

Photocatalytic destruction of VOCs for in-vehicle air cleaning

Wan-Kuen Jo^{a,*}, Jung-Hoon Park^a, Hee-Dong Chun^b

^a Department of Environmental Engineering, Kyungpook National University, Sankeok-dong, Bukgu, Taegu 702-701, South Korea

^b Environment and Energy Research Center, Research Institute of Industrial Science and Technology, Hyoja-Dong, Nam-Ku, Pohang, Kyungpook 790-330, South Korea

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Abstract

In order to reduce vehicle occupants' exposures to aromatic volatile organic compounds (VOCs), it is necessary to develop control strategies for in-vehicle VOCs. This study evaluated the technical feasibility of the application of TiO₂ photocatalysis for the removal of VOCs present in air at low parts per billion (ppb) concentrations commonly associated with in-vehicle air quality issues. The photocatalytic removal of five target VOCs was investigated: benzene, ethyl benzene, and *o*-, *m*-, *p*-xylenes. Variables tested for the current study included relative humidity (RH), hydraulic diameter (HD) and flow rate (FW). The fixed parameters included contaminant concentration, UV light source, photocatalytic oxidation (PCO) reactor material and the weight of TiO₂. Under the experimental conditions, the PCO destruction efficiencies were close to 100% for four different RH ranges that cover typical ambient air humidity ranges. The efficiency of PCO was dependent on the HD of the reactor and the stream FW. Some carbon monoxide was formed as a by-product in the oxidation albeit at low levels. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Aromatic VOCs; Humidity; Hydraulic diameter; In-vehicle cleaning; Photocatalytic destruction; Stream flow rate

1. Introduction

The vehicle cabin has recently been recognized as an important microenvironment that can lead to personal exposure to many volatile organic compounds (VOCs). Most VOCs are known or suspected carcinogens [1] such as benzene, while some are associated with acute effects [2,3]. Several studies [4–9] have found that individuals are exposed to elevated levels of 5–24 VOCs while commuting as compared to activities in which gasoline is not used. Some of the previous studies reported that in-vehicle concentrations of gasoline-derived VOCs were up to eight times higher than the corresponding ambient air nearby monitoring sites.

The elevated in-vehicle VOC levels warrant the development of control strategies to exposure. It has been reported that the use of UV illuminated titanium dioxide (TiO₂) catalytic surface can result in the overall reduction of VOCs in air [10–15]. However, previous studies on photocatalytic oxidation (PCO) of VOCs have dealt primarily with concentrations in the low to high parts per million (ppm) range, which is more typical of chemical process stream concentrations than that associated with indoor air quality (IAQ) [12,14–16]. Extrapolation of oxidation performance data

collected at concentrations much higher than the intended application may not be valid [12]. Even though typical in-vehicle VOC levels were much higher than typical ambient air VOC levels, they are still sub-ppm levels [4–9]. Accordingly, this study was conducted to establish the technical feasibility of application of TiO₂ photocatalytic technology for cleansing air aromatics in the low parts per billion (ppb, ≤100 ppb) concentration range commonly associated with in-vehicle air quality issues. The target VOCs investigated are markers of vehicle emissions: benzene, ethyl benzene, *o*-xylene, *m*-xylene and *p*-xylene.

The evaluation parameters included humidity, hydraulic diameter (HD) and stream flow rate (FW). Humidity is an important variable since photocatalytic technology should be applicable to the wide humidity range encountered in real in-vehicle environments. The influence of water vapor on the efficiency of the PCO air treatment process is unclear. Widely differing effects of water vapor have been reported [10–12,17–20]. Most previous research investigated pollutant levels well above 1 ppm. Obee and Brown [12] investigated the effect of water vapor on photocatalytic activity of a titania PCO reactor against sub-ppm formaldehyde, toluene and 1,3-butadiene, but not the target compounds of the present study. The effect of HD on PCO destruction efficiency was tested since UV intensity on the reactor surface is an important parameter [12,15] and varies with the HD of

* Corresponding author. Tel./fax: +82-53-950-6579.
E-mail address: wkJo@knu.ac.kr (W.-K. Jo).

the PCO reactor for an identical UV lamp. Here, HD is defined as the inside diameter of the annular reactor tube minus the outside diameter of the lamp. Stream FW is also tested for PCO destruction efficiency since it can influence mass transfer process of target VOCs in PCO reactors [12,16].

2. Experimental methods

2.1. PCO reactor and survey protocol

The concentrations of five target VOCs (benzene, ethyl benzene and *o*-, *m*-, *p*-xylenes) were measured at the PCO reactor inlet and outlet under different operating conditions. The schematic diagram of the experimental apparatus is shown in Fig. 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 TiO₂ photocatalyst. A cylindrical UV light source was inserted inside the glass tube and served as the inner surface of the annular reactor. The gas 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 [15,21]. 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, uniform TiO₂ 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 temperature and then baked for 30 min at 450 °C.

Variables tested for the current study included humidity, HD and FW (reactor residence time). The humidity range for these experiments was 10–80% (10–20, 30–40, 50–60 and 70–80%) relative humidity (RH) that covers dried and humidified environments. The humidity levels were adjusted by passing zero-grade air through a humidification

Table 1

Representative values of the variables employed for this study

Variable	Representative value
RH ^a (%)	50–60
HD ^b (mm)	5.0
FW ^c (dm ³ min ⁻¹)	1.0
Reactor residence time ^d (s)	2.0
Contaminant concentration ^e (ppb)	
Benzene	93
Ethyl benzene	21
<i>m</i> -, <i>p</i> -Xylene	78
<i>o</i> -Xylene	45
UV light source ^f	8 W fluorescent black light
Reactor material	Glass
Weight of catalyst film (mg cm ⁻²)	0.5

^a RH is within the ASHRAE comfort range (40–60%).

^b Near the HD (5.0 mm) employed in the present study, Jacoby et al. obtained nearly 100% of PCO of low-level formaldehyde and acetone.

^c This FW provides a face velocity of 12.1 cm s⁻¹ for the specified HD above.

^d Residence time is calculated by dividing the reactor volume by FW.

^e Contaminant concentration is nearly the maximum value of the concentration ranges for each target compound associated with in-vehicle air quality issues [9].

^f The UV light source provides UV intensities which are expected to be employed in a practical photocatalytic purifier [14,15].

device in a water bath (Cole-Parmer HAAKE W26) (Fig. 1). RH was measured just prior to the PCO reactor inlet using a humidity meter (Thermo Recorder TR-72S, T & D). The humidity level was incrementally increased from low to high to cover the range shown. Three different HDs (5.0, 20.0 and 45.0 mm) were also tested. These tests were conducted using the same residence time (5 s) calculated by dividing the volumetric FW by flow area of the reactor. The range of air FWs investigated was 0.4–3.0 dm³ min⁻¹ and is comparable to that reported previously by other researchers [12,15]. Measurements of FW were carried out using identical rotameters (0–10 dm³ min⁻¹) calibrated against a dry test meter (URG 3000-020C). For each variable test, the other variables were all fixed to the their representative values (Table 1). One exception was that in HD tests the

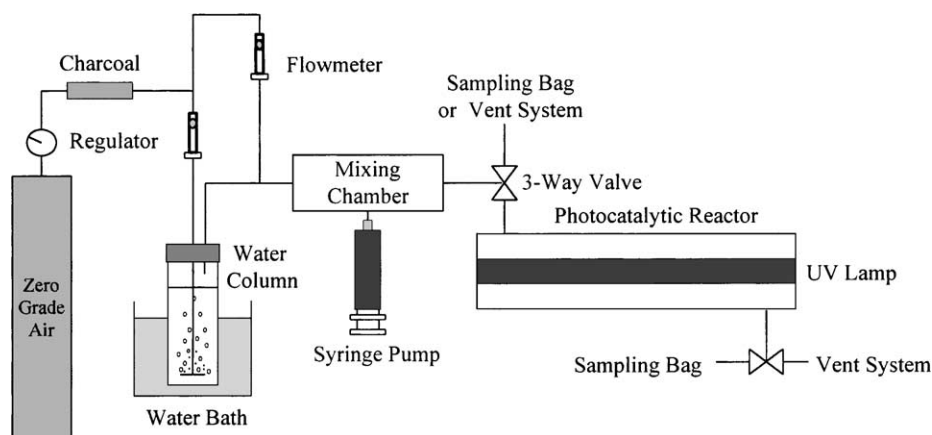


Fig. 1. Schematic diagram of the experimental apparatus.

FWs varied to provide the same residence time (5 s) in the three reactors with different HDs. The residence time was calculated using the FW and empty reactor volume.

The fixed parameters included contaminant concentration, UV light source, PCO reactor material and the weight of TiO₂ (Table 1). The concentrations surveyed were associated with in-vehicle air quality issues [9]: 4–93 ppb benzene, 2–21 ppb ethyl benzene, 3–78 ppb *m*-, *p*-xylene and 2–45 ppb *o*-xylene. The desired concentrations were achieved through 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. The UV light intensity was measured at the distance from the UV lamp equal to half of the HD of a reactor using a Black-Ray radiometer (Model J-221). The UV radiation intensities measured in the present study were 5.8, 3.8 and 2.2 mW cm⁻² for the 5.0, 20.0 and 45.0 mm HDs, respectively. The weight of the TiO₂ film coated inside reactor was 0.5 mg cm⁻².

The current experiments involved a catalyst pretreatment period of several hours, during which zero-grade air flowed through the illuminated photocatalytic reactor. When no contamination in the PCO reactor was measured for the target VOCs, the feed containing target compounds, water vapor and zero air was introduced. This study measured a series 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 were 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 in-vehicle CO levels.

2.2. Sampling and analysis

VOCs in the air stream were collected by filling an evacuated 51 Tedlar bag at a constant FW. Air from this bag was then drawn through a 0.64 cm o.d. and 18 cm long SS sorbent trap containing 0.5 g of Tenax TA using a constant flow-sampling pump (A.P. Buck Model I.H). Sampling times varied from 1 to 5 min depending on the FW. All samples were taken at ambient room temperature (19–25 °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 long fused silica column (Supelco SPB-5). Next, the adsorbent (Tenax TA) trap was thermally desorbed at 250 °C for 10 min, and the target compounds cryofocussed at -120 °C on a cryo trap (15.2 cm long, 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 refocus 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 5 min and ramped at 4 °C min⁻¹ to 200 °C for 5 min. The

present study identified the target VOCs by their retention times using GC/FID analysis. The quantitative analysis of the target compounds was performed using the calibration curves of a minimum of five concentrations.

The quality assurance/quality control (QA/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. When the quantitative response differed more than ±20% from that predicted by the specified calibration equation, a new calibration equation was determined. The method detection limits (MDLs) ranged from 0.1 to 0.4 ppb for the target VOCs.

CO concentrations were measured using portable monitors equipped with a data logging system (CMCD-10P, GASTEC). The QA/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 (IDL) was 0.1 ppm for CO.

3. Results and discussion

3.1. Effects of humidity on PCO destruction of aromatic VOCs

Figs. 2–5 show the VOC concentrations measured prior to and after turning on the UV lamp for four different RH ranges that cover typical ambient air humidity ranges. The experiments were repeated three times for each RH range and the repeated experiments showed very similar patterns. Thus, average values for each RH range are shown in the figures. 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 RH ranges the adsorption process reached equilibrium within 30 min for all target VOCs as indicated by the equality between the inlet/outlet pollutant concentrations. In addition, a series of VOC concentrations measured after turning on the UV lamp indicates that for all RH ranges, the outlet concentrations of the PCO reactor reached a steady state within 30 min 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.

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. For all RH ranges, the VOC concentrations measured after turning the UV lamp on were all below MDLs. Thus, the destruction efficiencies were close to 100%. This indicates that the PCO technology can be applied to in-vehicle air cleaning regardless of the

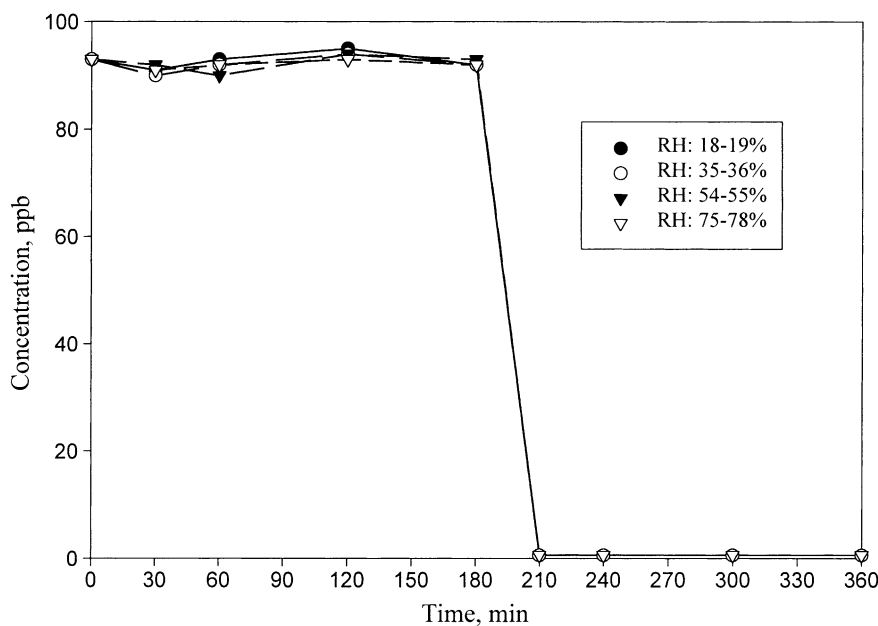


Fig. 2. PCO destruction of benzene for four different RH ranges. UV lamp was turned on at 181 min.

humidity of the polluted air. The current results are consistent with the Stevens et al. [15] results that reported complete, steady state conversions of lower concentrations of carbonyl compounds. It should be noted however that RH was not controlled in their study. 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 [10–16].

Obee and Brown [12] reported that under conditions of low humidity (ca. 8% RH) and a high toluene inlet level (ca.

2130 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. 8000 ppb). At moderate to high humidity (>20% RH) and low toluene levels (ca. 500 ppb) they found a decrease in the PCO efficiency with increasing humidity. This PCO rate dependence on humidity was also reported for *m*-xylene by Peral and Ollis [11]. The previous studies explained that

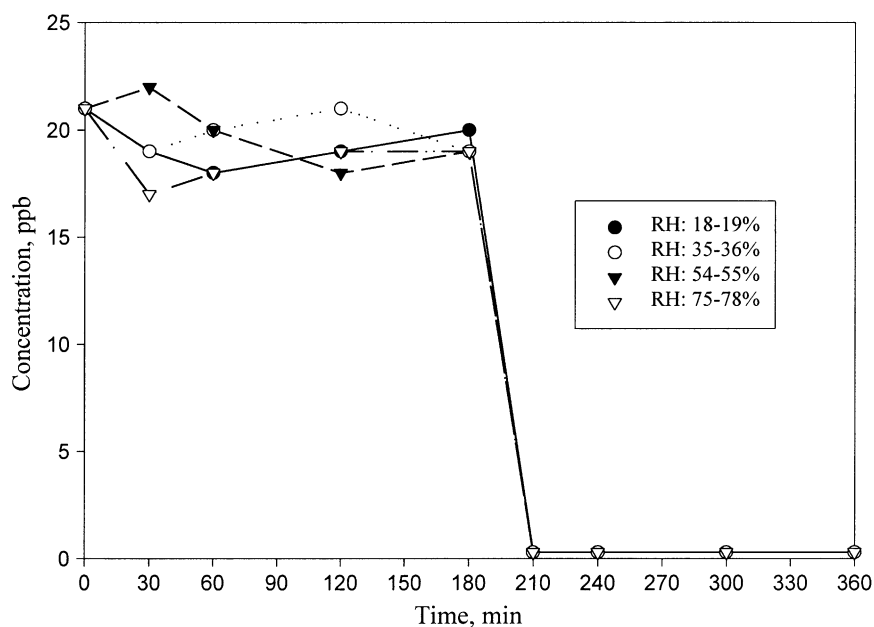


Fig. 3. PCO destruction of ethyl benzene for four different RH ranges. UV lamp was turned on at 181 min.

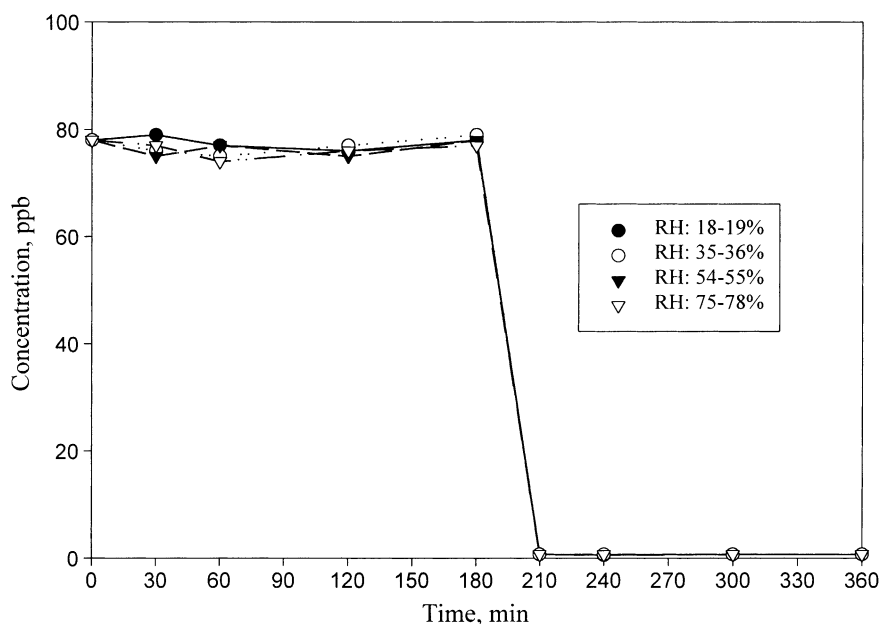


Fig. 4. PCO destruction of *m*-, *p*-xylene for four different RH ranges. UV lamp was turned on at 181 min.

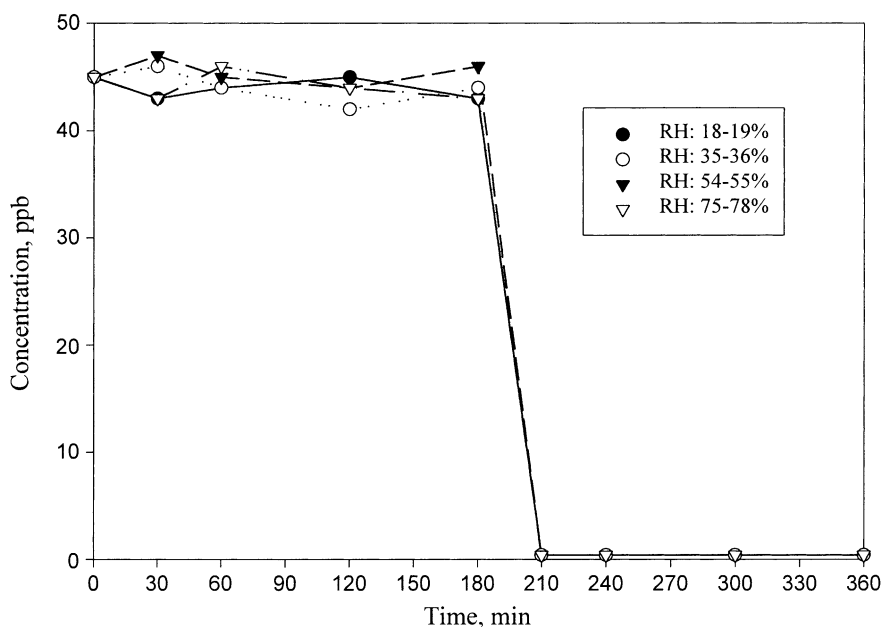


Fig. 5. PCO destruction of *o*-xylene for four different RH ranges. UV lamp was turned on at 181 min.

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 [12], 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 destruction 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. Effects of reactor HD on PCO destruction of aromatic VOCs

Three reactors with different HDs (5.0, 20.0 and 45.0 mm) were tested for PCO destruction efficiencies of the target compounds. To avoid the reactor residence time effect on

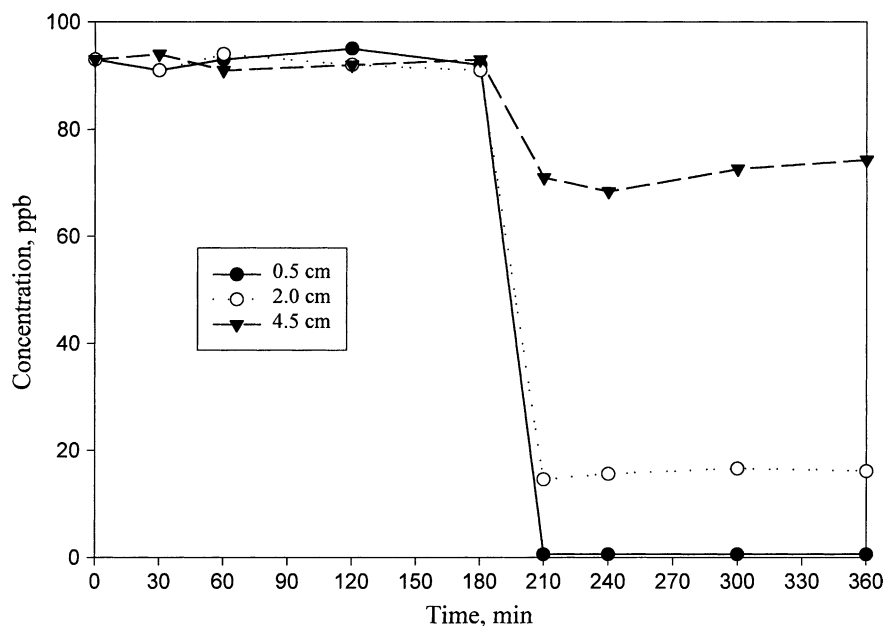


Fig. 6. PCO destruction of benzene for three reactors with different HDs. UV lamp was turned on at 181 min.

destruction efficiencies, these tests were conducted using the same residence time (5 s) for the three reactors by adjusting the stream FWs to 0.4, 2.3 and 7.8 dm³ min⁻¹ for the 5.0, 20.0 and 45.0 mm HDs, respectively. As discussed below, a FW increase can result in a mass transfer increase. Figs. 6–9 show the VOC concentrations measured during the course of the experiments using the reactors. As before, the experiments were repeated three times for each HD and the repeated experiments showed very similar patterns. Thus, average values for each HD are shown in the figures.

For all three reactors, the target VOCs showed the same results as the humidity study for both the adsorption and steady state processes. Accordingly, it is the steady state result that is discussed for the destruction efficiencies of the target VOCs here.

For the reactor with a 5.0 mm HD, the destruction efficiencies were nearly 100% for all target VOCs. However, the destruction efficiency decreased as the HD increased; the destruction efficiency ranged from 81 to 87% for the reactor with a 20.0 mm HD and ranged from 19 to 42% for the

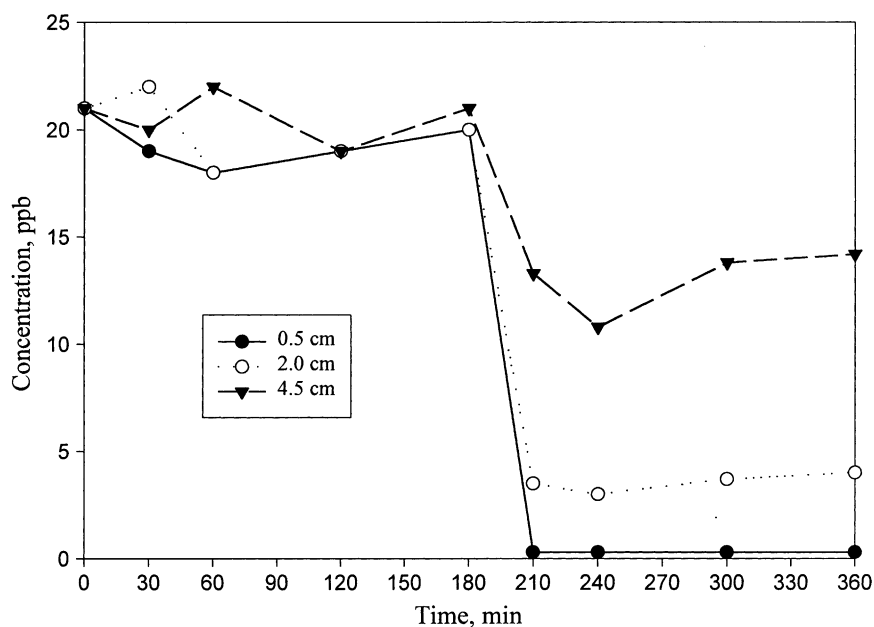


Fig. 7. PCO destruction of ethyl benzene for three reactors with different HDs. UV lamp was turned on at 181 min.

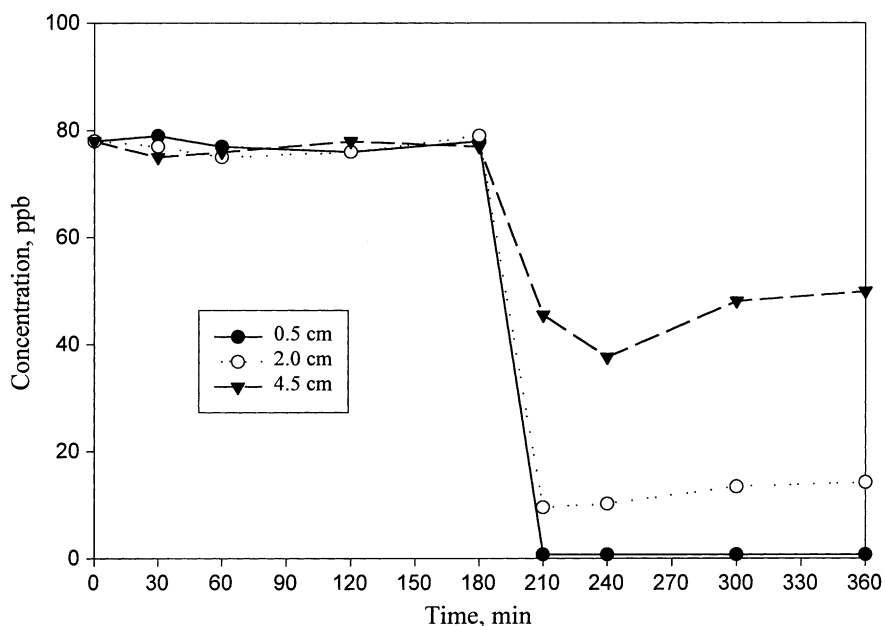


Fig. 8. PCO destruction of *m*-, *p*-xylene for three reactors with different HDs. UV lamp was turned on at 181 min.

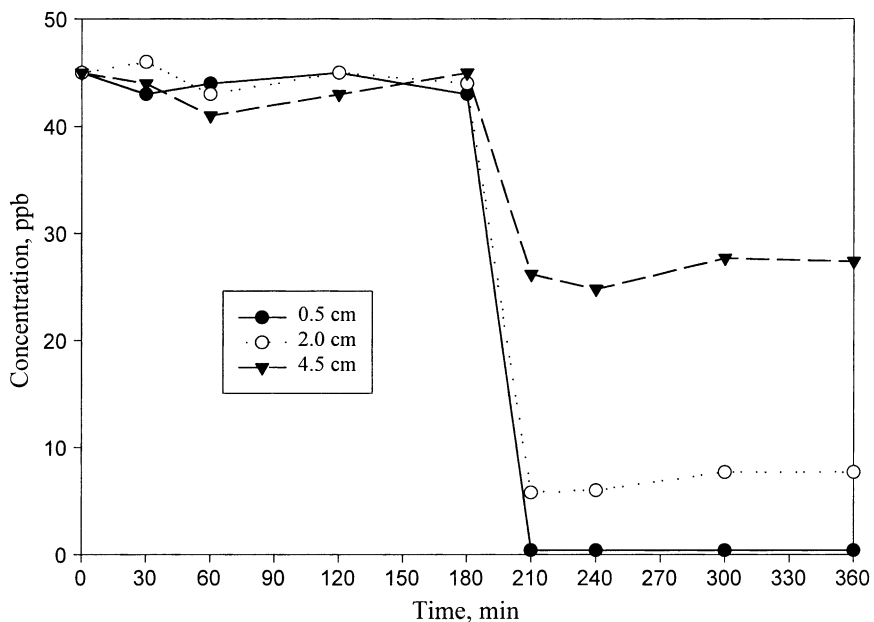


Fig. 9. PCO destruction of *o*-xylene for three reactors with different HD. UV lamp was turned on at 181 min.

reactor with a 45.0 mm HD, depending on the compound (Figs. 6–9). Accordingly, it is indicated that the HD of the PCO reactor is an important parameter for the application of TiO₂ photocatalytic technology for cleansing in-vehicle air. As the distance of the catalyst from the light source increases with increasing HD 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, 3.8 and 2.2 mW cm⁻² for the 5.0, 20.0 and 45.0 mm HDs, respectively. The effect of UV

radiation intensities is supported by Obee and Brown's study [12], which reported that the oxidation rate of toluene and formaldehyde increased with an increase in UV intensity. In addition, Peral and Ollis [11] reported the same results in the PCO study of acetone, 1-butanol, butyraldehyde, formaldehyde and *m*-xylene. In the present study, the FW 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 show that the mass transfer effects are not as

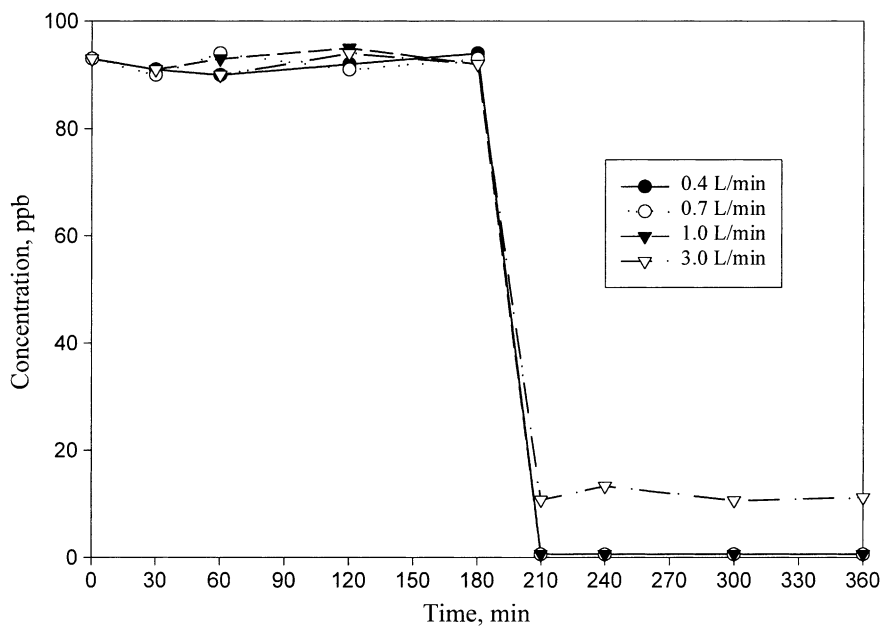


Fig. 10. PCO destruction of benzene for four different FWs. UV lamp was turned on at 181 min.

important as the light intensity effects on the PCO efficiency of the VOCs.

3.3. Effects of stream FW on PCO destruction of aromatic VOCs

The air FW (residence time) was tested for destruction efficiencies of the target compounds using the reactor with a 5.0 mm HD. Figs. 10–13 show the VOC concentrations measured using four different FWs (0.4, 0.7, 1.0 and

$3.0 \text{ dm}^3 \text{ min}^{-1}$). As before, these experiments were repeated three times for each FW and the repeated experiments showed very similar patterns. Thus, average values for each FW are shown in the figures. For all three FWs, the target VOCs showed the same results as the previous experiments for both the adsorption and steady state processes. Accordingly, it is the steady state result that is discussed for the destruction efficiencies of the target VOCs here.

For the three low FWs ($0.4, 0.7$ and $1.0 \text{ dm}^3 \text{ min}^{-1}$), the destruction efficiencies were nearly 100% for the entire

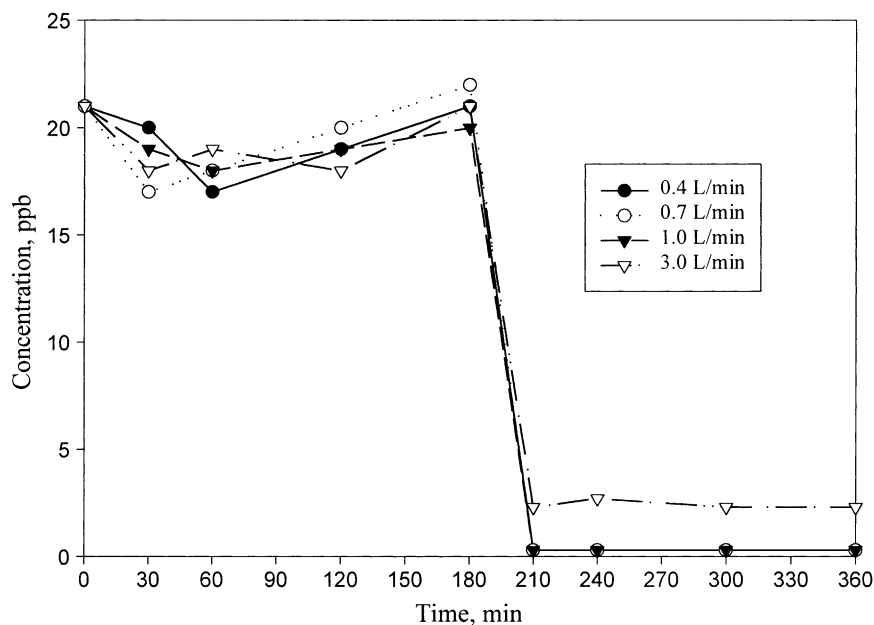


Fig. 11. PCO destruction of ethyl benzene for four different FWs. UV lamp was turned on at 181 min.

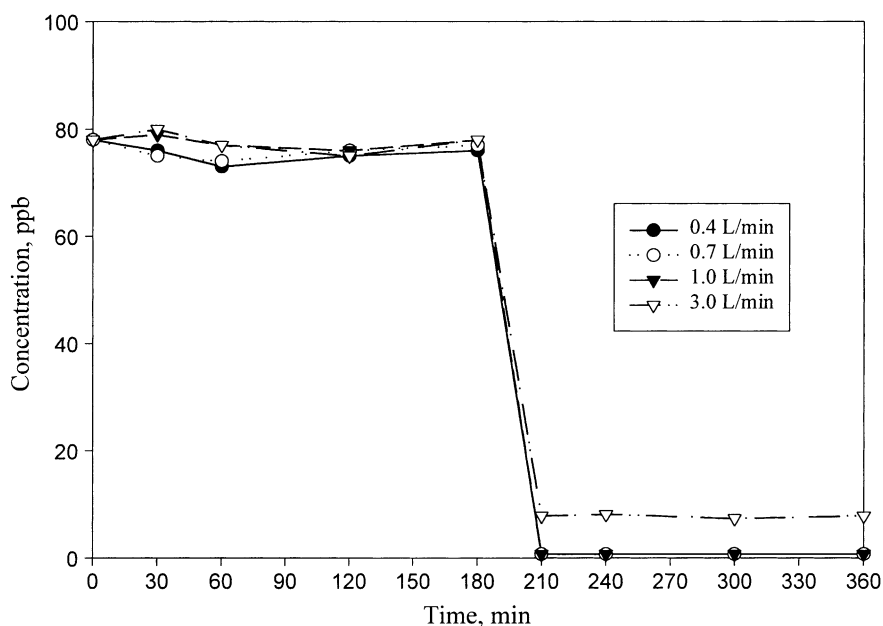


Fig. 12. PCO destruction of *m*-, *p*-xylene for four different FWs. UV lamp was turned on at 181 min.

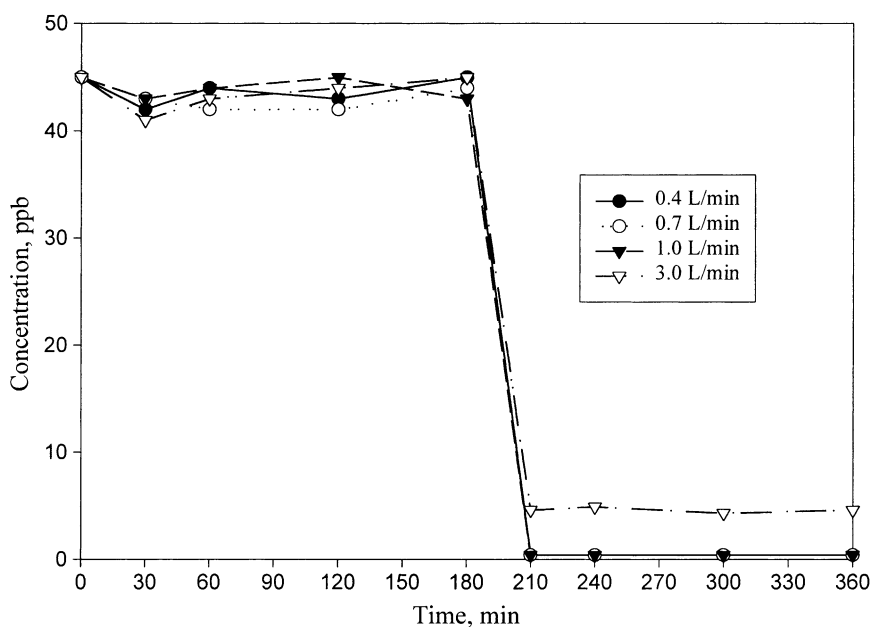


Fig. 13. PCO destruction of *o*-xylene for four different FWs. UV lamp was turned on at 181 min.

target VOCs. However, the destruction efficiency decreased for the highest FW ($3.0 \text{ dm}^3 \text{ min}^{-1}$), ranging from 86 to 89%, depending on the compound (Figs. 10–13). Accordingly, it is indicated that FW is an important parameter for the application of TiO_2 photocatalytic technology for cleansing in-vehicle air. The lower destruction efficiency for the high FW could be caused by an insufficient reactor residence time or mass transfer due to high FW. The residence times, which were calculated by dividing the reactor volume by FW, were 5.1, 2.9, 2.0 and 0.7 s for the FWs of 0.4, 0.7, 1.0

and $3.0 \text{ dm}^3 \text{ min}^{-1}$, respectively. The face velocities were 4.9, 8.5, 12.1 and 36.4 cm s^{-1} for the FWs of 0.4, 0.7, 1.0 and $3.0 \text{ dm}^3 \text{ min}^{-1}$, respectively. Bulk mass transport of the target compounds from the gas-phase to the surface of the catalyst particle due to convection and diffusion, an important heterogeneous catalytic reaction process [14], would be insufficient for a high FW (high face velocity for the same HD reactor). This explanation is supported by Obee and Brown's study [12], which reported that face velocity influences the mass transfer of formaldehyde, toluene and 1,3-butadiene.

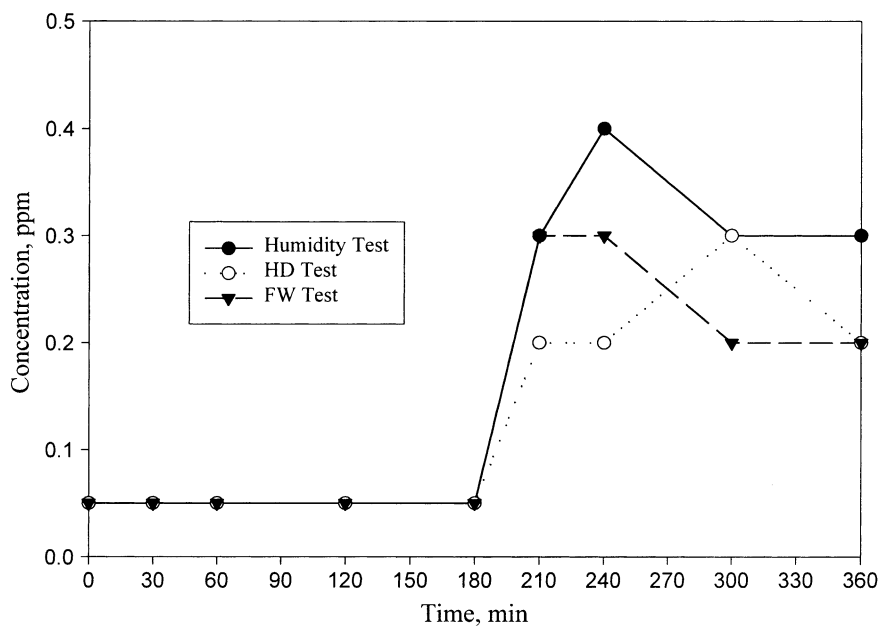


Fig. 14. Variation of CO concentrations at the PCO reactor outlet during the course of three tests (humidity, HD and FW tests). UV lamp was turned on at 181 min. The CO concentrations measured prior to turning on UV lamp were all below IDL and thus they were set equal to 1/2 of IDL.

3.4. CO levels at PCO reactor outlet

The CO concentrations were measured simultaneously with the VOC concentrations at the PCO reactor outlet during the course of the three tests previously described in this study (humidity, HD and FW tests). Fig. 14 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 min (prior to turning the UV lamp on) 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 min after the UV lamp was turned on as indicated by the equality among a series of the outlet concentrations.

As shown in Fig. 14, 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 [14], which reported that some portion of benzene was converted to CO. Nevertheless, considering that the occupational safety and health administration (OSHA) 8 h exposure limit for CO in the workplace is 35 ppm, under conditions relevant to the use of PCO, these elevations of CO concentrations (maximum 0.4 ppm) would be a negligible addition to the in-vehicle CO levels. Moreover, certain

CO, along with VOCs, contained inside vehicles can be destroyed through the PCO process [22–24].

Jacoby et al. reported that the primary product observed during the PCO process of benzene of 116 ppm was carbon dioxide (CO₂). In addition, several previous studies [12,25–27] reported that the PCO process of aromatic 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 mineralized to CO₂ during the course of the PCO process.

4. Conclusions

This study evaluated the technical feasibility of application of TiO₂ photocatalytic technology for cleansing in-vehicle air aromatics in the low ppb concentrations commonly associated with in-vehicle air quality issues. Within the conditions of the present study, the results clearly demonstrate that: (1) the destruction of aromatic VOCs at concentrations associated with in-vehicle air quality issues is nearly 100% using PCO techniques; (2) the destruction of aromatic VOCs was independent of humidity, but dependent on the HD of the reactor and stream FW (residence time); (3) the amount of CO generated during PCO would be a negligible addition to the in-vehicle 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 in-vehicle air quality. However, it is noted that the HD of the PCO reactor and stream FW should

be considered for better VOC removal efficiencies for the application of TiO₂ photocatalytic technology for cleansing in-vehicle air.

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