ORIGINAL ARTICLE

Toluene removal by oxidation reaction in spray wet scrubber: experimental, modeling and optimization

Juntima Chungsiriporn[1], Charun Bunyakan[2] and Roumporn Nikom[3]

Abstract

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Toluene, an important volatile organic compound (VOC), is used in many kinds of industries, such as painting, printing, coating, and petrochemical industries. The emission of toluene causes serious air pollution, odor problem, flammability problem and affects human health. This paper proposes the removal of toluene from waste air using a spray wet scrubber combining the absorption and oxidation reaction. Aqueous sodium hypochlorite (NaOCl) solution was used as the scrubbing liquid in the system. NaOCl, the strongest oxidative agent, presents an effective toluene removal. As the scrubbed toluene is reacted, recirculation of the scrubbing liquid could be operated with a constant removal efficiency throughout the operting time. The investigated variables affecting the removal efficiency were air flow rate, inlet toluene concentration, NaOCl concentration, scrubbing liquid flow rate and size of spray nozzle. Influence of the scrubbing parameters was experimentally studied to develop a mathematical model of the toluene removal efficiency. The removal model reveals that the increase of scrubbing liquid flow rate, toluene concentration, and NaOCl concentration together with the decrease of air flow rate and size of spray nozzle can increase the toluene

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removal efficiency. Optimization problem with an objective function and constraints was set to provide the maximum toluene removal efficiency and solved by Matlab optimization toolbox. The optimization constraints were formed from the mathematical model and process limitation. The solution of the optimization was an air flow rate of 100 m^3/h, toluene concentration of 1500 ppm, NaOCl concentration of 0.02 mol/l, NaOCl solution feed rate of 0.8 m^3/h, and spray nozzle size of 0.5 mm. Solution of the optimization gave the highest toluene removal efficiency of 91.7%.

Key words : VOCs, toluene, NaOCl, wet scrubber, optimization

บทคัดย่อ

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การกำจัดทูโลอีนโดยปฏิกิริยาออกซิเดชันในหอพ่นฝอยแบบเปียก: การทดลอง แบบจำลอง และการหาผลเลิศ

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ทูโลอีนเป็นสารประกอบอินทรีย์ระเหย (VOCs) ที่สำคัญที่ใช้ทั่วไปในอุตสาหกรรมสี อุตสาหกรรมการพิมพ์ อุตสาหกรรมการเคลือบผิว และอุตสาหกรรมปิโตรเคมี การปล่อยทูโลอีนจากอุตสาหกรรมเหล่านี้ทำให้เกิดมลภาวะทางอากาศ ก่อให้เกิดปัญหาเรื่องกลิ่น การเกิดไฟไหม้ และมีผลต่อสุขภาพของมนุษย์โดยตรง บทความนี้นำเสนอวิธีการกำจัดทูโลอีนจากอากาศเสียโดยอาศัยกระบวนการดูดซึมและการเกิดปฏิกิริยาออกซิเดชันในหอดูดซึมแบบเปียก ตัวออกซิแดนซ์ที่ใช้คือ โซเดียมไฮโปคลอไรด์ (NaOCl) ตัวแปรที่มีผลต่อประสิทธิภาพในการกำจัดทูโลอีนที่ศึกษาประกอบด้วย อัตราการไหลของอากาศ ความเข้มข้นของทูโลอีนในอากาศ ความเข้มข้นของ NaOCl อัตราการไหลของ NaOCl และขนาดของหัวพ่นฝอย ผลของตัวแปรที่ศึกษาต่อประสิทธิภาพการกำจัดทูโลอีนถูกนำมาพัฒนาเป็นแบบจำลองทางคณิตศาสตร์ โดยมีวัตถุประสงค์เพื่อหาสภาวะดำเนินการที่ให้ประสิทธิภาพในการกำจัดทูโลอีนสูงสุดภายใต้เงื่อนไขทั้งทางคณิตศาสตร์และข้อจำกัดของกระบวนการที่ศึกษา ผลเฉลยของการหาผลเลิศ พบว่าสภาวะที่เหมาะสมในการกำจัดทูโลอีนคือที่อัตราการไหลของอากาศ ความเข้มข้นของทูโลอีนในอากาศ ความเข้มข้นของ NaOCl อัตราการไหลของสารละลาย NaOCl และขนาดของหัวพ่นฝอย มีค่าเป็น 100 ลบ.ม./ชั่วโมง 1500 พีพีเอ็ม 0.02 โมล/ลิตร 0.8 ลบ.ม./ชั่วโมง และ 0.5 มม. ตามลำดับ โดยให้ประสิทธิภาพในการกำจัดทูโลอีนสูงสุด คือ 91.7%

ภาควิชาวิศวกรรมเคมี คณะวิศวกรรมศาสตร์ มหาวิทยาลัยสงขลานครินทร์ อำเภอหาดใหญ่ จังหวัดสงขลา 90112

Toluene, a kind of volatile organic compound (VOC), is a toxic but common compound used in many industry (painting, printing, coating, automotive, etc.). Emission of the toluene from these industries causes air pollution, and effects human health (Chang *et al*., 2000). Thus, air contaminated with toluene needs to be treated before it can be released to atmosphere. One of the most popular air treatments foreseen today is VOCs destruction through the technologies of oxidation treatment (Khan and Ghoshal, 2000). Wet scrubbers, add-on control device, are normally used to remove particulate matter and soluble gases from gas stream by counter current flow of waste air and scrubbing solution (Shabunya *et al*., 2003). Contacting and scrubbing of the counter current flows yields absorption in the wet scrubber. The absorption efficiency depends on solubility of pollutant matters in scrubbing liquid. This technique is not suitable for VOCs with low solubilty in water such as toluene. To improve the toluene treatment efficiency in the wet scrubber, an additional treatment step is required. Adding of strong oxidizing reagent in the scrubbing provides both absorbtion and oxidation that enhances the treatment efficiency. Sodium hypochlorite (NaOCl) is a strongly oxidative and fast-acting disinfectant agent (Tsai *et al*., 1999) and is suitable for oxidation of toluene (Bunyakan *et al*., 2004). The oxidation reaction yields the oxidation products including salt and chloride ions as given by equation (1)

$$C_6H_5CH_3 + 3NaOCl \rightarrow C_6H_5COOH + 3NaCl + H_2O \quad (1)$$

The response surface methodology (RSM), a mathematical and statistical technique, is a tool to determine the optimal values of variables without the necessity of testing all possible combinations (Sumnu *et al*., 2000). An important aspect of design of experiment (DOE) in the RSM is to select for the points where the response should be evaluated and developed for model fitting and surface plotting. The optimization problem consisting of objective functions and constraints is used to solve for the optimum operating condition for the process of interest. The constraints are formed from process limitation and mathematical model. Optimization toolbox in Matlab program package can easily solve for the optimization problem (Chungsiriporn *et al*., 2005).

The purposes of this work are: (1) to investigate the application of sodium hypochlorite (NaOCl) for treatment of waste air contained toluene using wet scrubber; (2) to develop a mathematical model and surface plotted for detemination of the effect of scrubbing parameters and (3) to form the optimization problem and solve for the optimum condition of toluene treatment using a wet scrubber. The results obtained can be used as guidance for the efficient VOCs treatment.

Experimental procedure

Experimental set up and procedure

The wet scrubber used in this investigation was custom designed and constructed to study toluene removal efficiency using NaOCl as oxidizing agent. The wet scrubber system consists of a waste air generation unit, spray chamber, liquid recirculation system and the sampling ports. Schematic diagram of the experimental apparatus is shown in Figure 1. Toluene- contaminated air was generated from the waste air generation unit consisting of compressed air stream, liquid toluene tank, blower, and air-mixing line. Compressed air was introduced into the toluene tank to bubble and generate a toluene gas stream. The air stream

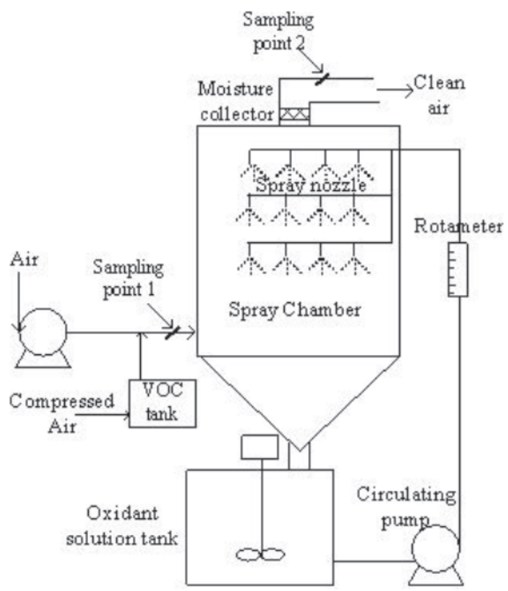

Figure 1. Schematic diagram of wet scrubber for removal toluene in waste air

from the air blower and the toluene gas stream were mixed in the air-mixing line to produce waste air containing toluene. Flow rate and concentration of the toluene-contaminated air were measured and controlled. The waste air was then introduced into the scrubbing spray chamber, an empty cylindrical chamber with a set of spray nozzles. NaOCl oxidizing liquid prepared in storage tank was pumped and sprayed through the spray nozzles to form small droplets in the chamber. Toluene in the waste air was scrubbed and oxidized by contact of the waste air and the NaOCl droplets. NaOCl liquid collected from the spray chamber was sent back to the storage tank and circulated in the wet scrubber system. Treated clean air vented out to atmosphere at the top of the chamber was analyzed for toluene concentration.

Sampling and analysis

Sorbent tubes were used to collect air samples at sampling points 1 and 2. The gas samples were analyzed using a gas chromatography (GC) model HP9806, Hewlett Packard. The GC equipped with capillary column, a flame ionization detector and an integrator (Model 9860) was operated at injection temperature of 180ºC, detector temperature of 200ºC and oven temperature of 40ºC. The GC column was HP-1 Hewlett Packard, 30 m capillary glass column with an inside diameter of 0.32 mm. Helium was used as the carrier gas at a flow rate of 1.5 ml/min. This analytical system gave an excellent resolution for toluene concentration measurement.

Experimental design

RSM with central composite design (CCD) was employed to optimize the toluene removal condition in the wet scrubber. Essential regression statistical package in Microsoft Excel was used as a tool for designing the experiment, model fitting, and surface plotting. In this work, 5 parameters of interest, consisting of toluene concentration, air flow rate, NaOCl solution flow rate, NaOCl concentration, and size of spray nozzle, were considered (Chu *et al.*, 2001). Operating ranges according to limitation and requirement of this study are shown in Table 1. Twenty six experimental runs were designed.

Model fitting

The regression model was used to represent the results of experiments (Sumnu *et al.*, 2000, Sin *et al.*, 2006). The response function (y) was partitioned into linear, quadratic, and interactive components as shown in equation (2).

$$y = \beta_0 + \sum_{i=1}^{k}\beta_i x_i + \sum_{i=1}^{k}\beta_{ii} x_i^2 + \left(\sum_{i=1}^{k-1}\sum_{j=i+1}^{k}\beta_{ij} x_i x_j\right)_{i<j} \qquad (2)$$

where β_0 is defined as the constant, β_i is the linear coefficient, β_{ii} is the quadratic coefficient and β_{ij} is the cross product coefficient. x_i and x_j are levels of the independent variable, while k equals to the number of tested factors.

The multiple regression started with all possible terms. The effects and regression coefficients of individual linear, quadratic and interaction terms were determined. The significances of all terms in the model were judged statistically by computing the F-value. Elimination of the non-significant terms was performed using criteria of level of significance (P-value > 0.05). A low P value of a particular term indicated its significant role in improving the model curve fitting.

Optimization problem formation

The optimization problem consists of objective function and constraints as given in equation (3). The problem statement aims to max-

Table 1. Interested range of experimental parameters

NaOCl solution flow rate, Q_L (m^3/h)	Air flow rate, Q_G (m^3/h)	NaOCl concentration, C_{NaOCl} (mol/l)	Toluene concentration, C_T (ppm)	Size of spray nozzle, r_d (mm)
0.5-0.8	100-300	0-0.04	150-1500	0.5-1.0

imize toluene removal efficiency $f(x)$ or minimize $1/f(x)$. The constraints were formed from limitation and mathematical model.

Objective function $\min_x (1/f(x))$
subject to

$$\begin{aligned} &c(x) \le 0, \quad ceq(x) = 0 \\ &A.x \le 0, \quad Aeq.x = beq, \quad lb \le x \le ub \end{aligned} \qquad (3)$$

where x, b, beq, lb, and ub are vectors, A and Aeq are matrices, $c(x)$ and $ceq(x)$ are functions that return vectors, and $f(x)$ is a function that returns a scalar. $f(x)$, $c(x)$, and $ceq(x)$ can be nonlinear functions.

Matlab Optimization Toolbox can effectively solve the optimization problem. The optimization toolbox consists of commands for option parameters, input and output arguments, and functions that perform minimization. The algorithm used to find the minimum of a constrained nonlinear multivariable function is fmincon. The objective function and constraints are written in function form of @myfun and ,@nonlcon, respectively. The syntax command line is written as shown below.

[*x*] = fmincon(@myfun,*x0*,*A*,*b*,*Aeq*,*beq*,*lb*,*ub*, @nonlcon,options)

Matlab constrained optimization routine that consists of starting guess, syntax command, options, lower bound and upper bound were invoked to solve the problem. The results of optimization were given by the maximum of removal efficiency.

Results and discussion

Toluene removal experiments in spray scrubber were carried out coupling with oxidation reaction using NaOCl as an oxidative reagent. The influence of air flow rate, inlet toluene concentration, NaOCl concentration, NaOCl solution flow rate and size of spray nozzle on toluene removal efficiency were investigated. The toluene removal efficiency, a response of the experiments, was calculated as equation (4).

$$\text{Removal efficiency (\%)} = \frac{C_i - C_o}{C_i} \times 100 \qquad (4)$$

where C_i and C_o are the toluene concentration, in ppm, at an inlet and outlet of wet scrubber, respectively.

The influences of the experiment parameters, model formation and optimization were investigated and detailed below:

Effect of air flow rate

The effect of air feed flow rate on toluene removal efficiency is presented in Figure 2. It is clearly shown from this figure that the removal efficiency decreased when the air feed rate was increased from 100 to 300 m^3/h. The volume of the scrubbing chamber was 0.4 m^3, so the corresponding retention time was in the range of 4.8-14.4 s. Since volume of the chamber was constant, the increase in air flow rate resulted in lower retention time. The retention time plays an important role in the absorption of toluene into scrubbing solution (Ebert and Buttner 1996), which was indicated by decrease in the toluene removal efficiency with the increase of air flow rate as shown in Figure 2.

Effect of toluene concentration

Difference inlet contaminant concentrations led to different absorption and reaction rate. Effect of toluene concentration on toluene removal efficiency is illustrated in Figure 3. It is evident that the trend can be separated into two regions. In the low toluene concentration range, 150-750 ppm, the toluene removal efficiency was low with a value of 57% and slightly increased with increasing of toluene concentration. At this concentration range, the absorption of toluene in the solution dominated over the oxidation reaction. In high concentration region, 750-1500 ppm, the toluene removal efficiency, however, significantly increased with increasing toluene concentration. Order of oxidation reaction with respect to toluene concentration and NaOCl concentration was 1.1 and 0.1, respectively (Bunyakan *et al.*, 2003). At high toluene concentration, the oxidation reaction

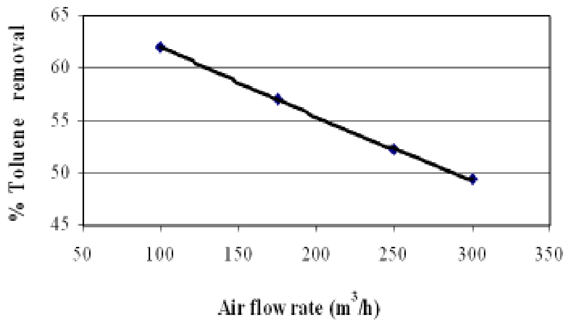

Figure 2. Effect of air feed flow rate on toluene removal in wet scrubber using NaOCl as oxidative reagent. (C_T = 350 ppm, Q_L = 0.65 mol/l, C_{NaOCl} = 13 mmol/l, and r_d = 1 mm)

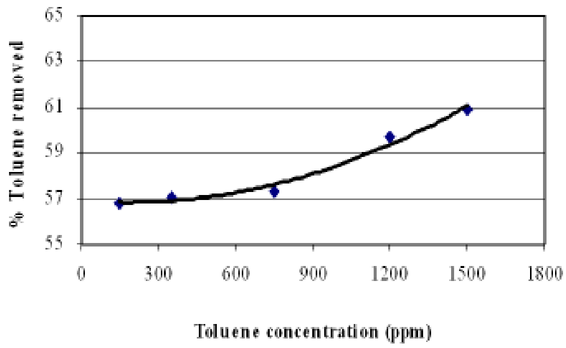

Figure 3. Effect of inlet toluene concentration on toluene removal in wet scrubber using NaOCl as oxidative reagent. (C_{NaOCl} = 13 mmol/l, Q_L = 0.65 mol/l, Q_G = 175 m^3/h, and r_d = 1 mm)

dominated over the absorption. Therefore, the toluene removal efficiency was high and significantly increased with increasing toluene concentration as shown in Figure 3.

Effect of NaOCl concentration

The influence of NaOCl concentration on toluene removal efficiency was carried out for C_{NaOCl} range of 0 to 0.03 mg/l, C_T = 350 ppm, Q_L = 0.65 mol/l, Q_G = 175 m^3/h, and r_d = 1 mm. It was found that, at C_{NaOCl} = 0 mg/l, the toluene removal efficiency was approximately 20%. Figure 4 presents the toluene removal efficiency for NaOCl concentration range of 0.006-0.03 mg/l. For the low concentration range of 0.006 to 0.02 mg/l, the toluene removal efficiency significantly improved with increasing NaOCl concentration. Huang *et al.* (2002) investigated the impact of oxidant concentration and found the same trend as this work. At high NaOCl concentration (> 0.02 mg/l), it was found that the NaOCl concentration tended to have less effect on toluene removal efficiency, most likely due to an excess amount of NaOCl. These results indicated that the optimum NaOCl concentration for the scrubbing system at this operating condition was 0.02 mg/l.

Effect of NaOCl solution flow rate

The effect of NaOCl solution flow rate on toluene removal efficiency is shown in Figure 5. The curve indicates that the NaOCl solution flow rate had significantly enhanced the toluene removal efficiency. Chien and Chu (2000), have reported that the higher the liquid flow rates, the greater the gas-liquid contacting surface area obtained. The higher solution feed flow rate can also increase the number of NaOCl solution droplets. A drop of NaOCl solution can be considered as one oxid-

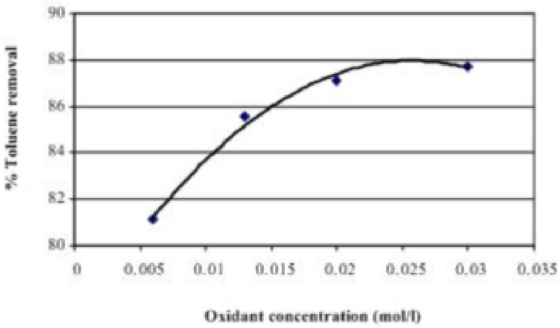

Figure 4. Effect of NaOCl concentration on toluene removal in wet scrubber using NaOCl as oxidative reagent. (C_T = 350 ppm, Q_L = 0.65 mol/l, Q_G = 175 m^3/h, and r_d = 1 mm)

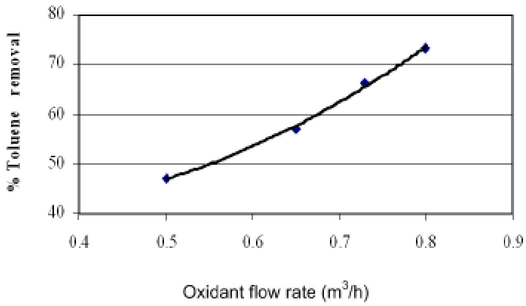

Figure 5. Effect of NaOCl solution flow rate on toluene removal in wet scrubber using NaOCl as oxidative reagent. (C_T = 350 ppm, C_{NaOCl} = 13 mmol/l, Q_G = 175 m^3/h, and rd = 1 mm)

ation reactor. The higher number of reactors, by increasing the NaOCl flow rate, the more toluene was removed from the air stream.

Effect of liquid drop size

The effect of scrubbing solution drop size on the toluene removal was studied by varying the opening size of spray nozzle. In this investigation, air flow rate, toluene concentration and NaOCl concentration were controlled at 175 m^3/h, 350 ppm, 0.013 mol/l, respectively. As shown in Table 2, liquid drop size or nozzle diameter strongly influence the toluene removal efficiency. The number of NaOCl droplets and surface area were increased with decreasing the drop size (Ebert and Buttner, 1996). Consequently, contacting opportunity and surface reaction was promoted. The absorption and oxidation reaction of toluene on the NaOCl droplets were then improved.

Mathematical model formation

Toluene removal model was obtained from data fitting using Essential Regression in Microsoft Excel package. The regression model given in equation 5 describes the influence of the operating parameters on the toluene removal efficiency.

$$x_1 = 123.22 + 292.46x_6^2 - 0.08792x_2x_5 + 2.272x_2x_3 - 282.51x_6 + 0.00479x_4 \quad (5)$$

where x_1 is toluene removal efficiency, x_2 is air flow rate (m^3/h), x_3 is oxidant concentration (mol/l), x_4 is toluene concentration (ppm), x_5 is nozzle diameter (mm) and x_6 is oxidant flow rate (m^3/h)

The significant operating parameters shown in the model are toluene concentration, air flow rate, NaOCl solution flow rate, NaOCl concentration, and size of spray nozzle. To improve the curve fitting, a particular significant term of P

Table 2. Toluene removal efficiency at different sizes of spray nozzle.

Diameter (mm)	NaOCl Solution Flow rate (m^3/h)	Toluene Removal eff. (%)
1	0.5	47.1
1	0.8	73.3
0.5	0.5	54.8
0.5	0.8	85.6

Table 3. Optimization problems and constraints of toluene removal system

Objective function	Min $f(x) = - x_1$
Subject to	Toluene removal model $x_1 = 123.22 + 292.46x_6^2 - 0.08792x_2x_5 + 2.272x_2x_3 - 282.51x_6 + 0.00479x_4$
	Boundary limit $100 < x_2 < 300$, $0 < x_3 < 0.4$, $150 < x_4 < 1500$, $0.5 < x_5 < 1.0$, $0.5 \le x_6 \le 0.8$,

Table 4. Optimum operating condition of toluene removal in wet scrubber according to the optimization problem.

NaOCl solution flow rate, (m^3/h)	Air flow rate, (m^3/h)	NaOCl concentration, (mol/l)	Toluene concentration, (ppm)	Size of spray nozzle, (mm)
0.8	100	0.02	1500	0.5

value < 0.05 was chosen and all insignificant terms of p value > 0.05 were eliminated. The most significant term of the regression model is interaction term of air flow rate and nozzle diameter (P = 2.76 x 10^{-6}). From the statistics test, F_0 = 65.54 and the critical value $F_{0.05,5,26}$ = 2.59. Since $F_0 > F_{0.05,5,26}$ the null hypothesis is rejected and we can conclude that at least one independent variable contributed significantly to the regression model. This implies that there was a statistically significant dependency of the removal efficiency on the independent variables. The value of R^2 was 0.94. In other words, 94% of the variance in is accounted by the model. Since the adjusted R^2 (0.92) was very close to R^2, all terms in equation 5 were significant. Surface plots of the regression model for various operating parameters are shown in Figure 6.

Optimization problem formation

The removal of toluene in wet scrubber was optimized with respect to objective function and their constraints as given in Table 3. The problem statement aims to maximize the removal efficiency.

Optimization results

The optimization problem was solved using optimization toolbox in Matlab program package that was written in function form of M-file. Constrained optimization routine that consists of starting guess, syntax command, options, lower bound and upper bound is invoked to solve the problem. Results of the optimization are presented in Table 4.

The maximum toluene removal efficiency of 91.7% was predicted from the optimum operating

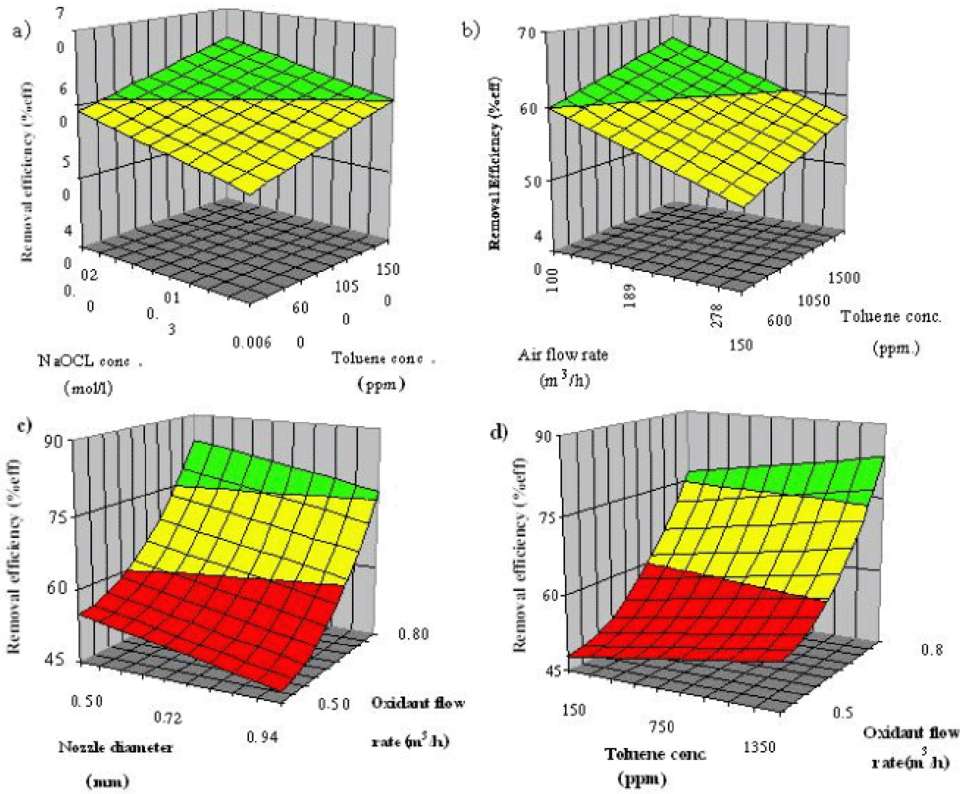

Figure 6. Effect of operating parameters on toluene removal in wet scrubber using NaOCl as oxidative reagent: (a) a surface plot of removal efficiency as a function of toluene and NaOCl concentration, (b) a surface plot of removal efficiency as a function of toluene concentration and air flow rate, (c) a surface plot of removal efficiency as a function of NaOCl concentration and nozzle diameter, and (d) a surface plot of removal efficiency as a function of toluene concentration and oxidant flow rate.

[Color figure can be viewed in the electronic version]

condition as suggested in Table 4. The optimum point indicated to control the wet scrubber at minimum of air flow rate and size of spray nozzle and maximum of oxidant flow rate and toluene concentration. The optimum concentration of NaOCl was 0.02 mol/l since the increase of the NaOCl beyond this point did not improve the toluene removal efficiency as clearly shown in Figure 4.

Conclusion

Wet scrubber coupling with oxidation reaction using NaOCl as oxidizing agent performed effectively in removing toluene from waste air. The steps of design of experiment, surface plotting, model formation, and optimization problem formation and solving were used to study toluene removal in the wet scrubber. A mathematical model of the toluene removal was constructed based on the experimental results. Five operating parameters including toluene concentration, air flow rate, oxidant flow rate, NaOCl concentration, and size of spray nozzle were considered as the significant parameters in the model. Optimization problem was formed with an objective function of maximum removal efficiency and constraints of mathematical model and process limitation.

Minimum control of air flow rate, small size of spray nozzle, maximum control of oxidant flow rate and toluene concentration, and NaOCl concentration of 0.02 mol/l were the optimum conditions of the wet scrubber system. Solution of the optimization showed the highest toluene removal efficiency of 91.7%.

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