

## Research Article

# Mucilage Extracted from Dragon Fruit Peel (*Hylocereus undatus*) as Flocculant for Treatment of Dye Wastewater by Coagulation and Flocculation Process

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Dye wastewater from textile industries shows very low biodegradability due to high molecular weight and complex structures of dyes. So far, the most simple method for treatment of this type of wastewater has been coagulation and flocculation. This study determined the removal of turbidity and other pollutants from dye wastewater by mucilage extracted from the peel of dragon fruit (*Hylocereus undatus*) and its effect in reducing synthetic chemical polyaluminum chloride (PACl) used in coagulation and flocculation (CF) process. The removal of turbidity in a sequent CF process using PACl and dragon fruit mucilage was investigated based on Jar tests. Maximum coagulation efficiencies of PACl were typically observed at pH 4.0-6.0 and PACl concentrations of about 100-150 mg/L depending on types of wastewater, whereas optimal settling times were 30-60 minutes, respectively. The addition of dragon fruit mucilage (0.5-50 mg/L) after PACl (75-245 mg/L) resulted in turbidity removal efficiencies up to 95%. The addition of mucilage extracted from dragon fruit peels was proven to increase turbidity removal efficiency and decrease PACl use. The increase of turbidity removal was often estimated at 10-32%, whereas PACl used was about 3-10% less compared to total PACl needed for obtaining comparable efficiency when used alone. The flocculation activity of mucilage was also compared to polyacrylamide (PAM)—a synthetically organic flocculant. Since the peel of a dragon fruit is an abundant agriculture waste in Vietnam, using its extracted mucilage as a flocculant is an environmentally friendly method.

## 1. Introduction

In water treatment techniques, coagulation-flocculation is an important process to remove turbidity in water as well as contributes to increasing the efficiency of subsequent treatment processes such as sedimentation, filtration, and disinfection. Synthetic coagulants and flocculants such as aluminum salts, iron salts, and polymers (PACl, polyacrylamide, polyacrylic acid) are still widely used in water treatment [1]. However, some researches have shown health and ecosystem concerns when using these chemicals. For examples, Alzheimer's disease is confirmed to be related to aluminum residues in domestic water [2]. Monomers of polymer flocculants cause neurotoxicity and cancer [3]. Moreover,

these chemicals are imported in developing country with a considered cost. Today more and more interests are drawn on natural coagulants and flocculants since they could minimize chemical residues, make use of various and diverse sources of waste, and reduce the risk of disease and are easily biodegradable [4].

Mucilages are polysaccharides, widespread in a number of plants, swell when dissolved in water, and form a viscous form similar to gelatin [5]. They have been used in the removal of pollutants from wastewater in both coagulant [6-8] and flocculant [9-11] roles with removal efficiency of suspended solids (SS) fluctuated from very low (about 26%) to quite high (about 89-97%). Mucilages extracted from fenugreek and okra have been shown to be as effective

as synthetic coagulant (polyacrylamide) in treatment of textile and tanning wastewaters. The SS removal was 95% with mucilage concentration of 0.04 mg/L (fenugreek) and 0.12 mg/L (orka) [4]. Experiments conducted on synthetic wastewater (kaolin and humic acid suspension, 63-73 NTU) resulted in turbidity removal of 96.3-97.4% and 93.0-97.3% when using mallow and okra mucilages as flocculants, respectively [9].

The textile industry is an industry with multistage technology lines, using a lot of materials as well as chemicals, and is one of the most polluted industries [12]. It uses a large amount of water to serve the production stages, and at the same time, discharges average 200-350 m<sup>3</sup> wastewater per ton of fabric products. In which, the disperse dyeing process generates about 91-129 m<sup>3</sup>/ton product [13]. According to the World Bank, about 17-20% of industrial wastewater comes from textile dyeing and finishing [14]. Wastewater from textile establishments is largely fluctuated both in the flow and the load of pollutants, depending on season, production capacity, and product quality. Basically, the use of many types of dyes, chemicals, and additives leads to wastewater often with high color, organic matters, etc., contributing to environmental pollution and diseases in humans [13]. About 40% of the colorants used globally contain organic chlorine, a carcinogen [15]. Chemicals used in dyeing could evaporate into the air hindering respiration or causing skin diseases. Characteristics of textile dyeing wastewater are varied by pH (1.9-14), COD (50-17900 mg/L), TSS (15-23900 mg/L), and strongly colored (50-2500 Pt/Co) as summarized by Freitas et al. [12] and Verma et al. [13]. Different mucilages (*Opuntia ficusindica*, *Cereus peruvianus*, and *Ocimum basilicum*) were found effective as flocculants in coagulation-flocculation treatment of industrial textile wastewater [8, 10, 11, 16].

Most *Hylocereus* species, being in the same family as cactus, originate principally from Latin America (probably from Mexico and Colombia), with others possibly from the West Indies, and have been a food source for inhabitants [17]. Today, they are distributed all over the world (in tropical and subtropical regions), and *Hylocereus undatus* (white flesh dragon fruit) is the most cosmopolitan species. In Vietnam, *H. undatus* has been widely grown for fruit harvesting in the provinces and cities across the country, with largest areas in the South [18]. Mucilage is present in some parts of the dragon fruit plant such as the stem, leaf, and fruit. The cactus mucilage contains galacturonic acid which is generally the predominant active coagulation agent regardless of the species [19]. High natural polymers such as polysaccharides (e.g., galacturonic acid) and proteins in dragon fruit provide active sites at the polymeric chain for particle adsorption and encourage the coagulation process [19-21]. So far, there has been only one investigation by Ismail et al. [17] on coagulation activity of the dragon fruit peel (considered an agricultural waste) for removal of turbidity from landfill leachate.

This study considered the turbidity removal efficiency of mucilage extracted from the dragon fruit peel as the role of a flocculant (PACl as coagulant) in removal of turbidity from dye wastewater.

## 2. Materials and Methods

**2.1. Materials.** The *Hylocereus undatus* peels were collected from a fruit juice shop for use as a material to extract mucilage. Three samples of textile wastewater were taken from Huy Phat dyeing company (Duong Noi, Ha Dong, Hanoi, Vietnam), which were characterized according to APHA [22] as in Table 1. PACl (AC100S) and PAM (A1110) were purchased from Grasim Industries Ltd. (India) and KMR (UK), respectively.

### 2.2. Methods

**2.2.1. Extraction of Mucilage from Dragon Fruit Peel.** The peels were washed to remove dirt and chopped to 5 mm, taken to dry at a temperature of 50°C until constant weight. The extraction process was executed in two steps.

Step 1: mucilage separation. Dried peels were mixed with distilled water (ratio 1:8 w/v) and heated at 60°C in water bath for 1 hour (the mixture was stirred regularly). After that, the mixture was taken out and cooled to room temperature within 1 hour to increase the amount of mucilage dissolved in distilled water. Thereafter, the mixture was filtered through 8 layers of muslin cloth to collect the filtrate containing the mucilage.

Step 2: mucilage precipitation and collection. The mucilage was precipitated from the filtrate by acetone with the volume ratio of 3:1. The precipitate was washed 3-4 times with concentrated alcohol to remove adhesives on its surface and dried at a temperature of 40°C until the constant weight. After being dried, the mucilage was grounded, put into a zip bag, and placed in a desiccator for storage.

**2.2.2. Characterization of Mucilage Extracted from Dragon Fruit Peel.** Fourier transform infrared spectra (FTIR) were obtained using the Nicolet Is50 Spectrometer in the resolution range of 4000-400 cm<sup>-1</sup>, data interval of 0.47 cm<sup>-1</sup> with 16 scans.

Zeta potentials were measured on mucilage suspensions of 20 mg/L at different pHs ranging from 4 to 9 using PCD-05, Muetek device.

### 2.2.3. Turbidity Removal from Dye Wastewater by Mucilage as Flocculant

**(1) Optimal Working Conditions of Coagulant PACl on Dye Wastewater.** For settling time and pH, experiments were conducted according to the Jar test model [9] under room temperature (27 ± 2°C). Initial pH was varied between 4 and 9 (adjusted with NaOH and HCl). PACl was added to wastewater at fixed concentrations, stirred quickly at 200 rpm within 1 minute, stirred slowly at 30-40 rpm for 10 minutes, and allowed to settle for 10, 20, 30, 40, 50, and 60 minutes. The turbidity removal was assessed by measuring of the turbidity of water after treatment at a water depth of 3 cm under the surface, using a Hach 2100Q turbidity meter.

For the PACl dosage, experiments were carried out similarly at optimal pH and settling time and PACl concentrations in the range of 50-900 mg/L, respectively.

TABLE 1: Characteristics of dye wastewater samples.

Wastewater sample	Dyes	pH	Turbidity (NTU)	TSS (mg/L)	Color index (Pt/Co)	COD (mg/L)
R	Red GS 1.8% Red FB 0.4%	7.04	319	800	1327	960
VB	Violet S3R 1.2% Blue 2BLN 0.6% red SB 0.4%	6.72	403	670	1027	1536
NB	Navy HGL 2.6% Black EXSF 0.7% Red GS 0.3%	7.10	469	511	1366	2496

(2) *Turbidity Removal in Sequent Coagulation and Flocculation Process Using PACl and Mucilage*. Similar experiments were employed, but mucilage was introduced in a slow mixing stage after the use of PACl in a flash mixing stage to increase the size and sedimentation capacity of colloidal particles, while reducing the amount of PACl used. The experiments were carried out at the optimal pH and settling time and PACl with lower concentrations than the optimal concentrations. Mucilage concentrations were varied between 0 and 50 mg/L for all dye wastewater samples.

### 3. Results and Discussion

3.1. *Characteristics of Mucilage Extracted from the Dragon Fruit Peel*. FTIR spectrum (Figure 1(a)) shows three major bands associated with the carbohydrate moieties (region  $\sim 1150\text{-}900\text{ cm}^{-1}$ ), protein (region  $\sim 1654\text{-}1635\text{ cm}^{-1}$ ), and hydroxyl (region  $\sim 3410\text{ cm}^{-1}$ ) bands, which were distinctly visible in case of mucilage extracted from the dragon fruit peel. Carboxyl and phenolic groups are recognized at  $1100.60\text{-}1018.74\text{ cm}^{-1}$  with C-OH stretching [23]. The absorption bands at  $1635.2\text{ cm}^{-1}$ , associating with ester carbonyl and carboxyl groups, suggested the possible presence of uronic acid in polysaccharides [24]. The band at  $1749\text{ cm}^{-1}$  is evidently not present, indicating a low degree of esterification of mucilage [25]. This is beneficial in enhancing properties of water absorption, viscosity, and texture formation of mucilage. Another band was found at  $2929.43\text{ cm}^{-1}$  due to C-H stretching and bending vibration, which corresponds to the vibrations of -CH and -CH<sub>2</sub> present in the mucilage molecule [26]. Additionally, a band at  $3411.91\text{ cm}^{-1}$  was found, which corresponds to O-H stretching of alcohol and carboxylic acid. -OH groups involved in intermolecular hydrogen bonding of mucilage molecules, as mentioned by Habibi et al. [27].

Zeta potentials of mucilage suspensions at 20 mg/L concentration show a negative charge nature (Figure 1(b)). Moreover, this value is rather stable in a pH range from 6 to 9, implying stable working ability at different pHs. At acidic pH of 4 and 5, negative-charged groups on the polysaccharide are protonated and therefore less negative. The ability to work stably at different pHs is the advantage of natural flocculants in comparison to chemical ones [6, 19].

#### 3.2. Efficiency of Mucilage as a Flocculant in Treatment of Dye Wastewater

##### 3.2.1. Appropriate Coagulation Conditions of PACl

(1) *Optimal Settling Time and pH*. The experiment was performed on all three types of dye wastewater using suitable PACl dosages for observation of the effect of settling times and pHs on turbidity removal efficiency (Figure 2).

Generally, a pH range from 4 to 6 showed high and stable turbidity removal, while a pH range from 7 to 9 showed much lower turbidity removal. According to the initial pH of wastewater (around 7), pH 6 was found most suitable for the reason of chemical saving regarding pH adjustment.

Settling times increasing from 10 to 60 (10 minutes interval) increased the turbidity removal. After 30 minutes of settling time, there were large changes in both turbidity removal (up to 6%) and sludge volume (up to 96%). However, for settling times from 30 to 60 minutes, small changes in turbidity removal (mostly around 1%) but no changes in sludge volume were observed. Balancing the three factors (turbidity removal, sludge volume, and hydraulic retention time), settling time of 40 minutes was selected for all three types of wastewater in further experiments.

The effect of initial pH on turbidity removal is correlated with speciation characteristics of PACl. Both monomeric and polymeric aluminum species are present when PACl is in water [28]. Of which,  $\text{Al}_{13} [(\text{AlO}_4\text{Al}_{12}(\text{OH})_{24}(\text{H}_2\text{O})_{12})]^{7+}$  and  $\text{Al}_{30} [(\text{AlO}_4)_2\text{Al}_{28}(\text{OH})_{56}(\text{H}_2\text{O})_{26}]^{18+}$  are generally believed to be the most effective coagulation species in poly-aluminum coagulants [29, 30]. At a pH range from 4 to 6, polymeric aluminum species are dominant [31, 32]. These positive species benefit flocs growing because they easily neutralize negative charges of dye wastewater causing destabilization of colloids and facilitate the physical or chemical adsorption of the destabilized colloids [30]. When working pH gets higher, PACl hydrolyzed faster [31] to form amorphous  $\text{Al}(\text{OH})_3$  and later  $\text{Al}(\text{OH})_4^-$ . The increase of pH also results in the decrease of charge neutralization capacity of PACl [31]. Furthermore, pH also affects the physical and chemical properties of colloids in dye wastewater. The lower pH improves protonation to make colloids easier to be charge-neutralized and charge-destabilized [30]. Besides, pH could affect the balance between the reaction of organic functional groups with hydrogen ions and aluminum hydrolyzates [33].

Textile wastewater has pH varied from acidic to basic, however typically neutral [13]. Effective coagulation condition at pH around a neutral pH is advantageous because no or less chemicals are needed to adjust the pH of wastewater.

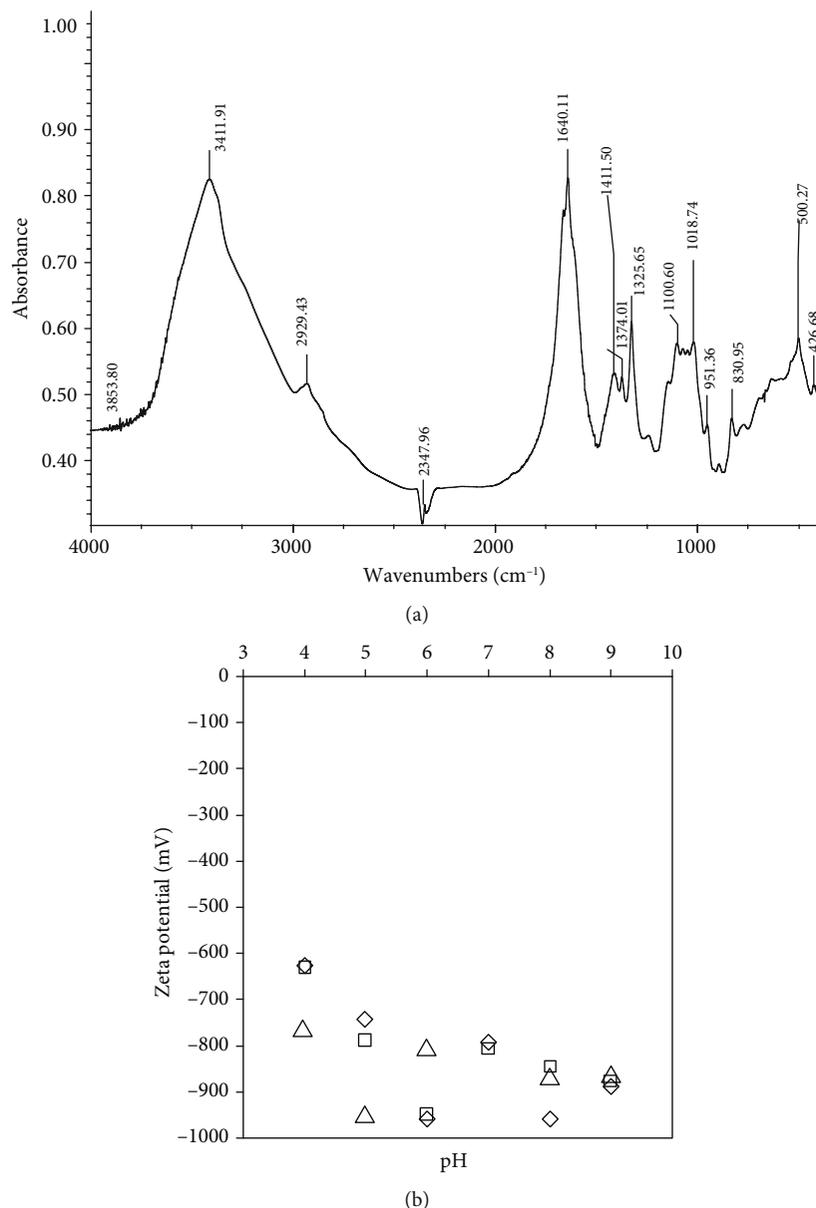


FIGURE 1: FTIR spectrum (a) and zeta potential (b) of mucilage extracted from the dragon fruit peel.

The settling capability of flocs produced after the coagulation process is greatly dependent on the type of coagulant, the type of wastewater, and also, the floc size generated during treatment [1]. Flocculants are capable of producing a larger floc size, mainly formed by adsorption and bridging involving long chain polymers during flocculation for rapid settling [1]. The results of this study are similar to the study of Lee et al. [34] when it was found that the effective removal of turbidity took place within 30 minutes. Longer than 30 minutes, the increase was not significant (1-2%). The settling time of 30-60 minutes is in line with researches listed in Lee et al. [4] on flocculation efficiencies of plant-based flocculants.

(2) *Appropriate Dosage of PACl*. The effect of PACl dosages on turbidity removal under optimal pH and settling time is

shown in Figure 3. When PACl was increased until optimal dosage, the turbidity removal was increased up to 97%. The optimal PACl dosages were dependent on the types of dye wastewater and ranged between 100 and 150 mg/L. The high turbidity removal was kept stable until it was going down at certain higher PACl dosages. To research mucilage as a flocculant when combined with PACl, the dosage of PACl was chosen at a value lower than the optimal dosage.

Optimal coagulant dosage is established as a significant and critical factor to control the performance, the cost, and the sludge formation [1]. It is believed that the major coagulation mechanism of PACl is charge neutralization, bridging, and sweeping [29, 35]. The higher the dosage of PACl, the higher its neutralization capacity is. Because more positive aluminum hydrolyzates are present in solution as counter

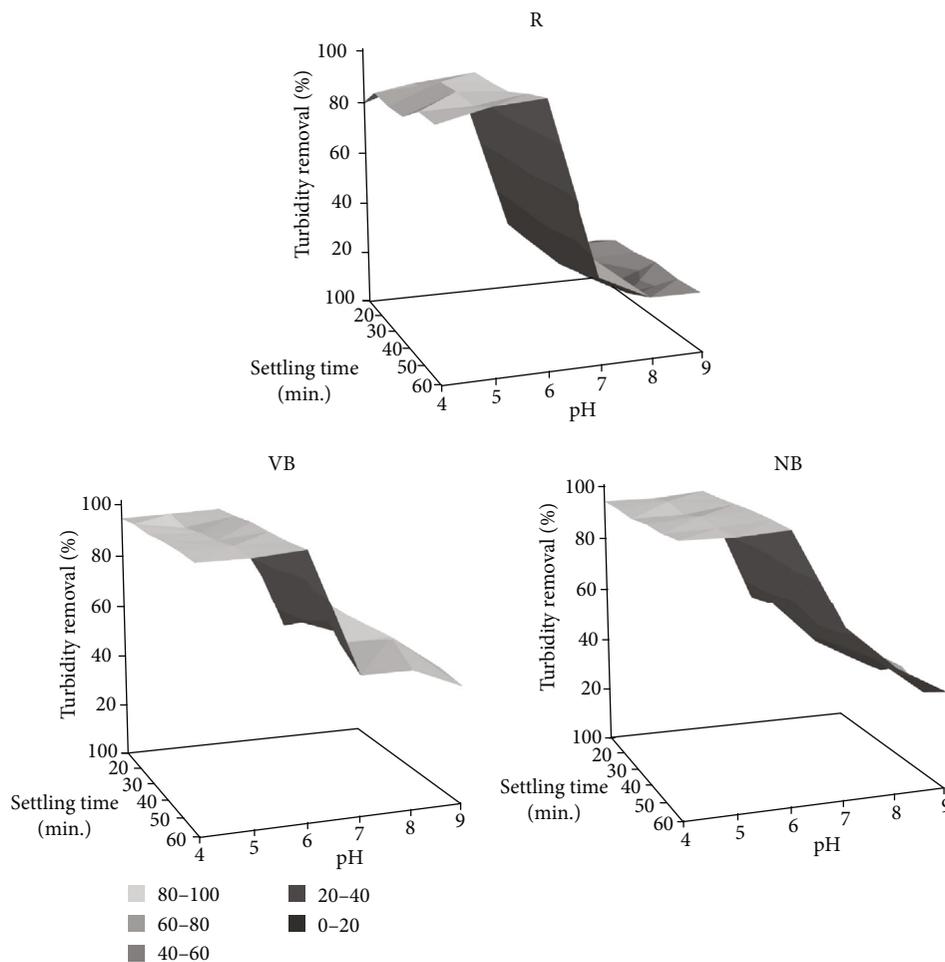


FIGURE 2: The effect of pH and settling time on turbidity removal by PACl.

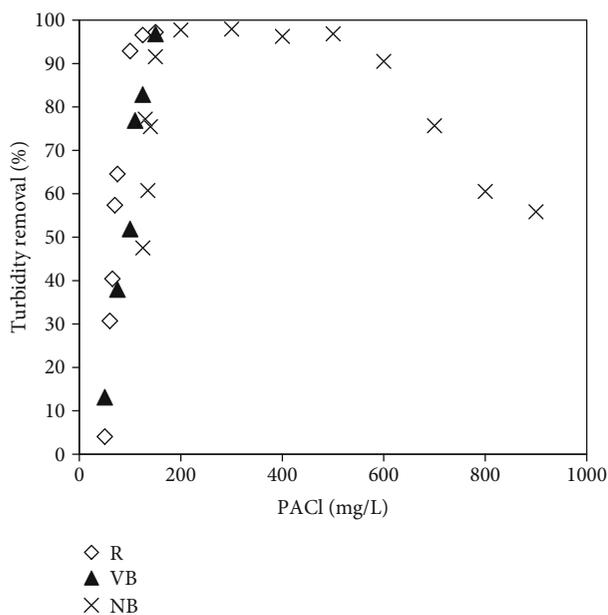


FIGURE 3: The effect of PACl dosages on turbidity removal.

ions of negative colloids present in dye wastewater. Similarly, the higher the PACl dosage, the more polymeric aluminum species appear in the solution, which in turn assist the bridging mechanism. Charge neutralization, leading to particle destabilization, is dominant with a smaller dosage of PACl, while sweep floc coagulation is dominant with a higher dosage of PACl [35]. In a suitable range, the higher the coagulant dosage, the higher the turbidity removal efficiency could be [36]. Excess dosages of coagulants cause resuspension of aggregated particles that results in reduction of coagulation efficiency [1]. In accordance with turbidity removal, COD removal also exhibits a similar trend [37].

**3.2.2. Treatment Efficiency of Mucilage as in Combination with Coagulant PACl for Dye Wastewater.** The turbidity removals of mucilage in the role of flocculant for PACl are shown in Figure 4. The addition of mucilages (5-50 mg/L) increased turbidity removal up to the highest value of about 95%. These increases often ranged between 10 and 32%. To obtain comparable turbidity removal for all three types of dye wastewater, around 10 mg/L PACl more was needed, accounting for 3-10% of total PACl used (100, 150, and 150 mg/L for wastewater R, VB, and NB, respectively). The mucilage concentrations which were most efficient in

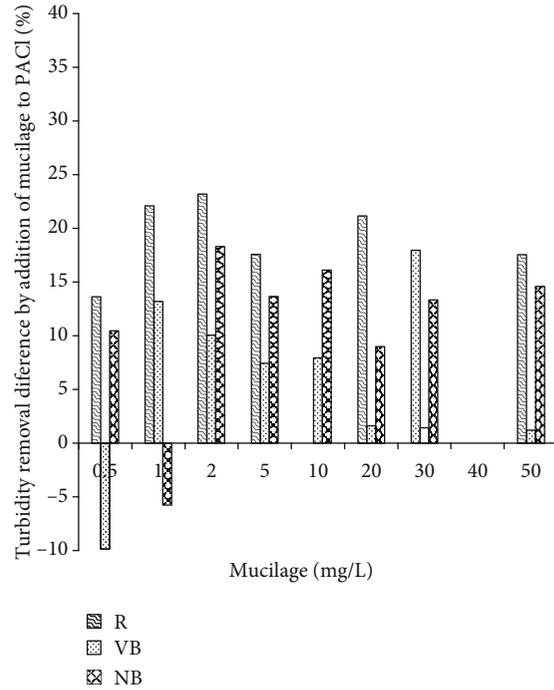
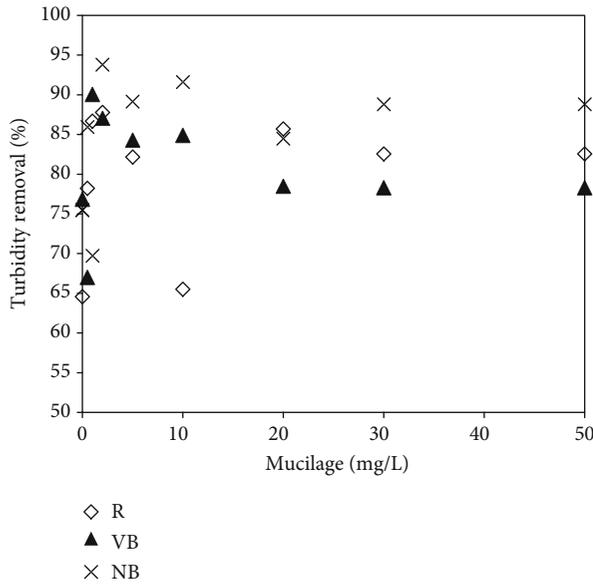


FIGURE 4: Turbidity removal efficiency of mucilage in combination with PACI.

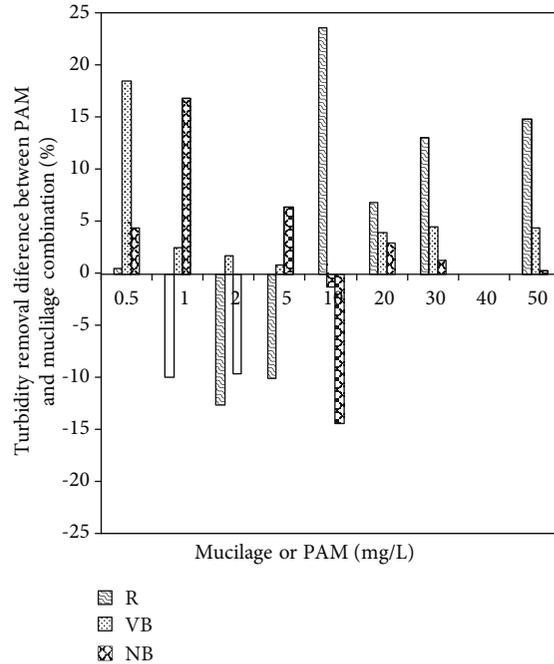
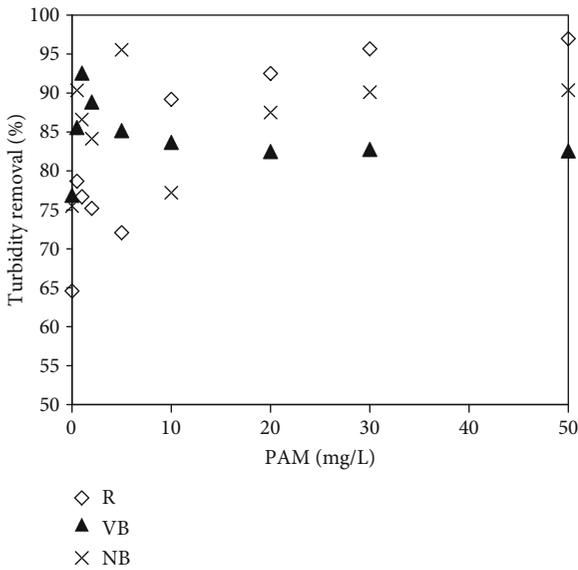


FIGURE 5: Turbidity removal of PAM in combination with PACI and its comparison with those of mucilage.

increasing the turbidity removal lied under 20 mg/L. Higher mucilage concentrations resulted in lower turbidity removal.

The flocculation activity of mucilage was compared to polyacrylamide (PAM) in Figures 5 and 6. PAM had more stable performance than mucilage and often showed continuous increase of turbidity removal when its dosage increased in investigated range (0.5-50 mg/L). The flocs formed by

PAM were much larger than those formed by mucilage. More often, its ability in flocculation was better than mucilage and the differences in removal efficiency could be more than 20%, but in many cases less than 5%. Sometimes, mucilage showed better activity as the differences went minus.

Overall, treatment efficiency of mucilage as a flocculant for PACI was high for turbidity (88-95%), color (87-93%), and TSS (53-97%) and lower for COD (20-50%) (Figure 6).

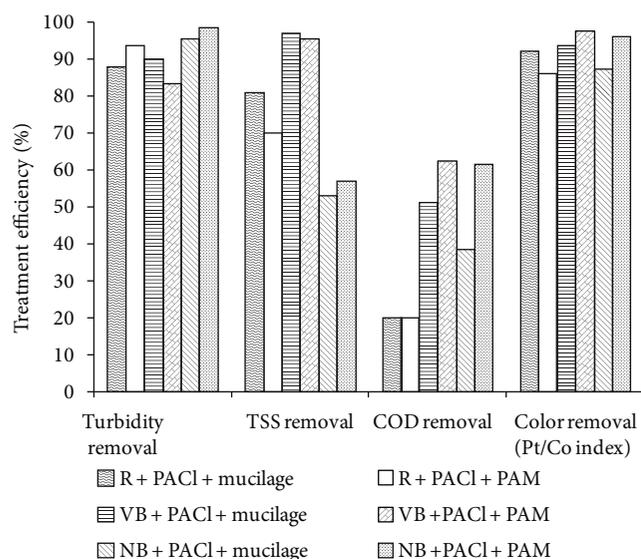


FIGURE 6: Pollutant removal of mucilage and PAM in combination with PACl.

Compared to those of PAM, these treatment efficiencies could be higher but more likely lower, especially in terms of turbidity and COD.

The hydrocolloid characteristics of mucilage are the basis for its utilization as bioflocculant. The mucilage from the dragon fruit peel is a heteropolysaccharide with rheological properties that are of great application [20]. A component of mucilage-galacturonic acid plays an important role in turbidity reduction, in combination with galacturonic acid, arabinose, galactose, and rhamnose also contribute to turbidity removal [21]. The turbidity removal efficiencies brought by the mucilage from the dragon fruit peel were similar to or higher than those achieved by mucilages from different cactuses on dye wastewaters in de Souza et al. [10, 11] and Bouatay and Mhenni [16], while its COD removals were lower. Likewise, the increase of PACl coagulation efficiency for turbidity by mucilage was comparable or even higher than that of PAM in combination with alum and PACl in treatment of pulp and paper mill wastewater [38]. Lower COD removal than turbidity, color, and TSS removals was also found in common [10, 11, 16, 39].

The mucilage concentrations which are higher than optimum concentration lead to the decrease of turbidity removal because of the restabilization of the solution. The optimum amount of flocculant causes the maximum amount of particles to aggregate and settle. Beyond the optimum dose, steric stabilization of the particle took place and increase the turbidity [7, 9].

#### 4. Conclusions

The mucilage obtained from the dragon fruit peel has shown a flocculation effect and the potential to save PACl and limit its harmful effects in the coagulation and flocculation process to remove turbidity and other pollutants from three different types of dye wastewater. The efficient operating dosages of

mucilage ranged in 0.5-20 mg/L, attaining turbidity removal efficiency at maximum of 95% with 10-32% increase compared to PACl coagulation alone, and the PACl saving could be about 3-10% for achieving comparable efficiency. Comparing to PAM, its efficiency was not as stable but sometimes even better or just similar with differences of less than 5%. Therefore, the mucilage from the dragon fruit peel is a potential green alternative in treatment of dye wastewater.

#### Data Availability

The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

#### Conflicts of Interest

The authors declare that they have no conflicts of interest.

#### Authors' Contributions

All authors commented on previous versions of the manuscript and read and approved the final manuscript. All authors contributed to the study conception and design with detailed contribution as the following: Oanh Thi Hoang Le is the first and corresponding author, who is responsible for conceptualization, experiment design, data synthesis, analysis, and interpretation, as well as writing and finalizing the article. Le Nhat Tran is a coauthor, who carried out experiments, especially jar tests, and partly contributed to the first draft of the article. Van Thi Doan is a coauthor, who carried out experiments and analyzed characterizing parameters of dye wastewater and the mucilage. Quang Van Pham is a coauthor, who carried out the sampling trips for obtaining dye wastewater and supervised analytical processes, as well as provided FTIR measurement and interpretation of the corresponding results. Anh Van Ngo is a coauthor, who contributed to experiment design and revised the content of the article. Huan Huu Nguyen is a coauthor, who carried out the sampling trips for obtaining dye wastewater and supervised analysis process.

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