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REMOVAL OF POLLUTANTS FROM DISPERSE BLACK DYE WASTEWATER BY MUCILAGE FROM DRAGON FRUIT PEEL

Tran Nhat Le, Doan Thi Van, Pham Van Quang, Ngo Van Anh, Nguyen Huu Huan, Le Thi Hoang Oanh*

Faculty of Environmental Sciences, University of Science, Vietnam National University,

334 Nguyen Trai, Thanh Xuan, Ha Noi, Viet Nam

*Email: *hoangoanh.le@hus.edu.vn*

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Abstract. Apart from the large revenue, the textile industry in general and the dyeing process in particular are releasing huge amounts of wastewater that pollutes the environment and adversely affects human health. Treatment of this type of wastewater by coagulation and flocculation process requires lots of synthetic chemicals, implying a chemical hazard. Thus, this study evaluated the efficacy of mucilage extracted from the peel of dragon fruit (*Hylocereus undatus)* towards partly substitution of Poly Aluminium Chloride (PAC) used in coagulation and flocculation process for removal of turbidity and other pollutants from disperse black dye wastewater. The effects of pH, settling time, coagulant dosage and mucilage dosage on removal of turbidity, chemical oxygen demand (COD) and color, etc. were analyzed based on Jar-tests. The results show that the addition of mucilage at 10 mg/L after PAC at 245 mg/L under optimal pH 7 and settling time 40 minutes removed turbidity up to 94.92 %. The amount of PAC used was 3 - 10 % less compared to total PAC needed for obtaining equal efficiency when used alone. The pollutant removal by mucilage was also comparable to Polyacrylamide (PAM) – a synthetically organic flocculants.

Keywords: dye wastewater, coagulation and flocculation, turbidity, dragon fruit, *Hylocereus undatus*, mucilage.

Classification numbers: 3.6.2, 3.4.2, 1.1.1.

1. INTRODUCTION

The textile industry is a strongly developing industry, that contributes about 15 % of the total export value of Viet Nam. From 2008 to 2014, Viet Nam was among the top 4 largest textile exporters in the world, after China, India and Bangladesh. With the increasing demand for apparel, as of 2017, there were about 6000 textile establishments across the country [1]. However, textile industry generates a huge amount of wastewater, especially from dyeing process. In total 200-350 m^3 wastewater per ton fabric product, about 91 - 123 m^3 is generated during dispersed dyeing [2]. In general, textile wastewater has a high color, chemical oxygen demand (COD), biological oxygen demand (BOD) and total suspended solids (TSS) [2, 3, 4]. Textile wastewater, if not treated, will cause environmental pollution, affect aquatic habitats and cause human diseases such as dermatitis, severe irritation of skin or tissue necrosis [2, 3].

There are methods to treat textile wastewater such as advanced oxidation process, fenton reagent, photochemical catalysis, adsorption, electro-coagulation, but these techniques have drawbacks such as high cost, volume sludge, and by-products [2]. Coagulation - flocculation (CF) is one effective solution applied popularly to treat wastewater [5]. This technology can remove colloidal particles, organic matters, microorganisms, color and soluble organic and inorganic pollutants. In the coagulation – flocculation process, synthetic salts (e.g. aluminium and ferric salts) or polymers (e.g. polyaluminium chloride, polyacrylamide, polyacrylic acid) are added in wastewater to destabilize colloidal particles and create conditions for colloidals to form larger particles and settle. However, using synthetic coagulants/flocculants has disadvantages. Some researches showed that aluminium residuals in domestic water can lead to Alzheimer's disease and residual monomers of polymer flocculants causes neurotoxicity and cancer [6, 7]. Nowadays, many coagulants and flocculants, which are biologically derived such as plant-based ones have been used to treat textile wastewater as well as wastewater because of their advantages including biodegradability, low cost, environmental friendliness and non-toxicity [5, 8].

Mucilage is a mixed polysaccharide commonly exists in many different parts of higher plants. It swells when dissolved in water and create viscous form. Due to the diversity of chemical composition, mucilage is expected to have many physiological functions in plants [7]. The extracted mucilage is slightly soluble in water, creating a brown, slimy solution and it was experienced to be insoluble in ethanol, acetone and chloroform [9]. Mucilage had been reported to remove at least 85 % TSS, 70 % turbidity, 60 % COD and 90 % color of different kinds of wastewater in both coagulant and flocculant role [5].

The *Hylocereus* species belongs to the family of *Cactaceae* and derived from Mexico and South America. There are three cultivars of dragon fruit: *Hylocereus undatus* (red skinned and white flesh), *Hylocereus polyrhizus* (red skinned and red flesh), *Selinecereus megalanthus* (yellow skinned and white flesh). *Hylocereus undatus,* namely "white dragon fruit", was planted in Viet Nam at least 100 years ago [10]. It is the most cosmopolitan cultivar, distributed all over the world because of its attractive red color and sweetness. Mucilage extracted from leaf, stem, peel and fruit of dragon fruit contains galacturonic acids which is generally the predominant active coagulation agent regardless of the species. High natural polymers such as polysaccharides (e.g. galacturonic acid) and proteins in dragon fruit provide active sites at polymeric chain for particle adsorption and encourage coagulation process [8, 11, 12]. Mucilage extracted from *Hylocereus undatus* peel has been reported working effectively in the role of flocculant for treatment of different dye wastewater [13].

This study determined the pollutant removal of mucilage extracted from dragon fruit peel as the role of a flocculant (PAC as coagulant) in coagulation and flocculation process for treatment of a disperse black dye wastewater.

2. MATERIALS AND METHODS

2.1. Materials

Black dye wastewater was collected from Huy Phat dyeing company in Ha Dong, Ha Noi, Viet Nam, named as B. Its dye ingredient contains black 4.5 % and GS 0.3 % and its characteristics are presented in Table 1.

Peels of white dragon fruit were collected from the kitchen of a school canteen in Ha Noi, Viet Nam for mucilage extraction.

Table 1. Characteristics of investigated dye wastewater sample.

Parameter	Unit	Value	Parameter	Unit	Value
pH		9.34	NO_3^-	mg/L	4.39
Turbidity	NTU	248	PO_4^{3-}	mg/L	8.56
TSS	mg/L	1000	TP	mg/L	169.37
COD	mg/L	1325	TN	mg/L	80.58
SO_4^{2-}	mg/L	112.70	Color index	Pt/Co	5794

2.2. Methods

2.2.1 Extraction of mucilage from dragon fruit peel

The extraction process followed the steps presented in Le *et al.* [13]. Dragon fruit peels were collected, washed and chopped to about 5 mm size and then dried at a temperature of 50 °C until constant weight. Thereafter, the dried peels were mixed with distilled water with a ratio of 1:8 (w/v) and heated at 60 ^oC in a water bath within 1 hour then cooled to room temperature. During the period of heating, the mixture was regularly mixed. After that, the solid-liquid mixture was filtered through 8 muslin layers to obtain the filtrate. Acetone was added to the filtrate with a 3:1 ratio (v/w) to precipitate mucilage. The collected mucilage was dried at 40 °C in an oven until constant weight. Finally, the dried mucilage (DFPM) was crushed and placed in a zip bag in a desiccator for later usage.

2.2.2. Jar tests

Jar tests were applied to examine treatment efficiency of coagulation – flocculation process using DFPM and PAC.

At first, optimal coagulation conditions (settling time, pH, and dosage) of PAC on dye wastewater were determined. Initial pH was varied between 4 and 9 by HCl or NaOH. After that, PAC was added at a fixed concentration, flashly mixed (200 rpm, 1 minute), then slowly mixed (30 rpm, 10 minutes) and finally settled in time range of 10 - 60 minutes. Turbidity was the primary parameter used for determination of optimum conditions. The measurement of water turbidity at 3 cm under the water surface was carried out using Hach 2100Q turbidity meter. Optimal PAC dosage was determined under optimal pH and settling time with PAC ranged from 100 to 300 mg/L.

To test the removal of pollutants when combine DFPM with PAC, optimal dose of DFPM was determined by evaluating turbidity removal. Suboptimal PAC dosage was added in the flash mixing stage (200 rpm, 1 minute) and DFPM (0.2-50 mg/L) was added in the slow mixing stage (30 rpm, 10 minutes). Under optimal combination, the removal of pollutants was calculated based on comparison between effluent and influent's parameters. Analyses were followed APHA [14].

3. RESULTS AND DISCUSSION

3.1. Appropriate conditions of coagulation process on disperse black dye wastewater by PAC

3.1.1. pH and settling time

Settling time and pH were investigated in the ranges of 10-60 minutes and 4-9, respectively (Figure 1). Generally, pH from 4-7 showed high turbidity removal while pH from 8-9 showed significant low turbidity removal. To save the amount of pH-adjusted chemicals, pH 7 was considered optimal for treatment of disperse black dye wastewater. This result matches with the researches of Lin & Peng [15] and Islam & Mostafa [16] in treating dye wastewater. Compared to research on three types of neutral dye wastewater of Le *et al*. [13], the effective pH range of PAC was larger. This could be explained by more protons added in pH adjustment process, which supplemented more positive charges and increased the charge neutralization capacity of PAC.

pH is a very important parameter because most chemical reactions in aquatic enviroment are controlled by changes in pH value. The turbidity removal is related to speciation characteristics of PAC. When PAC is added in water, monomeric (e.g. Al^{3+}, $Al(OH)^{2+}$, $Al(OH)_2^{+}$, etc.**)** and polymeric (e.g. Al_{13} $[(AlO_4Al_{12}(OH)_{24}(H_2O)_{12}]^{7+}$, Al_{30} $[(AlO_4)_2Al_{28}(OH)_{56}(H_2O)_{26}]^{18+}$) aluminium species are formed [8, 17]. The positive species of aluminium neutralize negative charges of dye wastewater leading to destabilization of colloids [17, 18, 19]. pH = 8 is the isoelectric point of aluminum hydroxide, therefore at lower pH values, the precipitate has positive charge and it would neutralize the negative charge of dye wastewater [20]. On the other hand, colloidal particles would be also removed from colloidal suspension through sweep coagulation mechanism by enmeshing in the precipitate. Under alkaline conditions, $Al(OH)^{4-}$ is formed and decreases charge neutralization capacity of PAC.

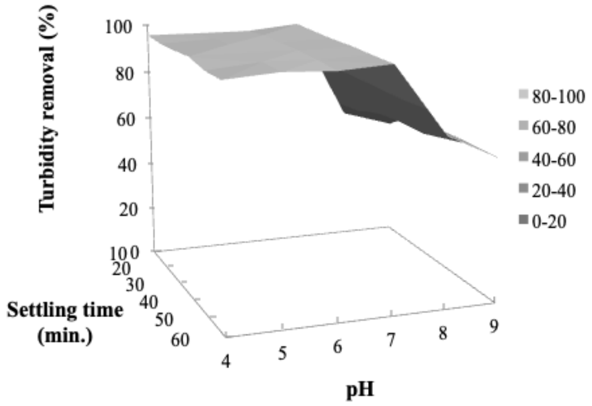

Figure 1. Turbidity removal under different pH and settling time conditions.

Figure 1 also indicated that settling time didn't show significantly different turbidity removal (maximum difference of 3 %); but the optimal settling time was found around 30-40 minutes. Thus, 40 minutes was chosen for assuring maximum effect of coagulation process in further experiments. The results of the study are similar with studies of Zawawi *et al*. and Arulmathi *et al*., which showed that turbidity removal was effective within 30-35 minutes [21, 22].

3.1.2 PAC dosage

Under optimal pH and settling time, PAC dosage was varied from 100 to 300 mg/L in order to study the effect of PAC dosage on turbidity removal (Figure 2). Turbidity removals by PAC dosage between 100 and 260 mg/L were high and stable and peaked at PAC 260 mg/L with 94.86 % removal efficiency. With a further increase in the PAC dosage, turbidity removal was reduced strongly.

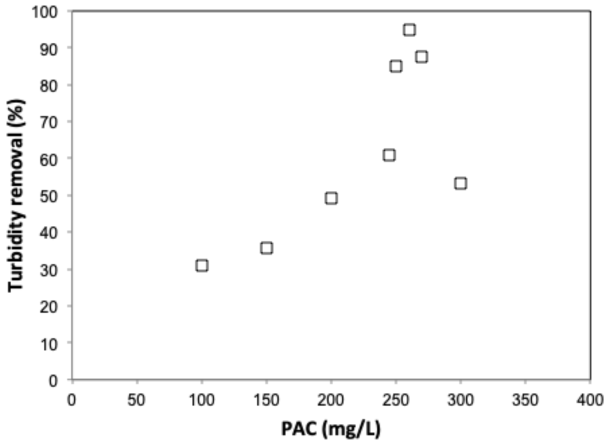

Figure 2. Turbidity removal under different PAC dosages.

Appropriate PAC dosage for treatment of textile wastewater in general and dye wastewater in particular is usually high. This ranged in 150-300 mg/L in Freitas *et al*., 100-400 mg/L in Islam & Mostafa, and 100 - 150 mg/L in Le *et al*. [13, 16, 23]. Optimal dosage is the most important factor for determining coagulation performance and computing the treatment cost. It is also a factor influencing charge neutralization process. The increase of turbidity removal when increasing PAC dosage can be explained by more positive aluminium hydrolysates formed and reacted with negative colloidals in dye wastewater. At higher PAC dosage, polymeric aluminium species are present in solution and assist bridging mechanism [5, 16, 24]. When precipitate formed, sweep floc coagulation mechanism is dominant. Exceeding the optimal concentration will re-stabilize colloidal particles and increase turbidity in the water, leading to reduced turbidity removal efficiency [16, 24].

To research mucilage at the role as a flocculant to combine with PAC, the lower dosage of PAC than optimal dosage was chosen.

3.2. Pollutants removal by coagulation and flocculation process using PAC and mucilage from dragon fruit peel (DFPM)

3.2.1 Turbidity removal

Figure 3 shows the turbidity removal by DFPM with a fixed dosage of PAC at 245 mg/L, under constant pH 7 and settling time 40 minutes. The addition of DFPM (0.2 - 50 mg/L) increased turbidity removal up to the highest value of 93.04 %. Compared with using PAC of the same dosage, turbidity removal of the combination increased 32.29 %. About 10 % PAC was saved in achieving similar efficiency when PAC was used independently, respectively. The effective range of DFPM dosage was less than 10 mg/L. At a DFPM dose greater than 10 mg/L, the turbidity removal tended to decrease.

Adsorption and bridging were reported to be the predominant flocculation mechanisms of mucilage [11, 25]. Natural polymers such as polysaccharides (e.g. 20 % pectin) in DPFM provide active sites at polymeric chain for particle adsorption [4, 11, 12]. Pectin is the methylated ester of polygalacturonic acid containing 1,4-linked α-D-galacturonic acid residues with 300 - 1,000 chains of galacturonic acid units [26, 27]. In dragon fruit, pectin was found with high content (up to 20.14 % dw); of which the fruit peel presents up to one third of the fruit weight [28, 29]. However, an over optimal dose of flocculants will lead to settlement of redispersed and disturbed particles due to increasing in the repulse energy between the flocculant and the dye in solution. Beyond the optimum dose, steric stabilization of the particles take place and increase the turbidity [23].

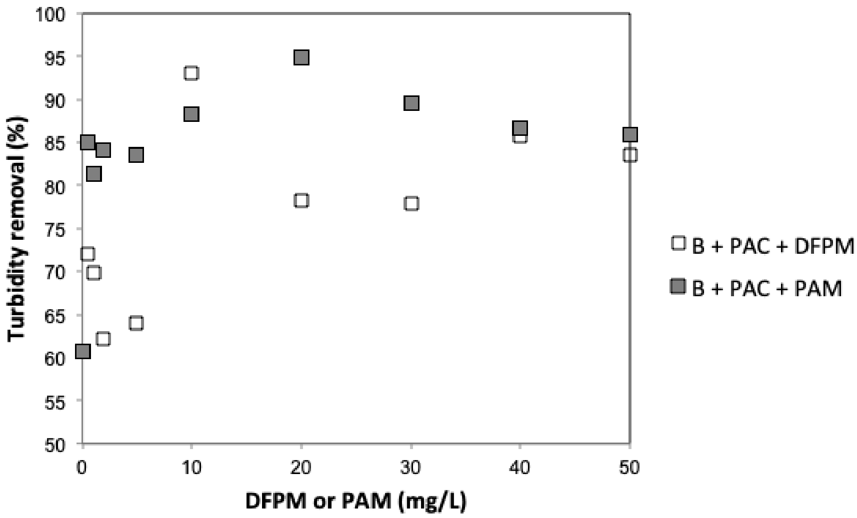

Figure 3. Turbidity removal by coagulation and flocculation process using PAC and DFPM or PAM.

The turbidity removal of DPFM was compared with polyacrylamide (PAM). The highest treatment efficiency of PAM was 94.91 %. Generally, the turbidity removal of PAM was higher than mucilage by 13 - 17 % and the flocs formed by PAM were bigger than flocs formed by mucilage, indicating more efficient and faster settling. However, at some cases, PAM resulted in 5 - 10 % lower efficiency. Investigated on different types of dye wastewater, Le *et al.* obtained compatible results [13]. The results were also in agreement with those of Sellami *et al.* on cactus juice as flocculants, which had similar or slightly lower efficiency (3 - 5 %) than PAM in almost assays [30].

3.2.2 Pollutants removal

Under optimal working conditions, the combination of PAC with DFPM or PAC with PAM showed significant decrease of pollution parameters (Table 2 and Figure 4). Treatment efficiency of mucilage as flocculants for PAC were high for turbidity (94.92 %), TSS (77.6 %), COD (85.51 %), PO_4^{3-} (83.64 %), and color (83.53 %). Lower treatment efficiency was obtained for SO_4^{2-} (12.12 %), NO_3^- (38.87 %), TN (32.87 %) and TP (49.87 %). Comparing DFPM to PAM, the treatment efficiencies were equal (turbidity, PO_4^{3-}, TSS), higher (COD, TN) or lower (NO_3^-, TP, color). The results were similar to the research comparing the coagulation-flocculation effect of mucilage extracted from cactus juice with PAM [30].

The pH is one of the most significant effluent discharge quality parameters of an industry. After treatment, the pH value was within the permitted range regulated by QCVN 13-MT: 2015/BTNMT for B class (5.5-9.0). The investigated dye wastewater had a very high initial pH of 9.34 because dyed fiber is polyester and black. At the final stage of polyester dyeing, there is a need to decrease the amount of residual dye on the fiber surface by alkalinizing the dye solution. However, parameters such as TSS, COD and color index were higher than regulated values, indicating further treatment after coagulation - flocculation process for treatment of this wastewater.

Table 2. Characteristics of dye wastewater treated by coagulation - flocculation process using PAC and DFPM or PAM.

Parameter	Unit	Influent	Effluent		QCVN 13-MT: 2015/BTNMT (B class)
			PAC+DFPM	PAC+PAM	
pH		9.34	8.18	8.07	5.5 – 9.0
Turbidity	NTU	248	12.6	10.2	-
TSS	mg/L	1000	224	228	100
COD	mg/L	1325	192	292	200
SO_4^{2-}	mg/L	112.70	99.04	97.34	-
NO_3^-	mg/L	4.39	2.68	2.30	-
PO_4^{3-}	mg/L	8.56	1.40	1.47	-
TP	mg/L	169.37	84.91	75.92	-
TN	mg/L	80.58	54.09	64.16	-
Color index	Pt/Co	5794	954	961	200

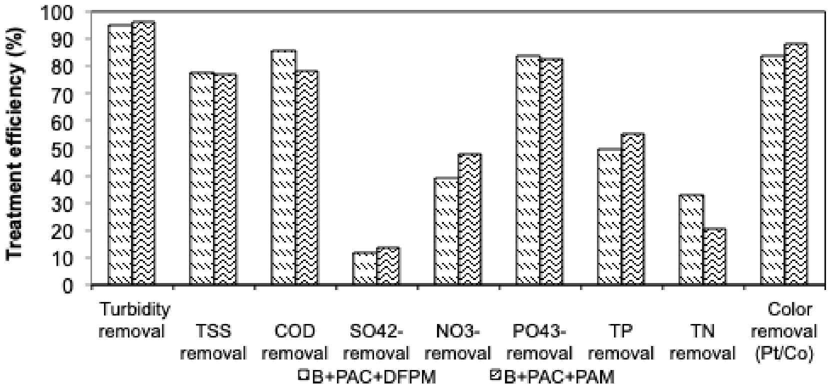

Figure 4. Pollutants removal by coagulation and flocculation process using PAC and DFPM or PAM.

The pollutants removal efficiency achieved by DFPM was higher than mucilage of *Ocimum basilicum* L. on textile wastewater [31]. Compared with study of Bouatay *et al.*, turbidity removal by DFPM was also higher, but COD and color removal were about 3-8 % lower [32]. Okra mucilage had COD removal efficiency equal to DFPM, but turbidity and color removal were 3-10% higher [23].

4. CONCLUSIONS

Effective removal of turbidity and pollutants from disperse black dye wastewater could be achieved by using *Hylocereus undatus* mucilage (DFPM) as a plant-based flocculants in coagulation and flocculation process with PAC as coagulant. The combination of PAC

(245 mg/L) with DFPM (10 mg/L) was able to remove 94.42 % of turbidity, 77.64 % of TSS, 85.51 % of COD, 83.64 % of PO_4^{3-}, 83.53 % of color and other parameters such as TN, TP, NO_3, SO_4^{2-}. The addition of DFPM to the coagulation and flocculation process was proved to save PAC (10 %), indicating a reduction of negative effects on human health and the environment. Comparing with PAM, DFPM was not as stable but in compatible. Therefore, DFPM is a promising natural flocculant to substitute commercial synthetic chemicals as PAC and PAM, and at the same time, its production will take advantage of abundant sources of dragon fruit waste in Viet Nam.

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