

Optimization Studies on Textile Wastewater Decolourization by Fe³⁺/Pectin

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Abstract. The existence of dyes in wastewater is harmful to the environment if it is released without proper treatment. The aim of this study was to determine the application of pectin extracted from banana pseudostem for coagulation-flocculation of textile wastewater in Penang. Pectin was applied with the addition of Fe³⁺ on textile wastewater and the optimum of colour removal efficiency was measured. The optimization study through central composite design (CCD) was conducted in jar test to determine the interaction effect between pH, coagulant dose, and flocculant dose. Results showed there is a significant effect between pH and flocculant dose on the colour removal, followed by the effects between coagulant dose and flocculant dose and no interaction between pH and coagulant dose ($p > 0.05$). In conclusion, 74.2% of colour removal can be obtained at the optimum treatment conditions (pH 7.5, coagulant dose 250 mg/L, and flocculant dose 35 mg/L). This demonstrates the benefits of the approach based on the response surface method (RSM) in achieving good predictions especially for treating actual textile waste.

Keywords: Pectin, Textile wastewater, Coagulation- flocculation, Central composite design (CCD)

1. Introduction

Textile industries are one of the biggest polluter on the earth which generates wastewater not only high in biochemical oxygen demand (BOD), chemical oxygen demand (COD) but also high in colour. Due to complexity of the dye compound, the colour may still remain in the effluent even after extensive treatment (Che Noraini et al., 2012). Therefore the effluents have to be treated carefully before discharge to nearby water body.

Al³⁺ and Fe³⁺ based coagulants, such as polyaluminum chloride (PAC) and polyferric chloride (PFC) , are two principal inorganic coagulants used in wastewater treatment (Chen et al., 2010). The coagulation-flocculation mechanisms of most biopolymeric flocculants are based on electrostatic attraction of the pollutants to cationic functional groups of the coagulants in aqueous solutions, indicating that coagulation capabilities depend heavily on the charged state of the pollutants (Jeon et al., 2009). Chao et al. (2004) state that organic flocculant shows efficiency on anionic dye removal but modification needs to be done when involving cationic dye. Decolorization of reactive dyes using inorganic coagulants (1g/L) only can remove less than 20% of color removal compared by addition with synthetic polymer showed almost 100% of color removal (Joo et al., 2007).

In general, organic polymers are used as flocculant after the addition of inorganic coagulants. Chitosan, pectin, tannin and a range of microbial flocculants are few examples of naturally occurring flocculating agent. Fe³⁺ demonstrates good coagulant properties in inorganic kaolin suspensions. It has long been known that the addition of cations to suspension is necessary to induce the effective flocculating activity of negatively charged polymers (Ho et al., 2010).

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Coagulation-flocculation treatment of extracted pectin on synthetic wastewater implied that the performance of extracted pectin is excellent compared to the commercial pectin (Ho et al., 2010). Qiu et al. (2010) stated that banana could be one of the potential flocculant as alternative pectin.

Even though coagulation-flocculation has been used widely from the past, the time consuming procedure is crucial to be minimize. Thus response surface method (RSM) is an alternative for conventional method to minimize the required number of experiment. Therefore, the main objective of the study is to see the interaction effect between the selected factors (pH, coagulant dose and flocculant dose) from preliminary experiment by using CCD.

2. Materials and methods

2.1. Preparation of Pectin

Raw banana pseudo-stem was cut into smaller pieces and dried at 60°C for 48 hours. Extraction of pectin was carried out at pH 2, temperature 80°C and time 4 hours. Isopropyl Alcohol (IPA) was added to precipitate the pectin before undergo freeze dried. Finally the homogenous pectin powder will be collected and stored at room temperature.

2.2. Experimental procedure

A standard jar-test apparatus equipped with 6 stainless steel paddles and stirrer was used for the coagulation and flocculation tests. Wastewater effluent is collected from one of the textile company in Penang. The characteristics of the wastewater are shown in Table 1. 250 mL of textile wastewater was transferred into the jar. The wastewater pH was adjusted with 1M H₂SO₄ and 1M NaOH. Jar test was conducted at 150 rpm rapid mixing for 3 minutes, 45 rpm slow mixing at 15 minutes and the samples was left to settle for 30 min. Apparent colour concentration was reported in Platinum–cobalt (pt co) using Method No. 2120C, APHA 1992. The equation for removal efficiency of colour is shown below, where C_i and C_f are the initial and final colour concentration of dyes wastewater.

$$\text{Removal (\%)} = [(C_i - C_f) / C_i] * 100 \quad (1)$$

The central composite design (CCD) (Table 2), which is the standard RSM, was used to evaluate the effect of pH, coagulant dose and flocculant dose in the optimization of colour removal.

Table 1: The characteristics of textile wastewater

Parameters	Values
pH	7.89
Chemical Oxygen Demand	372 mg/ L
Biological Oxygen Demand	41 mg/ L
Colour	550 pt co
Temperature	34.3 °C
Disolved Oxygen	1.05 mg/L

3. Result and Discussion

3.1. Interaction Effect between Independent Variables (pH, coagulant dose and flocculant dose) on Colour Removal

The response variable was fitted by a second-order model in the form of quadratic polynomial equation models for colour removal in terms of coded variable is given by Eqs.(2)

$$79.25 + 7.12x_1 + 0.30x_2 - 1.20x_3 - 8.97x_1^2 - 4.51x_2^2 - 8.90x_3^2 - 3.36x_1x_2 - 5.99x_1x_3 - 4.76x_2x_3 \quad (2)$$

3.2. Regression Analysis of Relationship between Colour Removal and Independent Variables (pH, coagulant dose and flocculant dose)

Regression analysis of colour removal showed the significance of each coefficient determined using the F-test and p-value at significance level of 0.05. The corresponding variables would be more significant if the absolute p-value is smaller while F-value is greater. It showed that the variables with the largest effect were the quadratic term of flocculant dose (X₃²), the quadratic term of pH (X₁²), the linear term of pH (X₁), the interaction effect of pH and flocculant dose (X₁X₃), the quadratic term of coagulant dose (X₂²) and the interaction effect of pH and flocculant dose (X₂X₃). The rest of the source of variance showed insignificant

effect with p-value more than 0.05 (coagulant dose: X_2 , flocculant dose: X_3 , the quadratic term of coagulant dose: X_2^2 , the interaction effect of pH and coagulant dose X_1X_2).

Table 2: Central composite design (CCD) with the experimental responses values for colour removal.

Std	X_1 : pH	X_2 : Coagulant dose (mg/L)	X_3 : Flocculant dose (mg/ L)	Colour removal (%)
1	9	500	20	66.5
2	6	350	35	82.5
3	3	500	50	45.2
4	6	350	35	77.4
5	9	200	50	54.7
6	3	200	20	30.9
7	6	350	35	81.3
8	6	350	35	77.8
9	3	200	50	58.8
10	9	500	50	51.4
11	9	200	20	74.5
12	3	500	20	60.1
13	6	350	10.5	56.6
14	6	105.05	35	70.2
15	1.1	350	35	45.1
16	6	350	35	75
17	6	594.95	35	70
18	6	350	59.49	60.2
19	6	350	35	80.8
20	10.9	350	35	71.3

Table 3: Analysis of variance (ANOVA) for colour removal

Source of variance	Sum of Squares	DF	Mean square	F-value	p-value
Block	217.38	2	108.69		
Model	3368.62	9	374.29	12.98	0.0007
X_1	675.23	1	675.23	23.42	0.0013
X_2	1.18	1	1.18	0.041	0.8445
X_3	19.25	1	19.25	0.67	0.4375
X_1^2	1062.87	1	1062.87	36.86	0.0003
X_2^2	268.46	1	268.46	9.31	0.0158
X_3^2	1045.17	1	1045.17	36.25	0.0003
X_1X_2	90.45	1	90.45	3.14	0.1145
X_1X_3	286.80	1	286.80	9.95	0.0135
X_2X_3	181.45	1	181.45	6.29	0.0364
Residual	230.66	8	28.83		
Lack of fit	194.71	5	38.94	3.25	
Pure error	35.95	3	11.98		
Cor total	3816.67	19			

Analysis of variance (ANOVA) for the quadratic model was also carried out to validate the model (Table 3) showed the regression model was significant p-value < 0.05. The second-order regression models obtained for colour removal is satisfactory since the value of the coefficient of determination (R^2) is high and close to 1. The value of R^2 for colour removal is 0.9359 and the correlation coefficient indicates that only 6.41% of the total variation could not be explained by the empirical model. The Lack of Fit F-value for Colour removal showed 3.25 which implies the Lack of Fit is not significant relative to the pure error. It implies that the second-order polynomial model fitted the experimental results well.

3.3. Analysis of response surface

Fig. 1 and Fig. 2 illustrated the relationship between colour removal and the independent variables (pH, coagulant dose and flocculant dose) through three-dimensional response surface plots.

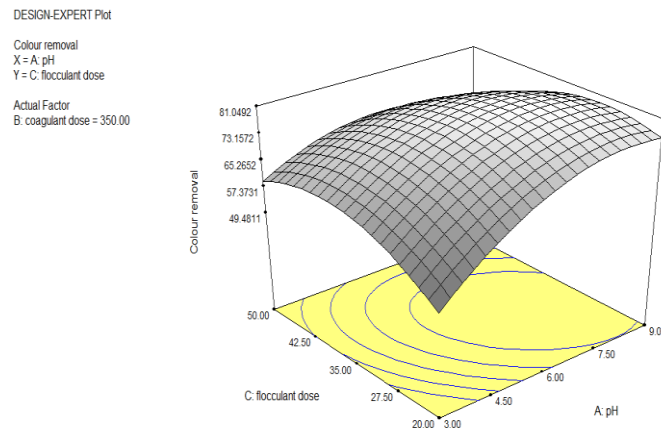


Fig. 1: Interaction effect of pH and flocculant dose on colour removal.

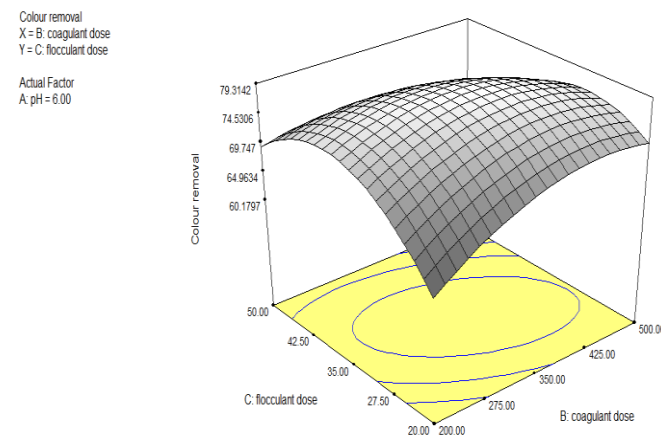


Fig. 2: Interaction effect of coagulant dose and flocculant dose on colour removal.

3.4. Interaction effects of pH and flocculant dose on colour removal efficiency

The best results for colour removal was obtained at pH 7.5 and flocculant dose levels at 35 mg/ L and as can be observed by the stationary point of maximum. On the other hand, the mound shape at alkaline pH indicates that the highest percentages can be obtained at intermediate values of the factors. Colour removal is higher when the pH around pH 7.5 with lowers addition of flocculant. Otherwise the removal rate is decreasing after flocculant dose is more than 35 mg/ L. This implies that over dosing happened in the reaction solution (Chen et al., 2010) which contribute to lower efficiency of colour removal. The response surface plot reveals that the pH had significant effect on colour removal. The colour removal response surfaces in Fig. 1 indicate 76.4% removal efficiency.

3.5. Interaction effects of coagulant and flocculant dosage on colour removal efficiency

The correlative effects of coagulant dose and flocculant dose on colour removal is demonstrated in Fig. 2, indicating parabolic relationships between coagulant/flocculant dose and color rate. A higher coagulant dose may lead to an increase of colour removal, while increase of the flocculant dose may rapidly improve colour removal and then decrease it when excessive of flocculant being added into the wastewater. The possible explanation is hydrolysis reaction take place when coagulant (cation) is added into the wastewater which leads to the adsorption on the negatively charged particles to neutralize the charged around the particles. It might need high dose of coagulant and at the same time lower the alkalinity of the water to make the coagulation and flocculation process efficient. However, an increase in both coagulant dose and flocculant dose beyond the optimum region resulted in a decrease in the removal efficiency. Overdosing deteriorated supernatant quality, referring to the “restabilization” of the colloidal particles (Ho et al., 2010), and therefore

the particles could not be coagulated well. The highest removal of colour can be achieved by changing the coagulant dose and flocculant dose to 250 mg/ L and 35 mg/ L, respectively.

4. Conclusion

Pectin from banana pseudo-stem proved to be a useful anionic flocculant with help from Fe³⁺ for colour removal of textile wastewater. The effect of pH, coagulant dose and flocculant dose on colour removal of textile wastewater was investigated. The ANOVA tests also demonstrate the effect between pH and flocculant dose and the effect between coagulant dose and flocculant dose influenced the efficiency of colour removal. The results reveal that the optimal colour removal was obtained at coagulation dose of 250 mg/ L, flocculant dose of 35 mg/ L and pH 7.5.

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6. References

- [1] A. C. Chao , S. S. Shyu, Y. C. Lin, F. L. Mi. Enzymatic grafting of carboxyl groups on to chitosan-to confer on chitosan the property of a cationic dye adsorbent. *Bioresource Technology*. 2004, **91**(2), 157–162.
- [2] T. Chen, B. Gao, Q. Yue. Effect of dosing method and pH on color removal performance and floc aggregation of polyferric chloride–polyamine dual-coagulant in synthetic dyeing wastewater treatment. *Colloids and Surfaces A: Physicochem. Eng. Aspects*. 2010, **355**(1-3), 121–129.
- [3] C. H. Che Noraini, N. Morad, , I. Norli, T. T. Teng, C. J. Ogugbue. Methylene blue degradation by *Sphingomonas paucimobilis* under aerobic conditions. *Journal of Water Air and Soil Pollution*. 2012, doi: 10.1007/s11270-012-1264-8.
- [4] D.J. Joo, W. S. Shin, J. H. Choi, S. J. Choi, M. C. Kim, M.H. Han, T. W. Ha, Y. H. Kim. Decolorization of reactive dyes using inorganic coagulants and synthetic polymer. *Dyes and Pigments*. 2007, **73**(1), 59–64.
- [5] Y.C. Ho, I. Norli, F.M., Abbas Alkarkhi, N. Morad. Characterization of biopolymeric flocculant (pectin) and organic synthetic flocculant (PAM): A comparative study on treatment and optimization in kaolin suspension. *Bioresource Technology*. 2010, **101**(4), 1166-1174.
- [6] J. R. Jeon, E. J. Kim, Y. M. Kim, K. Murugesan, J.H. Kim, Y. S. Chang. Use of grape seed and its natural polyphenol extracts as a natural organic coagulant for removal of cationic dyes. *Chemosphere*. 2009, **77**(8): 1090–1098.
- [7] L. P. Qiu, G. Zhao, H. Wu, L. Jiang, X. Li, J. Liu. Investigation of combined effects of independent variables on extraction of pectin from banana peel using response surface methodology. *Carbohydrate Polymers*. 2010, **80**: 326–331.
- [8] APHA, AWWA, WPCF. Standard Methods for the Examination of Water and Waste Water, 19th edition, 1992.
- [9] D. C. Montgomery. Design and analysis of experiments, 5th edition. New York: Wiley, 2004.