

ORIGINAL ARTICLE

Control of Airborne Organic Pollutants Using Plug-Flow Reactor Coated With Carbon Material-Titania Mixtures Under Visible-Light Irradiation

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Abstract

Graphene oxide (GO)-titania composites have emerged as an attractive heterogeneous photocatalyst that can enhance the photocatalytic activity of TiO₂ nanoparticles owing to their potential interaction of electronic and adsorption natures. Accordingly, TiO₂-GO mixtures were synthesized in this study using a simple chemical mixing process, and their heterogeneous photocatalytic activities were investigated to determine the degradation of airborne organic pollutants (benzene, ethyl benzene, and *o*-xylene (BEX)) under different operational conditions. The Fourier transform infrared spectroscopy results demonstrated the presence of GO for the TiO₂-GO composites. The average efficiencies of the TiO₂-GO mixtures for the decomposition of each component of BEX determined during the 3-h photocatalytic processes were 26%, 92%, and 96%, respectively, whereas the average efficiencies of the unmodified TiO₂ powder were 3%, 8%, and 10%, respectively. Furthermore, the degradation efficiency of the unmodified TiO₂ powder for all target compounds decreased during the 3-h photocatalytic processes, suggesting a potential deactivation even during such a short time period. Two operational conditions (air flow entering into the air-cleaning devices and the indoor pollution levels) were found to be important factors for the photocatalytic decomposition of BEX molecules. Taken together, these results show that a TiO₂-GO mixture can be applied effectively for the purification of airborne organic pollutants when the operating conditions are optimized.

Key words : Heterogeneous photocatalyst, Airborne organic pollutant, Graphene-TiO₂ mixture, FTIR spectra

1. Introduction

Photocatalytic oxidation over semiconductors has emerged as an attractive advanced oxidation technology for environmental treatment (Fujishima et al., 2007; Paz, 2010). Among the different semiconductors, titania (TiO₂) is commonly employed for water and air purification owing to its ability to oxidise photocatalytically, strong thermal and chemical resistance, and low cost (Fujishima et al., 2007; Paola et al., 2012). However,

the application of TiO₂ has been limited by its low photon efficiency, low adsorption potential, high recombination rate of electron-hole pairs, and narrow absorption wavelength range (Henderson, 2011). To overcome these disadvantages, several studies have coupled TiO₂ powder to various carbon materials such as powder-type activated carbon, fibrous activate carbon, carbon nanotubes, and graphene oxide (GO) (Ao and Lee, 2003; Chen and Oh, 2010; Jiang et al., 2011; Jo et al., 2011; Pastrana-Martinez et al., 2012).

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Among these carbon materials, GO has received special attention as a supporting substrate that can improve the photocatalytic activity of TiO₂ particles because of their synergistic nature of electronic and adsorption characteristics (Leary and Westwood, 2011). GO, which can be prepared using graphite with a two-dimensional monolayer of fused sp² carbon bonds, has been frequently utilized in photocatalyst-supporting substrates, nanoelectronic products, drug-delivery media, polymer mixtures, and for hydrogen-gas production and storage (Eda et al., 2008; Kamat, 2010). The electronic property of GOs is noted by the potential that the electrons in TiO₂ generated by photons can be mobilized through the GO sheets, which decreases the recombination of photon-produced electron-hole pairs (Lightcap et al., 2010). Moreover, GOs represent high-adsorption sites that increase the photocatalytic activity of TiO₂ nanoparticles (Stankovich et al., 2006). The coupling of TiO₂ to GO can extend light absorption into the visible range, thus activating the TiO₂-GO mixture under this range (Leary and Westwood, 2011). These superior characteristics of GOs have prompted their coupling to TiO₂ for application in environmental treatment.

Though several studies (Zhang et al., 2010; Jiang et al., 2011; Pastrana-Martínez et al., 2012) have attempted to couple TiO₂ to GO for treating environmental pollutants, they primarily dealt with the treatment of water pollutants such as methylene blue and methyl orange. In contrast, only a few studies on the application of TiO₂-GO mixtures on air pollutants were published. It is noteworthy that the photocatalytic results obtained from a water-solid conjunction may differ from those obtained from a gas-solid conjunction because the photon kinetics and reaction mechanism differ between the two conjunctions (Ochiai and Fujishima, 2012). To confirm this assertion, TiO₂-GO mixtures were synthesized in the present study by a simple chemical blending process, and their heterogeneous photocatalytic efficiencies were investigated for the

control of three major air organic pollutants—benzene, ethyl benzene, and *o*-xylene (BEX)—under visible-light irradiation. Because the application of such photocatalyst powder in air necessitates a support tool to minimize the amount of the catalyst from being blown away from the treated air, some photocatalytic tests were performed using a continuous-flow cylindrical glass tube as a supporting substrate (Jo and Kim, 2009; Shan et al., 2010). These tests were carried out under different operation conditions by varying the flow of the air being treated and the concentration of the target pollutants. The photocatalytic activity of the commercially available P25 TiO₂ was also investigated for comparing it with that of the TiO₂-GO mixture under the same operational conditions. The target pollutants (i.e., BEX) belong to one of the most important air pollutant groups frequently detected at elevated concentrations in both indoor and outdoor urban air environments (Schlink et al., 2010; de Blas et al., 2012). These compounds found in an urban atmosphere originate primarily from motor vehicle emissions, industrial combustion processes utilizing fossil fuels, petroleum handling, solvent usage, and other industrial processes (de Blas et al., 2012).

Major indoor sources involve cigarette smoke, building finishing materials, furnishings, cleaning compounds, dry cleaning agents, paints, glues, cosmetics, textiles, and combustion appliances (de Blas et al., 2012). They are also carcinogenic and/or toxic to the nervous system and liver (USEPA, 2008). These characteristics of the target pollutants have led to the application of TiO₂-GO mixtures for air-pollutant control.

2. Methodology

Fig. 1 shows a schematic diagram of the experimental setup. The photocatalytic degradation efficiencies of TiO₂-GO mixtures were investigated for the control of airborne BEX under different operational conditions

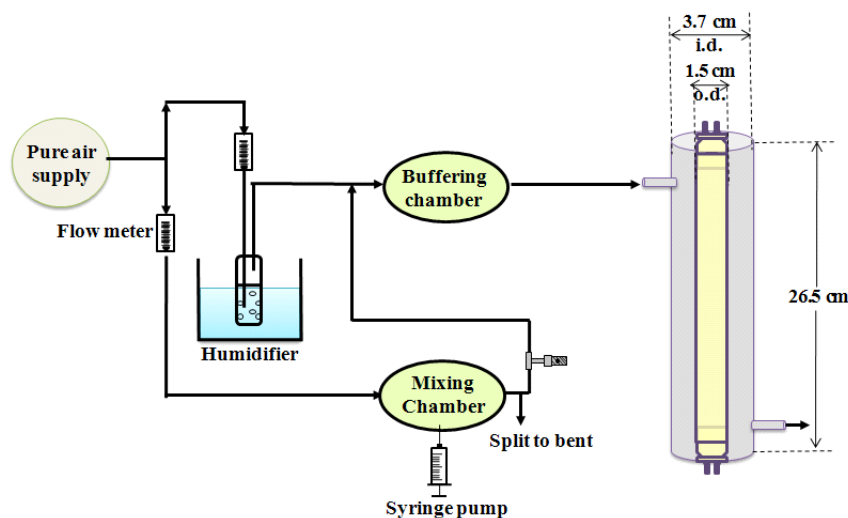


Fig. 1. Schematic diagram of experimental setup.

using a plug-flow glass tube reactor. In addition, the photocatalytic degradation efficiencies of pure P25 TiO_2 with the same weight were also determined for a comparison. The inner wall of the glass tube-type photocatalytic reactor was coated with a thin film of the TiO_2 -GO mixtures or pure TiO_2 powder. A cylindrical visible-light lamp (8-W fluorescent daylight lamp, F8T5DL, Youngwha Lamp, Co.) was installed inside the glass tube and served as the inner surface of the cylindrical reactor through which the gas flowed. Two primary operational factors, i.e., the flow rate (FR) and input concentration (IC), were investigated for the control of BEX. The FRs ranged from 1 to 4 L min^{-1} , which were controlled using identical rotameters ($0\text{--}0 \text{ L min}^{-1}$) calibrated using a mass-flow meter. The ICs ranged from 0.1–1.0 ppm, which included typical indoor air-quality levels. The desired concentrations were adjusted by varying the injection rates of standard pollutants using a syringe pump (KdScientific Model 210). Other operational factors were fixed to their representative values: FR, 1 L min^{-1} ; IC, 0.1 ppm. In addition, the relative humidity was fixed to 50% by passing clean dried air through a humidifier immersed in a water bath, which was placed immediately

adjacent to the photocatalytic reactor inlet.

TiO_2 -GO mixtures were synthesized using GO and TiO_2 powder with an anatase phase of 70% and a rutile phase of 30%. Graphite powder was dispersed in concentrated sulphuric acid and pure potassium permanganate in an ice-cooled bath while stirring vigorously. This solution was then heated to 40°C for 0.5 h, and deionized water was added to start an oxidation reaction. After 0.5 h, deionized water and hydrogen peroxide were slowly added to obtain a light-yellow solution. This suspended solution was centrifuged at a rate of 3000 rpm, after which it was washed with hydrogen chloride and acetone in sequence. This solid product was dried at 70°C for 12 h to fabricate GO powder, which was then added to deionized water and sonicated for 0.5 h, after which it was diluted with more deionized water. This solution was added to TiO_2 dispersed in deionized water, after which it was sonicated for 2 h, and then stirred for 3 h under lab conditions to obtain a homogeneous solution. This product was then filtered and dried in a vacuum at 50°C for 4 h. The characteristics of the TiO_2 -GO mixture were determined using Fourier transform infrared (FTIR) spectroscopy with a PerkinElmer

Spectrum GX spectrophotometer.

Air samples were collected periodically at the inlet and outlet of the photocatalytic reactor prior to and after turning on the light source. New light sources were employed every day of the experiment to minimize any confounding factors that could affect the photocatalytic results owing to different light-source ages. The coated photocatalysts were purified overnight by flowing clean air through the visible-light irradiated photocatalytic reactor. When no contamination with toluene or other gaseous compounds was monitored in the reactor, toluene was introduced into the photocatalytic reactor. Air sampling was carried out by filling an evacuated Tedlar bag, after which air was drawn from this bag through a Tenax-containing trap. Air pollutants collected in the Tenax trap were analysed through the coupling of a thermal desorption system (Perkin Elmer ATD 350) to a gas chromatograph (Agilent 7890A) with a flame ionization detector. The quality assurance program for the measurement

of gaseous compounds involved blank and spiked sample tests, as well as calibration calculations.

3. Results and discussion

The photocatalytic efficiencies of the TiO_2 -GO mixture, along with a reference TiO_2 photocatalyst, were determined under visible-light irradiation for the control of BEX. As shown in Fig. 2, the photocatalytic decomposition efficiencies of the TiO_2 -GO mixture were much higher than those of the reference TiO_2 particle. The average efficiencies of the TiO_2 -GO mixture for the decomposition of each component of BEX determined during 3-h photocatalytic processes were 26%, 92%, and 96%, respectively, whereas the corresponding average efficiencies of the TiO_2 powder were 3%, 8%, and 10%, respectively. Furthermore, the degradation efficiency of the TiO_2 powder for all the target compounds decreased during the 3-h photocatalytic processes, suggesting the presence of

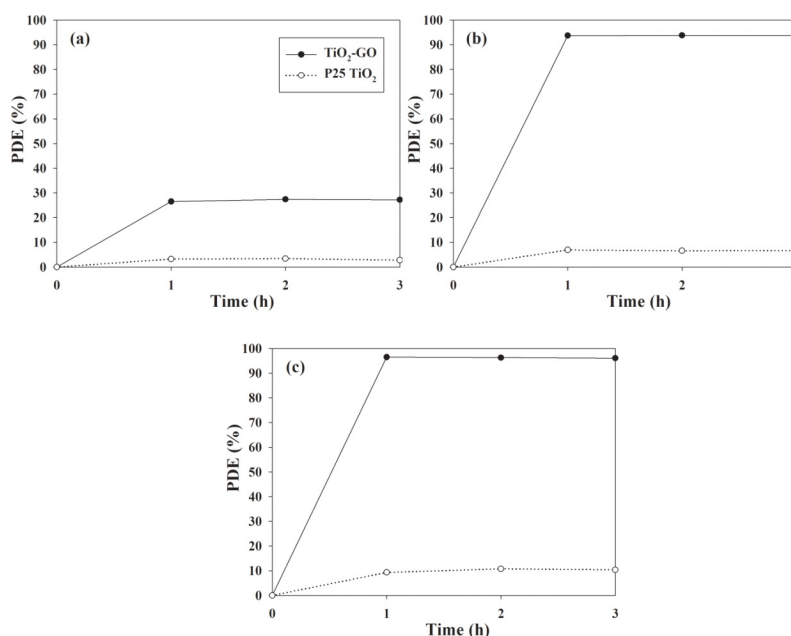


Fig. 2. Control photocatalytic decomposition efficiency (PDE, %) of (a) benzene, (b) ethyl benzene, and (c) *o*-xylene determined via TiO_2 -GO mixture, and P25 TiO_2 particles under visible-light irradiation.

potential deactivation even during such a short time period. As mentioned briefly in the Introduction section, the improved photocatalytic activity of the TiO₂-GO mixture was primarily attributed to the unique function of GO sheets in the composite. As shown in Fig. 3, the FTIR spectra demonstrated the presence of GO for the TiO₂-GO composite. A peak at 1630 cm⁻¹ was observed for the TiO₂-GO mixture, which was attributed to the stretching of carboxylic functional groups at the surface of GO (Zhang et al., 2010; Nguyen-Phan et al., 2011). Another peak appearing at 1230 cm⁻¹ was likely associated with the carboxyl C-O group at the GO surface (Nguyen-Phan et al., 2011). However, the peak at a low frequency (690 cm⁻¹), which was also observed for the uncoupled P25 TiO₂ and attributed to Ti-O-Ti vibration (Zhang et al., 2010), was not observed for the TiO₂-GO composite. Alternatively, a wide and deep peak at 730 cm⁻¹ appeared for the TiO₂-GO composite, which was attributed to the coupled effects of Ti-O-C and Ti-O-Ti vibrations, demonstrating the impregnation of GO into TiO₂ (Sakthive and Kisch, 2003; Zhang et al., 2010). In addition, two major peaks at 3400 cm⁻¹ appearing in the TiO₂-GO composite were assigned to an O-H stretching of water

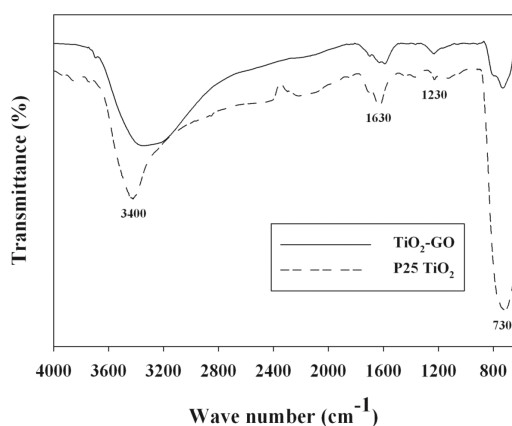


Fig. 3. Fourier transforms infrared of TiO₂-GO mixture and P25 TiO₂ particles.

vapours absorbed on the composite surface (Zhang et al., 2010; Nguyen-Phan et al., 2011).

GO acts as an excellent support for the adsorption of toluene, enhancing the photocatalytic activity of the TiO₂-GO mixture. Furthermore, GO acts as an electron acceptor that facilitates the interfacial electron transfer from TiO₂, owing to its 2-dimensional π conjugation nanostructure (Lightcap et al., 2010). Such electronic properties of GO can minimize the recombination of electron-hole pairs, improving the photocatalytic activity of the TiO₂-GO mixture. GO can also show photosensitizing characteristics, which can extend light absorbance into the visible range, activating the TiO₂-GO mixture under visible-light irradiation.

Fig. 4 illustrates the photocatalytic efficiencies of the TiO₂-GO mixture for the decomposition of BEX based on its FRs (1, 2, 3, and 4 L min⁻¹) under visible-light irradiation. The average efficiency of the TiO₂-GO mixture for the decomposition of BEX determined during the 3-h photocatalytic processes decreased as the FR increased. As the FR increased from 1 to 4 L min⁻¹, the average efficiencies of the TiO₂-GO mixture for the decomposition of BEX decreased from 29% to 12%, 95% to 58%, and 97% to 64%, respectively. These results are consistent with those obtained by Kontos et al. (2012), who used an undoped TiO₂ photocatalyst and found that the degradation efficiency of gaseous nitrogen oxide decreases when the FR is increased. In particular, at higher FRs, the gas-residence time in a continuous-flow photocatalytic reactor may be too short to for the sufficient transfer of toluene from the gas phase to the catalyst surface (Kontos et al., 2012). The residence times in the present study, which were calculated by dividing the reactor volume by the SFR, were 7.6, 3.8, 2.5, and 1.9 s for FRs of 1, 2, 3, and 4 L min⁻¹, respectively. Accordingly, the lower efficiencies at higher FRs suggest that an insufficient reactor residence time effect outweighs the bulk mass

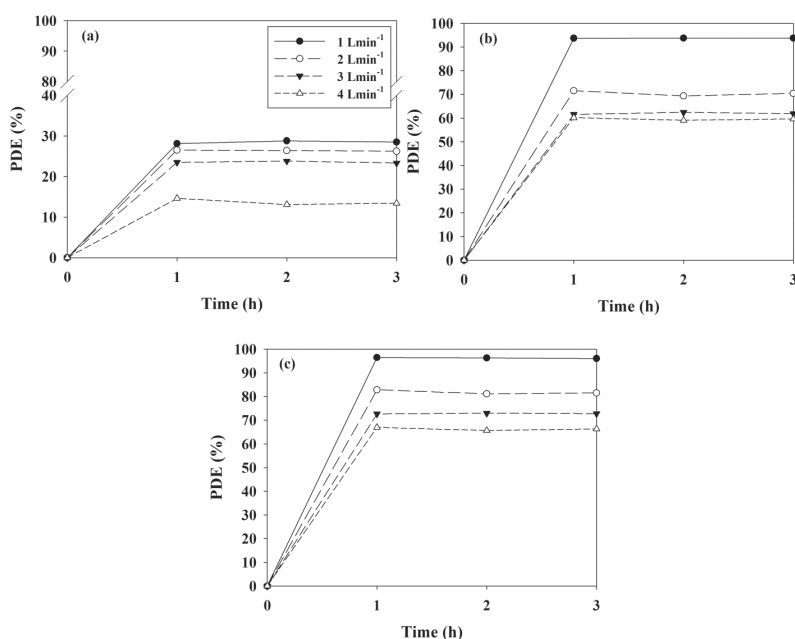


Fig. 4. Control photocatalytic decomposition efficiency (PDE, %) of (a) benzene, (b) ethyl benzene, and (c) *o*-xylene determined using TiO₂-GO mixture according to the stream flow rate (1, 2, 3, and 4 L min⁻¹).

transport effect of toluene degradation on the catalyst surfaces.

These findings indicate that FR is still a critical factor in the photocatalytic application of a TiO₂-GO mixture. However, Table 1 shows that, in contrast to the photocatalytic efficiencies of the TiO₂-GO mixture for the decomposition of BEX, the photocatalytic decomposition rates of BEX increased from 1.6×10^{-3} to 3.1×10^{-3} $\mu\text{-mole m}^{-2} \text{s}^{-1}$, 1.8×10^{-3} to 4.2×10^{-3} $\mu\text{-mole m}^{-2} \text{s}^{-1}$, and 1.8×10^{-3} to 4.5×10^{-3} $\mu\text{-mole m}^{-2} \text{s}^{-1}$, respectively, as the FR increased from 1 to 4 L min⁻¹. A likely explanation for these results is that the increased FRs increases the bulk mass transport of BEX molecules from the gas-phase to the surface of the photocatalyst particles owing to convection and diffusion phenomena, which are important heterogeneous catalytic reactions (Kontos et al., 2012). In the present case, the photocatalytic oxidation rate will increase with an increase in the FR, indicating that BEX removal is limited to the catalyst surface.

Table 1. Photocatalytic reaction rates (mole m⁻² s⁻¹) of BEX based on stream flow rate

Compound	Stream flow rate (L min ⁻¹)			
	1	2	3	4
Benzene	1.6×10^{-3}	2.0×10^{-3}	2.5×10^{-3}	3.1×10^{-3}
Ethyl benzene	1.8×10^{-3}	2.6×10^{-3}	3.3×10^{-3}	4.2×10^{-3}
Xylene	1.8×10^{-3}	2.9×10^{-3}	3.4×10^{-3}	4.5×10^{-3}

The photocatalytic degradation efficiencies of toluene obtained from a TiO₂-GO mixture under visible-light irradiation, according to the IC, are presented in Fig. 5. The degradation efficiency of toluene exhibited a decreasing trend with an increase in the IC. The average degradation efficiencies of toluene during a 3-h process decreased from 86% to 8% as the IC increased from 0.1 to 1.0 ppm. These findings are consistent with those reported in other studies (Pengyi et al., 2003; Jo et al., 2011) using undoped TiO₂ under UV irradiation. The IC dependence was ascribed

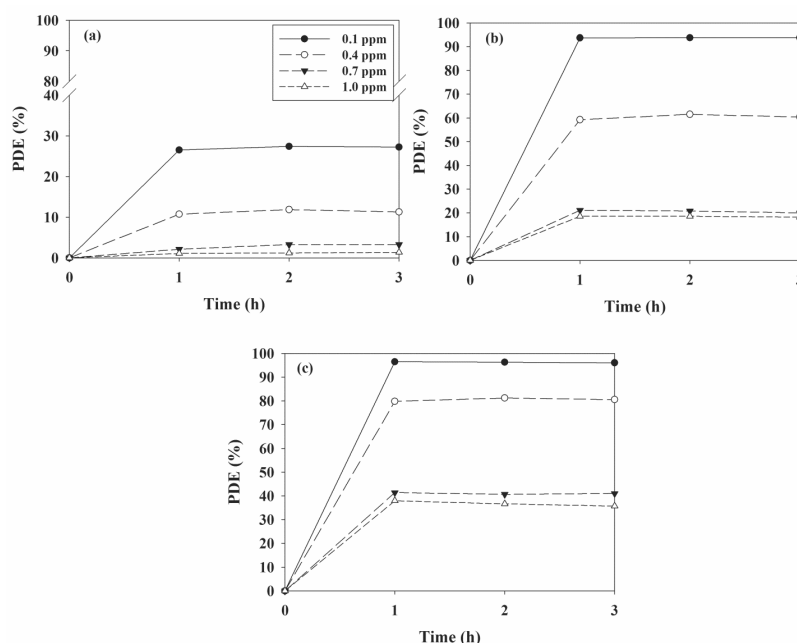


Fig. 5. Control photocatalytic decomposition efficiency (PDE, %) of (a) benzene, (b) ethyl benzene, and (c) *o*-xylene determined via TiO₂-GO mixture according to initial concentration (0.1, 0.4, 0.7, and 1.0 ppm).

to adsorptive competition between toluene molecules for the active adsorption sites on the surface of the TiO₂-GO mixture. In terms of higher ICs, the active adsorption sites on the photocatalyst surface might be more limited for the adsorption of toluene molecules. However, Table 2 shows that, in contrast to the photocatalytic efficiencies of the TiO₂-GO mixture for the decomposition of BEX, the photocatalytic reaction rates for the decomposition of BEX increased from 1.6×10^{-3} to $3.6 \times 10^{-3} \mu\text{-mole m}^{-2} \text{s}^{-1}$, 1.8×10^{-3} to $6.1 \times 10^{-3} \mu\text{-mole m}^{-2} \text{s}^{-1}$, and 1.8×10^{-3} to $6.9 \times 10^{-3} \mu\text{-mole m}^{-2} \text{s}^{-1}$, respectively, as the IC increased from 0.1 to 1.0 ppm. These results are related to the Langmuir-Hinshelwood (LH) adsorption isotherm model that is most commonly used to link the photocatalytic reaction rate of VOCs to their ICs (Demeestere et al., 2007). Based on the LH model, the increasing order of the photocatalytic reaction rates indicates that the concentration range used in the current study corresponds to the intermediate

regime, in which the photocatalytic reaction rate increases slowly but the degradation efficiency decreases rapidly. However, for a high regime, the photocatalytic oxidation might be independent of the ICs (zero-order reaction kinetics), while the degradation efficiency would decrease.

Table 2. Photocatalytic reaction rates (mole m⁻² s⁻¹) of BEX based on initial concentration

Compound	Input concentration (ppm)			
	0.1	0.4	0.7	1.0
Benzene	1.6×10^{-3}	2.5×10^{-3}	2.8×10^{-3}	3.6×10^{-3}
Ethyl benzene	1.8×10^{-3}	3.0×10^{-3}	4.3×10^{-3}	6.1×10^{-3}
Xylene	1.8×10^{-3}	3.2×10^{-3}	4.5×10^{-3}	6.9×10^{-3}

4. Conclusions

This study explored the photocatalytic activities of a TiO₂-GO mixture for the purification of airborne organic pollutants (BEX) under different operational

conditions. The FTIR spectra showed that the surface characteristics of the as-prepared TiO₂-GO mixture differed from those of the Degussa P25 TiO₂ photocatalyst, demonstrating the presence of GO in the TiO₂-GO composite. A major finding of this study is that the TiO₂-GO mixture exhibits superior photocatalytic activity compared to a reference undoped TiO₂ powder for the decomposition of BEX, which is mainly ascribed to the unique function of the GO sheets in the TiO₂-GO composite. In addition, two operational conditions (air flow entering into the air-cleaning devices and the indoor pollution levels) were found to be important factors for the photocatalytic decomposition of BEX molecules. Overall, the TiO₂-GO mixture can be effectively applied for the purification of airborne organic pollutants when the operational conditions are optimized.

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