Nature Environment and Pollution Technology
An International Quarterly Scientific Journal
p-ISSN: 0972-6268
e-ISSN: 2395-3454
Vol. 16 No. 2 pp. 591-597 2017

Original Research Paper
Open Access

Optimizing Advanced Oxidation Process for Industrial Textile Wastewater Treatment

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Nat. Env. & Poll. Tech.
Website: www.neptjournal.com

Received: 16-03-2016
Accepted: 16-04-2016

Key Words:
H_2O_2/pyridine/Cu(II)
Optimization
Industrial dye wastewater
Full factorial design
Central composite design.

ABSTRACT

The H_2O_2/pyridine/Cu(II) advanced oxidation system is a treatment method involving a combination of a transition metal ion with ligand molecules which decomposes H_2O_2 to produce hydroxyl radicals (OH*), which will then lead to the mineralization of organic compounds to produce CO_2 and H_2O. Most researches used this system on synthetic dye wastewater and reported removal in colour only. This study was carried out to assess and to optimize the treatment efficiency in terms of COD reduction and colour removal using this system in the treatment of industrial dye wastewater. Full factorial design and central composite design (CCD) from the response surface methodology (RSM) were utilized in the screening and optimization of the system. Results obtained indicated that this system was found to be capable in reducing the concentration of COD of the dye wastewater up to 80% and removing 94% of the colour at the optimal concentrations of 0.0023 M [H_2O_2], 0.0073 M [pyridine], 0.0123 M [Cu(II)] and at pH 10. Final COD concentration was recorded at 102 mg/L, and colour point at 55 PtCo. Amount of sludge produced at the end of this treatment system was 260 mg/L.

INTRODUCTION

Wastewaters generated from the textile industries and other industries which used dyes and pigments often contained high colour variations and appreciable quantities of organic compounds (Gomez et al. 2007, Lee et al. 2012, Lee et al. 2014, Lee et al. 2015). Other pollutants present in textile effluent include recalcitrant organics, toxicants and inhibitory compounds, surfactants, chlorinated compounds, highly alkaline pH, and salts with the main sources of water pollution coming from the dyeing, desizing, and scouring processes in a textile industry (Manu & Chaudhari 2002, Sena & Demirerb 2003). Each of this process offers its own series of environmental issues and they required the development of effluent treatment method to ensure protection to the environment and human health.

Among the treatment methods applied in the treatment of dye containing effluent are coagulation and flocculation, adsorption, membrane technology, chemical oxidation processes using Fenton reagent, ozone, UV plus H_2O_2 or NaOCl, aerobic biological treatment and anaerobic bioremediation each with their own advantages and disadvantages (Dhaouadi & Henni 2008, Won & Yun 2008). The flow chart of effluent treatment plant of a typical textile mill in Malaysia is outlined in Fig. 1. Recently, advanced oxidation processes (AOP) is gaining popularity in the treatment of refractory compounds in textile effluent especially Fenton oxidation. However, the Fenton process is limited by its acidic pH requirement (pH 2-4) and the high amount of sludge produced during the coagulation step (Dantas et al. 2006).

A modified classical Fenton process involving the combinations of copper and organic peroxides in the presence of pyridine, a fungal metabolite were found not only able to depolymerized lignin, but also able to decolourize wide ranges of synthetic dyes (Watabe et al. 1998, Nerud et al. 2001). Based on this information, this study was aimed to evaluate the efficacy of this system in the treatment of a real textile effluent, more specifically in the reduction of chemical oxygen demand (COD) concentration, colour removal, and the amount of sludge produced at the end of the treatment process.

MATERIALS AND METHODS

Materials: Industrial textile effluent was collected from the equalization pond of a textile plant. Effluent characteristics are provided in Table 1. The permissible discharge limit for COD and BOD of effluent in Malaysia is 100 mg/L and 50 mg/L, respectively. H_2O_2 (35%) was obtained from R&M chemicals, pyridine from Merck, and $CuSO_4.5H_2O$, NaOH and HCl were obtained from Systerm ChemAR. All chemicals were of analytical reagent grade. Distilled water was used in the preparation of all stock solutions.

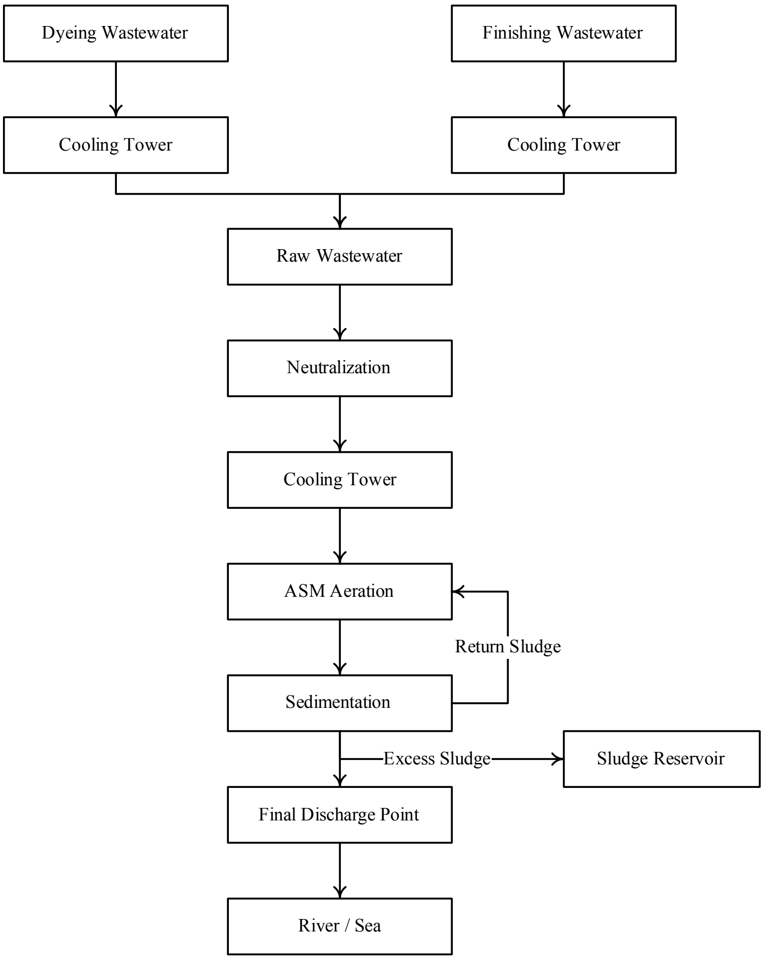

Fig. 1: Flow chart of effluent treatment plant of a typical textile mill in Malaysia.

Methods: All experiments were performed in a standard jar-test apparatus (Velp Scientifica JLT 6). Six 500 mL beakers each filled with 150 mL textile effluent were used in each run. The desired pH was adjusted using NaOH and HCl before the addition of H_2O_2, pyridine and $CuSO_4.5H_2O$. pH was measured using a pH meter (CyberScan 20).

Mixing time and mixing speed were fixed at 6 minutes and 60 rpm, respectively. Reaction time was kept constant at 60 minutes and all samples were filtered using Sartorius filter paper (pore size 20-25 µm). Filtrates were used in the measurement of COD and colour point.

COD, colour point, and sludge production was determined according to procedures in Standard Methods, method no. 5220D, 2120C, and 2540D, respectively (APHA et al. 2005). Results for colour point were expressed in Platinum-Cobalt (PtCo), the unit of colour being produced by 1 mg platinum L^{-1} in the form of the chloroplatinate ion. Reduction and removal efficiency of COD and colour were obtained using the following equation:

$$\% \text{ Removal} = \frac{C_i - C_f}{C_i} \times 100 \quad ...(1)$$

Where C_i and C_f represent the initial and final concentrations of textile effluent, respectively.

Statistical Design of Experiment: ***2^k Full factorial design:*** This design was utilized to study the joint effect of several factors on a response. Without the use of this design, important interactions among factors may not be detected and thus, lead to misleading conclusions (Montgomery 2001).

Independent factors affecting this study include pH, $[H_2O_2]$, [pyridine], and [Cu(II)] while COD reduction was selected as the main response in the screening of factors.

Table 1: Characteristics of textile wastewater.

Parameters	Value
pH	11.5
Temperature (°C)	44.2
Colour Point (PtCo)	860
COD (mg/L)	510
BOD5 (mg/L)	51.4
TSS (mg/L)	22.5
Turbidity (NTU)	28.8
Conductivity (μS/cm)	5700

Table 2: Input factors of H_2O_2/pyridine/Cu(II) System and experimental ranges and levels of 2^4 factorial design.

Experiment	Experimental Factors			
	ApH	BH_2O_2 (M)	Cpyridine (M)	DCu(II) (M)
1	3	0.005	0.008	0.015
2	3	0.005	0.010	0.013
3	3	0.003	0.008	0.015
4	3	0.003	0.008	0.013
5	9	0.005	0.008	0.013
6	6	0.004	0.009	0.014
7	3	0.005	0.010	0.015
8	9	0.003	0.008	0.013
9	6	0.004	0.009	0.014
10	3	0.003	0.008	0.013
11	9	0.003	0.010	0.015
12	9	0.005	0.010	0.015
13	3	0.003	0.010	0.013
14	9	0.003	0.010	0.013
15	9	0.005	0.008	0.015
16	9	0.003	0.008	0.015
17	9	0.005	0.010	0.015
18		0.005	0.008	0.013

Factors	Levels	
	- (Low Level)	+ (High Level)
A: (pH)	3	9
B: ([H_2O_2])	0.003	0.005
C: ([pyridine])	0.008	0.010
D: ([Cu (II)])	0.013	0.015

COD was chosen as the main response since the concentration of COD can serve as an indication of the concentration of all organic compounds that could be oxidized by strong oxidizing agents and therefore, could provide an idea of the level of pollution of the effluent (Chakinala et al. 2008).

Central composite design, response surface method: Response surface methodology, RSM is useful in modelling and analysis of problems in which a response of interest is influenced by several factors and the objective is to optimize the response. The first step in RSM is to find a suitable approximation for the true functional relationship between the yield of the process, *y* and the set of independent factors either by the first order model or the second order model. When a curvature is detected, the second order model was required to approximate the response (Montgomery 2001):

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i=1, i<j}^{k-1} \sum_{j=2}^{k} \beta_{ij} x_i x_j \quad ...(2)$$

Where, Y is the predicted response, β_O the offset term, β_i the linear effect, β_{ii} the squared effect and β_{ij} represents the interaction effect.

After performing the screening of factors with the factorial design, a response surface analysis was employed to optimize the highest COD reduction and colour removal of textile effluent. Coded variables were converted to natural variables according to the following relationship (Montgomery 2001):

$$x_k = \frac{\xi_k - x_0}{\delta x} \quad ...(3)$$

Where, x_k is the coded value of the k^{th} independent variable, ζ_k the natural variable of the k^{th} independent variable, x_O the natural value of the k^{th} independent variable at the centre point, and δx is the value of step change.

RESULTS AND DISCUSSION

Screening of factors: Effect of factors and their interactions were determined by performing the experiment according to Table 2 in triplicate. Factor levels were coded as – (low level) and + (high level). Low level represents the lowest concentration of chemicals used and high level, the highest concentration used (Tanja et al. 2003).

The normal probability plot of effects is presented in Fig. 2 to evaluate the statistical significance of the effect. The blue line serves as an indication of where the individual and interactions among effect points were expected to fall if they were not significant and significant effects were located farther away from the line (Montgomery 2001). Apart from identifying significant effects, this plot also reveals the direction of the effect. Positive effects are located to the right of the line and negative effect on the left. Positive effects mean that by increasing the level of the effects would result in the increase to the response while negative effects mean that by reducing the level of the effects would result in the increase to the response (Montgomery 2001).

From Fig. 2, the most significant effects were A (pH), followed by interaction between ACD (pH, pyridine, Cu (II)), individual effect of B (H_2O_2), and interaction between ABC (pH, H_2O_2, pyridine). Fig. 2 also indicates positive direction of effect A (pH) which means that higher pH lead to higher percentage COD reduction. This result was in contrast with research results obtained by researchers working

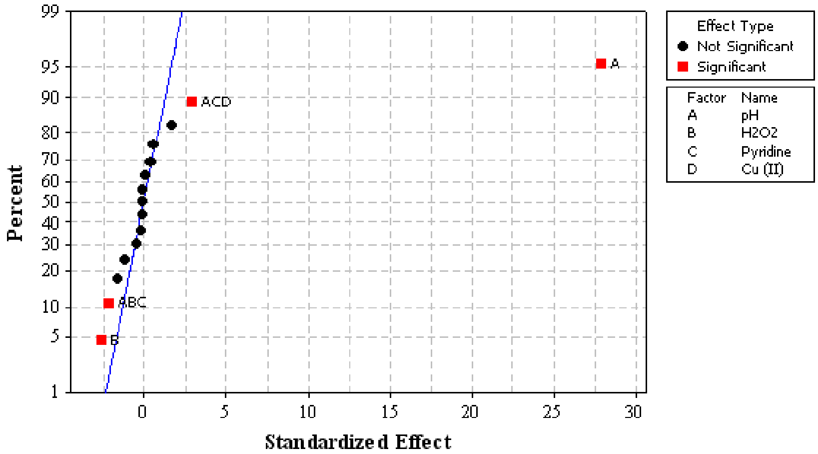

Fig. 2: Normal probability plot of effects.

on the decolourization of various synthetic dyes which found that this system was unaffected by pH within the range 3-9 (Nerud et al. 2001, Bali & Karagözoglu 2007a, Bali & Karagözoglu 2007b).

The reason could be due to the composition of the dye wastewater used in the study. pH was found unimportant by researchers working on synthetic dye wastewater while in this study, real textile wastewater was used. Real effluent contained various compositions which could influence the screening result of the study as the behaviour of this oxidation system could be affected by the structure of dye as well as the presence of other compounds in relation to pH (Guimarães et al. 2008).

Response optimization: Central composite design (CCD), a response surface method was used to obtain and to optimize the relationship between factors and response. A full 2^4 factorial design plus 4 center points in cube, 8 axial points and 2 center points in axial as presented in Table 3 was carried out in three replicates and six blocks in order to fit the second-order polynomial model allowing the estimation of the linear, quadratic and interactive effects of this system on the percentage of COD reduction and colour removal.

The quadratic regression model for the percentage COD reduction and colour removal are given by Eq. 4 and Eq. 5 respectively:

COD reduction (%):

$$Y = 64.69 + 9.95x_1 - 0.65x_2 + 0.12x_3 - 0.09x_4$$
$$-4.32x_1^2 + 0.79x_2^2 + 0.87x_3^2 + 1.28x_4^2$$
$$+0.18x_1x_2 - 0.26x_1x_3 - 0.55x_1x_4 + 0.20x_2x_3$$
$$-0.26x_2x_4 - 0.05x_3x_4 \quad ...(4)$$

Colour removal (%):

$$Y = 89.66 + 2.33x_1 + 0.90x_2 + 0.93x_3 + 0.59x_4$$
$$-3.53x_1^2 + 0.75x_2^2 + 0.81x_3^2 + 0.96x_4^2$$
$$-0.80x_1x_2 - 0.76x_1x_3 + 0.37x_1x_4 - 0.48x_2x_3$$
$$+0.49x_2x_4 - 0.05x_3x_4 \quad ...(5)$$

Where Y is the predicted response, x_1, x_2, x_3 and x_4 are the coded values of the respective treatment system factors; pH, H_2O_2, pyridine and Cu(II).

Effects of treatment system factors: The regression models (Eq. (4) and Eq. (5)) of factors on the treatment responses were represented graphically by contour plots as shown in Figs. 3-6. These plots show the responses of different operating parameters and thus, can be used in establishing desirable operating conditions (Bhatia et al. 2007). Contour plot in Fig. 3 shows the effect of [H_2O_2] and pH on percentage COD reduction. From the plot, COD reduction increased with pH and the highest COD reduction at 73% was found in the region [H_2O_2] of -1.64 to -1.55 and pH of 0.87 to 1.37 (coded level). Contour plot in Fig. 4 shows the effect of [Cu (II)] and pH on percentage COD reduction. It was found that high COD reduction, 74% was obtained at high pH level (from 0.78 to 1.64) and low [Cu(II)] level (from -1.65 to -1.41).

Based on Fig. 3 and Fig. 4, we conclude that higher pH is recommended for higher treatment efficiency in this system. At high pH, the probability of multi-azo groups being attacked by free radicals is much higher. Chu & Ma (1998)

Table 3: Input factors of H_2O_2/pyridine/Cu(II) System and experimental ranges and levels of process variables for % COD reduction.

Experiment	Experimental Factors			
	A	B (M)	C (M)	D (M)
1	3	0.0030	0.0100	0.0130
2	6	0.0040	0.0090	0.0140
3	3	0.0030	0.0080	0.0150
4	9	0.0030	0.0100	0.0150
5	3	0.0050	0.0080	0.0130
6	9	0.0050	0.0080	0.0150
7	3	0.0050	0.0100	0.0130
8	3	0.0030	0.0100	0.0150
9	3	0.0050	0.0080	0.0150
10	9	0.0030	0.0080	0.0130
11	3	0.0030	0.0080	0.0130
12	3	0.0050	0.0100	0.0150
13	9	0.0030	0.0100	0.0130
14	6	0.0040	0.0090	0.0140
15	9	0.0030	0.0080	0.0150
16	6	0.0040	0.0090	0.0140
17	6	0.0040	0.0090	0.0140
18	9	0.0050	0.0100	0.0150
19	9	0.0050	0.0073	0.0130
20	9	0.0050	0.0100	0.0130
21	6	0.0040	0.0090	0.0140
22	1	0.0040	0.0090	0.0140
23	11	0.0040	0.0090	0.0140
24	6	0.0040	0.0090	0.0123
25	6	0.0040	0.0107	0.0140
26	6	0.0040	0.0073	0.0140
27	6	0.0057	0.0090	0.0140
28	6	0.0040	0.0090	0.0157
29	6	0.0040	0.0090	0.0140
30	3	0.0023	0.0090	0.0140

Factors	Levels				
	$-\alpha$	-1	0	+1	$+\alpha$
A: pH	1	3	6	9	11
B: $[H_2O_2]$	0.0023	0.0030	0.0040	0.0050	0.0057
C: pyridine]	0.0073	0.0080	0.0090	0.0100	0.0107
D: [Cu (II)]	0.0123	0.0130	0.0140	0.0150	0.0157

suggested 2 mechanisms in which the decolourization of polyazo dyes at low pH was incomplete:

I Since the amount of free radicals in solution were insufficient for the simultaneous attack of the multi-azo bond of the dye molecule.

II At low pH, the interference of H^+ towards the conjugated system of polyazo dye forming positively charged central amine decreased the reactivity of electrophilic free-radical mechanism as shown in Eq. 6:

$$\ddot{N} = \ddot{N} \xrightarrow{H^+} \overset{\overset{H}{\downarrow}}{N^+} - \ddot{N} \quad ...(6)$$

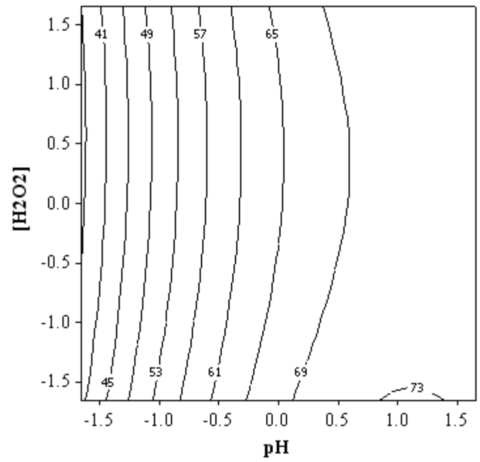

Fig. 3: Contour plot of % COD reduction versus $[H_2O_2]$ and pH.

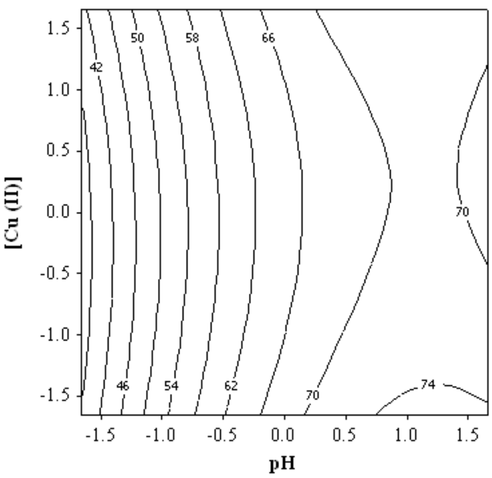

Fig. 4: Contour plot of % COD reduction versus [Cu(II)] and pH.

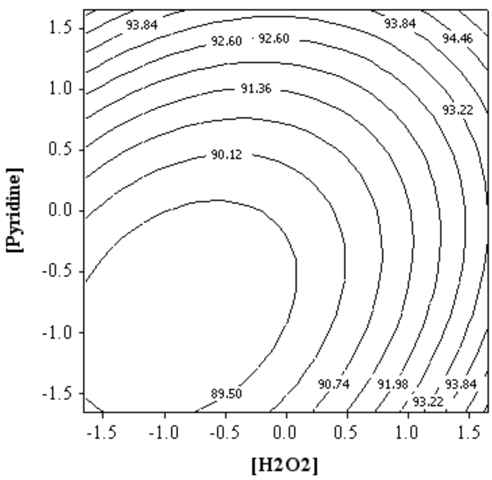

Fig. 5: Contour plot of % Colour removal versus [pyridine] and $[H_2O_2]$.

Table 4: Values from general solutions of process variables.

Run	pH	$[H_2O_2]$ (M)	[Pyridine] (M)	[Cu(II)] (M)	COD reduction (%)		Colour removal (%)		Sludge Produced (mg/L)
					Experimental	Predicted	Experimental	Predicted	
1	1	0.00	0.0073	0.012	79.71	80.46	93.58	92.30	260
2	0	23	0.0073	3	76.78	79.86	94.57	90.01	435
3	1	0.00	0.0107	0.012	77.41	80.00	93.21	90.65	910
4	1	23	0.0073	3	75.10	79.72	93.46	93.90	360
5	9	0.00	0.0107	0.012	75.52	78.56	94.07	93.22	555
	9	57		3					
	1	0.00		0.015					
	0	23		7					
		0.00		0.012					
		23		3					

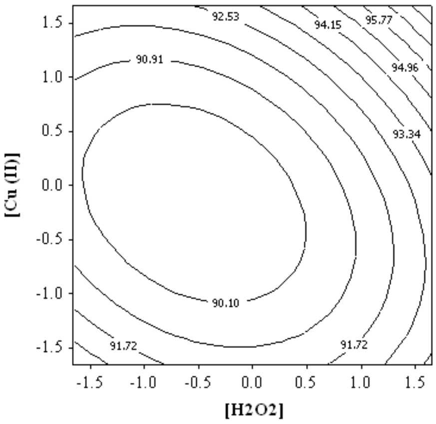

Fig. 6: Contour plot of % Colour removal versus [Cu(II)] and $[H_2O_2]$.

Contour plot in Fig. 5 shows the effect of [pyridine] and $[H_2O_2]$ on percentage colour removal. This plot studies the influence of $[H_2O_2]$, an oxidant and [pyridine], a ligand. In this figure, the regions which led to high colour removal, 95% were region where both the level of [pyridine] and $[H_2O_2]$ were high, region where the level of [pyridine] was high and $[H_2O_2]$ was low and region where the level of [pyridine] was low and $[H_2O_2]$ was high.

Oxidation of the aromatic substrates ensues along with decomposition of H_2O_2 in the production of OH* in the presence of pyridine in which the OH* is responsible in the decomposition of the dye (Kuznetsova et al. 2007). The enhancement of decolourization with an increase in $[H_2O_2]$ is because of the increase in OH* and H_2O_2 is unable to generate enough OH* at low concentration (Muruganandham & Swaminathan 2004). At high $[H_2O_2]$ however, instead of producing more OH*, these radicals react with the excessive H_2O_2 resulting in undesirable competition with the destruction of the chromophoric groups (Galindo & Kalt 1998). Therefore, it was important to have a proportionate amount of $[H_2O_2]$ and [pyridine] in order to obtain desirable treatment efficiencies.

Fig. 6 shows a contour plot of percentage colour removal versus [Cu(II)] and $[H_2O_2]$. High colour removal (97%) was observed at the high level region of $[H_2O_2]$ (1.48 to 1.65) and [Cu(II)] (1.49 to 1.65). Even at low level region of $[H_2O_2]$ (-1.65 to -1.35) and [Cu(II)] (-1.65 to -1.45), 93% colour removal was recorded. Though decolourization of textile wastewater using this system was high (>90%), the main aim of this present work is to maximize percentage COD reduction. Therefore, the optimal operating conditions were selected based on combinations which give the highest COD reduction.

Optimization analysis: An additional 5 experiments were carried out as shown in Table 4 by using the proposed numerical optimization solution generated by the Minitab software in order to determine the optimum condition for this system. Comparison between actual experimental results and predicted results from the regression model was made. Operating conditions which give the highest percentage of COD reduction was selected as the optimal conditions. Based on results tabulated in Table 4, the optimal operating conditions were determined at pH 10, 0.0023 M $[H_2O_2]$, 0.0073 M [pyridine], and 0.0123 M [Cu(II)] in which 80% COD reduction and 94% colour removal was achieved with 260 mg/L amount of sludge produced at the end of the treatment process.

CONCLUSIONS

Experimental design was used to evaluate the efficacy of the H_2O_2/pyridine/Cu(II) system in treating industrial dye wastewater and obtained the optimum operating conditions of the process variables; pH, $[H_2O_2]$, [pyridine], and [Cu(II)] on the percentage of COD reduction and colour removal. In the selection of the optimal operating conditions, we should also consider the economy feasibility apart from looking at the treatment efficiencies. In this study, we select the conditions which used the lowest concentration of materials and at the same time yield high COD reduction and colour removal and produced low amount of sludge at the end of the treatment. Therefore, the optimal conditions obtained from this study were pH 10, 0.0023 M [H_2O_2], 0.0073 M [pyridine], and 0.0123 M [Cu(II)]. At this condition, 80% COD reduction and 94% colour removal was reported with the final COD concentration at 102 mg/L, and final colour point at 55 PtCo. Amount of sludge produced at the end of this treatment system was 260 mg/L.

ACKNOWLEDGEMENT

The authors would like to acknowledge the financial support provided by Universiti Kebangsaan Malaysia through Geran Universiti Penyelidikan (GUP-2014-034) and Dana Impak Perdana (DIP-2015-008).

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