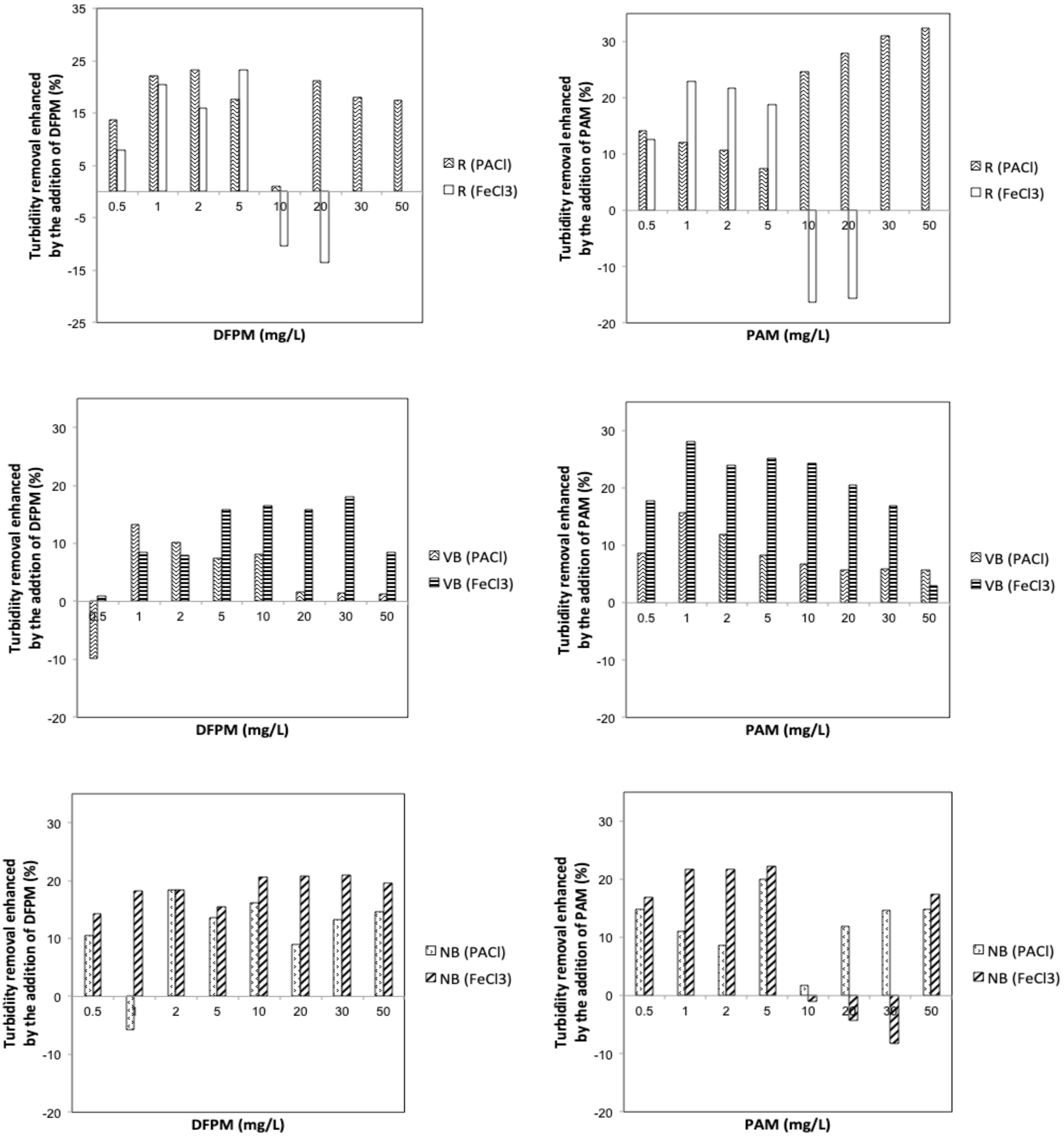

Fig. 3. Increase of turbidity removal by the use of DFPM or PAM in combination with $FeCl_3$ (this study) or PACl [14].

For the same kind of dye wastewater, optimal dosages of PACl were considerably higher (about 0.74 – 1.18 mM Al^{3+} or 125–200 mg/L PACl) in Le et al. [14]. Additionally, the changes in turbidity removal by PACl was not as sharp as those observed by $FeCl_3$. The optimum $FeCl_3$ dosages observed in this study were much lower compared to those for textile and dye wastewater studied by Kim et al. [30] and Rana and Suresh [31].

Technically, the optimum dosage of a coagulant is the value below which there is no significant increase in removal efficiency with further addition of coagulant. At lower $FeCl_3$ dosage than the optimum value, a sequent transformation of $FeCl_3$ occurs to facilitate turbidity removal. These steps include hydrolysis and polymerization of metal ion, adsorption of hydrolysis products at the interface, charge neutralization, and then sweep floc (or precipitation and enmeshment) [29]. The sweep floc is conditioned at high enough dosage of $FeCl_3$ by the nucleation of the insoluble precipitate on the surface of particulates, leading to the growth of amorphous precipitate, which is capable of entrapping particles. The coagulation efficiency increases when the coagulant dosage increases up to optimum value; Whereas it is reduced at excess coagulant dosage due to resuspension of aggregated particles [32]. Selecting an optimum coagulant dosage is significant and critical to control the performance, the cost and the sludge formation.

Lower coagulation efficiency of $FeCl_3$ was reported for blends from surface, ground water, treated sewage water, rainwater, seawater, and brackish water [33]; and pharmaceutical wastewater [27]; Whereas, higher performance was reported for reverse osmosis concentrate [25,34], and textile wastewater [31].

The greater efficiency of $FeCl_3$ than PACl can be attributed to the large floc sizes, greater adsorption of organic matter and the ability to remove organic compounds over a wider size range [25,34]. Additionally, $FeCl_3$ has higher charge density compared with the aluminum-based coagulants [34,35]. Furthermore, ferric-based coagulants are more insoluble than aluminum-based ones in high salinity water such as dye wastewater; Thus, results in better floc formation [34,36]. Possibly, the ability of Fe^{3+} to form complexes increases its coagulation performance also [28].

3.1.3. Optimal dosage of DFPM

The effect of DFPM on the coagulation efficiency of $FeCl_3$ was examined and compared to that of PAM. The results are also compared to those achieved by Le et al. [14] when PACl was employed instead of $FeCl_3$ (Fig. 3). The addition of DFPM (5–50 mg/L) after using $FeCl_3$ increased turbidity removal by $FeCl_3$ alone at about 10%–20%. These increases were comparable to the cases when PAM was used as flocculant. DFPM addition also reduced 10%–50% $FeCl_3$ used in order to achieve the similar turbidity removal, which $FeCl_3$ achieved alone. To meet economic benefit while maintaining reasonable performance, DFPM dosage should be kept between 1 and 5 mg/L. Compared to PACl [14], the combination of flocculants (DFPM or PAM) with $FeCl_3$ tended to show better performance, while saving more coagulant.

The increase of coagulation efficiency of $FeCl_3$ by DFPM were also comparable to those achieved by plant-based flocculants in Anatasakis et al. [13] and Hamandani et al. [37]. This effect demonstrated flocculation activity of DFPM. Mucilage has a nature of polysaccharides with hydrocolloid characteristics [17]. Studies suggested that galacturonic acid component of mucilage plays an important role in its adsorption and bridging capacity, which are the typical flocculation mechanisms [20].

3.2. Treatment efficiency of dye wastewater by the combination of DFPM with $FeCl_3$

Besides turbidity, the removal of TSS, COD and color of dye wastewater were also determined for the coagulation–flocculant process using $FeCl_3$ and DFPM under optimal pH (6.0), settling time (40 min.) and appropriate dosages of $FeCl_3$ and DFPM (5 mg/L) (Fig. 4). The removal of pollutants was the reduction of their contents in the influent (Table 1). Overall effect of $FeCl_3$ and DFPM resulted in higher removal of turbidity (89%–98%), color (87%–91%) and TSS (63%–95%) and lower removal of COD (38%–46%). Compared

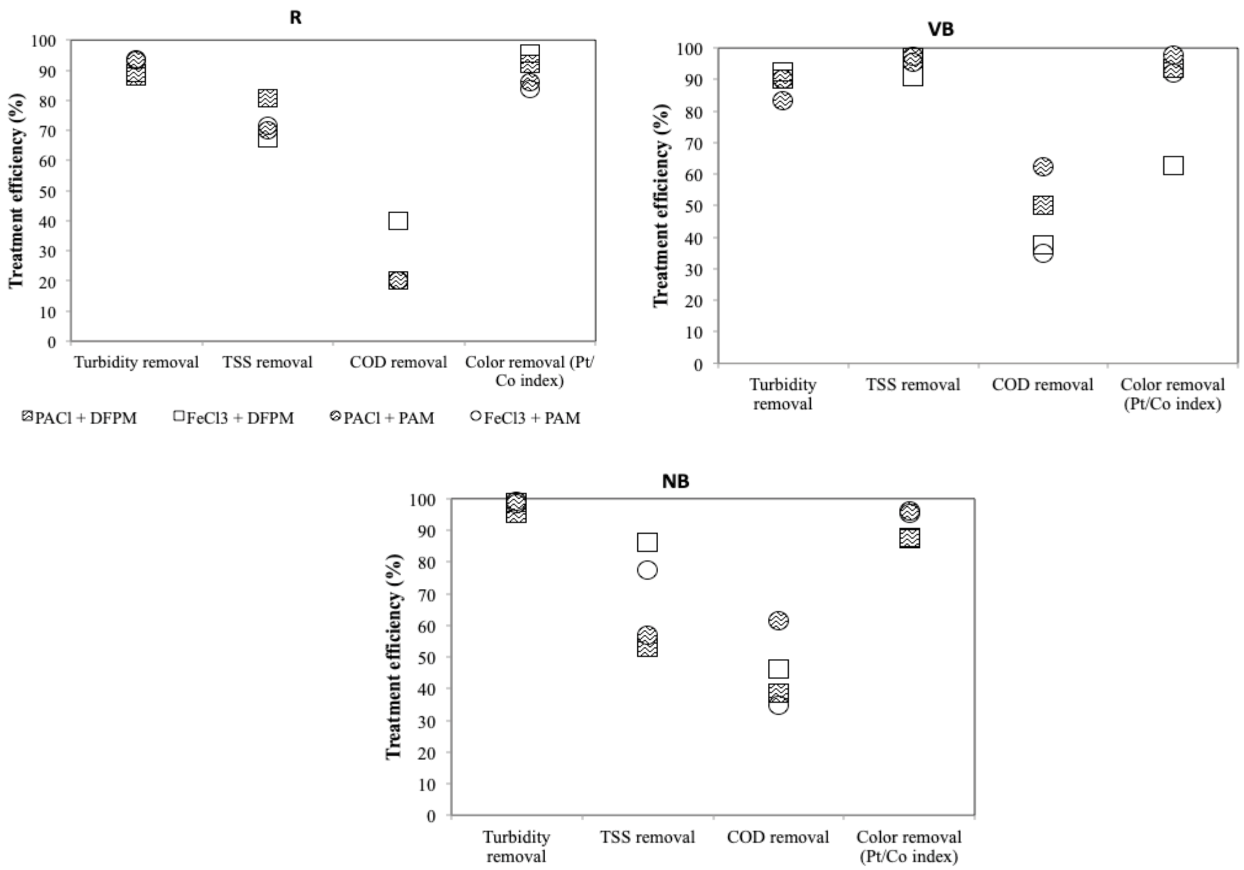

Fig. 4. Pollutant removal of DFPM and PAM in combination with $FeCl_3$ (this study) and PACl [14].

to those of PAM, these treatment efficiencies could be also compatible. As for the combination of DFPM with PACl [14], the treatment efficiencies were also similar in term of value and type of pollutants (turbidity (88%–95%), color (87%–93%), TSS (53%–97%), and COD (20%–50%)).

Combined with alum or $FeCl_3$, mucilages from *Abelmoschus esculentus* [11], *Cereus peruvianus* [12], *Ocimum basilicum* [38], *Opuntica ficus-indica* [5,16] performed similar effect on turbidity and color removal but higher effect on COD removal (58.30%–88.76%) when compared to *Hylocereus undatus*'s. Despite of the coagulant used, all of these mucilages resulted in better turbidity and color removal than COD removal.

The results showed a comparable effect of plant-based flocculant to the commercial flocculant – PAM. This has been observed by different researchers as well [10,14–16].

4. Conclusions

The combination of $FeCl_3$ and DFPM showed an effective coagulation and flocculation performance in treatment of dye wastewater. Optimal coagulation conditions of $FeCl_3$ were typically found in pH 4.0–6.0 and settling time of 30–60 min, whereas $FeCl_3$ dose varried as 0.15, 0.26, and 0.22 mM for 0.15–0.26 mM R, VB, and NB dye wastewater, respectively. The addition of DFPM (0.5–50 mg/L) after $FeCl_3$ (0.04–0.74 mM) resulted in turbidity removal up to 97%, corresponding to 10%–20% increase compared to those obtained with $FeCl_3$ alone and the potential to decrease 10%–50% the amount of $FeCl_3$ used to obtain the similar turbidity removal. DFPM showed a comparable effect to the comercial flocculant – PAM. In comparison to PACl, $FeCl_3$ showed better coagulation performance on investigated dye wastewater. The removal of turbidity, TSS, color and COD were similar when combined either $FeCl_3$ or PACl with DFPM; However, in the combination with $FeCl_3$, DFPM could reduce more coagulant amount than in the case with PACl.

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