J. KOSAE Vol. 24, No. E2 (2008) pp. 83~91
Journal of Korean Society for Atmospheric Environment

Methodology to Simultaneously Optimize the Inlet Ozone Concentration to Oxidize NO and Relative Humidity Composition for the NO_x Degradation using Soil Bio-filter

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(Received 2 October 2008, accepted 10 December 2008)

Abstract

This work investigated the methodology to simultaneously optimize the ozone and relative humidity composition for the NO_x degradation using soil biofilter. Experiments were made as a function of inlet ozone concentration (0 ~ 1,770 ppb) and relative humidity (38 ~ 81%). Factorial design (2^2+3) and response surface methodology by central composite designs were used to examine the role of two factors and optimal response condition on NO_x degradation. It was found that a second-order response surface model can properly interpret the experimental data with an R^2-value of 0.9730 and F-value of 71.83, based on which the maximum NO_x degradation was predicted up to 92.8% within our experimental conditions.

Key words : Soil biofilter, NO_x degradation, Response surface methodology, Central composite designs, Full factorial design

1. INTRODUCTION

As modern times come, humans are spending more than 90% of their time indoors. Indoor air pollution is not only undermining the pleasantness of indoor environment but also threatening the health of residents. This is because the polluted indoor air is not cleaned easily due to the indoor sources of air pollution and inducement of outdoor polluted material (Nararoff and Weschler, 2001; Burge and Hoyyer, 1977). Thus, in order to identify the influence on the body of modern people spending most of their time indoors and provide them with pleasant environments. We must begin by examining the materials contaminating the indoor environment and indoor air quality. The materials contaminating indoor environments should be managed and effective prevention measures are needed. Among the methods to improve indoor air quality, ventilation, the control of the use of indoor burning instruments, the development of air cleaners using catalysts and so on are being researched. Recently, research related to the improvement of indoor air quality using indoor plants and the control of materials contaminating indoor air through bio-filter methods using environmentally friendly filtration materials has been carried out (Cho *et al.*, 2006; Joseph *et al.*, 1999; Matteau and Ramsay, 1997; Leson and Winer, 1991).

The technology using catalysts has good efficiency but also has the problems of the toxic effect of catalysts, high cost to set up the equipment, the de-

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crease in efficiency at high concentration, the replacement of catalysts and so on (Broer and Hammer, 2000; Mok and Nam, 1999). Thus, the technology using environmentally friendly filtration materials to process contaminated material continuously has a very important meaning. The bio-filter method is the technology that lets polluted gases pass through materials such as soil and manure which are very active biologically. This technology was proved to be effective to eliminate volatile organic compounds and odors etc. which are generated in industrial areas and public places. In addition, the merits of biological processing method have been reported to be that it is not only more economical than physical and chemical processing methods in terms of installation and operation costs but also doesn't release secondary contaminated materials (Kim, 1997; Warren and Raymond, 1997; Medina and Devinny, 1995; Speitel and Mclay, 1993).

In the 1980's, the bio-filter method was used to process volatile organic compounds and toxic materials in the air in Germany (Senior and Kasali, 1990). Afterwards, as the serious effects of nitric oxides in the air due to industrialization and the increase in the number of automobiles and so on became evident, nitric oxides were processed using the bio-filter method (Chris *et al.*, 1997; Barnes *et al.*, 1995; Shanmugasundram *et al.*, 1993; Baumgarter and Conrad, 1992).

In the case of domestic areas, a lot of contaminated materials are generated downtown, around main street etc., and these things exert bad influences on the quality of indoor air. Therefore, the development of environmentally friendly filtration materials to reduce contaminated materials induced from outside such as NO, NO_2, SO_2, CO etc. and control contaminated materials generated from indoor environment such as VOCs, HCHO, PM_{10}, Rn etc. is in reality very urgent.

The preceding research was carried out in order to eliminate nitric oxides at the same level found in contaminated indoor air using soil bio-filters. Through this preceding research, an optimal mixing rate of loess, soil, briquette ash, and compost to configure soil-based bio-filters was drawn up (Cho and Lee, 2007). Based on this research, after choosing two parameters for induced ozone concentration to oxidize NO and relative humidity of soil bio-filter as a factor affecting the NO_x degradation, an experiment to draw optimal induced ozone concentration and relative humidity to maximize the NO_x degradation was carried out and the mathematical models using Full factorial analysis, Stepwise regression, and Response surface design were suggested.

2. EXPERIMENT APPARATUS AND METHODS

2.1 Apparatus

The experimental apparatus consist of a gas-providing unit, an ozone-oxidation unit, mixer-tank, reactor unit, and a measuring unit of discharged gases. The soil bio-filter reactor for eliminating NO to be used for this experiment was made as a diameter of 50 mm and to the height of 100 mm.

The gas-providing unit was designed to inject NO gas diluted with air. Mixed gases were set up to flow upward after being injected through the lower end of the filtration materials. The ozone-oxidation unit has the role of oxidizing NO to NO_2 from the injected standard gas using the ozone generator and the NO_2 mixed with the standard gas in the mixer tank and is injected into the bio-filter equipment. To look over the removal rate of contaminated air (NO, NO_2) by the bio-filter, a nitrogen oxides analyzer was used. To measure the concentration of O_3, an O_3 analyzer was used. This equipment used for analysis can send analyzed data every 5s to the automatic successive analysis equipment so that the changes of nitrogen oxides in the bio-filter can be monitored.

2.2 Experimental conditions and procedures

In Table 1, the physical properties of biofilter and the experiment conditions related to the initial concentration of O_3 and NO, flow rate, and relative humidity are shown. The initial injected concentration of NO was maintained around 100 ppb and the initial O_3 injected to oxidize the NO gas was changed within the range of 0 ~ 1,770 ppb and the injected flow

Table 1. Design and operation parameters.

Parameters	Condition of experiment
Initial concentration of NO (ppb)	100 ± 2
Initial concentration of O_3 (ppb)	0~1,770
O_3 gas flow rate (mL/min)	1,000
Total flow rate [Air+NO+O_3] (mL/min)	2,000
Mixture gas residence time (s)	5.89
Moisture content of packing material (%)	38~81
Size of used mixture reactor ($\Phi \times H$)	50 mm × 100 mm
Field capacity (%)	70
pH	5.4
Particle density (g/cm^3)	0.90
Particle size (mm)	2

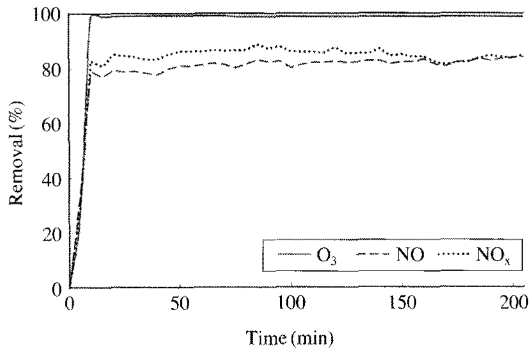


Fig. 1. A typical removal rate of O_3, NO, and NO_x using the soil bio-filter.

rate of O_3 was 1,000 mL/min. The charged content of water put into the soil bio-filter was adjusted within the range of 38~81% and the relative humidity of the injected mixed gases was adjusted to the same level of water content as the soil within the bio-filter and injected into the reactor.

The soil bio-filter in the reactor is charged with a total volume of 196 cm^3. Once the initial injected concentration of O_3 is determined and stabilized using a separate pipe, while maintaining the concentration of initial injected NO as 100 ppb with pure air, the O_3 is injected into the mixer tank and made to react with the NO. And then after injecting the mixed gases (Air+NO gas+O_3 gas) into the reactor, the removal rate can be calculated by measuring the concentration of ejected NO, NO_2, O_3 at the exit of the reactor.

2.3 Analysis

O_3 concentration at the influent and effluent of the reactor was determined by an ozone analyzer with internal and external zero and span gas (Model: ML 9811, Ecotech, Australia). NO, NO_2 and NO_x concentration was determined by a nitrogen oxides analyzer (Model: EC 9841, Ecotech, Australia). This analysis equipment can send data every 5s to the equipment for automatic successive analysis and as for the measuring principles, the chemiluminescence method was used for NO and NO_2. The U.V. Photometric method was used for O_3.

3. RESULTS AND DISCUSSION

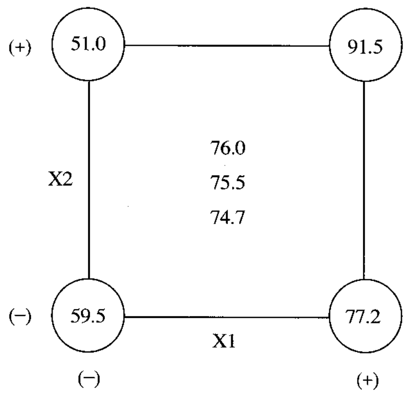
3.1 Nitrogen oxides degradation

As a preceding research to eliminate the nitrogen oxides occuring at the same concentration level of polluted indoor air, the optimal mixing rate of materials for the bio-filter using loess, soil, briquette and compost was configured (Cho and Lee, 2007). Based on the preceding research, the configuration materials in the bio-filter were fixed at a constant ratio, the temperature in the reactor was maintained at a normal temperature and the initially injected concentration of NO gas was set as 100 ± 2 ppb for this study. According to a statistical design of experiments, the concentration of injected ozone to oxidize NO gas was changed in to vary between 0 to 1,771 ppb and the relative humidity of injected mixed gases was controlled within the range of 38% to 81%. In Fig. 1, the typical degradation characteristic of each component according to the elapsed time of this research is shown. As the first stage in the existing research, the response characteristic of NO gas with ozone before being injected into the reactor was reviewed, but this study left out this stage and the injected mixed gases were put into the reactor simultaneously (Cho *et al.*, 2006). We then measured the differences between the inlet and the outlet concentration, and reviewed the degradation characteristics of each gas. This research was focused on identifying the optimal conditions between the concentration of injected ozone to oxidize NO gas and relative humidity, so the omission of the first stage is considered to have no influence on the final results. As shown in Fig. 1, injected ozone to oxidize NO gas was removed at the rate of more than 99% in almost all of the experimental conditions, and the removal rate of NO_x was shown to vary from 36.5 % to 91.5% according to experimental conditions.

3.2 Full factorial analysis

In this study, we investigated the methodology to simultaneously optimize ozone concentration and relative humidity to maximum NO_x degradation by considering the injected ozone concentration to oxidize NO and the relative humidity of mixed gases as factors to influence the removal rate of NO_x. These two factors were examined at two levels. Using full factorial analysis, the two levels can be decided as the lower and the higher level and then the experiment was repeated 3 times at the center point of the experimental design of 2^2+3, this was decided and analyzed by 7 experimental conditions. At that time, ozone and relative humidity were decided and experimented as 200 ppb (−) for the lower ozone level, and 1,500 ppb (+) for the higher ozone level and at 45% (−) for the lower humidity level, and 75% for the higher humidity level respectively. In Fig. 2, the analyzing conditions for full factorial analysis and the removal rate of NO_x according to each condition are shown. Each corner point and center point of the rectangle shows experimental conditions and the higher and lower levels were distinguished as + and − respectively. The upper surface of the rectangle shows higher relative humidity (X2) and the lower surface shows the lower conditions and the right side of the rectangle shows the higher ozone concentration and the left side shows the lower conditions.

Based on the experimental results according to the above condition for analysis, the standardized graph of normal probability distribution is shown in Fig. 3 in order to identify the significant difference of the effects of each factor using the normal probability plot from the estimated value of the main effect and interaction. If there was no significant difference in the effects, the estimated values of the effect tend to be a linear line and the effects with significant difference are located far from the linear line, which means the absolute values are high. As shown in Fig. 3, ozone, in this research, humidity, the inter-



Run	X1	X2	Y
1	(−)	(−)	59.5
2	(−)	(+)	51.0
3	(+)	(−)	77.2
4	(+)	(+)	91.5
5	(0)	(0)	76.0
6	(0)	(0)	75.5
7	(0)	(0)	74.7

Fig. 2. Experimental levels and results of full factorial design (2^2+3, X1: O_3 concentration, X2: relative humidity).

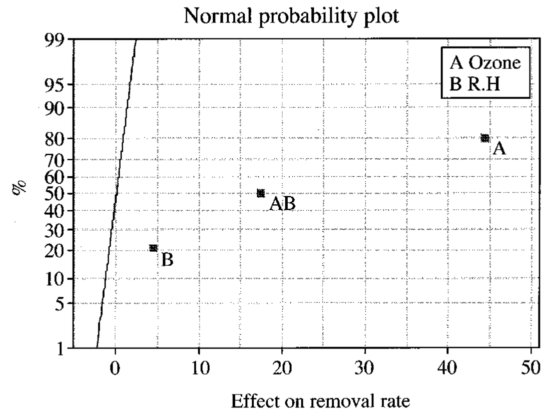


Fig. 3. Normal probability plot.

Table 2. Coefficients of the model [Eq. (1)] for NO_x degradation using the soil bio-filter.

Coefficient	Value	Standard error	T-value	P-value
Constant	69.800	0.3279	212.89	0.000
Ozone	14.550	0.3279	44.38	0.001
R.H.	1.450	0.3279	4.42	0.048
Ozone-R.H.	5.700	0.3279	17.38	0.003
C_tP_t	5.600	0.5008	11.18	0.008
S=0.655744	R^2=99.92%		R^2 (revision)=99.75%	

action of ozone and humidity were all shown to influence NO_x degradation and the most influential factor to NO_x degradation was the ozone concentration to be injected to oxidize NO, and the lowest influential factor was relative humidity.

In Table 2, the analyzed results of model coefficient by T-test are shown and as a result of the analysis, ozone concentration, relative humidity, the interaction of ozone and relative humidity were shown to be significant at the level of 5% and the P value at the center point (*CtPt*) was also shown to be significant at the value of 0.008, as it was less than significant level. The results from regressing model by full factorial design at two levels are represented by Eq. (1).

$$Y=69.8005+14.550\ \text{ozone}+1.450\ \text{R.H.} +5.700\,(\text{ozone}\times\text{R.H.}) \quad (1)$$

As the coefficients of ozone and relative humidity were all positive values, we can know that the greater the positive values of ozone and relative humidity were, the higher removal rate was. But to predict the removal rate with this equation outside of the experimental range can induce errors because the estimated coefficients here were obtained only by 3 levels (minimum, middle, maximum) of each factor. In this research, to solve the above problem, central composite design of response surface methodology was carried out in order to simultaneously optimize the composition of ozone concentration and relative humidity for the maximum NO_x degradation.

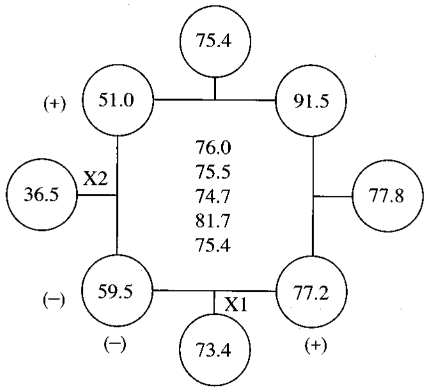
3.3 Response surface methodology

In this study, central composite design of response surface methodology was used to draw the optimal conditions for the concentration of injected ozone and relative humidity in order to maximize the removal rate of NO_x. Response surface methodology is the most widely used statistical technique for bioprocess optimization (Mead and Pike, 1975; Box, 1954; Box and Wilson, 1951). Response surface experiments identify the response of a system as a function of explanatory variables (Annadurai *et al.*, 2002). Central composite design is a method generally used among response surface methodology and is used very often to estimate the 1st and 2nd orders and model curvature reaction variables in further experiments after the experiment by factorial design. As the equation for response surface design is not known generally, the validation of the model equation is evaluated through a lack-of-fit test after deciding a closely estimated model. The mathematical relationship of the response Y and these two variables can be approximated by the quadratic (second order) polynomial equation (Box and Behnken, 1960, 1959):

$$Y=\beta_0+\beta_1 x_{1i}+\beta_2 x_{2i}+\beta_{11} x_{1i}^2+\beta_{22} x_{2i}^2 +\beta_{12} x_{1i}\, x_{2i}+\varepsilon_\varepsilon-N(0, \sigma^2) \quad (2)$$

Central composite design consists of an axial point and the experimental point (corner point) of factorial design including a center point, and if the number of independent variable is k, the number of factorial experimental points becomes 2^k and the number of axial points becomes 2k. So, in this case when the repeated times at the center point is n_c, the total times of experimentation becomes 2^k+2k+n_c. In Fig. 4, the experimental conditions by central composite design and the removal rate of NO_x according to each experimental condition were shown. In this study, using two independent variables of the concentration of injected ozone and relative humidity, the total 13 experiments (4 corner points, 4 axial points and 5 center points) were carried out.

For each parameter and parameter combination, the t-statistics value and the individual significance probability of t-value greater than t are shown in Table 3. As a result of the analysis, 2nd order between relative humidity · relative humidity was shown



Run	X1	X2	Y
8	−1.414	(0)	36.5
9	1.414	(0)	77.8
10	(0)	−1.414	73.4
11	(0)	1.414	75.4
12	(0)	(0)	81.7
13	(0)	(0)	75.4

Fig. 4. Experimental levels and results of central composite design ($2^2+2\times 2+5$, X1: O_3 concentration, X2: relative humidity).

Table 3. Coefficients of the model [Eq. (3)] for NO_x degradation using the soil bio-filter.

Coefficient	Value	Standard error	T-value	P-value
Constant	76.574	1.039	73.689	0.000
x_1	14.576	1.017	14.329	0.000
x_2	1.079	1.017	1.060	0.320
x_1x_1	−8.733	1.082	−8.074	0.000
x_1x_2	5.700	1.439	3.962	0.004
S=2.877	R^2=97.3%		R^2(revision)=95.9%	

to be insignificant and excluded. In Table 3, although P-value of relative humidity was shown to be 0.320 which is higher than the significance level and seems to be insignificant and even though the primary factor was insignificant, it can not be omitted if the interaction factor including the primary factor or the 2nd order was significant. Thus the following equation related to the estimated 2nd response surface shape from the result of this analysis was obtained and it was revealed that injected ozone to oxidize NO had

Table 4. Regression analysis and response surface model fitting (ANOVA) for degradation of NO_x using the soil bio-filter.

Source	Sum of squares	Degree of freedom	Mean squares	F-value	P-value
Model	2378.59	4	594.648	71.83	0.000
Residual	66.23	8	8.279		
Lack of fit	33.62	4	8.404	1.03	0.489
Pure error	32.61	4	8.153		
Total	2444.82	12			
S=2.877		R^2=97.3%		R^2(revision)=95.9%	

a curvature effect.

$$Y=76.574+14.576x_1+1.079x_2-8.733x_1^2+5.7x_1x_2 \quad (3)$$

In Table 4, the result of regression analysis and ANOVA to validate the estimated model equation to discover the optimal concentration of injected ozone and relative humidity to eliminate NO_x using a biofilter is shown. If P-value of lack-of-fit test is smaller than the significance level (α), the chosen mathematical model is considered to lack validation but in this research, the P-value of the lack-of-fit test was 0.489 higher than 5% of the significance level, which shows the chosen 2nd model is proper.

In order to confirm that the model of this analysis was established properly, the review of the assumptions of the model equations was carried out using the graphs of residual normal probability distribution, residual vs. adequacy and residual vs. data order as in Fig. 5. In the above process, the assumptions of any error items of the model were confirmed.

As shown in Fig. 5(a), using the graph of residual normal probability distribution, whether or not the residual deviates significantly from the normal distribution was identified. If residuals were within normal distribution, points would appear close to a linear line. By this graph the assumption that data residuals of response values do not deviate from normal distribution can be verified. In Fig. 5(b), as data of response values in the graph of residuals vs. adequacy were distributed evenly in both the upper side and lower side around 0 (zero), it can be known that evidence to contradiction the assumptions does not exist. In Fig. 5(c) which shows residuals vs. data order to

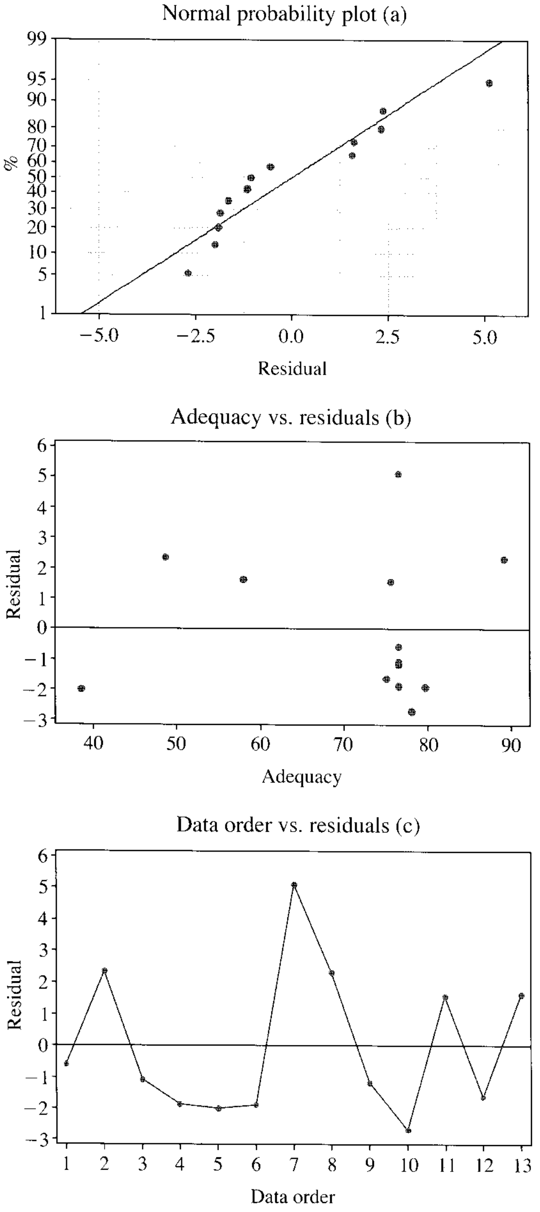


Fig. 5. Residual analysis results (normal probability plot, adequacy, data order).

check residuals are independent and random, if residuals are not influenced by data order, it can be known that the residuals are scattered randomly around 0 (zero) and any evidence that residuals are influenced by time and order in the data of response values

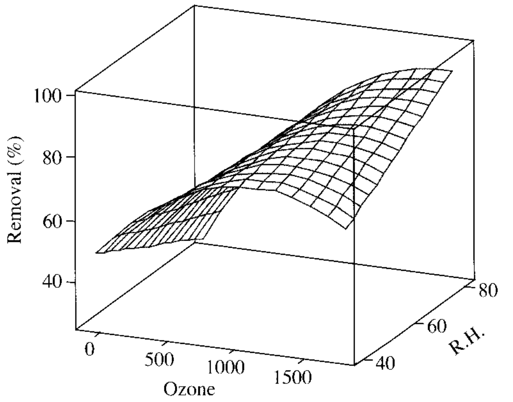


Fig. 6. Response surface optimization of NO_x degradation vs. ozone and relative humidity using the soil bio-filter.

doesn't exist.

In Fig. 6, the graph of response surface optimization of optimal ozone concentration and relative humidity to eliminate NO_x using soil bio-filter was shown. As shown in Fig. 6, when the levels of ozone concentration and relative humidity were high, the removal rate of NO_x was shown to be high. In the case when relative humidity was 81.2% and the concentration of injected ozone was 1691.5 ppb, the maximum removal rate through response surface optimization was estimated 92.8% as the following equation.

$$X_1=([O_3]-850)/650,\ X_2=([R.H.]-60)/15,$$

From, calculated for experimental design,

$$\begin{aligned} Y&=76.574+14.576x_1+1.079x_2-8.733x_1^2 \\ &\quad+5.7x_1x_2 \\ &=76.574+14.576(1.2946)+1.079(1.4133) \\ &\quad-8.733(1.2946)^2+5.7(1.2946)(1.4133) \\ &=92.8\% \end{aligned}$$

However, in the case of using soil bio-filter, as the growth of microorganisms in the soil can influence the elimination to a great extent, under the optimal growth condition for microorganisms where moisture content should be adjusted to around 60%, the review result according to the model type it was estimated that the removal rate was about 82.6% at

1,381 ppb in ozone concentration and 60% in relative humidity. To carry out the validation test of the estimated value, a replication test under the same conditions of 1,381 ppb of the concentration of injected ozone and 60% of the relative humidity was performed. A very close experimental result to the estimated value by the mathematical model, the removal rate of nitrogen oxides was shown to be 81.5% and the mathematical model used in this experiment was a very useful tool to explain the experiment result.

Afterwards, based on the result of this research, we are planning to draw the optimal response time of injected gases and soil bio-filter, the correlation with the concentration of injected NO gases, the optimization of the injected amount of ozone to oxidize NO and the concentration of injected NO gases etc., in order to maximize NO_x degradation.

4. CONCLUSIONS

This research had the purpose of suggesting a mathematical model by finding the optimization of the concentration of ozone and relative humidity in order to maximize the NO_x degradation by using soil bio-filter. In this study, we used full factorial analysis and response surface analysis which are useful to optimize the concentration of injected ozone to oxidize NO gas and the relative humidity of both mixed gases injected into the bio-filter and within the bio-filter.

In this study, central composite design among response surface analysis was used to draw the optimization and as a result of this research, it was shown that ozone concentration, relative humidity, and the interaction of ozone concentration · relative humidity were all influential to the removal rate of NO_x, and the most influential factor to the NO_x degradation was the concentration of injected ozone to oxidize NO.

The optimization result of ozone concentration and relative humidity to remove NO_x using central composite design was that when the levels of concentration of injected ozone and relative humidity were high, the removal rate of NO_x was high. And the maximum removal rate of NO_x was estimated to be 92.8% as a response surface optimization under 81.2% for relative humidity and 1691.5 ppb of ozone concentration. In the process, the following mathematical model was obtained:

$$Y=76.574+14.576x_1+1.079x_2-8.733x_1^2 +5.7x_1x_2$$

As a result of review by the model where moisture content was adjusted to 60% as the optimal growth condition of microorganisms, the removal rate of NO_x was estimated to be about 82.6% under 1,381 ppb of ozone concentration and 60% of relative humidity. To confirm the validation of this estimated value, when the replication test was performed under the above same conditions, the removal rate by the experiment was shown to be 81.5% which is similar to the estimated value by mathematical model. Therefore we can conclude that the mathematical model used in this research is valid.

Afterwards, based on the result of this research, the mathematical models related to discover the optimal response time of injected gases and soil bio-filter, the correlation with the concentration of injected NO gases, the optimization of the amount of injected ozone and the concentration of injected NO gases etc. will be drawn up.

ACKNOWLEDGEMENTS

The work presented in this paper was supported by the DongNam Health College Funding.

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