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Photo-catalytic oxidation of individual and mixture of benzene, toluene and *p*-xylene

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Abstract Photo-catalytic degradation of volatile organic compounds [VOCs: benzene, toluene and p-xylene (BTX)] was investigated using a batch reactor having a TiO₂ (catalyst)-coated aluminum sheet and a source of UV light (sunlight or UV lamp). To study the photo-oxidation, experiments were conducted under the following configurations: (1) TiO₂ (m): microparticle $(0.32-3.31 \mu m)$ and sunlight (2) TiO_2 (n): nanoparticle (0.80–4.70 nm) and sunlight, (3) TiO_2 (m) and UV lamp and (4) TiO_2 (n) and UV lamp. Degradation of BTX followed first-order decay for individual compounds. The degradation rate constant in $min^{-1} cm^{-2}$ (coated surface area) was the highest for configuration (4) (benzene 1.07×10^{-3} , toluene 1.36×10^{-3} and *p*-xylene 2.93×10^{-3}) followed by configuration (2), thus indicating the importance of particle size of the catalyst in degradation. Degradation of BTX mixture did not follow first-order decay. Benzene was an intermediate product of oxidation of toluene. Benzene and toluene were intermediate products of oxidation of p-xylene. For degradation of BTX mixture, a mathematical model was proposed to predict concentrations as a function of time. Experimental and model results showed similar trends in BTX degradation. The model accounted for increases in mass of toluene and benzene due to the degradation of *p*-xylene.

Keywords Photo-degradation \cdot Benzene \cdot Toluene $\cdot p$ -Xylene \cdot Nano-sized TiO₂ \cdot Volatile organic compounds

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Introduction

Volatile organic compounds (VOCs) are important air pollutants, emitted largely by industries, vehicles and domestic cooking that use fossil fuels, especially biofuels. Many VOCs (e.g., benzene, ethylbenzene and n-decane) are toxic, and some are carcinogenic, mutagenic or teratogenic (Edgerton et al. 1989; Burstyn et al. 2007; You et al. 2008). Besides health effects, VOCs can combine with nitrogen oxides leading to production of secondary air pollutants (ozone, peroxy-acetyl nitrates and secondary organic aerosols; Carp et al. 2004; USEPA 2006; Song et al. 2007). Out of several VOCs, the common VOCs which are toxic and present in appreciable quantities in ambient air include benzene, toluene and *p*-xylene (BTX; Lau and Chan 2003; Lü et al. 2006; Zhang et al. 2012). The major sources of BTX are vehicular exhausts and evaporation losses from handling, distributing and storing of solvents (Choi et al. 2009; Srivastava et al. 2005; Dutta et al. 2009).

Increasing levels of VOCs in ambient air, indoor air and industrial premises (Dhada et al. 2012; Sharma et al. 2010; Mo et al. 2009) necessitate that VOC control technologies be developed and further improved. Earlier studies (Rene et al. 2005; Deshusses and Webster 2000) have reported that VOCs from polluted air streams can be controlled by traditional methods such as adsorption, condensation and incineration. Although these methods use simple techniques, they have certain implementation constraints which include high cost, unpredictable recoveries and difficulty in separating one or more VOCs for reuse/recycle (Jeong et al. 2005; Miller and Fox 1993).

Advanced oxidation processes such as thermal oxidation (Everaert and Baeyens 2004) and photo-catalytic oxidation (Carp et al. 2004) are promising technologies for control of VOCs. Thermally catalytic oxidation requires high



temperatures of 200–1200 °C for efficient operation, and this technique is economically not feasible at low VOC concentrations (Wang et al. 2007). Ultraviolet (UV)-induced photocatalytic oxidation (PCO) techniques have been extensively studied and were found effective in controlling VOCs even at room temperature (Jeong et al. 2005; Ao et al. 2003).

In the PCO systems, accelerated oxidation of chemicals is achieved with the help of UV light and semiconductors as catalyst (Hager and Bauer 1999). Usually, semiconductor materials enhance the photo-catalytic reaction by lowering the required activation energy of the reaction due to their special electronic band structure (Puddu et al. 2010). The desirable properties of titanium dioxide (TiO₂) in terms of catalytic activity, chemical stability, nontoxicity, relative inexpensiveness and availability make it a suitable photo-catalyst (Hong 2006). VOCs are converted to CO_2 and H_2O during PCO, with TiO_2 as catalyst (Collins 2012). The humidity is an important constituent for effective PCO, as humidity is the sole supplier of OH⁻ that is needed during photo-catalysis to maintain a reasonable population of OH° (Demeestere et al. 2007; Sun et al. 2010). The following reactions have been suggested for PCO (Collins 2012) of VOCs:

Activation reaction $TiO_2 + h\nu \rightarrow h^+ + e^-$ Oxidation reaction $h^+ + OH^- \rightarrow OH^\circ$ Reduction reaction $O_2 + 2e^- + 2H_2O \rightarrow H_2O_2$ $+ 2OH^ H_2O_2 + e^- \rightarrow OH^\circ + OH^-$ Net reaction $OH^\circ + CnHm + O_2 \xrightarrow{TiO_2}$ $nCO_2 + mH_2O$

Review of earlier studies (Peral and Ollis 1992; Obee and Brown 1995; Jacoby et al. 1996; Obee 1996; Stevens et al. 1998) suggests that UV-illuminated TiO₂ catalytic surface can produce an overall reduction in levels of VOCs. However, these studies have not suggested the degradation mechanism under various combinations of irradiated sources (e.g., artificial UV and natural sunlight) and different particle sizes of TiO₂ (nano and micro). This study has attempted to investigate the probable mechanisms of photo-degradation of common VOCs (BTX) under natural sunlight and artificial source of UV radiation with micro-sized TiO₂ [TiO₂ (m); average size 1.8 μ m] and nano-sized TiO₂ [TiO₂ (n); average size 1.6 nm] in a batch reactor. This study was carried out at the Air Quality Laboratory, Indian Institute of Technology Kanpur, India, during late 2006 and early 2010.

Materials and methods

Materials

Chemicals used in this study were of AR (analytical reagent) grade (Merck, India): methanol anhydrous



(99.8 % purity), toluene anhydrous (99.8 % purity), benzene anhydrous (99.8 % purity), *p*-xylene anhydrous (99.5 % purity), tetrabutylorthotitanate (98 % purity), *n*propanol (99.5 % purity) and acetyl acetone (99.55 % purity). The photo-catalyst TiO₂ was procured from Loba Chemie, India (Art no 6325). In the initial stage of this study, various media like water, acetyl acetone and methanol were considered separately to obtain dispersed TiO₂ particles. Dispersion normally depends on temperature, polarity of solvent, active surface area and crystalline phase (brookite, anatase and rutile) of TiO₂. It was found that TiO₂ was more uniformly dispersed in methanol than other media, possibly due to high polarity of methanol (Parish et al. 1985).

Aluminum sheets of size 3 cm \times 15 cm were used as substrate for coating TiO₂ on it. The choice of aluminum was based on its useful properties like nontoxic and nonmagnetic nature, light weight and high reflectance of radiation in the range of 200–400 nm (Seisler machine 2013). Further, aluminum sheets can withstand operating conditions such as temperature and pressure and provide resistance to corrosion (Linsebigler et al. 1995). TiO₂ (n) particles were prepared from TiO₂ (m) particles using a ball mill (Pulverisette Fristsch, Germany). TiO₂ (m) was milled for 16 h at about 450 rotations per minute with six quartz balls. Size distribution of TiO₂ particles was carried out using the Master Sizer 2000 (Malvern Instruments, UK). The size range for TiO₂ particles was as follows: TiO₂ (m): 0.32–3.31 μ m and TiO₂ (n): 0.80–4.70 nm.

Sonicated suspension of nano- and micro-sized TiO_2 particles with methanol as a dispersive medium was used for the physical coating of TiO_2 on aluminum sheet (Jo and park 2004). The coated sheets were dried at room temperature and were placed in an oven at 100 °C for 6 h to evaporate methanol. A mass of 0.096 g of TiO_2 was coated per cm² surface area of aluminum sheet. The band gap between valence and conduction bands of coated TiO_2 was estimated as 3.27 eV (using UV spectroscopy, Varian, USA), which was equivalent to band gap of 3.23 eV for the anatase phase (Mo et al. 2009). Thus, it is expected that the major part of TiO_2 used in this study was in anatase phase. Uniformity of TiO_2 coating could not be checked due to limitation of profilometry sensor, which cannot be operated on aluminum surface.

Experimental setup and chemical analysis

A cylindrical batch reactor (made of glass B-50) having a dimension of 50 mm internal diameter and 370 mm height (actual volume 700 cm³ and effective volume 676 cm³) was used in this study (Fig. 1). The TiO₂-coated aluminum sheet was placed inside the reactor. For all experiments on degradation of individual and mixture of VOCs, the reactor

along with VOCs was filled with the ambient air. The ambient oxygen was the main oxidant for oxidizing the VOCs through formation of OH radical. A UV lamp (30 cm length and 1 cm diameter; C-UV, Kipp and Zonen, G \in Japan) of 10 W was placed coaxially in the reactor as the UV source. The peak intensity of the UV source was estimated to be $1223 \pm 5 \,\mu$ W/cm² at a wavelength of 365 nm near the coated surface. The reactor was made airtight and had the facility to draw samples and to inject the known quantities of BTX vapors.

Concentrations of BTX were analyzed using gas chromatography (GC; Buck Scientific model 910, USA) with a photo-ionization detector. The liquid standards (in methanol, Merck) of BTX were used for calibration of GC. The GC was calibrated at 2, 20, 200 and 2000 μ g of BTEX (R^2 : 0.99). The gas samples were collected periodically from the reactor using a 5-mL SGE gastight syringe, and 1 mL of each sample was injected into the GC (column: Restek MXT; having an internal diameter of 0.53 mm, length of 30 m). The range of BTX mass (in the sample) was 2–200 μ g which was in the calibration range of GC. The operating conditions of GC were set as carrier gas (helium) at 2.5 kg/cm² pressure, column oven temperature at 60 °C and detector temperature at 150 °C. Injector temperature was held at 100 °C.

Details of the experiment

In the preliminary stage of the experiment, degradation of BTX was assessed under dark conditions without any source of external UV radiations with and without TiO₂-coated aluminum sheet; the degradation was less than 3 % in 4 h. In the second test, BTX degradation was assessed in the presence of sunlight (11:00 am to 2:00 pm) without TiO₂ coating; the degradation was slightly higher (about 6 ± 1.3 %). From these preliminary tests, it was concluded that BTX degradation process was very slow in dark and in the presence of sunlight (without TiO₂ coating). Thus, there was a need to have TiO₂ as catalyst and an external source of UV light for producing photons in the desired wavelength range.



Fig. 1 Experimental setup comprised of coating material in the batch reactor in: a open atmosphere with natural sunlight, b laboratory with external UV radiation source

Table 1	Overall	initial	condition	during	the	experiments
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Experiment	Benzene			Toluene			<i>p</i> -Xylene		
	$C_0 (\text{mg/m}^3)$	Temp (°C)	RH (%)	$\overline{C_0 \text{ (mg/m}^3)}$	Temp (°C)	RH (%)	$C_0 \text{ (mg/m}^3)$	Temp (°C)	RH (%)
Sunlight $+ \text{TiO}_2(m)$	52.4	46.2	41.3	132.9	47.1	39.2	207.9	47.2	40.9
UV light + TiO_2 (m)	53.8	45.4	40.2	138.9	45.2	40.7	44.1	42.3	39.4
$Sunlight + TiO_2(n)$	21.1	46.3	40.6	116.3	45.6	41.3	44.5	46.6	37.6
UV light + $TiO_2(n)$	57.4	45.7	41.8	56.6	44.3	38.6	72.8	42.4	41.1



Table 1. A concentration range of VOCs used in this study.

20-80 ppm, was approximately in the observed range

(30-350 ppm) in field at Arti Industries, Vapi, India

(Sharma and Dhada 2013). Gas samples (1 mL) were taken

from the reactor at regular intervals (about 15 min) for

analysis on the GC; these experiments were repeated six

times for each compound. Each experiment was

90-150 min long. After three experiments (one each for

benzene, toluene and xylene with total exposure period of

270-450 min), the catalyst was visibly examined and there

The following four sets of experiments were conducted to examine the degradation rates of BTX under varying configurations of catalyst and UV Source: (1) TiO_2 (m) and sunlight [referred to as 'sunlight + TiO_2 (m)'], (2) TiO_2 (m) and external UV lamp [referred to as 'UV lamp + - TiO_2 (m)'], (3) TiO_2 (n) and sunlight [referred to as 'sunlight + TiO_2 (n)'] and (4) TiO_2 (n) and UV lamp [referred to as 'UV lamp + TiO_2 (n)']. In all these experiments, BTX were separately injected in the reactor. The initial conditions for the experiments are presented in

Fig. 2 Photo-catalytic oxidation of **a** benzene, **b** toluene and **c** xylene under various experiment categories





Experiment	Benzene		Toluene		<i>p</i> -Xylene	
	Degradation constant \times 10 ⁻⁴ (min ⁻¹ cm ⁻²)	R^2	Degradation constant \times 10 ⁻⁴ (min ⁻¹ cm ⁻²)	R^2	Degradation constant \times 10 ⁻³ (min ⁻¹ cm ⁻²)	R^2
1. Sunlight + TiO_2 (m)	5.78 ± 0.91	0.974	6.67 ± 0.74	0.988	1.00 ± 0.67	0.981
2. Sunlight + TiO_2 (n)	9.33 ± 0.78	0.957	10.00 ± 0.82	0.998	2.31 ± 0.53	0.934
3. UV +TiO ₂ (m)	8.22 ± 0.62	0.968	8.67 ± 0.97	0.986	1.47 ± 0.48	0.971
$4. \text{ UV} + \text{TiO}_2 (n)$	10.7 ± 0.89	0.984	13.6 ± 0.78	0.99	2.93 ± 0.59	0.968

Table 2 First-order degradation constant per unit surface area of TiO₂ coating for BTX (min⁻¹ cm⁻²) with correlation coefficient (R^2)

was no change in color which suggests that catalyst was stable during the experiments.

degrade VOCs, in the situations where external UV source is not feasible.

Results and discussion

Photo-degradation of BTX

The PCO of BTX was studied separately for each compound for four varying configurations (1–4), described above. The degradation pattern followed the first-order decay for BTX for all configurations (Fig. 2).

The degradation rate constants of BTX per unit coated surface area $(\min^{-1} \operatorname{cm}^{-2})$ have been estimated and are presented in Table 2. These estimated constants can be used to design a reactor for specific conditions and desired efficiency of removal. It can be concluded that the decay of all three compounds was fastest under UV lamp and TiO₂ (n) (the fourth row of Table 2) than any other configuration. The second fastest decay was under the condition of sunlight and TiO_2 (n) (the second row of Table 2). This accelerated degradation can be attributed to larger surface area of TiO₂ (n) and the external source of UV radiation of intensity around 40 W/m² (integrated over all UV wavelengths) which is much higher than the intensity of sunlight (16 W/m^2) . In addition, the high energy radiations from sun (i.e., UV-B \sim 3.94 to 4.43 eV and UV-C \sim 4.43 to 12.4 eV) do not reach earth surface, whereas the external UV source provides energy radiations in UV-A, B and C bands. It can be seen from Table 2 that use of TiO_2 (n) makes the reaction about 30 % faster for degradation of benzene than the degradation using TiO_2 (m) with UV lamp as source of radiation.

This study also signifies that PCO of VOCs with nanoparticles of TiO_2 in presence of sunlight is about 1.13 times faster than by TiO_2 microparticles in the presence of external UV source. The major radiations in sunlight are UV-A and UV-B, which also produce OH^o (Seinfeld and Pandis 1997) and can degrade VOCs on large surface area provided by nanoparticles. The outcome of this research provides an option of using TiO_2 nanoparticles and the sunlight as the sole source of radiation to effectively

Degradation of BTX mixture

BTX is emitted as a mixture from many sources. Therefore, ideally, their degradations must be studied as a mixture. In such cases, the degradation of one compound may affect the degradation of other compounds. PCO of BTX mixture was studied under the same conditions as those of individual compounds. The results of degradation of BTX mixture are shown in Fig. 3, and it is observed that the degradation does not strictly follow the first-order decay. The degradation of BTX mixture is a complex process, and there is a need to investigate pathways of degradation which might be influenced by competitive reactions and presence of intermediate products of degradation.

To specifically examine the formation of intermediate products, presence of other compounds is examined during the degradation of toluene and p-xylene (Fig. 4). As the oxidation of toluene and p-xylene progresses, the concentration of parent compounds decreases and the concentration of intermediate products (toluene and benzene) increases. From Fig. 4, it was concluded that (1) benzene is an intermediate compound during the oxidation of toluene and benzene are intermediate compounds during the oxidation of p-xylene. These intermediate formations indicate interlinking among these three species.



Fig. 3 Photo-catalytic oxidation of BTX mixture under various experiment categories





Fig. 4 a Photo-catalytic oxidation of *p*-xylene showing formation of toluene and benzene as intermediate product. b Photo-catalytic oxidation of toluene showing formation of benzene as intermediate product

Hung (2001) have also reported formation of benzene as an intermediate compound during oxidation of toluene. The degradation of p-xylene through formation of toluene exists and can be understood from the oxidation pathways of aromatics in the environment (Dhada 2008). We could not measure the intermediates (other than toluene and benzene) due to limitations of the GC column.

A mathematical modeling needs to be developed so that concentrations of BTX can be predicted as a function of time. In building the model for degradation of mixture of BTX, we have assumed that the pathway of degradation of xylene is through toluene and that of toluene is through benzene. Formation of other intermediates is assumed to be negligible. This assumption is not irrational because the intermediates as seen in Fig. 4a, b are toluene and benzene. The degradation pathway of BTX is considered as:

$$X \xrightarrow{k_{\rm X}} T \xrightarrow{k_{\rm T}} B \xrightarrow{k_{\rm B}}$$

In view of the observed degradation pathways (xylene to toluene to benzene) and the assumption that formation of other intermediates is negligible, the values of k_X , k_T and k_B (degradation rate constants) can be taken from Table 2 and used in the model.

$$-r_{\rm X} = \frac{-\mathrm{d}C_{\rm X}}{\mathrm{d}t} = k_{\rm X}C_{\rm X} \tag{1}$$

$$-r_{\rm T} = \frac{\mathrm{d}C_{\rm T}}{\mathrm{d}t} = k_{\rm X}C_{\rm X} - k_{\rm T}C_{\rm T} \tag{2}$$





Fig. 5 Modeling value versus observed value for degradation of BTX for UV light with TiO_2 (n) particle and sunlight with TiO_2 (n) particle



$$-r_{\rm B} = \frac{\mathrm{d}C_B}{\mathrm{d}t} = k_{\rm T}C_{\rm T} - k_{\rm B}C_{\rm B} \tag{3}$$

where subscripts X, T and B represent xylene, toluene and benzene, r_X , r_T , r_B are the rate of degradation, C_X , C_T , C_B are the concentration at any time and k_X , k_T , k_B are the firstorder degradation rate constants. Assuming that degradation of *p*-xylene, toluene and benzene to be first order, their concentrations as a function of time can be estimated as:

Solving the above first-order linear differential equations (Eqs. 1-3)

$$C_{\rm X} = C_{\rm X0} \, e^{-k_{\rm X} t} \tag{4}$$

$$C_{\rm T} = C_{\rm T0} \, e^{-k_{\rm T}t} + k_{\rm X} \, C_{\rm X0} \left[\frac{e^{-k_{\rm X}t}}{k_{\rm T} - k_{\rm X}} - \frac{e^{-k_{\rm T}t}}{k_{\rm T} - k_{\rm X}} \right] \tag{5}$$

$$C_{\rm B} = \left[C_{\rm T0} \frac{k_{\rm T} e^{-k_{\rm T} t}}{k_{\rm B} - k_{\rm T}} + \frac{k_{\rm X} k_{\rm T} C_{\rm X0}}{k_{\rm T} - k_{\rm X}} \left(\frac{e^{-k_{\rm X} t}}{k_{\rm B} - k_{\rm X}} - \frac{e^{-k_{\rm T} t}}{k_{\rm B} - k_{\rm T}} \right) \right] + e^{-k_{\rm B} t} \left[C_{\rm B0} - \frac{k_{\rm T} C_{\rm T0}}{k_{\rm B} + k_{\rm T}} - \frac{k_{\rm X} k_{\rm T} C_{\rm X0}}{k_{\rm T} - k_{\rm B}} \left(\frac{1}{k_{\rm B} - k_{\rm X}} - \frac{1}{k_{\rm B} - k_{\rm T}} \right) \right]$$
(6)

where, $C_{\rm X0}$, $C_{\rm T0}$, $C_{\rm B0}$ are the initial concentration.

As an example, the observed concentrations of BTX [under the configurations (1) Sunlight + TiO_2 (n) and (2) UV + TiO_2 (n)] were compared with model's computed concentrations (from Eqs. 1–6) as a function of time (Fig. 5).

Although the model does not seem to fit well for toluene and benzene, the general trends in modeling and experimental results are similar. The model's performance could be improved by considering the formation of intermediates and accounting for them in future. In addition, difference between the experimental data and the model could be due to the fact that rate constants in the degradation mixture may not follow first order. It is suggested that for better insight into the PCO of BTX mixture, experiments should be undertaken to account for formation of intermediate compounds and CO₂; carbon balance should be carried out for the above process.

Conclusion

BTX degradation follows a first-order decay when its constituents are studied separately. However, BTX degradation studied as a mixture does not obey the first-order decay. A quick degradation of benzene, toluene and pxylene (approx 95 %) with TiO_2 (n) coating under external source of UV radiation (UV lamp) was achieved in about 1 h. This accelerated degradation can be attributed to the large surface area of TiO_2 (n) and a higher UV radiation intensity in all wavelengths ranges (UV-A, UV-B and UV-C). There were consistent results that showed formation of toluene as an intermediate product of oxidation of *p*-xylene and the formation of benzene as an intermediate product of oxidation of toluene. In the BTX mixture, p-xylene degrades to form toluene and this toluene along with toluene initially present (at t = 0) degrades to form benzene. A model was developed to predict concentrations of BTX as a function of time in the BTX mixture. The photo-catalytic oxidation technology has shown promise for VOC removal, and the estimated degradation rate constants can be used for designing reactors for specific conditions and desired efficiency of VOC removal.

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