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# Optimization of Fenton Oxidation Process for Degradation of 1-Butyl-3 Methyl Imidazolium Chloride (BMIMCL) Using Response Surface Methodology

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## ABSTRACT

The degradation of 1-butyl 3-methyl imidazolium chloride (BMIMCl) ionic liquid (IL) by Fenton oxidation has been studied. The optimization of operating parameters for maximum degradation of BMIMCl has been carried out using the Central Composite Design (CCD) of Response Surface Methodology (RSM). The three independent input parameters selected were the dosage of Hydrogen Peroxide ($H_2O_2$), the dosage of iron ($Fe^{2+}$), and the pH of the output or response selected was Total Organic Carbon (TOC) removal efficiency. Experiments were carried out according to the experimental design provided by CCD. For TOC Degradation, the model's $R^2$ and $R^2_{adj}$ correlation coefficients between experimental and model-predicted values were 0.9769 and 0.9561, respectively. This indicates a satisfactory correlation of experimental results with model-predicted values. The optimum values of operating parameters for maximum degradation were found to be $H_2O_2$=307 mM (X1), $Fe^{2+}$=1.1 mM (X2), and (pH)=3.3 (X3), for a reaction time of 120 min. For these operating parameters, the experimental result for TOC removal efficiency was found to be 72.89% as compared to the model-predicted value of 73.67%. These results indicate that the values were closely correlated with each other and thus the model was validated satisfactorily. Overall, the results indicate that the BMIMCl ionic liquid can be effectively degraded by the Fenton oxidation process.

## INTRODUCTION

Ionic liquids are molten salts having melting points below 100°C. Their structure constitutes a complex and asymmetric bulky cation with a weakly coordinating anion. Generally, the structural variations in molecular organic solvents also known as volatile organic compounds (VOCs) are very much limited to the innumerable structural variations in ionic liquids. This possibility of a large variation in the structures of ionic liquids, due to the availability of an enormous number of combinations of cations and anions, makes them the "designer solvents" (Siedlecka et al. 2008). Due to their immeasurable low vapor pressure, non-flammability, and tailor-made Physico-chemical properties, ILs have found wide applications in various fields like organic synthesis, solvent catalysis, electrochemistry, separation and extraction processes, fuel cells, solar cells, sensors, and nanochemistry. For example, ILs can extract benzene derivatives and metal ions from the aqueous phase (Yue et al. 2011) The physical features of ILs can be adjusted and made suited for certain applications by making appropriate adjustments in the alkyl chain, its branching, and the anion. Significant increases in reaction efficiency, selectivity, yield, and process economics can be achieved by executing the reactions in an IL media with target-oriented physico-chemical properties (Erdmenger et al. 2008).

The commonly used molecular or volatile organic compounds, due to their toxicity, flammability, and potential explosion hazard, create a greater concern for the workers in chemical industries. Apart from these properties, the emissions of solvent vapors in the atmosphere, and their release into water bodies through effluent discharge cause serious environmental concerns. Due to all these reasons, the use of volatile organic compounds in industries is getting restricted. In this context, ILs are gaining more popularity in industries as environmentally friendly "green solvents". The present status of research indicates that ILs provide a better alternative for organic solvents in many industries (Clarke et al. 2018).

However, the wider use of ILs in various industries may also cause harmful effects on the environment. For example, due to their non-biodegradability or persistency, ILs may get escaped from wastewater treatment processes and appear in the treated effluent before discharge in sufficient concentration. Due to the toxic nature of ILs, this treated effluent when discharged into the water bodies causes harmful effects on the aquatic ecosystem (Docherty et al. 2015). The very physico-chemical properties like non-volatility, and high chemical and thermal stability, which make the ILs most attractive for industrial applications, cause a lot of problems due to their

toxicity, higher solubilities in water, non-biodegradation or persistency, and bioaccumulation, in various environmental compartments (Pham et al. 2010). Also, the recovery of ILs from very dilute solutions or the waste streams is not an economically feasible option as it needs a large amount of energy. Hence it becomes very much essential to have an effective and economically feasible option for the degradation and removal of ILs from the aquatic solutions (Zhou et al. 2018). In this context, the advanced oxidation processes (AOPs) provide an attractive and effective option for the degradation of toxic and persistent ILs from the aquatic phase. The hydroxyl radicals($^{\bullet}OH$) generated in AOPs like Ozonation, Photo-Fenton's process, Photocatalytic reaction, Fenton's process, etc., are highly reactive and non-selective and hence oxidize the toxic and persistent organic compounds like ILs at a faster rate (Huang et al. 2007). Due to their simple operation and the high potential for degrading the highly toxic and refractive compounds, AOPs are gaining wider acceptance in industries and research as an effective means for the treatment of waste (Oller et al. 2011).

In the present work, a study has been carried out on the degradation of an ionic liquid namely, 1-butyl-3methyl imidazolium chloride (BMIMCl) using the Fenton oxidation process. This process involves the oxidation of organic compounds with hydrogen peroxide ($H_2O_2$) and employing Iron ($Fe^{2+}$) as a catalyst in acidic conditions. As compared to other methods of AOPs, Fenton's process is more advantageous in terms of process economy and ease of operation and maintenance. It is more effective for the effluents containing a high amount of suspended and dissolved solids as the penetration of photons across the depth of solution in the photo-oxidation process is limited (Anilkar et al. 1988). The hydrogen peroxide in Fenton's process gets decomposed by ferrous ions under acidic pH conditions (Qiu et al. 2010). Fenton process has been applied for the oxidation of a wide range of chemicals, organic and inorganic compounds such as phenolic compounds, atrazine, pesticides, toxic chemicals from the pharmaceuticals industry, and dyes from the textile industry (Pliego et al. 2012).

Experiments in the present study were performed as per the design of experiments using Central Composite Design (CCD) of Response Surface Methodology (RSM). Using RSM, a statistical and mathematical tool, optimization of operating parameters in industrial processes can be effectively carried out. This technique is used for the assessment of individual and interactive effects of various operating parameters on process output or response. In RSM a mathematical model is generated which can be used to find the optimum values of independent operating parameters. Through RSM the research results can be obtained at a faster rate than the time-consuming one variable at a time (OVAT) process (Alim et al. 2008). The individual and interactive effect of operating parameters (pH, dosage of $H_2O_2$, and $Fe^{2+}$) on the response (removal efficiencies of COD and TOC) has been assessed. The model predicted parameters were experimentally validated.

## MATERIALS AND METHODS

### Chemicals

All the chemicals and reagents including hydrogen peroxide ($H_2O_2$, 30% w/w) and ferrous sulfate heptahydrate, $FeSO_4.7H_2O$ (99.0%) used in the present work were of analytical grade procured from Fisher Scientific manufactured in India. Sulfuric acid, $H_2SO_4$ (98-99%), and sodium hydroxide, NaOH (>98%) were used for the adjustment of pH of the solution.

The ionic liquid, 1-butyl 3-methyl imidazolium chloł ride (BMIMCl), used in the present study was purchased from Sigma-Aldrich. The synthetic solution of BMIMCl was prepared using doubled distilled water (TOC<5 ppb), obtained by circulating tap water through a carbon filter, then subjecting it to reversed osmosis, and finally filtering it through 0.45-μm filter paper.

### Fenton Oxidation process of BMIMCl ionic liquid

Fenton experiments were carried out in an Erlenmeyer flask of 1L capacity with a continuous stirring at 200 RPM. The sample size was kept at 500 mL and the ionic liquid (BMIMCl) concentration selected was $1mM.L^{-1}$. The oxidation reaction was conducted at room temperature 25±1°C for a designed reaction time of 120 min. A total of twenty experiments, as per the design of experiments by CCD of RSM, were conducted. The ionic liquid solution was placed in the glass reactor and a designed dose of hydrogen peroxide ($H_2O_2$) ranging from 200 to 400 mM and ferrous ions ($Fe^{2+}$) ranging from 0.5 to 1.5 mM were added to a reactor and a pH value of 3 to 4 was maintained. After a reaction time of 120 min, the reaction was stopped by adding 0.5 mL of methanol and the samples were analyzed for TOC. All the experiments were conducted in duplicate.

### TOC Analysis

The Total Organic Carbon (TOC) was determined with a Shimadzu TOC-L analyzer with potassium phthalate as a standard calibration solution. Orion pH meter was used for measuring the pH values.

### Experimental Design and Statistical Data Analysis.

Optimization and statistical data analysis have been carried out by using the Central Composite Design (CCD) of response surface methodology (RSM). The experimental

design was constituted of three factors considered in five levels and 20 runs were conducted to optimize the level of the selected factors that are hydrogen peroxide ($X_1$), $Fe^{2+}$ concentration ($X_2$ ), and pH ($X_3$) (Table 1).

State-Ease Design expert 11.00 software was used to analyze model parameters and for analysis of variance (ANOVA), a second-order quadratic model is useful for correlating the predicted response and the independent variable in the coded values Equation 1

$$Y = A_0 + A_1X_1 + A_2X_2 + A_3X_3 + A_{12}X_1X_2 + A_{13}X_1X_3 + A_{23}X_2X_3 + A_{11}X_1^2 + A_{22}X_2^2 + A_{33}X_3^2 \quad \ldots(1)$$

Where y is the response (dependent variable) of percentage removal of TOC by the degradation of BMIMCl compound. In coded units, $A_1$, and $A_2$ are regression coefficients for linear effects, $A_{11}$, and $A_{22}$ are quadratic coefficients. and $A_{12}$, $A_{13}$, and $A_{23}$ are the interaction coefficients. According to Table 2, the CCD includes eight factorial points, six axial points, and center points with six additional experimental trials as the duplicates of this point. The term coded value was used to present independent variables at three levels: + $\alpha$ and - $\alpha$ (star points), -1 (minimum), 0 (central), and +1 (maximum). The specific value of $\alpha$ depends on certain properties looked for in the design and a number of factors. The accuracy of the quadratic model was explained by the coefficient of determination $R^2$. Also, model terms were chosen or rejected based on the

Table 1: Levels of the independent variables and experimental ranges.

| Variables | Code | Variables | -$\alpha$ | -1 | 0 | +1 | +$\alpha$ |
|---|---|---|---|---|---|---|---|
| $H_2O_2$ [mM)] | $X_1$ | $H_2O_2$ [mM] | 131.82 | 200 | 300 | 400 | 468.18 |
| $Fe^{2+}$(mM) | $X_2$ | $Fe^{2+}$ [mM] | 0.16 | 0.5 | 1 | 1.5 | 1.84 |
| pH | $X_3$ | pH | 2.7 | 3 | 3.5 | 4 | 4.34 |

Table 2: The CCD experimental design with three independent variables of 20 runs.

| Run | Factor 1 | Factor 2 | Factor 3 | Experimental | Predicted |
|---|---|---|---|---|---|
| | $X_1$: Hydrogen peroxide mM | $X_2$: Fe mM | $X_3$: pH | TOC(%) | TOC(%) |
| 1 | 300 | 1 | 3.5 | 71.55 | 71.36 |
| 2 | 300 | 1 | 4.3409 | 9.02 | 8.45 |
| 3 | 200 | 1.5 | 3 | 40.21 | 40.09 |
| 4 | 200 | 1.5 | 4 | 15.59 | 17.05 |
| 5 | 300 | 1 | 3.5 | 71.56 | 71.36 |
| 6 | 400 | 0.5 | 3 | 36.72 | 35.25 |
| 7 | 300 | 1 | 2.6591 | 35.00 | 35.59 |
| 8 | 200 | 0.5 | 4 | 23.27 | 23.54 |
| 9 | 400 | 1.5 | 3 | 62.11 | 61.83 |
| 10 | 200 | 0.5 | 3 | 30.88 | 31.75 |
| 11 | 300 | 1 | 3.5 | 71.50 | 71.36 |
| 12 | 400 | 1.5 | 4 | 38.65 | 37.76 |
| 13 | 300 | 1 | 3.5 | 71.53 | 71.36 |
| 14 | 300 | 0.159104 | 3.5 | 38.65 | 38.78 |
| 15 | 300 | 1.8409 | 3.5 | 55.78 | 55.67 |
| 16 | 131.821 | 1 | 3.5 | 30.95 | 39.47 |
| 17 | 300 | 1 | 3.5 | 71.52 | 71.36 |
| 18 | 300 | 1 | 3.5 | 71.56 | 71.36 |
| 19 | 468.179 | 1 | 3.5 | 48.32 | 49.82 |
| 20 | 400 | 0.5 | 4 | 25.89 | 26.00 |

P-value with 95% confidence levels. Surface plots (three-dimensional) and respective contour plots (two-dimensional) were obtained for the degradation of BMIMCl ionic liquid.

Where y is the response variable of TOC (%) degraa dation of BMIMCl compound. In coded units, A*1*, and *A2* are regression coefficients for linear effects, *A11,* and *A22* are quadratic coefficients., and *A12* is the interaction coefficient (Simsek et al. 2013). According to Table 2, the CCD includes eight factorial points, six axial points, and a center point with six additional experimental trials as the duplicates of this point. The term coded value was used to present independent variables at three levels: + $\alpha$ and - $\alpha$ (star points), -1 (minimum), 0 (central), +1 (maximum), the specific value of $\alpha$ depends on certain properties looked for the design and number of factors. The accuracy of the quadratic model was explained by the coefficient of determination $R^2$. Also, model terms were chosen or rejected based on the P-value with 95% confidence levels. Surface plots (three-dimensional) and respective contour plots (two-dimensional) were obtained for the degradation of BMIMCl ionic liquid.

## RESULT AND DISCUSSION

### Development of the Quadratic Model and Statistical Analysis

Using a model software that is design expert 11.0, the following quadratic model for the experimental response of TOC degradation was obtained from equations 2.

$$(Y_1) = +71.36 + 6.19 * H_2O_2 + 5.67 * Fe^{2+}$$

$$- 7.72 * pH + 4.96 * H_2O_2 * Fe^{2+} + 0.1375$$

$$* H_2O_2 * pH - 4.10 * Fe^{2+} * pH - 11.78$$

$$* (H_2O_2)^2 - 9.16 * (Fe^{2+})^2 - 16.59$$

Table 3: ANOVA result for Fenton oxidation.

| | Source | Ss | DF | Ms | F-Value | P-Value |
|---|---|---|---|---|---|---|
| TOC (%) | Model | 8204.04 | 9 | 911.56 | 2826.23 | 0.001 |
| | Residual | 3.23 | 10 | 0.32 | | |
| | Lack of Fit | 2.89 | 5 | 0.57 | 8.65 | |
| | Pure Error | 0.33 | 5 | 0.29 | | |
| | $R^2$ | 0.9769 | | 0.9561 | | |

$$* (pH)^2 \quad \ldots(2)$$

The value 71.36 shown in equation 2 is the intercept value for TOC (Y), These results indicate a positive influence on the model. The values of coefficients for the variables such as $Fe^{2+}$* pH, $H_2O_2$*$Fe^{2+}$and $H_2O_2$*pH have an optimistic effect on the degradation of TOC of BMIMCl by Fenton reaction. The analysis of variance (ANOVA) of the model for the degradation of BMIM is given in Table 3.

Results of the ANOVA of the empirical second-order quadratic model for TOC reduction of BMIMCl shows P-values less than 0.0500. This indicates that model terms are significant. If P values are greater than 0.1000, it is inferred that the model terms are not significant.

The correlation coefficient $R^2$ describes the variability in the response values by the experimental factors and their interactions. $R^2$ and $R^2_{adj}$ values in the present work were found to be 0.9769 and 0.9561 The variance between the correlation coefficient $R^2$ and $R^2_{adj}$ were sensibly satisfactory and hence this model was fit for determining the optimum condition for degradation of BMIMCl using Fenton oxidation. A plot of experimental values versus predicted values resulting from the model is shown in Fig. 1.

### Response Contour Plots and Response Surface Plots for the Fenton Process

#### *Effect of pH and $H_2O_2$*

The interactive effects of $H_2O_2$ concentration and initial pH are depicted in Fig. 2 (a) by 3D response surface plots and (b) by contour plots. As can be seen in the graph, as the pH rises, so does the rate of TOC breakdown. At a pH of 3.5, a

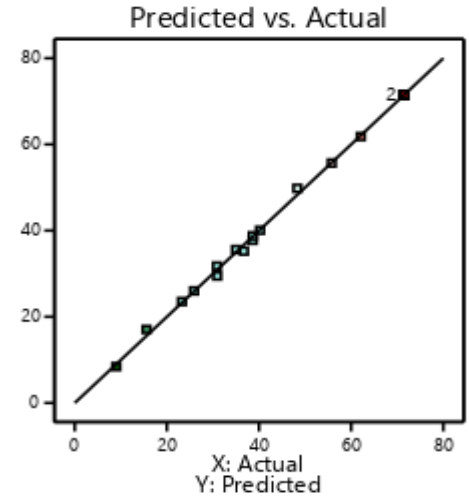



Fig. 1: Plot of experimental values against predicted values.

maximum of 71.55 TOC degradation appears with an oxidizing concentration of $H_2O_2$ 300 mM in the presence of $Fe^{2+}$ 1 mM catalyst concentration with a reaction time of 120 mins. Moreover, the decreased degradation efficiency was also detected at lower pH (<2.6). When the pH was reduced from 3 to 2.6, for example, the breakdown efficiency of BMIMCl decreased due to the production of the oxonium ion ($H_3O_2^+$), which is less reactive (Siedlecka et al. 2008). According to the equation (3), excessive hydrogen ion concentration at a high pH level has a probability of scavenging hydroxyl radicals (Devi et al. 2009)

$$H^+ + OH^\bullet + e \rightarrow H_2O \quad \ldots(3)$$

### *Effect of $H_2O_2$ Concentration and $Fe^{2+}$ Concentration*

The oxidation efficiency of ionic liquids is strongly dependent on the concentration of the oxidant ($H_2O_2$) and catalyst [$Fe^{2+}$] in a Fenton process. Generally, the more [$H_2O_2$] and [$Fe^{2+}$] the faster the degradation rate of IL Behnajady et al. 2007). Similar observations were found in this experiment. As the $Fe^{2+}$ concentration increased from 0.5 to 1 mM [Fig. 3 (a) and (b)], the TOC degradation efficiency of BMIMCl increased from 23.27% to 71.55%. This may be attributed to the fact that increasing the concentration of $Fe^{2+}$ accelerates the decomposition of $H_2O_2$ and consequently induces an increase in the availability of hydroxyl radicals (Vallejo et al. 2015). However, overconsumption of Fe(II) and $H_2O_2$ will also consume the hydroxyl radicals [equation (4) and (5)] and increases the chances of hydroxyl radical recombination [equation (6)]. Besides, the excess utilization of Fe(II) would cause the development of an abundance of ferric-based sludge, thereby increasing the disposal cost (Munter et al. 2006).

$$OH^\bullet + Fe^{2+} \rightarrow Fe^{3+} + OH^- \quad \ldots(4)$$

$$OH^\bullet + H_2O_2 \rightarrow OOH^\bullet + H_2O \quad \ldots(5)$$

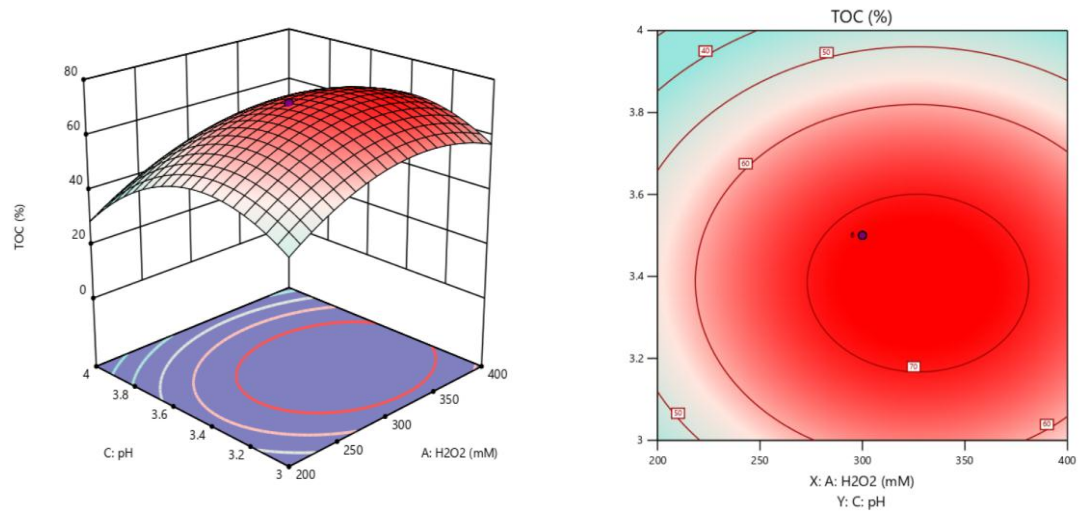
$$H^\bullet + OH^\bullet \rightarrow H_2O_2 \quad \ldots(6)$$

### Effect of $Fe^{2+}$ Concentration and pH

The effect of catalyst concentration and pH on the TOC (%) degradation is shown in Fig. 4 (a, b) pH value 3.5 and iron concentration 1 mM shows the development of active iron species of $Fe^{2+}$ ions due to which the production of hydroxyl radicals ($^\bullet$OH) increases, Also, at a very small pH, the reaca tion of $Fe^{2+}$ with $H_2O_2$ is hindered, and hydroxyl radical is scavenged by $H^+$ ions (Tang & Huang 1996). A comparable observation is found in the present experiment also. For these values of parameters, the percentage removal of TOC was found to be 71.55%.

### Optimization of Inducing Parameters

Optimization of inducing parameters was achieved based on the desirability function to decide the optimal conditions for the degradation of BMIMCl ionic liquid. Numerical optimization was used to recognize the specific ranges that maximize the desirability function. The program uses five




(a) (b)

Fig. 2: Three-dimensional surface plot (a) and two-dimensional contour plot (b) for the effect of catalyst concentration and pH, on TOC (%) degradation.

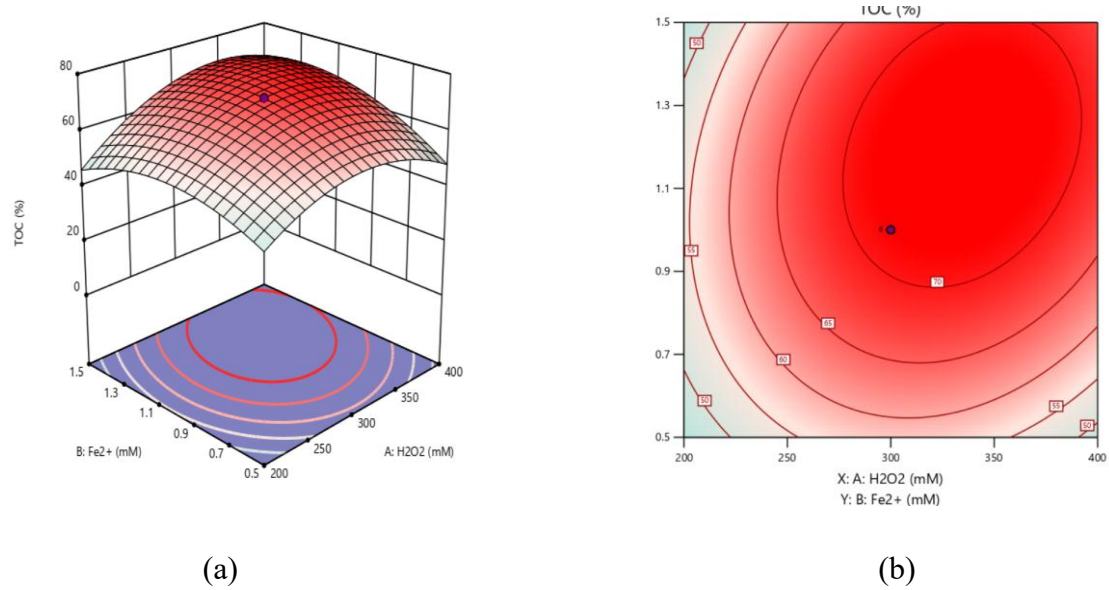



Fig. 3: Three-dimensional surface plot (a) and two-dimensional contour plot (b) for the effect of $H_2O_2$ concentration and $Fe^{2+,}$ on TOC (%) degradation.

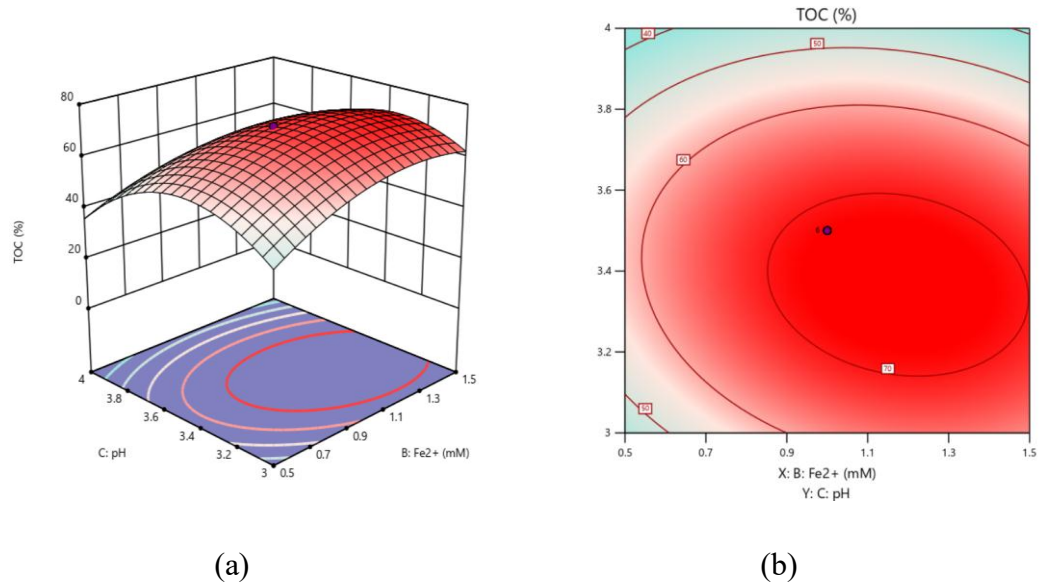



Fig. 4: Three-dimensional surface plot (a) and two-dimensional contour plot (b) for effect of $Fe^{2+}$ concentration and pH on TOC (%) degradation.

probabilities as a goal to construct desirability indications: none, maximum, minimum, target, and within range. The criteria for all variables in correspondence with degradation percentages are revealed in Table 4.

Based on the boundaries constraints, described by numerical optimization, the optimum conditions for BMIMCl ionic liquid maximum condition were found that be TOC (73.67%). in presence of a hydrogen peroxide concentration of 307 mM and $H_2O_2$ concentration of 1.1 mM and pH value of 3.3.

## Validation of the Model and Confirmation

To confirm the appropriateness of the model for predicting the maximum percentage of TOC degradation of BMIMCl, the experiment was conducted in a 1L capacity glass reactor using the optimum conditions which were obtained from model software. An average maximum TOC of 73.79 % was obtained from three duplicate experiments, as shown in Table 5. The good agreement between the predicted value and the experimental value confirms the validity of the model

Table 4: Optimization of variables under limits.

| Name | Goal | Lower Limit | Upper Limit | Lower Weight | Upper Weight | Importance |
|---|---|---|---|---|---|---|
| $H_2O_2$ | is in range | 200 | 400 | 1 | 1 | 3 |
| -$Fe^{2+}$ | is in range | 0.5 | 1.5 | 1 | 1 | 3 |
| pH | is in range | 3 | 4 | 1 | 1 | 3 |
| TOC | maximize | 9.02 | 71.58 | 1 | 1 | 3 |

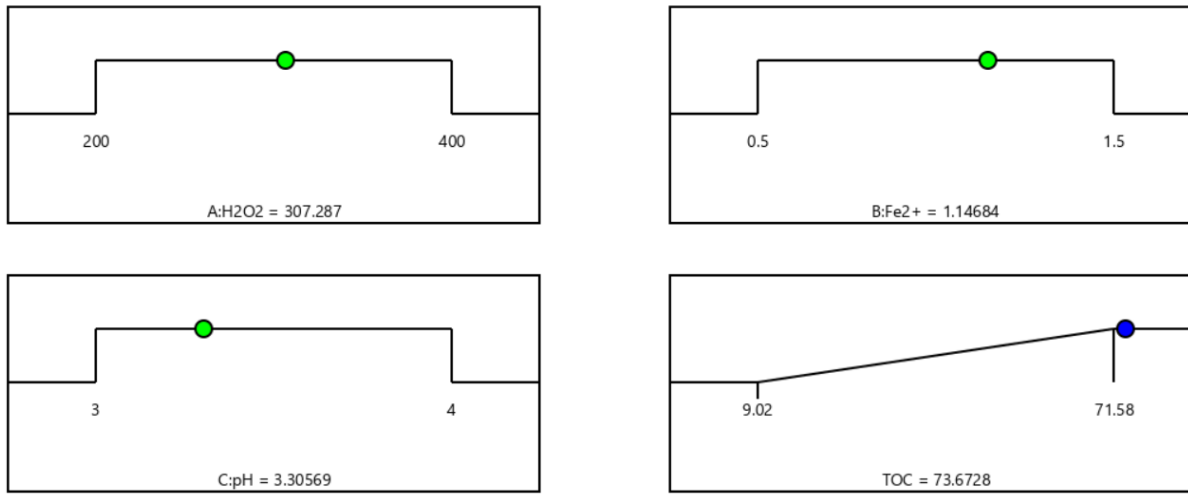



Fig. 5: Numerical optimization constraints by using ramps.

for simulating the degradation of BMIMCl by the Fenton oxidation process.

## CONCLUSION

The degradation of 1-butyl 3-methyl imidazolium chloride (BMIMCl) ionic liquid (IL) by Fenton oxidation was very effective for a reaction time of 120 min. The following parameters affected the entire Fenton process: $H_2O_2$ dosage, $Fe^{2+}$ dosage, and pH. The central composite design (CCD) model was selected to attain the optimal conditions for BMIMCl degradation in the Fenton oxidation experiment to minimize the number of experimental runs. This statistical design was found suitable for this study with a regression coefficient $R^2$ equal to 0.9769 and Adj-$R^2$ equal to 0.9561 for TOC degradation. ANOVA and the response surface led to the optimization of the degradation of 1-butyl-3methyl imidazolium chloride (BMIMCl) ionic liquid (IL) The optimum parameters for degradation by the Fenton oxidation process were found to be: $H_2O_2$=307 mM (X1), $Fe^{2+}$=1.1 mM (X2) and (pH)=3.3 (X3). Overall it can be concluded that the Fenton process is very much effective in degrading BMIMCl ionic liquid as per the above-mentioned operating conditions.

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Table 5: Comparison of predicted and experimental results for optimum working conditions in Fenton oxidation.

| Variables | Optimum Value | TOC Degradation % | |
|---|---|---|---|
| | | Predictive | Experimental |
| $H_2O_2$ ($X_1$) mM | 307.287 | 73.67 | 72.89 |
| $Fe^{2+}$ ($X_2$) mM | 1.146 | | |
| pH ($X_3$) | 3.30 | | |

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