Photocatalytic Oxidation of Indoor Air Volatile Organic Compounds (VOCs) in ppb Level

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Abstract

This study evaluated the technical feasibility of the application of titanium oxide (TiO_2) photocatalysis for the removal of VOCs in low ppb concentrations commonly associated with non-occupational indoor air quality issues. A series of experiments were conducted to evaluate four parameters (relative humidity (RH), hydraulic diameter (HD), photocatalytic oxidation (PCO) reactor material (RM), and inlet port size (IPS) of PCO reactor) for the PCO destruction efficiencies of the selected target VOCs. None of the target VOCs presented significant dependency on the RH, which is inconsistent with a few previous studies. However, it is noted that the three parameters (HD, RM and IPS) should be considered for better VOCs removal efficiencies for the application of TiO_2 photocatalytic technology for cleansing non-occupational indoor air. The PCO destruction of VOCs at concentrations associated with non-occupational indoor air quality issues can be up to nearly 100%. The amount of CO generated during PCO would be negligible in comparison to the indoor CO levels. These results can make the PCO reactor an important tool in the effort to improve non-occupational indoor air quality.

Key words : Indoor air, Parameter, Photocatalytic oxidation, Titanium oxide, VOCs

1. INTRODUCTION

Non-occupational indoor air quality has caused increasing concern in the community because individuals spend large portions of their time in the indoor environments. For those compounds having indoor sources, the indoor exposures can be high. VOCs are well known to have their indoor sources, e.g., cigarette smoke, building materials, furnishings, cleaning compounds, dry cleaning agents, paints, glues, cosmetics, textiles and combustion sources (Schneider et al., 1999; Wallace et al., 1989; Higgins et al., 1983). They have received significant attention mainly due to their toxicity (U.S. EPA, 1990; Tancrede et al., 1987) and prevalence in indoor air (Chan et al., 2002; Ilgen et al., 2001; Heavner et al., 1995; Wallace et al., 1991). This characteristic of indoor VOCs warrants the develop-

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ment of control strategies to non-occupational indoor exposures in order to minimize health risks.

It has been reported that the use of ultraviolet (UV) illuminated titanium dioxide (TiO$_2$) catalytic surface can result in the overall reduction of VOCs in air (Cho et al., 2001; Kim et al., 2001; Lee et al., 2001; Jeong et al., 2000; Park et al., 2000; Stevens et al., 1998; Anderson et al., 1996; Jacoby et al., 1996; Obee and Brown, 1995; Peral and Ollis, 1992). However, these previous studies on photocatalytic oxidation (PCO) of VOCs have dealt primarily with concentrations in the low to high ppm range, which is more typical of chemical process stream concentrations than that associated with indoor air quality (IAQ). Extrapolation of oxidation performance data collected at concentra-tions much higher than the intended application may not be valid (Obee and Brown, 1995). Even though typical indoor air VOC levels were higher than typical ambient air VOC levels, they are still in low-ppb levels (Chan et al., 2002; Ilgen et al., 2001; Heavner et al., 1995; Wallace et al., 1991). Accordingly, this study was conducted to establish the technical feasibility of application of TiO$_2$ PCO technology for cleansing air aromatics in the low ppb (≤ 100 ppb) concentration range commonly associated with indoor air quality issues. The target compounds that are particularly abundant in the non-occupational indoor environment were five aromatic VOCs (benzene, ethyl benzene and o, m, p-xylenes) (Chan et al., 2002; Ilgen et al., 2001; Heavner et al., 1995; Wallace et al., 1991). These VOCs are known or suspected carcinogens, which have no safety threshold dose for the carcinogenic effects, so that any exposure, however small, poses a finite risk (USEPA, 1990; Tancrede et al., 1987).

The evaluation parameters included relative humidity (RH), hydraulic diameter (HD), PCO reactor material (RM), and inlet port size (IPS) of PCO reactor. Humidity is an important variable since photocatalytic technology should be applicable to the wide humidity range encountered in real indoor environments. The influence of water vapor on the efficiency of the PCO air treatment process has not been clearly discovered.

Widely differing effects of water vapor have been reported (Obee and Brown, 1995; Murabayashi et al., 1994; Weedon, 1994; Suzuki, 1993; Peral and Ollis, 1992; Ibusuki and Takeuchi, 1986). Most previous researches investigated pollutant levels well above 1 ppm. The effect of HD on PCO destruction efficiency was tested since stream flow rate (FR) and UV intensity on the reactor surface that are important parameters (Obee and Brown, 1995) vary with the HD of the PCO reactor. Here, HD is defined as the inside diameter of the annular reactor tube minus the outside diameter of the lamp. Two parameters also, RM and IPS, are possible parameters that influence on the PCO destruction efficiency.

2. EXPERIMENTAL

2. 1 Experimental apparatus

The schematic of the experimental apparatus is presented in Figure 1. The PCO reactors used in this investigation had annular geometries. A reactor consisted of a glass tube coated on the inner surface with a thin film of TiO$_2$ photocatalyst. A cylindrical UV light source was inserted inside the glass tube and served as the inner surface of the annular reactor. The gas was flowed through the annular region. This design is well adapted to the research environment because it provides a well-characterized reactive catalyst surface along the length of the reactor body and allows uniform light distribution (Stevens et al., 1998; Jacoby et al., 1995). Moreover, the reactor was designed to direct the flow of incoming air normally to the UV light to increase air turbulence inside the reactor, thus enhancing distribution of target compounds onto the catalytic surface of the reactor. The application of a thin and uniform TiO$_2$ coating on the inside of the glass reactor tubes was essential. The Pyrex surface was coated using 20% Degussa P-25 slurry. The coated reactor was dried for an hour at room tempera-ture and then baked for 30 minutes at 450°C. The humidity levels were adjusted by passing zero-grade air through
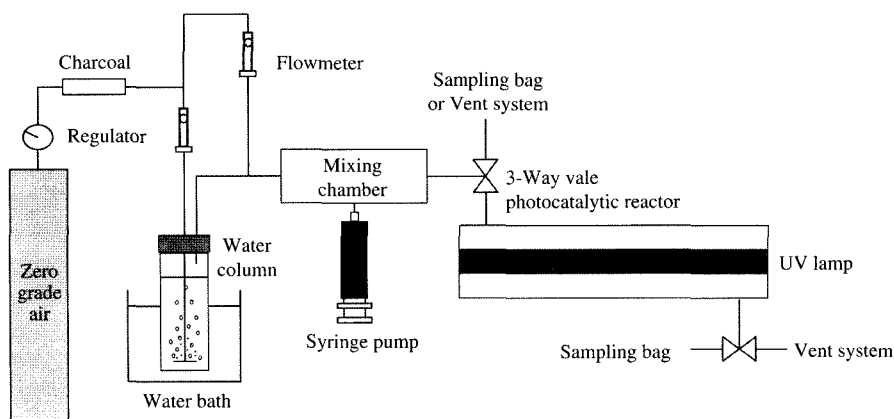
Fig. 1. Schematic diagram of the experimental set-up.

a charcoal filter and then, a humidification device in a water bath (Cole-Parmer HAAKE W26). RH was measured just prior to the PCO reactor inlet using a humidity meter (Thermo Recorder TR-72S, T & D Co.). Measurements of flow rate were carried out using identical rotameters $(0 \sim 10 \ dm^3 \ minutes^{-1})$ which had been calibrated by a dry test meter (URG 3000-020C). The desired concentrations were obtained in a mixing chamber by the use of a syringe pump (KdScientific Model 210). The UV radiation was supplied by an 8-W fluorescent black light (SANKYO DENKI F8T5/BLB) with a maximum spectral intensity at 352 nm.

2. 2 Experimental parameters

Parameters tested for the current study included humidity, HD, RM, and IPS. The humidity in this experiment varied $10 \sim 100\%$ ($10 \sim 20$, $40 \sim 60$ and $80 \sim 100\%$) covering dry and wet environments. The humidity level was incrementally increased from low to high to cover the range shown. Two different hydraulic diameters (5.0 and 20.0 mm) of tubular reactor were also tested. The residence time (5 s) was fixed for 5 seconds throughout every experiment. The range of air FR investigated was 0.4 to 2.3 $dm^3 \ minutes^{-1}$ and is comparable to that previously reported by other researchers (Stevens et al., 1998; Obee and Brown, 1995). Two RMs (stainless steel (SS) and glass) and two IPSs

Table 1. Representative operating parameters.[a]

Parameter	Representative value
RH, %	$40 \sim 60\%$
HD, mm	20.0
FR, $dm^3 \ min^{-1}$	2.3
Reactor residence time, sec	5.0
Contaminant concentration, ppb	
Benzene	34
Ethyl benzene	10
m, p-Xylene	32
o-Xylene	4
UV light source	8-W fluorescent black light
RM	Glass
Reactor IPS, mm	4.0
UV light intensity, $mW \ cm^{-2}$	3.8
Weight of catalyst film, $mg \ cm^{-2}$	0.5

[a]RH is within the ASHRAE comfort range ($40 \sim 60\%$); residence time is calculated by dividing the reactor volume by flow rate; contaminant concentration is nearly the maximum value of the concentration ranges for each target compound associated with indoor air quality issues (Wallace et al., 1989 & 1991); and the UV light source provides UV intensities which are expected to be employed in a practical photocatalytic purifier (Stevens et al., 1998; Jacoby et al., 1996).

(2 and 4 mm inside diameter (id)) also were tested focusing on the PCO destruction efficiency. For each parameter test, the other parameters were all fixed to the their representative values (Table 1).

The representative operating parameters are summarized in Table 1. RH is within the ASHRAE comfort range ($40 \sim 60\%$). The HD (20.0 mm) and the FR

(2.3 dm^3 min^{-1}) employed in the present study provide a residence time of 5 s. The residence time was calculated by dividing the reactor volume by FR. The concentration of pollutants is nearly the maximum value found in the non-occupational indoor air levels (Chan et al., 2002; Jo and Oh, 2001; Baek et al., 1997; Heavner et al., 1995). The UV light source provides UV intensities that are expected to be employed in a practical photocatalytic purifier (Stevens et al., 1998 Jacoby et al., 1996). The UV radiation intensities measured in the present study were 5.8 and 3.8 mW cm^{-2} for the 5.0 and 20.0 mm HDs, respectively. The UV light intensity was measured at the distance from the UV lamp equal to half of the hydraulic diameter of a reactor using a Black-Ray radiometer (Model J-221). The weight of the TiO$_2$ film coated inside reactor was about 0.5 mg cm^{-2}. Mixture of test VOCs was introduced into the PCO reactor.

2. 3 Procedure controls

An individual test was carried out as follows: first, the air flows and humidity levels are set. After the rector outlet humidity levels reach equilibrium, the PCO reactor is pretreated for several hours, while the humidified zero-grade air is flowing through the illuminated PCO reactor. After flushing out the reactor out by dry air, test VOCs is introduced. After adsorption process between the titania catalyst and the VOCs has reached equilibrium as indicated by equality between the inlet and outlet VOC concentrations, the UV lamp is turned on (Obee and Brown, 1995). The present procedure includes a series measurements of VOC concentrations at the outlet of the PCO reactor prior to and after turning on the UV lamp. In addition, carbon monoxide (CO) concentrations are measured at the PCO reactor inlet and outlet during the course of this experiment in order to examine if this PCO application can create a significant addition to indoor CO levels.

In order to determine PCO conversion efficiency, E (%), the following equation was employed:

$$E = (C_o - C_i) \times 100/C_o \qquad (1)$$

where C$_i$ is the reactor inlet contaminant concentration at equilibrium state and C$_o$ is the reactor outlet concentration at photocatalytic steady state. The outlet concentrations were calculated by averaging the four outlet concentrations at photocatalytic steady state.

2. 4 Sampling and analysis

Target VOCs in the air stream were collected by filling an evacuated 5 L Tedlar bag at a constant flow rate. Air from this bag was then drawn through a 0.64-cm-outside diameter (o.d.) and 18-cm-length SS sorbent trap containing 0.5 grams of Tenax TA using a constant flow-sampling pump (A.P. Buck Inc. Model I.H). Sampling times varied from one to five minutes depending on the flow rate. All samples were taken at ambient room temperature (18~26°C).

The VOCs collected on the Tenax TA trap were analyzed by coupling a thermal desorption system (TDS, Tekmar Model Aerotrap 6000) to a gas chromatograph (GC, Varian 3400CX) with a flame ionization detector (FID) using a 0.32-mm-i.d. by 60-m length fused silica column (Supelco Co. SPB-5). Next, the adsorbent (Tenax TA) trap was thermally desorbed at 250°C for 10 minutes, and the target compounds concentrated at (120°C on a cryo trap (15.2-cm-length, 0.32-cm-o.d. tube packed with glass beads). The cold trap was rapidly heated to 250°C, flushed to the Cryofocusing Module (CM) of the TDS and then cooled to −120°C to re-concentrate the target compounds. The CM was then heated to 225°C and flushed to transfer the target compounds to a GC. The initial oven temperature was set at 35°C for five minutes and ramped at 4°C minutes^{-1} to 200°C for five minutes.

The quality control (QC) program included laboratory blank traps and spiked samples. At the beginning of the day, a laboratory blank trap was analyzed to check for any trap contamination; however, no trap contamination was identified. An external standard was analyzed daily to check the quantitative response. The method detection limits ranged from 0.2 to 0.7 ppb for the target VOCs. Meanwhile, CO concentrations were measured using portable monitors equipped with a data

logging system (CMCD-10P, GASTEC Co.). The QC procedure for CO measurements included a daily check of zero and span. The procedure was done with the monitors powered by a primary electrical outlet instead of using charged batteries. The instrumental detection limit was 0.1 ppm for CO.

3. RESULTS AND DISCUSSION

A series of experiments was conducted to evaluate four parameters (RH, HD, RM, and IPS) for the PCO destruction efficiencies of the selected target VOCs. The experiments were repeated three times for each experimental condition and the repeated experiments showed very similar patterns. Thus, average values for all experimental conditions are presented in this paper. The VOC concentrations shown at 'zero' minute in the figures indicate the inlet VOC concentrations. A series of VOC concentrations measured prior to turning on the UV lamp shows that for all experimental conditions the adsorption process reached equilibrium within 30 minutes for all target VOCs as indicated by the equality between the inlet/outlet pollutant concentrations. The outlet VOC concentrations sharply dropped when the UV lamp was turned on, primarily due to the PCO process (Obee and Brown, 1995; Peral and Ollis, 1992). Moreover, a series of VOC concentrations measured after turning on the UV lamp indicates that the outlet concentrations of the PCO reactor reached a steady state within 30 minutes after the UV lamp was turned on for all target VOCs. Accordingly, it is the steady-state result that is discussed for the destruction efficiencies of the target VOCs here. The destruction of VOCs takes place through reactions with molecular oxygen or through reactions with hydroxyl radicals and super-oxide ions formed after the initial production of highly reactive electron and hole pairs when TiO_2 is UV-irradiated as follows (Stevens et al., 1998; Anderson et al., 1996; Jacoby et al., 1996; Obee, 1996; Obee and Brown, 1995; Peral and Ollis, 1992):

$$OH^- + h^+ \rightarrow \cdot OH \qquad (2)$$

$$O_2 + e^- \rightarrow O_2^- \qquad (3)$$

3. 1 RH effects

The humidity effects were tested on the PCO efficiency employing three RH ranges (10~20, 40~60 and 80~100%) that cover dried and humidified environments. Figure 2 exhibits the VOC concentrations measured prior to after turning on the UV lamp for the different RH ranges. For each RH range, the inlet VOC concentrations measured prior to turning on the UV lamp were compared with the outlet VOC concentrations measured after turning the UV lamp on in order to evaluate the PCO destruction efficiencies of the target VOCs. None of the target VOCs presented significant dependence on the RH (Table 2). The current results are inconsistent with the Obee and Brown results (1995) that reported that under conditions of low humidity (ca. 8% RH) and a high toluene inlet level (ca. 2,130 ppb), there was a drop in the PCO efficiency with decreasing humidity. They proposed that the drop in PCO efficiency was probably due to a decrease in the hydroxyl radical population on the catalyst surface. They reported an increase in the PCO efficiency with increasing humidity under conditions of high humidity (>80% RH) and high toluene levels (ca. 8,000 ppb). At moderate to high humidity (>20% RH) and low toluene levels (ca. 500 ppb) they found a decrease in

Table 2. PCO conversion efficiency (%) of all target VOC according to experimental condition.

Compound	RH[a], %			HD[b], mm	RM[c]	IPS[d], mm
	10~20	40~60[a]	80~100	5.0	SS	2.0
Benzene	68	71	67	99	79	77
Ethyl benzene	79	81	80	99	88	86
m, p-Xylene	84	82	83	97	87	88
o-Xylene	75	79	80	99	85	85

[a] All experimental conditions shown in Table 1 except RH were employed: HD, 20 mm; RM, glass; and IPS, 4.0 mm.
[b] HD employed is 5.0 mm; RH range employed for the tests of RM and IPS was 40~60%, which is within the ASHRAE comfort range.
[c] RM employed is SS; RH range employed for the tests of RM and IPS was 40~60%, which is within the ASHRAE comfort range.
[d] IPS employed is 2.0 mm; RH range employed for the tests of RM and IPS was 40~60%, which is within the ASHRAE comfort range.
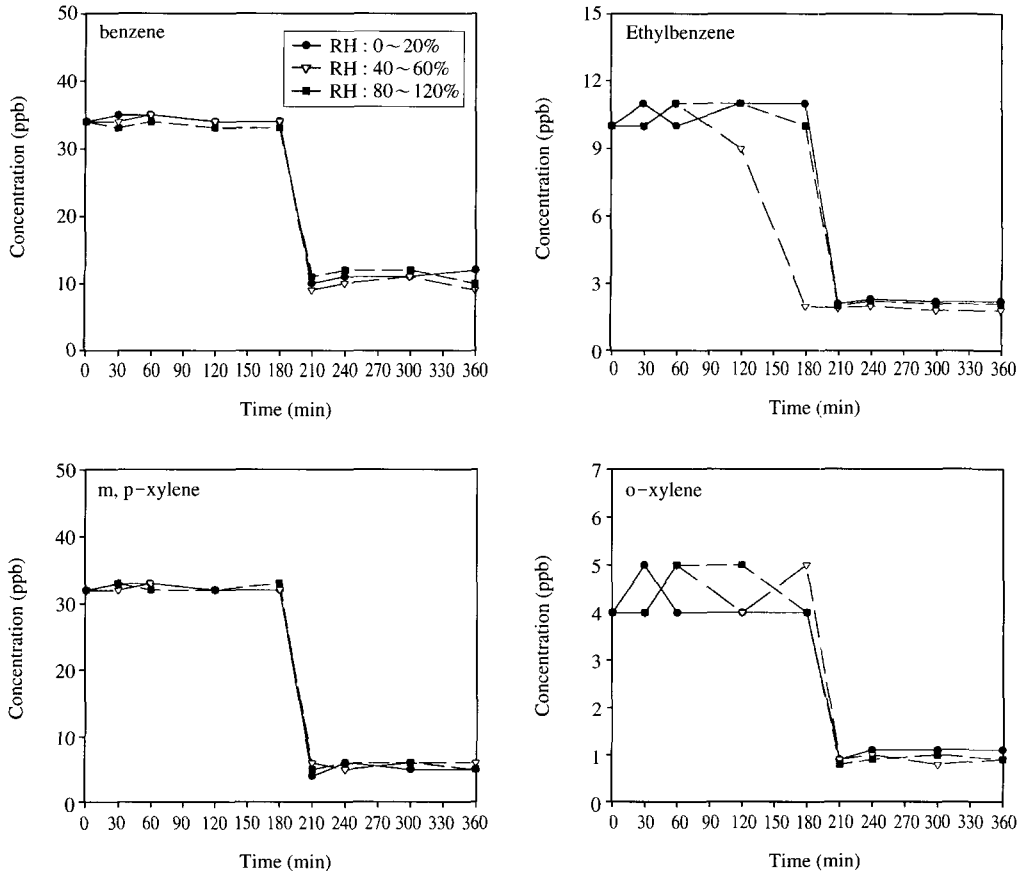
Fig. 2. Concentrations of benzene, ethylbenzene, m, p-xylene, and oxylene measured prior to and after turning on UV lamp for three different RH ranges. UV lamp was turned on at 181 min.

the PCO efficiency with increasing humidity. This PCO rate dependence on humidity was also reported for m-xylene by Peral and Ollis (1992). The previous studies explained that the PCO rate dependence on humidity is likely the result of competitive adsorption between water and the contaminant on the catalyst surface. However, under conditions of the present study there were no recognizable effects of competitive adsorption of water and target compounds or hydroxyl radical population on the PCO destruction efficiencies. As indicated by Obee and Brown's results (1995), the PCO destruction efficiencies would be a reflection of the combined effects of RH and inlet concentrations. Thus, the difference of RH dependence on PCO de-

struction efficiencies between the present study and the two previous studies might be due to inlet concentration differences, although further research is needed to confirm this.

3. 2 HD effects

Two reactors with different HDs (5.0 and 20.0 mm) were tested for PCO efficiencies of the target compounds. Figure 3 displays the VOC concentrations measured during the course of the experiments using the reactors. To avoid the reactor residence time effect on destruction efficiencies, these tests were conducted using the same residence time (5 s) for the three reactors by adjusting the stream flow rates to 0.4 and 2.3
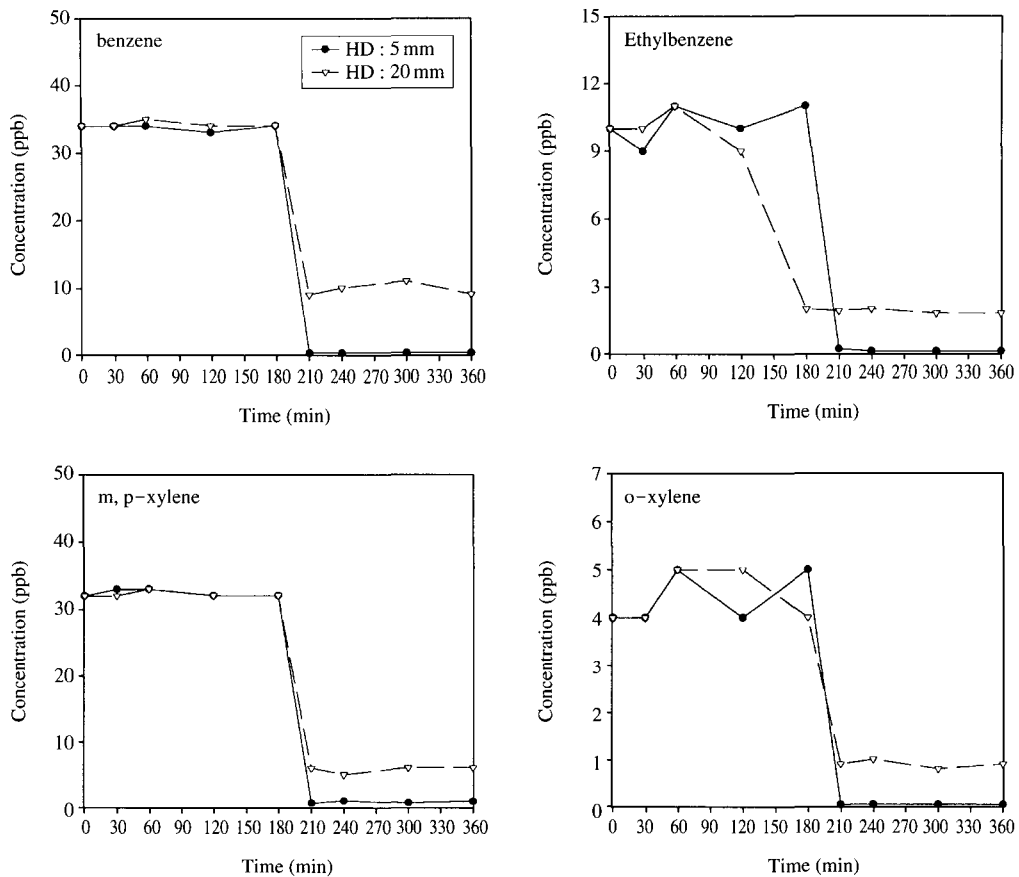
Fig. 3. Concentrations of benzene, ethylbenzene, m, p-xylene, and oxylene measured prior to and after turning on UV lamp for two reactors with different HD. UV lamp was turned on at 181 min.

dm³ minutes⁻¹ for the 5.0 and 20.0 mm HDs, respectively. For the reactor with a 5.0 mm hydraulic diameter, the destruction efficiencies were close to 100% (Table 2). This indicates that the PCO technology can be applied to non-occupational indoor air cleaning with high efficiency. The current results are consistent with the Stevens *et al.* results (1998) that reported complete, steady-state conversions of lower concentrations of carbonyl compounds. However, the destruction efficiency decreased as the hydraulic diameter increased: the destruction efficiency ranged from 71 to 82% for the reactor with a 20.0 mm HD, depending on the compound (Table 2). Accordingly, it is indicated that the hydraulic diameter of the PCO reactor is an

important parameter for the application of TiO₂ photocatalytic technology for cleansing indoor air. As the distance of the catalyst from the light source increases with increasing hydraulic diameter the decrease in light intensity appears to be the most obvious reason for the drop in the PCO of the VOCs. The UV radiation intensities measured in this study were 5.8 and 3.8 mW cm⁻² for the 5.0 and 20.0 mm HDs, respectively. The effect of UV radiation intensities is supported by Obee and Brown's study (1995), which reported that the oxidation rate of toluene and formaldehyde increased with an increase in UV intensity. In addition, Peral and Ollis (1992) reported the same results in the PCO study of acetone, 1-butanol, butyraldehyde, formaldehyde and

m-xylene. In the present study, the flow rate was increased with increasing reactor volume to give the same residence time. Therefore, mass transfer would also increase for the larger HDs. However, the current results shows that the mass transfer effects are not as important as the light intensity effects on the PCO efficiency of the VOCs.

3. 3 RM and IPS effects

The RM and IPS were tested for the PCO destruction efficiencies of the target compounds. Figure 4 shows the VOC concentrations measured prior to after turning on the UV lamp for the different RM (glass and SS). The destruction efficiencies were higher for SS

than for glass for all target VOCs (Table 2). They ranged from 79 to 88% for SS, whereas for glass 71 to 82%, depending on the compounds. One possible explanation for this difference is the effectiveness of UV light use in the PCO reactor. It is likely that certain portion of UV light is transmitted through the glass reactor surface, although the present study did not attempt to quantify this. Accordingly, the higher effectiveness of UV light use in the SS PCO reactor would cause elevated PCO destruction efficiencies compared to the glass PCO reactor.

Figure 5 presents the VOC concentrations measured prior to after turning on the UV lamp for the different IPS (2 and 4 mm i.d.). The destruction efficiencies
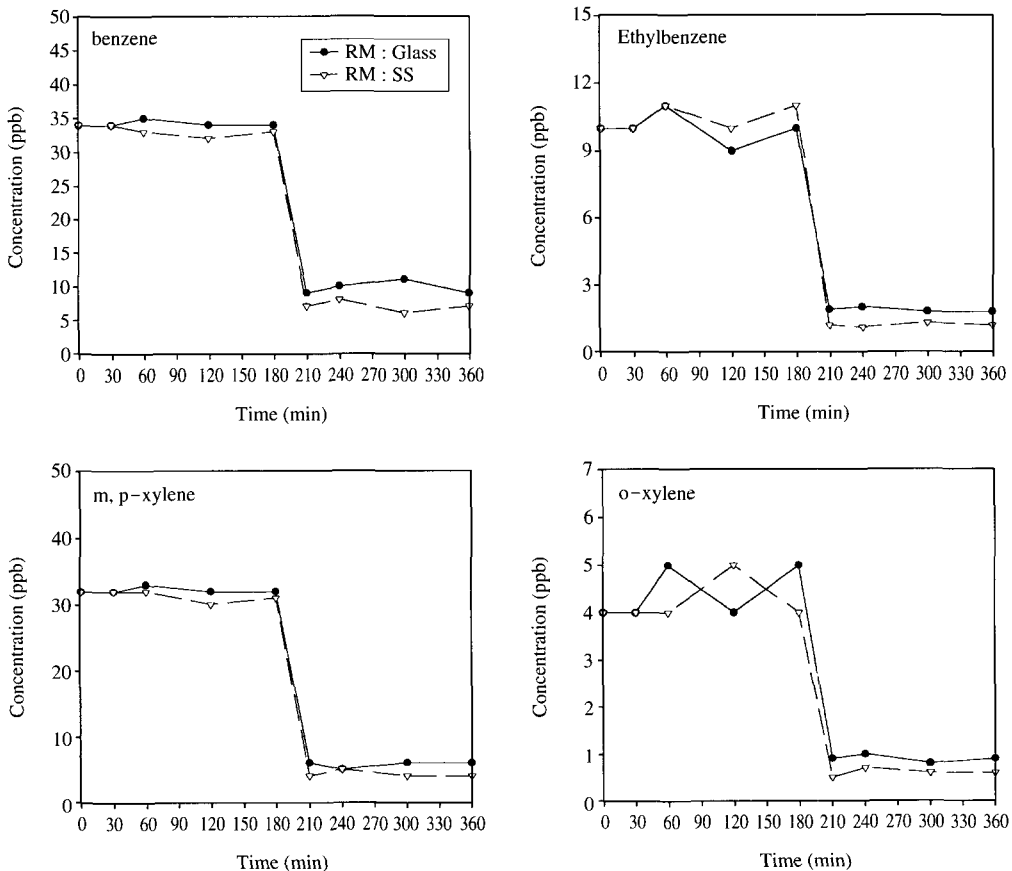

Fig. 4. Concentrations of benzene, ethylbenzene, m, p-xylene, and oxylene measured prior to and after turning on UV lamp for two reactors with different RM. UV lamp was turned on at 181 min.
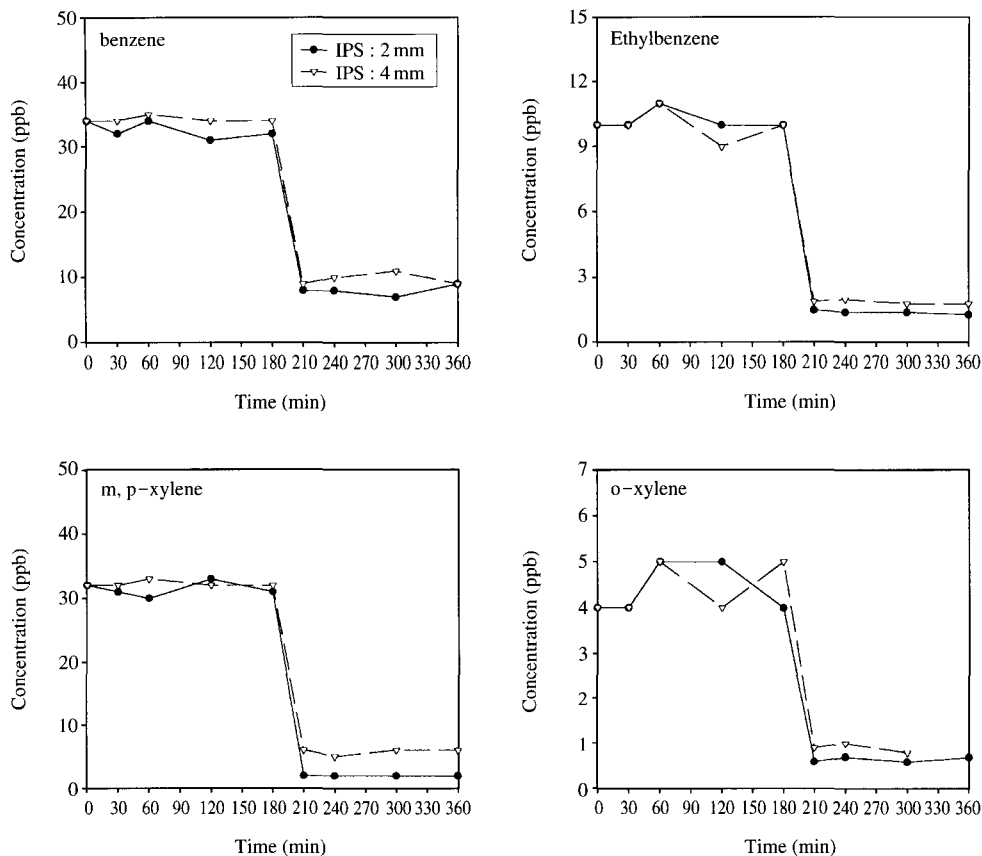
Fig. 5. Concentrations of benzene, ethylbenzene, m, p-xylene, and oxylene measured prior to and after turning on UV lamp for two reactors with different IPS. UV lamp was turned on at 181 min.

were higher for the 2 mm-IPS than for the 4 mm-IPS for all target VOCs (Table 2). They ranged from 77 to 88% for the 2 mm-IPS, whereas for the 4 mm-IPS glass 71 to 82%, depending on the compounds. For the smaller IPS (2 mm i.d.), more fast flow of incoming air increases air turbulence inside the reactor, thus enhancing distribution of target compounds onto the catalytic surface of the reactor. Then, the enhanced VOCs distribution in the smaller IPS reactor would elevate PCO destruction efficiencies compared to the larger IPS reactor.

3. 4 CO levels at PCO reactor outlet

CO is generated during the course of PCO process (Jacoby et al., 1996). The CO concentrations were

measured simultaneously with the VOC concentrations at the PCO reactor outlet during the course of the four tests previously described in this study (humidity, HD, RM and IPS tests). Figure 6 shows average values for each test. The CO concentrations shown at 'zero' minute in the figure indicate the inlet CO concentrations. No CO adsorption on the catalyst surface occurred during the course of the first 180 minutes (prior to UV irradiation) as indicated by the equality between the inlet/outlet concentrations. In addition, as with the VOC concentrations, the CO concentrations measured at the outlet of the PCO reactor reached a steady state within 30 minutes after the UV lamp was turned on as indicated by the equality among a series of the outlet concentrations.
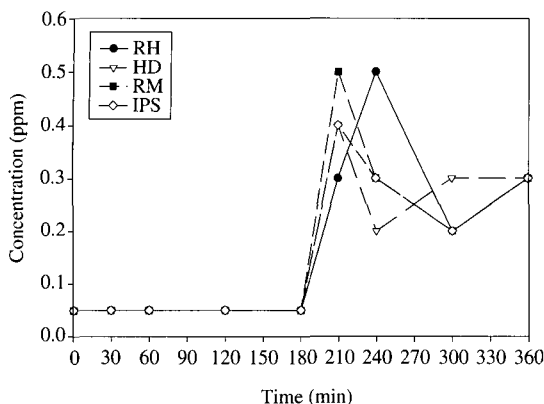
Fig. 6. Variation of CO concentrations at the PCO reactor outlet during the course of four tests (RH, HD, RM, and IPS tests). UV lamp was turned on at 181 min. The CO concentrations measured prior to turning on UV lamp were all below IDL and thus, they were set equal to 1/2 of IDL.

As shown in Figure 6, the CO levels measured after turning the UV lamp on were somewhat elevated compared to those measured prior to turning the UV lamp on. This indicates that some CO was generated through the oxidation reaction of surface-bound reactants and adsorbed intermediates, illustrating the function of the PCO reactor as a self-cleaning filter with respect to adsorbed organic compounds. The current results are consistent with those of a previous study (Jacoby *et al.*, 1996), which reported that some portion of benzene was converted to CO. Nevertheless, considering that the occupational safety and health administration (OSHA) eight-hour exposure limit for CO in the workplace is 35 ppm, under conditions relevant to the use of PCO, these elevations of CO concentrations (maximum 0.5 ppm) would be a negligible addition to the indoor CO levels. Moreover, certain CO, along with VOCs can be destroyed through the PCO process (Vorontsov *et al.*, 1997; Linsebigler *et al.*, 1996; Damme and Hall, 1981). Jacoby *et al.* (1996) reported that the primary product observed during the PCO process of benzene of 116 ppm was carbon dioxide (CO_2). In addition, several previous studies (d'Hennezel *et al.*, 1998; Blanco *et al.*, 1996; Fu *et al.*, 1995; Obee

and Brown, 1995) reported that the PCO process of many VOCs did not form any detectable by-products in the reactor effluent. Accordingly, the low CO generation observed in the present study indicates that the target VOCs would mostly be mineralised to carbon dioxide during the course of the PCO process.

4. CONCLUSIONS

This study evaluated the technical feasibility of application of TiO_2 photocatalytic technology for cleansing VOCs in the low ppb concentrations commonly associated with non-occupational indoor air quality issues. Within the conditions of the present study, the results clearly demonstrate that (1) the destruction of VOCs was independent of humidity, but dependent on the HD, RM, and IPS; (2) the PCO destruction of VOCs at concentrations associated with non-occupational indoor air quality issues can be up to nearly 100%; and (3) the amount of CO generated during PCO would be a negligible addition to the indoor CO levels. These abilities, combined with operational attributes such as a low pressure drop across the reactor and ambient temperature operation, can make the PCO reactor an important tool in the effort to improve non-occupational indoor air quality. However, it is noted that the three parameters (HD, RM and IPS) should be considered for better VOC removal efficiencies for the application of TiO_2 photocatalytic technology for cleansing non-occupational indoor air.

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