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Assay of p-Chlorophenol Compliance Monitoring in Textile Wet Processing Industry Effluent Using Fenton Oxidation Process

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ABSTRACT

The textile industries utilize number of dyes, chemicals, and other materials to suffuse the characteristic of fabric qualities. A huge quantity of effluents is produced during the process. However, toxicity from synthetic dyes has become a cause of severe environment concern. Chlorophenols are mostly present in synthetic dyes which are proven carcinogenic and therefore undesirable. A number of techniques were used to remove p-Chlorophenol up to the ZDHC MRSL limit. However, none of them found to be up to mark. Fenton oxidation process was selected for its suitability to degrade the p-Chlorophenol up to 5 ppm or less from the textile wet processing industry effluent. In the present study cotton fiber was selected, as medium considering its common use in textile industry. The impact of Ferrous ion (Fe^{+2}), Hydrogen peroxide (H_2O_2) and pH on the removal of p-Chlorophenol was examined. The Box-Behnken Design (BBD) of (RSM) was employed to achieve optimum desirable condition for the removal of p-Chlorophenol from effluent. A quadratic model is suggested to relate the independent variables for maximum removal of p-Chlorophenol at the optimal process condition. Results suggest that removal efficiency under the optimum condition $[Fe^{+2}] = 6.5\times10^{-3}$ M, $[H_2O_2] = 2.9\times10^{-2}$ M, and [pH] = 3.5 was >90% in 15 minutes. It can be summarized that Fenton oxidation process as the promising potential for removal of p-Chlorophenol from textile wet processing industry effluent. This research work helps to address for the general knowledge gap in the textile wet processing industry effluent treatment and provide a plate form for further research.

Keywords: p-chlorophenol, textile wet process, fenton oxidation process, cotton fiber, BBD

INTRODUCTION

Due to the fast track progress regarding the worldwide economy, the productivity of textile wet processing industry has revealed tremendous development at the same time poses sever environmental issues (Cai et al., 2019b, 2019a; Chen et al., 2015; Zhou et al., 2019). Textile wet processing industry is one of the oldest industries occupies an important place in terms of foreign exchange earnings and providing employment resources globally. However, on other hand this industry is responsible in contributing an enormous amount of industrial waste water which has a huge concentration of pollutants, including organic dyes, dispersing agents, surfactants, detergents, organic solvents, heavy metal content and intractable mixtures (Vanhulle et al., 2008; Wang et al., 2019) responsible in detreating the environment containing the persistent organic pollutants in effluent, such as chlorophenols, require additional attention due to their hazardous ecological profile on the environment and human health. Chlorophenols has an easy oxidation characteristic inheriting property in their chemical structures and are multipurpose intermediates in synthesis of different chemical (Jing et al., 2023).

Considering the health effect of Chlorophenols not only have direct toxicological impact but also pose the potential impact as carcinogen, teratogen and mutagen (Jing et al., 2023). Moreover, the toxicological impacts of Chlorophenols in the environment are much prominent apart from phenolic compounds catalogued as priority organic contaminants in the European Union, United States and China. Lower concentration of Chlorophenols at ppb level can also cause harm to the environment (Wei et al., 2019). Owing to the adverse impact of Chlorophenols, three type of Chlorophenols, mono, di and tri Chlorophenols have been listed as priority control persistent organic pollutants by the United States Environmental Protection Agency (US EPA) and European Chemical Agency (ECHA) (Nguyen et al., 2020).

Several classical methods such as guar gum, activated charcoal etc. have been suggested for the removal of chlorophenols from different industrial wastewater. However, none of them proved successful as per globally sustainable standards (Zero Discharge of Hazardous Chemicals Manufacturing Restricted Substances List (ZDHC MRSL)) (Aruoja et al., 2011; Faludi et al., 2015; Igbinosa et al., 2013; Kuch et al., n.d.; Nguyen et al., 2020; Olaniran and Igbinosa, 2011; Patra and Pariti, 2022; Pera-Titus et al., 2004). Additionally, biodegradation of phenols has also been reported to remove chlorophenol by Fenton Oxidation. However, Fenton is one of the well-known effluent treatment processes. When aqueous ferrous ion react with hydrogen peroxide produces hydroxyl radical (▪OH) which can degrade persistent and hazardous organic pollutant in effluent (Dąbrowski et al., 2005).

Presently, Fenton has become a prevalent process to degrade the calcitrant organic pollutants from textile wet processing industry effluent. Electro Fenton and Photo Fenton become more effective for color removal and mineralizing textile wet processing industry effluent when it is compared with classical Fenton method (Soto et al., 2011).

Number of advantages that makes Classical Fenton process makes more efficient by photo radiation with external sources including Solar and UV light (Patra and Pariti, 2022), no exterior energy sources is required and make this process low cost that help the biodegradation of complex organic based textile wet processing industry effluent (Kuch et al., n.d.), Fenton reaction take place at room temperature and normal atmospheric pressure that can be adapted in normal environmental conditions (Lucas et al., 2007). Textile wet processing industry effluent is in alkaline range of pH and reaction is take place in acidic pH range between 2 to 4 that makes limitation of Fenton process (Titchou et al., 2021).

It was frequently used to treat effluent by oxidation and flocculation. Ferrous ions catalysis hydrogen peroxide into hydroxyl radical (▪OH) and to generate the other radical production, which oxides organic matter completely. standard electrode potential of hydroxyl radical (▪OH) is 2.80 V which possess high oxidization capacity (Brillas, 2020). Hydroxyl radical (▪OH) is oxidized refractory organic pollutants effectively in effluent and decompose into CO_2, inorganic salt and water (Zhang et al., 2021).

Fenton mechanism is shown as represented by following equations (Zhen et al., 2017):

$$Fe^{+2} + H_2O_2 \rightarrow Fe^{+3} + \blacksquare OH + OH^- \quad (1)$$

$$\blacksquare OH + H_2O_2 \rightarrow Fe^{+3} + HO\blacksquare_2 + H_2O \quad (2)$$

$$Fe^{+2} + \blacksquare OH \rightarrow Fe^{+3} + OH^- \quad (3)$$

$$Fe^{+3} + HO\blacksquare_2 \rightarrow Fe^{+2} + O_2 + H^+ \quad (4)$$

$$\blacksquare OH + \blacksquare OH \rightarrow H_2O_2 \quad (5)$$

The oxidation activity of hydroxyl radical (▪OH) generated by Fenton process, usually operated under the solution of pH 3.5 this process removed the chlorophenol from textile wet processing industry effluent in desirable limit. This research work has been carried out to investigate the removal of p-Chlorophenol compound and derivatives from textile wet processing industry effluent by Fenton oxidation process to obtain desirable results as per guideline of ZDHC MRSL sustainable standard for textile wet processing industry. The existing literature is silent about this study.

Response surface methodology (RSM) can be used to optimize the process variables and maximize the efficiency of p-Chlorophenol removal by systematically varying the input variables and measuring the response variable (Kermet-Said and Moulai-Mostefa, 2022; Lacson et al., 2022; Masouleh et al., 2022; Zahmatkesh et al., 2022). box-behnken design (BBD) is a statistical experimental design method used to explore the relationship between multiple variables and their effect on a response (Dbik et al., 2022; Kavitha et al., 2022). It is a type of response surface methodology that uses a series of designed experiments to determine the optimum values of process variables that lead to the best response. The mathematical expression for BBD can be represented as follows:

$$Y = \beta0 + \beta1X1 + \beta2X2 + \beta3X3 + \beta12X1X2 + \beta13X1X3 + \beta23X2X3 + \beta11X1\text{^}2 + \beta22X2\text{^}2 + \beta33X3\text{^}2 + \varepsilon$$

where: Y – response variable (such as p-Chlorophenol removal); β0, β1, β2, β3, β12, β13, β23, β11, β22, β33 = regression coefficients; X1, X2, X3 – coded independent variables (representing low, intermediate, and high levels of the variable); ε – error term.

The three-level factorial design involves three values for each independent variable (-1, 0, and 1) and uses a balanced number of experiments at each level. By systematically varying the values of the independent variables and measuring the response variable, a quadratic model can be developed to determine the optimal values of the independent variables that lead to the best response.

The main goal of BBD is to reduce the number of experiments required to determine the optimal process variables and to identify any curvature in the response surface (Naseer et al., 2021; Zaidi et al., 2023). The regression coefficients in the above equation can be estimated using a least squares method or other regression techniques. The resulting quadratic model can be used to predict the response variable for any combination of the independent variables, allowing for the identification of the optimal process conditions.

This technique has been applied for the determination of a number of pollutant removal efficiency as a single or multiple parameters in different industrial waste water. However, literature is silent for the determination of chlorophenol removal efficiency though this technique. Therefore, this study has been carried out to find out chlorophenol removal efficiency through surface response methodology by using BBD.

MATERIALS AND METHODS

Sampling

The experimental samples were taken from discharge point of a Textile Wet Processing Industry, which is located in Korangi Industrial Area Karachi, Pakistan. Samples were collected from May to June 2022. To avoid light influence, all samples were collected from 12 inches depth and kept in plastic sealed bottles which were prewashed with deionized water. These samples were stored at 4.0 °C in a refrigerator until analysis.

Fenton conditioning experiments

Specific amount of effluent was treated with Hydrogen peroxide (H_2O_2) and Ferrous Sulphate ($FeSO_4$) with dosage of 490–1100 and 500–1000 respectively mg/l mix thoroughly using magnetic Stairer up to 180 minutes sample were analyzed after 15 minutes interval. pH value of textile wet processing industry effluent was maintain 3.5 by using 1.0 N Sulphuric acid (H_2SO_4) and 0.5 N sodium hydroxide (NaOH) if required.

Standards and chemicals

A solution of 100 mg/l in methylene chloride of 19 Chlorophenols (mono through penta) was used as a standard solution in this research study. Potassium Carbonate (K_2CO_3) analytical grade supplied by Daejung was used in the experiments. Hexane HPLC grade supplied by Fisher Chemicals and Acetic Anhydride used of commercial grade because authorities have posed bane on it due to this reason analytical grade is not available. A 99.5% Triethylamine of HPLC Grade is used in the experiment. In each set of the experiment Potassium carbonate (K_2CO_3) solutions were prepared. The solution (0.1 M) was hold in 500 ml. All the solutions were prepared in distilled water. Ferrous sulphate ($FeSO_4{\cdot}7H_2O$) and Hydrogen Peroxide (H_2O_2) of analytical grades from LabChem are used to treat the effluent samples in diluted with distilled water.

Sample preparation

All effluent samples are prepared according to following sequence: Take 1.0 g of treated effluent sample in 25 ml capacity vial and add 10 ml of 0.1 M of Potassium carbonate (K_2CO_3) solution in it that enhance the solubility. Sonicate the above sample at room temperature for 25 minutes. After sonication, add 5 ml of Hexane that help the extraction of chlorophenols from effluent sample. Add 4 ml of Acetic Anhydride that help the acetylation and 0.5 ml of Triethylamine to adjust the pH. Sealed the vial and shake the samples at Horizontal shaker at 160–170 RPM for 1 hour. Puncture the vial with the help of needle in order to remove CO_2 and take the upper layer of hexane with the help of 2.5 ml of syringe. Make the vial for the injection and analyze on GCMS of Shimadzu from Japan.

Measurement apparatus

In this study p-Chlorophenols measurements were done by using Shimadzu gas chromatograph-mass spectrometer (GCMS-Model QP2010 Ultra).

Analytical protocol

The analytical protocols followed in this research study of p-Chlorophenols analysis according to BS EN ISO 17070:2015 method by using Shimadzu gas chromatograph-mass spectrometer (GCMS-Model QP2010 Ultra).

Optimization of process parameters using RSM and experimental design

Response surface methodology was employed to optimize the reduction of p-Chlorophenol in textile wet processing industry effluent, treatment time 10–15 minutes and employing Fe^{+2}/H_2O_2 reaction. Design Expert Software Version-13.0 was used for this purpose. Design of experiments with a four-factor composite design was utilize and experiment performed to construct model. p-Chlorophenol removal was selected as response variable, while pH (A), $FeSO_4$ (B), H_2O_2 (C) and

Table 1. RSM independent variables

Independent variable	Parameter	Low	High
A	pH	2.0	9.0
B	$FeSO_4$ (mg/L)	500	1000
C	H_2O_2 (mg/L)	490	1100
D	p-Chlorophenol	0.03	5.05

Table 2. Experimental design for p-Chlorophenol

Std	Run	Factor A, pH	Factor B, $FeSO_4$	Factor C, H_2O_2	Factor D, p-CP
1	22	2	500	907.5	2.54
2	8	9	500	907.5	2.54
3	7	2	1000	907.5	2.54
4	23	9	1000	907.5	2.54
5	27	5.5	750	490	0.03
6	2	5.5	750	1325	0.03
7	17	5.5	750	490	5.05
8	3	5.5	750	1325	5.05
9	21	2	750	907.5	0.03
10	10	9	750	907.5	0.03
11	20	2	750	907.5	5.05
12	6	9	750	907.5	5.05
13	24	5.5	500	490	2.54
14	18	5.5	1000	490	2.54
15	19	5.5	500	1325	2.54
16	16	5.5	1000	1325	2.54
17	9	2	750	490	2.54
18	15	9	750	490	2.54
19	25	2	750	1325	2.54
20	5	9	750	1325	2.54
21	26	5.5	500	907.5	0.03
22	1	5.5	1000	907.5	0.03
23	11	5.5	500	907.5	5.05
24	13	5.5	1000	907.5	5.05
25	12	5.5	750	907.5	2.54
26	14	5.5	750	907.5	2.54
27	4	5.5	750	907.5	2.54

p-Chlorophenol (D) were acted as independent variable as shown in Table 1.

The corresponding experimental results and experimental design are represented in the Table 2 and the obtained results are for removal of p-Chlorophenol are after treatment.

RESULTS AND DISCUSSION

The hydroxyl radical (▪OH) is formed by the action of Hydrogen peroxide (H_2O_2) with Ferrous ion (Fe^{+2}) the reaction is as under:

$$Fe^{+2} + H_2O_2 \rightarrow Fe^{+3} + \cdot OH + OH^-$$

The Fenton oxidation reaction is well known reaction, having high oxidation potential value, however its restoration in implementation to textile wet processing industry start merely recently. The main task of complete degradation of pollutants to alter in less toxic final yield CO_2 and inorganic salts is significant for the development of efficient textile wet processing industry effluent treatment method. To achieve the desire concentration, various concentration of H_2O_2 and Fe^{+2} were used for this reaction including operating pH value.

Optimization of p-chlorophenol removal process parameters by RSM

Numbers of studies have employed using various experimental design techniques including response surface method (Feng et al., 2018; Zaidi et al., 2021). Response surface method is one of the powerful techniques to determine the optimization condition necessary to enhance the efficiency of experimental performance. Furthermore it will help towards less experimental trails, and this will lead to an outcome in the form of cost reduction, in term of reagents cost and process time (Alwan, 2012).

To determine the range for optimizing the H_2O_2 and $FeSO_4$ doses by using RSM. Prior experiments were performed using different doses of Fe^{+2} and H_2O_2 concentration to acquire optimum dose for efficient removal of p-Chlorophenol. For this purpose various concentration were used for of H_2O_2 (490–1100 mg/l) and $FeSO_4$ (500–1000 mg/l). A series of designed experiment were conducted with RSM and compared with observed results with expected values. Removal of p-Chlorophenol achieved from Fenton oxidation process using different dosages of $FeSO_4$ and H_2O_2.

In previous studies lack of fit (LOF) model was widely used for model validation. LOF indicates the deviation between the actual observed values and for the fitted surface (Yates et al., 2023). LOF model is used to validation in according with the established studies as indicated in Table 3. The LOF F-value of 1.66 implies the Lack of Fit is not significant relative to the pure error. There is a 43.46% chance that a LOF F-value this large could occur due to noise. Non-significant lack of fit is good – we want the model to fit.

Table 3. ANOVA for quadratic model of p-CP removal

Specification	Sum of squares	df	Mean square	F-value	p-value
Source model	888.75	14	63.48	17.06	< 0.0001
A-pH	2.03	1	2.03	0.5441	0.4749
B-$FeSO_4$	85.39	1	85.39	22.94	0.0004
C-H_2O_2	17.71	1	17.71	4.76	0.0498
D-CP	82.06	1	82.06	22.05	0.0005
AB	360.62	1	360.62	96.88	< 0.0001
AC	164.22	1	164.22	44.12	< 0.0001
AD	0.5476	1	0.5476	0.1471	0.7080
BC	53.80	1	53.80	14.45	0.0025
BD	1.77	1	1.77	0.4752	0.5037y
CD	19.98	1	19.98	5.37	0.0390
A^2	29.97	1	29.97	8.05	0.0150
B^2	9.29	1	9.29	2.50	0.1402
C^2	36.94	1	36.94	9.92	0.0084
D^2	17.94	1	17.94	4.82	0.0485
Residual	44.67	12	3.72		
Lack of fit	39.85	10	3.99	1.66	0.4346
Pure error	4.81	2	2.41		
Cor total	933.42	26			

High response is observed with H_2O_2 for the removal of p-Chlorophenol in Fenton oxidation process. It is concluded that $FeSO_4$ is required to activate the H_2O_2 to produce Hydroxy radicals (–OH). our study and reported results are in-close agreement with each other.

The determination of factor R^2 and adjusted R^2 were obtained as 0.9521 and 0.8963, subsequently. According to ANOVA results the P-value (Prob > F) is 0.0001, which shows the relationship between the free variables and the response factor that was examined in this process is significant and system is suitable. Due to other parameters in this process on the p-Chlorophenol removal, the term A^2 Prob > F value of ratio is 0.0150, meaning that A^2 had significant influence in the p-Chlorophenol removal. The term C^2 also has significant impact on p-Chlorophenol removal. Evaluating the P-value with A, B and C, additionally it is observed that the effect of these 4 parameters such as pH > H_2O_2 Dosage > $FeSO_4$ >p-Chlorophenol.

The values of R^2 and adjusted R^2 were 95% and 89% respectively as indicated in Table 4. This indicates the experiment data for p-Chlorophenol removal efficiency fitted with the predicted value of the model. For verification of R^2 value comparable data graph was plotted between the observed and predicted data presented in Figure 1.

Table 4. Fit statistics

Std. Dev.	1.93	R^2	0.9521
Mean	82.5	Adjusted R^2	0.8963
C V %	2.34	Predicted R^2	0.7425
		Adeq. precision	16.9158

The regression equation (eq^{-1}) in term of coded factors suits the experimental data of p-Chlorophenol removal:

$$CP_{Removal} = +80.12 + 0.4108 \cdot A - 2.67 \cdot B + 1.21 \cdot C + 2.62 \cdot D + 9.49 \cdot A \cdot B - 6.41 \cdot A \cdot C + 0.3700 \cdot A \cdot D - 3.67 \cdot B \cdot C + 0.6650 \cdot B \cdot D + 2.23 \cdot C \cdot D + 2.37 \cdot A^2 - 1.32 \cdot B^2 + 2.63 \cdot C^2 + 1.83 \cdot D^2$$

The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. By default, the high levels of the factors are coded as +1 and the low levels are coded as -1. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients. Outcome of response surface design in three-dimensional form response curves are mentioned in Figure 2.

CONCLUSIONS

This study was carried out to increase the p-Chlorophenol removal with advance oxidation process using hydrogen peroxide with Fenton oxidation process to remove p-Chlorophenol in textile wet processing industry effluent. As the worst case in term of p-Chlorophenol contamination we have selected cotton fiber and apply the Fenton Oxidation process on cotton dyeing industry effluent. The results of this study showed that the Fenton oxidation process was found the most suitable and efficient for the removal of p-Chlorophenol up to 5 ppm or less from textile wet processing industry effluent.

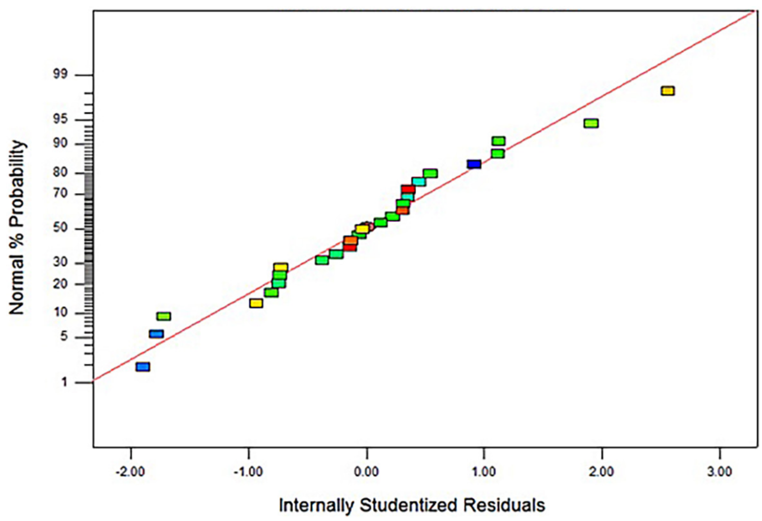

Fig. 1. Normal plot of residuals

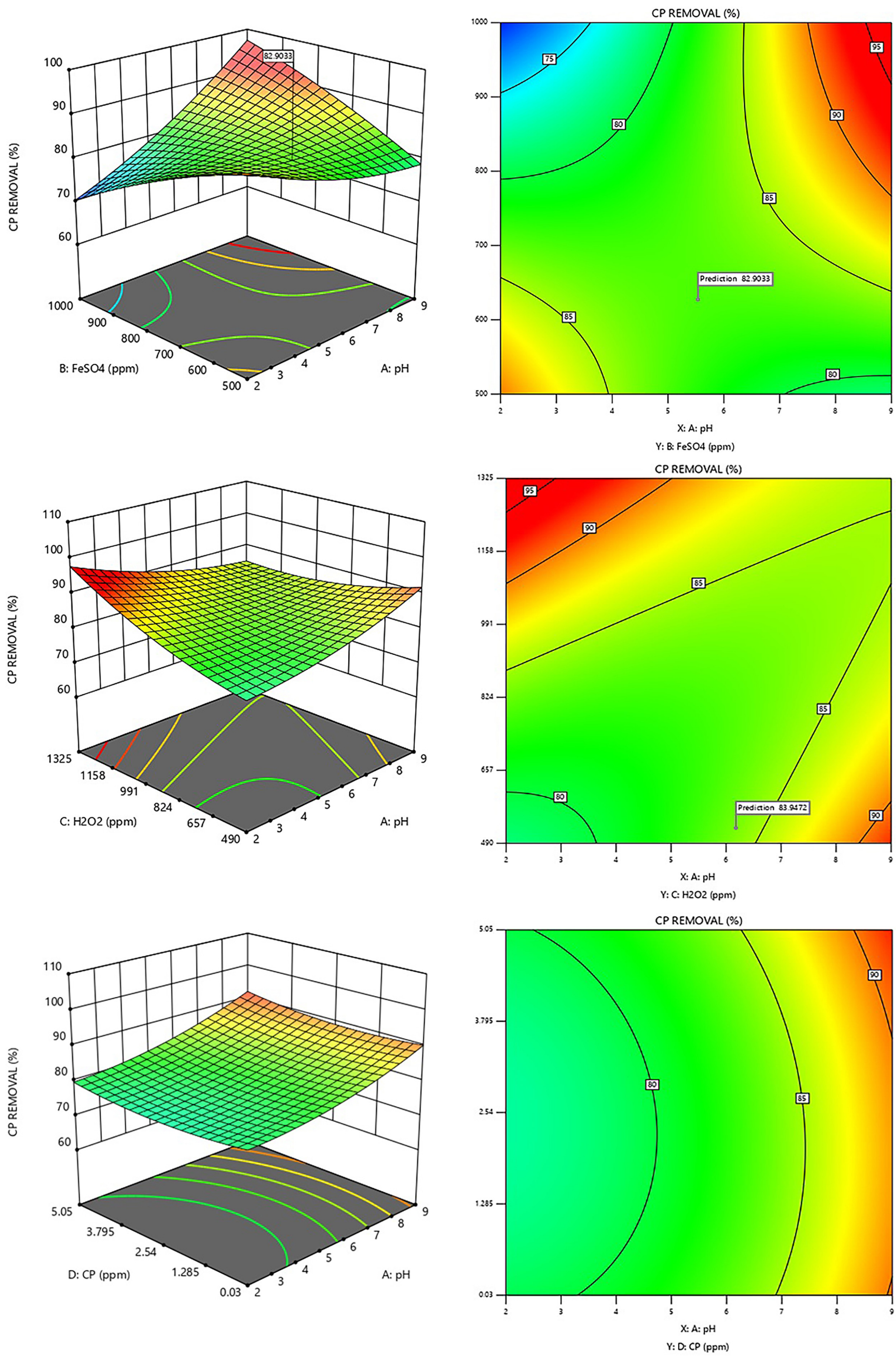

Fig. 2. Comparing the factor coefficients

However, this process can be carried out only in the pH range of 3 to 4 with doses of 1000 mg/l Hydrogen Peroxide (H_2O_2) and 1000 mg/l Fe^{+2} ions as Ferrous Sulphate ($FeSO_4$) with normal environmental conditions of room temperature and atmospheric pressure. It has been concluded that using hydrogen peroxide in Fenton oxidation process, assisted p-Chlorophenol removal efficiently up to ZDHC MRSL effluent requirements and color removal then in simple anaerobic digestion of the textile wet processing industry effluent. Based on obtained results, it is highly recommended to use the Fenton Oxidation process for treatment of textile wet processing industry effluent containing p-Chlorophenol and other complex organic pollutants. The suitability of response, P-value and F-value indicates that the analysis parameters are significant influence on the treatment process. Optimization of the process is likely by using BBD design of RSM variables and the model was well fitted. This technique can be advantageous for p-Chlorophenol removal from different industries effluent.

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