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Coagulation and flocculation of printing ink effluent using polyaluminium chloride (PAC): optimization and phytotoxicity study

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ABSTRACT

The aim of the current study was to optimize the efficiency of polyaluminium chloride (PAC) for reducing color from the ink effluent. The optimization process was conducted based pH, coagulant dose, mixing time and mixing speed. The phytotoxicity in treated dye effluent using green bean seeds to use on seed germination test. The optimal reduction efficiency for dye samples color was 98.53%, while was 71.02% for chemical oxygen demand, 85.89% for turbidity and 88.01% for total suspended solid. The phytotoxicity concentration was 43.24%. Scanning electron microscopy images indicated that the use of PAC in coagulation–flocculation had led to settle the particles.

Keywords: Polyaluminium chloride (PAC); Color; Printing ink wastewater; Scanning electron microscopy (SEM); Phytotoxicity

1. Introduction

The printing ink industry is one of the ancient and lucrative industries in society, which has added values to the economic growth of the nation [1]. The ink industry has experienced significant growth in Malaysia during recent years that estimated at around 93% [2]. The generated ink effluent from the printing processes considers one of the most colored and contaminated wastewater due to the high content of organic matter and inks, which require pre-treatment before the disposal stage [2,3]. The effluents of printing ink are characterized by high chemical oxygen demand (COD), turbidity and total suspended solid (TSS). Printing effluent is also associated with chemicals and dye which are capable of damaging the environment and natural water systems [3]. Various technologies have been used to remove the color, organic matters and COD from the ink and industrial effluents, including the microorganisms' biodegradation, the adsorption using activated carbon and the coagulation and flocculation [4–6]. These technologies are frequently used in the treatment of printing effluent due to their high efficiency during limited numbers of operations besides the ease of handling with them [7]. The application of coagulation/flocculation process using organic or inorganic flocculants/coagulants such as aluminum, iron(III) salts or organic polyelectrolytes have achieved satisfactory results in wastewater treatment in the literature [8]. The polyaluminium chloride (PAC) has been extensively used as a flocculent for treating dyes wastewater where it has shown high removal efficiency of dyes, and polymeric Al_{13} stability [9]. Ghafari et al. [9] have reported a high performance of PAC in leachate treatment where the removal efficiencies of color, COD, turbidity and TSS are estimated by 90.7%, 43.1%, 94.0% and 92.2%, respectively.

Although several studies have been conducted on the treatment of ink effluent discharged from the printing

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industry, there is a lack of studies on the optimization of the independent factors such as the pH and dosage of PAC to obtain the optimal parameters for reducing color and the other parameters [10–12]. Moreover, previous studies have not detailed out on the optimization of maxing speed and mixing time in coagulation–flocculation, besides that, most of the literature have focused on laboratory tests as the removal ratio of color, COD, TSS, etc., while there is a lack of sources that test the treated ink wastewater as a water source for irrigation or for other human uses.

The ink effluents contain a relative amount of heavy metals, aromatic dyes and chemical nutrients which contribute to the increasing the phytotoxicity. Therefore, the phytotoxicity test of the treated ink effluent should be conducted before the final disposal of these effluents into the environment. Mitelut and Popa [13] revealed that the seed germination (SG) could be used for the phytotoxicity test. Hassan et al. [14] used country bean to test the phytotoxicity of the dye effluent. In the present study, the optimization of the coagulation and flocculation process of ink effluent by using PAC was investigated, the efficiency of the PAC was evaluated based on the reduction in COD, TSS, and color. The safe disposal was confirmed based on the phytotoxicity test using green bean seeds.

2. Materials and methods

2.1. Sampling of ink effluents

Dye samples were collected from the printing ink center at UTHM, Parit Raja, Johor, Malaysia. 10 L was collected in various periods during the working days to investigate the water characteristics. 30 L was collected triple into a plastic bottle (50 L) on different days to conduct 30 experimental runs, followed by 60 confirmatory experimental runs. The sample was brought from the printing ink center to the laboratory at the icebox (approximately 3°C) to preserve the water quality and contents. The characteristics of the ink effluents were investigated, as described by APHA [15].

2.2. Polyaluminium chloride

PAC (174.75 MW) (basicity: 45%–55%) in the form of white powder produced by R&M Chemical Company (UK) was used as a treatment coagulant in the current study without further purification or modification.

2.3. Optimization study

The reduction of color, COD, turbidity and TSS of the ink effluents was optimized using response surface methodology (RSM). The experimental runs and the independent variables were designed by Design Expert 11.1.2.0, central composite design (CCD) (Stat-Ease, Inc., Minneapolis, USA) software. The optimizing of the coagulation and flocculation process was performed using CCD to observe the reflection of coagulant dosage (X_2) at a different range of pH (X_1), mixing time (X_3), and mixing speed (X_4). The maximum (+1), intermediate (0), and minimum (–1) values of each independent variable are illustrated in Table 1. The range of the independent factors was selected as described by Ashtekar et al. [16]. Thirty (30) experimental runs were performed to assess the interaction between independent factors and for the optimization of the variables and responses which included the removal of color, COD, turbidity, TSS and phytotoxicity concentration.

2.4. Coagulation and flocculation process using a jar test

The jar test apparatus consists of six spindle steel paddles working simultaneously with the same speed and time set up. The experiments were conducted under different conditions of various doses of PAC, pH values, mixing time and rate as the provided design of the Design-Expert software (RSM). A fixed volume (1,000 mL) of the ink-water sample was filled in 1,000 mL volume beakers. The pH value of each run was adjusted with 0.1 M NaOH or 0.1 N of HCl following the provided experimental design by RSM. The processed sample was left in the jar test equipment for 15 min under 45 rpm then for 30 min under 0 rpm for settling. The water quality and color tests were performed immediately after the settlement time as described by APHA [15].

2.5. Sludge height test

The sludge height test is one of the most useful tests in proving the removal efficiency of the contaminated particles from the wastewater where the increase in height is accompanied by an increase in pollutions removal [17].

The sludge height was determined using a ruler after every experimental run in the current study that was shown a sediment layer which proved the efficiency of the experiments.

2.6. Phytotoxicity test

Germination index (GI) was used as a phytotoxicity test for the toxicity evaluation of processed printing effluents. The tests were carried out with ten green bean seeds placed in a Petri dish containing cotton wool. Each Petri dish was fed with 10 mL of the treated samples, besides one Petri dish was fed with the control sample (deionized water). All the Petri dishes were incubated for 5 d under temperature between 20°C to 28°C. The length of the growing plant was measured daily at a fixed time (every 24 h) for 5 d, while the SG and the root length after the 5th day were considered. The relative SG, relative root elongation and GI were determined as described by Jagadabhi et al. [17]:

$$\mathrm{SG}(\%) = \frac{\begin{array}{c}\text{Number of seeds germinated in}\\ \text{the treated sample}\end{array}}{\begin{array}{c}\text{Number of seeds germinated in}\\ \text{the control sample}\end{array}} \times 100 \tag{1}$$

$$\mathrm{RE}(\%) = \frac{\text{Mean root length in the treated sample}}{\text{Mean root length in the control sample}} \times 100 \tag{2}$$

$$\mathrm{GI} = \frac{\mathrm{SG}(\%) \times \mathrm{RE}(\%)}{100} \tag{3}$$

Table 1
Coded and un-coded levels of the independent variables used in the current study

Factor	Symbol	Level		
		Low (–1)	Middle (0)	High (+1)
Value of pH	X_1	4 (1[a])	7	10 (13[a])
Polyaluminium chloride dosage	X_2	100 (0[a])	300	500 (700[a])
Mixing time, (min)	X_3	2 (0[a])	10	15 (22[a])
Mixing rate, (rpm)	X_4	40 (0[a])	100	150 (205[a])

[a]Single run was suggested by Design-Expert software among of the 30 experimental runs.

The experimental runs were laid out in a regular design with 30 different samples (treated samples), one control sample (deionized water) and one seed type (green bean seeds). GI data were analyzed by RSM using a quadratic model for all the experimental runs.

3. Results and discussion

3.1. Characteristics of ink effluents

The parameters of ink effluents are presented in Table 2. The COD and TSS concentrations were 577 ± 7 and 208 ± 10 mg Al/L, respectively, which are exceeded the Malaysian standards of sewage and industrial effluent discharge that stated by 100 mg Al/L for both parameters. Color concentration was also high (657 ± 20 Pt-Co) compared to the allowed concentration for disposal wastewater prepared by APHA [15], 2120B method, which was estimated by 300 Pt-Co. The high concentrations of COD, TSS and color in the ink effluents are due to the high content of organic matter and inks which are represented in the form of a complex matrix of dyes [18].

3.2. Optimization study

The parameters optimization in the present study achieved a high removal efficiency of color ranged from 43 to 97%, using various concentration of PAC (100, 300 and 500 mg Al/L) (Table 3). The obtained results of the current study were shown that the best removal efficiency of color (y_1) was observed with the average dosage of PAC (X_2) (300 mg Al/L) (Fig. 1), while there were no effects of pH value (X_1), mixing time (X_3), and mixing speed (X_4) on the color removal process (Table 3). According to Mahmudabadi et al. [19], the optimal removal of color was with a PAC concentration of 300 mg Al/L, while the removal performance has reduced in the high concentration of PAC (500 and 600 mg Al/L) which can be caused due to the excessive usage of dosage which leads to resuspend the flocs and decrease the removal efficiency [20]. The results were in agreement with the study performed by Daud et al. [21] showed that the best removal efficiency was in the average dosage of PAC (350 mg Al/L).

The analysis of variance (ANOVA) provided by Design-Expert software showed that the model of color removal was significant, with a P-value of 0.0418 (Table 4). According to the ANOVA analysis (Table 4), there were no effects of pH (X_1), mixing time (X_3), and mixing speed (X_4) factors ($P > 0.05$) on color removal, while the concentration of the PAC dosage (X_2) had a quadratic effect on color removal with a P-value of 0.0001.

Table 2
Characteristics of ink effluents compared to Malaysian sewage and industrial effluent discharge standard (samples, $n = 3$, Mean ± Stdev)

Parameter	Mean ± Stdev	Standard effluent
Color, Pt-Co	657 ± 20	300[a]
COD, mg/L	557 ± 7	100[b]
Turbidity, NTU	91 ± 3	6.5–8.5[b]
TSS, mg/L	208 ± 10	100[b]
Phytotoxicity concentration, %	92 ± 1	NR

[a]APHA 1995, method 2120B;
[b]Malaysian sewage and industrial effluent discharge standard;
NR – Not recorded.

COD removal efficacy was estimated between 41.22% and 74.63% (Table 3). The best removal ratio of COD was recorded with the average concentration of PAC (300 mg Al/L), the highest dosage of PAC (500 mg Al/L) was shown the lowest removal efficiency of COD, besides with increased the PAC concentration in the out of range sample (700 mg Al/L) the removal ratio reduced to 37.34% (Table 3 and Fig. 2).

Several studies have been performed about COD removal using PAC. Daud et al. [21] reported that COD reduction achieved the optimum ratio estimated by 75% with a PAC dosage of 300 mg Al/L. Mahmudabadi et al. [19] stated that 47.5% of COD had removed with a PAC concentration of 400 mg Al/L.

The ANOVA analysis was indicated the significance of the COD removal model with a P-value of 0.0452 (Table 4). The Importance of each factor was represented by the P-value, where pH value (X_1) and PAC dosage (X_2) factors were showed a quadratic effect on the removal of COD with the P-value of 0.0108 and 0.0003, respectively (Fig. 2 and Table 4). While there were no effects of the mixing time (X_3), and mixing speed (X_4) on the removal of COD (P-value < 0.05) (Table 4).

Turbidity reduction indicated that the PAC dosage effects strongly in the removal efficiency. Fig. 3a clarifies that the turbidity removal ratio increased by increasing the dosage

Table 3
Summary results of all experimental runs of the study (central composite design arrangement and responses)

Run	X_1	X_2	X_3	X_4	y_1	y_2	y_3	y_4	y_5
					Observed	Observed	Observed	Observed	Observed
1	7	300	9	95	96.35	72.48	85.57	85.67	42.80
2	4	500	2	150	43.53	43.65	91.87	89.61	91.60
3	13	300	9	95	75.02	56.37	86.25	78.37	100.00
4	7	0	9	95	16.29	19.34	27.36	36.06	46.77
5	10	500	2	150	44.61	43.66	90.74	84.23	74.00
6	7	300	9	95	96.35	73.04	85.04	86.03	42.80
7	7	300	9	0	82.73	58.21	60.19	66.92	48.30
8	10	500	2	40	45.06	49.84	87.54	87.15	35.80
9	7	700	9	95	45.51	37.34	92.97	90.38	93.20
10	7	300	9	95	97.08	72.33	85.94	86.10	42.80
11	10	100	2	40	59.56	50.83	60.02	66.42	10.20
12	7	300	22	95	96.65	74.63	86.32	87.60	34.80
13	10	500	15	40	46.35	41.22	86.11	87.60	60.80
14	4	100	2	150	61.11	50.81	70.43	78.56	60.10
15	4	100	15	40	63.91	59.61	61.21	64.60	89.70
16	4	500	15	150	47.98	40.63	92.66	94.71	96.85
17	4	500	2	40	45.39	42.01	85.86	83.75	82.60
18	7	300	9	95	95.98	72.22	87.43	85.67	42.80
19	10	500	15	150	45.43	48.94	99.78	96.08	33.10
20	7	300	0	95	83.76	63.35	71.02	78.56	40.40
21	7	300	9	95	96.22	72.90	86.12	86.40	42.80
22	7	300	9	205	94.21	71.29	90.64	94.81	33.00
23	10	100	15	40	60.43	55.40	65.12	63.27	55.10
24	4	500	15	40	42.34	41.89	89.30	82.79	92.80
25	4	100	15	150	65.59	64.81	75.98	78.08	0.00
26	1	300	9	95	80.02	37.88	88.71	81.25	100.00
27	4	100	2	40	60.89	61.81	59.02	56.04	57.40
28	10	100	2	150	61.54	52.68	67.87	72.31	0.00
29	7	300	9	95	96.19	72.78	85.39	85.39	42.80
30	10	100	15	150	64.78	58.58	74.23	74.71	47.70

X_1 (pH value); X_2 (PAC dosage, %); X_3 (mixing time, min); X_4 (mixing speed, rpm); y_1 (color removal, %); y_2 (COD removal, %); y_3 (turbidity, %); y_4 (total suspended solid, %); y_5 (phytotoxicity concentration, %).

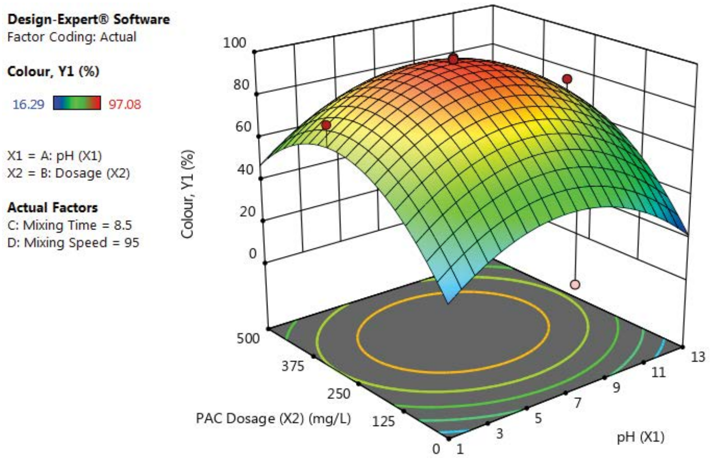

Fig. 1. Design-Expert plot, a 3D surface graph showing the effect of affected factors on color removal.

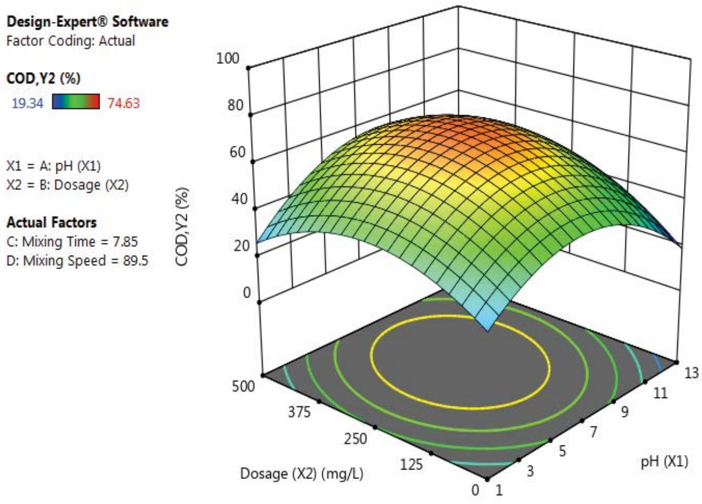

Fig. 2. Design-Expert plot, a 3D surface graph showing the effect of affected factors on COD removal.

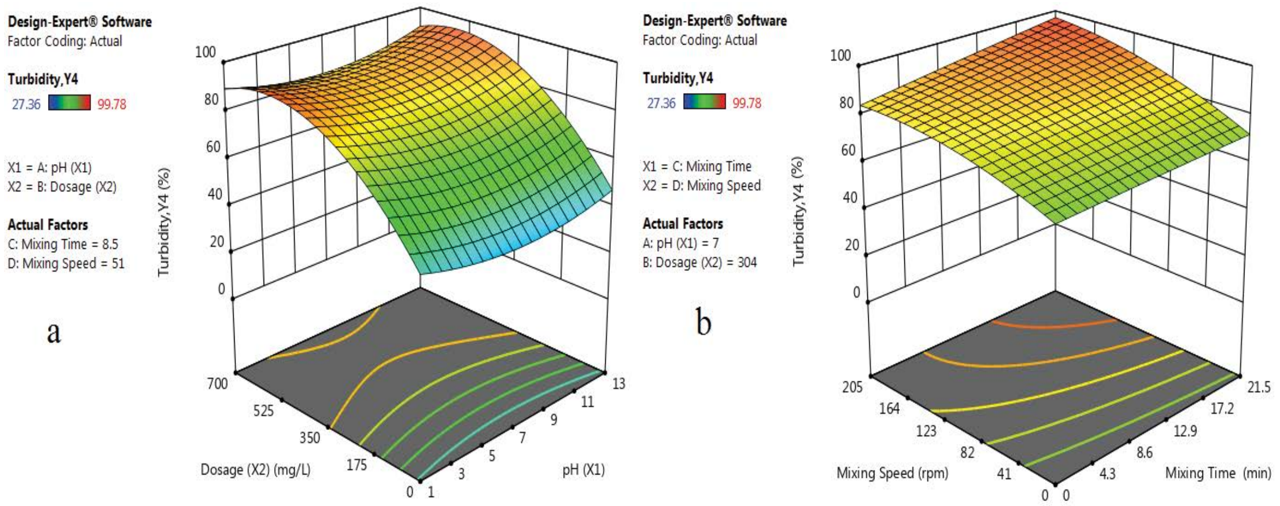

Fig. 3. Design-Expert plot, a 3D surface graph showing the effect of affected factors on turbidity removal.

of PAC, which is strongly related to the precipitation of the ink particles at the bottom [22]. On the other hand, the mixing speed (X_4) factor was effected on the turbidity removal where when the mixing speed increased the removal ratio increased which can be due to the potent combination occurred between the molecules of the inks and PAC dosage because of the rapid maxing speed (Fig. 3b).

The turbidity removal ratio was increasing with the increase of PAC as it reached the optimal removal ratio of 99.78% with a PAC dosage of 500 mg Al/L (Table 3). Mahmudabadi et al. [19] showed similar findings, the turbidity removal percentage improved by increasing the PAC dosage and reached 98% with a dosage of 200 mg Al/L and increased to 99.6% with a PAC dosage of 400 mg Al/L.

The model of turbidity removal was significant with a *P*-value of 0.0003, and the lack of fit was not significant with a *P*-value of 0.0229. The turbidity removal response was effected by PAC dosage (X_2) and mixing speed (X_2) factors as shown in Fig. 3. PAC dosage (X_2) and mixing speed (X_4) has a direct (linear) effect on turbidity removal with a *P*-value of 0.0001 and 0.0035 respectively. Furthermore, PAC dosage (X_2) has an indirect (quadratic) effect on turbidity removal with a *P*-value of 0.0025. According to the ANOVA analysis, pH value (X_1) and mixing time (X_3) factors have no effects on turbidity removal response (P-value > 0.05) (Table 4).

The TSS removal results showed a high removal efficiency reached 96.0% with the highest dosage of PAC (500 mg Al/L) (Table 3). PAC dosage (X_2) and mixing speed (X_4) was effected strongly on TSS where according to the 3D surface graph shown in Fig. 4, TSS removal is affected by the increase of the PAC concentration cumulatively, where the increase of PAC leads to an increase of TSS removal. Mixing speed (X_4) has a similar effect of X_2 on TSS removal, where the effects of both factors are presented in Fig. 4b.

The obtained results were compatible with previous studies, where Mahmudabadi et al. [19] displayed that TSS removal has achieved 97% with a PAC dosage of

100 mg Al/L, and improved to 99% at the sample processed by PAC dosage of 200 mg Al/L.

The TSS removal model was significant with a *P*-value of 0.0002. PAC dosage (X_2) and mixing speed (X_4) factors were effected the TSS removal efficiency directly according to the analysis of ANOVA with a *P*-values estimated by 0.0001 and 0.0008 respectively. Furthermore, PAC dosage (X_2) has a quadratic effect on TSS removal by 0.0012. The factors of pH (X_1) and mixing time (X_3) was not affected by the removal efficiency of the TSS.

3.3. Phytotoxicity evaluation

The treated samples were subjected to the GI tested to evaluate the phototoxicity as reported in Table 3. Phytotoxicity concentration differed with different samples where the alkaline (pH = 13) and acidic (pH = 1) samples were recorded a high concentration of the toxic estimated by 100.00% which consider invalid to reused for human purposes where according to Jagadabhi et al. [17] classified sample with a toxicity concentration < 50% as a typical toxic sample. While the water samples with a toxicity concentration from 60% to 100% were considered as highly toxic samples.

These results revealed that raw water was showed a high toxin level which estimated by 92%. However, the level of toxic was decreased after the treatment where the phytotoxicity concentration of the optimal run was 43.24% which is considered as valid water for irrigation.

According to the obtained results, the phytotoxicity concentration was effected by all factors, where the ANOVA analysis was displayed that the phytotoxicity concentration has a linear effect by pH value (X_1) and PAC dosage (X_2) with *P*-value of 0.0127 and 0.0027 respectively. In addition to that, the pH value factor (X_1) has a quadratic effect on phytotoxicity with a *P*-value of 0.0016. Moreover, there was an interaction effect between the mixing time (X_3) and mixing speed (X_4) factors which effected the phytotoxicity concentration with a *P*-value of 0.0450. Figs. 5a and b explain the effects of each factor on the phytotoxicity and the relationship between interaction factors. The model of phytotoxicity response was significant with a *P*-value of 0.0064.

3.4. Validation of the optimal parameters

The optimal run was provided by the Design-Expert software 11.1.2.0 from the "point prediction" option where the software in this option suggested an additional run to be tested for ensuring the results of the conducted runs. The values of independent factors X_1 (pH value), X_2 (Iron chloride dosage, g/L), X_3 (Mixing time, min), X_4 (Mixing speed, rpm) given by the software were 6.80, 295, 11 and

Table 4
ANOVA analysis for optimization response surface quadratic model of color, COD, turbidity and TSS removal

Source model	*P*-value				
	y_1	y_2	y_3	y_4	y_5
Model	0.0418	0.0452	0.0003	0.0002	0.0064
X_1	0.8784	0.5408	0.9967	0.9468	0.0127
X_2	0.7905	0.4628	<0.0001	<0.0001	0.0027
X_3	0.4606	0.4971	0.1680	0.2782	0.3993
X_4	0.6092	0.5900	0.0035	0.0008	0.2508
X_1X_2	0.9149	0.4273	0.9009	0.8548	0.3830
X_1X_3	0.9691	0.9720	0.8199	0.9795	0.2437
X_1X_4	0.9917	0.7838	0.9560	0.2466	0.3767
X_2X_3	0.9069	0.5133	0.8134	0.7271	0.3879
X_2X_4	0.9330	0.9509	0.5893	0.2601	0.1012
X_3X_4	0.8755	0.5164	0.6905	0.5936	0.0450
X_1^2	0.0661	0.0107	0.2467	0.6409	0.0016
X_2^2	<0.0001	0.0003	0.0025	0.0012	0.2140
X_3^2	0.2364	0.8754	0.9724	0.7088	0.3063
X_4^2	0.2391	0.4993	0.4177	0.8019	0.6651

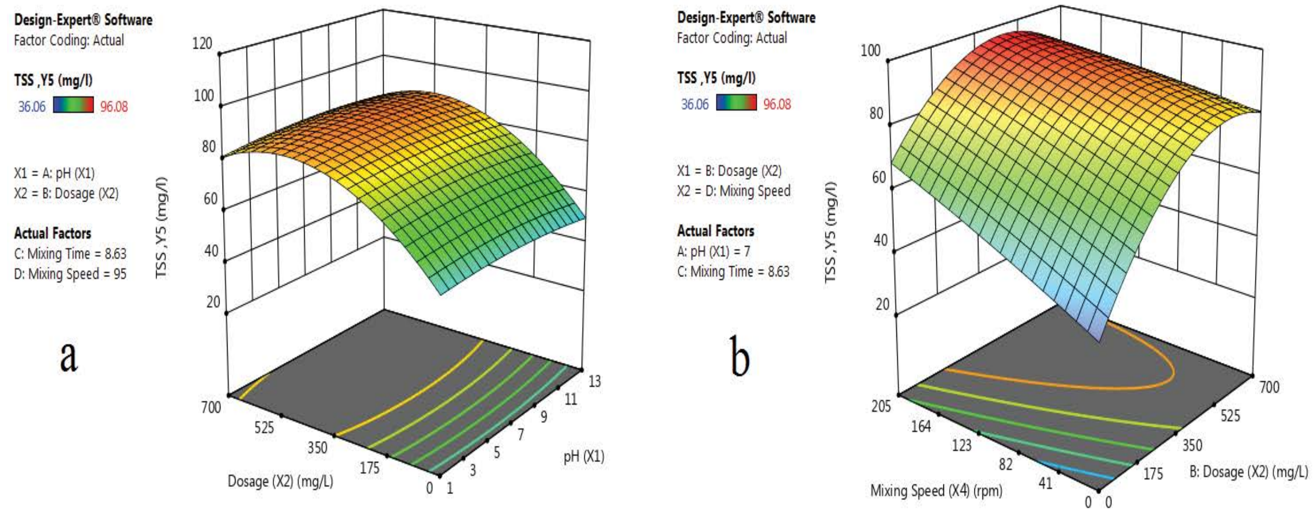

Fig. 4. Design-Expert plot, a 3D surface graph showing the effect of affected factors on TSS removal.

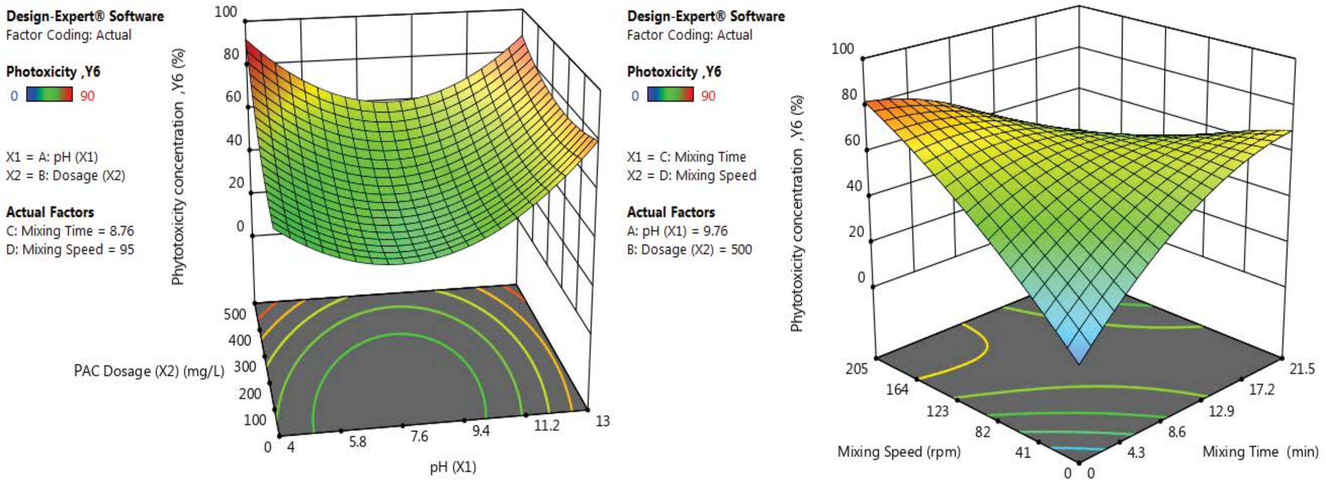

Fig. 5. Design-Expert plot, a 3D surface graph showing the effect of affected factors on the phytotoxicity concentration.

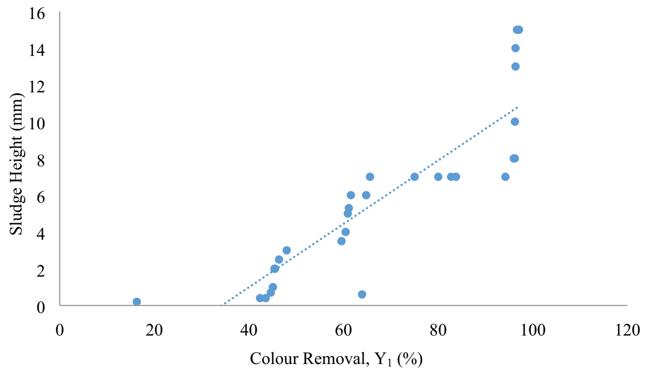

Fig. 6. Effects of color removal on sludge height.

108 respectively (Table 6). The optimal parameters designed by Design Expert 11.1.2.0 showed that a convergence between obtained and predicted values (Table 6) which support the results of performed runs. Color removal ratio (y_1), COD (y_2), turbidity removal (y_3), TSS removal (y_4), and the phytotoxicity concentration (y_5), were compatible to the predicted value given by Design-Expert software which indicates that the analytical of the software was successful with a standard error of ±5%.

3.5. Affecting of color removal on sludge height

The sludge height was compared with the color removal response where the obtained results showed that the sludge height increase with the increase of color removal ratio due to the high precipitation of the inks particles at the bottom, as stated by Govindan et al. [23]. Furthermore, Fig. 6 summarized and clarify the obtain results of the 30 experimental runs which indicate the relationship between color removal and sludge height with an R^2 value of 0.78 (Table 5).

Table 5
Effects of color removal response on sludge height

Run	Color removal ratio (y_2)	Sludge height (mm)
1	96.35	14
2	43.53	0.4
3	75.02	7
4	16.29	0.2
5	44.61	0.7
6	96.35	13
7	82.73	7
8	45.06	1
9	45.51	2
10	97.08	15
11	59.56	3.5
12	96.65	15
13	46.35	2.5
14	61.11	5.3
15	63.91	0.6
16	47.98	3
17	45.39	2
18	95.98	8
19	45.43	2
20	83.76	7
21	96.22	10
22	94.21	7
23	60.43	4
24	42.34	0.4
25	65.59	7
26	80.02	7
27	60.89	5
28	61.54	6
29	96.19	8
30	64.78	6

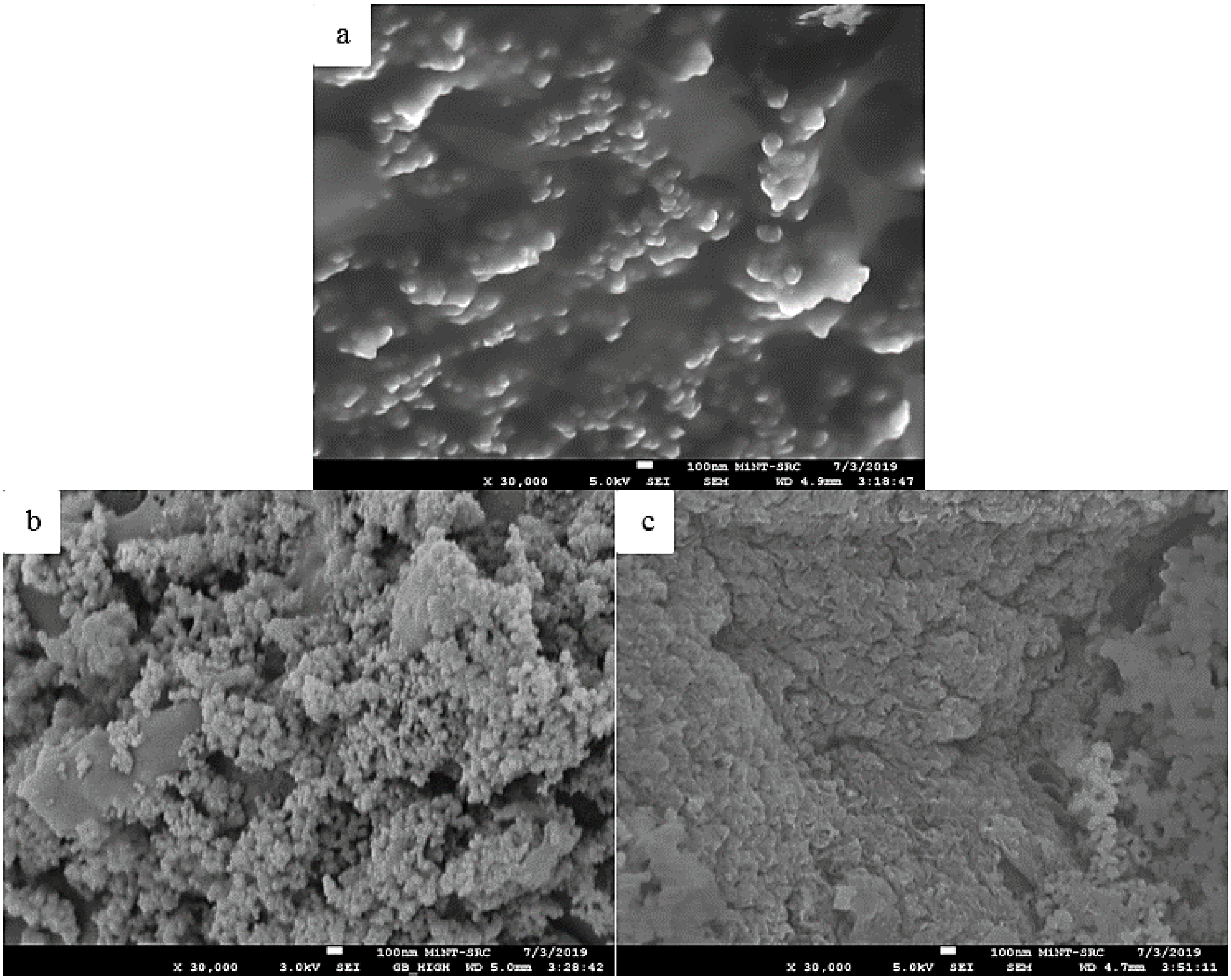

Fig. 7. PAC material (a), dyeing wastewater before the treatment (b) and dyeing wastewater after treatment using PAC (c).

Table 6
The best operating for color treatment using PAC

Responses	X_1	X_2	X_3	X_4	Experimental result	
					Tested	Predicted
y_1	6.80	295	11	110	98.53	95.00
y_2	6.80	295	11	110	71.02	70.83
y_3	6.80	295	11	110	85.89	84.83
y_4	6.80	295	11	110	88.01	85.54
y_5	6.80	295	11	110	43.24	43.17

y_1 (color removal %); y_2 (COD removal, %); y_3 (turbidity removal, %); y_4 (TSS removal); y_5 (phytotoxicity ratio, %); X_1 (pH value); X_2 (PAC dosage, mg/L); X_3 (mixing time, min); X_4 (mixing speed, rpm).

3.6. Coagulation and flocculation mechanism

Scanning electron microscopy technology was used to investigate the mechanism of color removal using the PAC. Three images were selected to investigate the removal mechanism, the first image was for PAC material (Fig. 7a), the second image was for the dyeing wastewater before the treatment (Fig. 7b), and the third image was for the dyeing wastewater after treatment using PAC (Fig. 7c). Fig. 7 shows the conversion of the dyes matters, where Fig. 7b shows the spread of dyes particles before the addition of PAC and the coagulation/flocculation processes. After the treatment of coagulation/flocculation processes with the addition of PAC, the dyeing particles were gathered together to become one plate as shown in Fig. 7c which led to sediment the particles at the end of treatment.

4. Conclusion

The independent factors exhibited high efficiency in the removal of color, COD, turbidity, and TSS by 98.53%, 71.02%, 85.89% and 88.01%, respectively in the optimal run under pH value of 6.80, PAC dosage of 295, mixing time of 11 min and mixing speed of 108 rpm. Phytotoxicity test of raw water was showed a high toxin level which estimated by 92%, while the level of toxic was decreased after treatment where the toxicity concentration of the optimal run was 43.24% which considered as a valid water for human uses.

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