



Enhancement effect of TiO₂ immobilized on activated carbon filter for the photodegradation of pollutants at typical indoor air level

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Abstract

The competition effect between water vapor and pollutants at ppb level for adsorption sites has been previously reported. It was found that at high humidity levels, the pollutant photodegradation rate reduced drastically. To improve the photodegradation of pollutants at ppb level and at high humidity levels, TiO₂ is immobilized on an activated carbon (AC) filter. 200 ppb of nitrogen oxide (NO) and 20 ppb of benzene, toluene, ethylbenzene and *o*-xylene (BTEX) were chosen as target pollutants. Sensitivity analyses were conducted for NO and BTEX under different residence times and levels of humidity. Results showed that the combination of TiO₂ and AC significantly increased NO and BTEX removals at short residence time and high humidity levels. TiO₂ immobilized on AC filter was less affected by the increasing humidity levels. NO₂, as an intermediate generated from the photodegradation of NO, was also successively suppressed from exiting the system. Deactivation from the photodegradation of NO was suppressed by immobilizing TiO₂ on an activated carbon filter. The use of activated carbon acted as a local pollutant concentrator by adsorbing pollutants from the air stream and thereby diffused to the TiO₂ for photodegradation.

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1. Introduction

People generally spend more than 80% of their time in an indoor environment [1] and the quality of indoor air has a vital impact on human health [2]. Due to the energy crisis in the 1970s, buildings were designed to be more airtight to save energy. With less fresh air intake and pollutants generated from building materials, occupants complained about sickness associated

with working in an indoor environment [3]. This phenomenon is known as sick building syndrome (SBS). In general, there are three mitigation measures to reduce SBS, namely source control, ventilation and air cleaning. The first two methods are usually impossible as the source is unreachable and the ventilation is often ungovernable by building occupants. Thus, air cleaning is a feasible and convenient method for the individual occupant to improve indoor air quality.

Photocatalysis is an emerging and promising technology for pollution remediation in the gaseous and aqueous phase [4–13]. Recently, studies [14–17] showed that the combination of photocatalyst TiO₂

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with adsorbents appeared to have higher photocatalytic efficiency. Takeda and coworkers [18,19] showed that the combination of TiO₂ with mordenite has a higher photodegradation rate for gaseous pyridine and propionaldehyde by concentrating pollutant on adsorbent and diffused to TiO₂ for photodegradation. The above studies, however, were all conducted at ppm levels and the effects of humidity levels are not investigated.

In the present study, the objective is to investigate the photodegradation of indoor air pollutants at the ppb level using TiO₂ loaded on activated carbon (AC) filter. It is of special interest to use TiO₂ with adsorbent for indoor pollutant removal as the concentration is extremely low and the competition for adsorption sites between water vapor and pollutants is most critical. Nitrogen oxide (NO), benzene, toluene, ethylbenzene and *o*-xylene (BTEX) were chosen as target pollutants as they are most commonly found in many urbanized cities such as Hong Kong [20–22]. Previously, we reported [23] the water competition effect of NO and BTEX at ppb level. In this study, the effect of humidity levels and residence times are investigated by using TiO₂ immobilized on activated carbon filter. To the best of our knowledge, no detailed investigation has been reported on the photodegradation of NO and BTEX at ppb level under different levels of humidity, by TiO₂, loaded on an activated carbon filter.

2. Experimental

2.1. Reagents and catalyst preparation

The experimental procedure was described previously, and the only change was the use of a smaller size reactor [23]. BTEX (spectra gases) with a mixing ratio of 1:1:1:1 and NO (BOC gases) were used as reactant gas and acquired from compressed gas cylinder at a concentration of 1 ppm ± 2% and 50 ppm ± 2% with nitrogen as balanced gas with traceable National Institute of Standards and Technology (NIST) standard. TiO₂ (Degussa P-25) was used as a photocatalyst. The catalyst was used as received without any pretreatment. Water suspension with 5% of TiO₂ was coated on a glass fiber filter (Whatman) as a supporting substrate (TiO₂ filter). It was then calcinated at 120 °C for 1 h with a temperature gradient of 5.5 °C per minute.

The same procedure was followed for TiO₂ loaded on activated carbon filter (TiO₂/AC), except an activated carbon filter (23.085 g ± 0.5%) was used instead of a glass fiber filter. The surface area of the glass fiber filter is identical to the activated carbon, which is 20 cm × 21 cm. The amount of TiO₂ imposed is determined by the weight difference before and after the coating procedure. In all experiments, the weight of TiO₂ imposed is 1.64 g ± 5%.

2.2. Reactor and experimental setup

A reactor with a volume of 18.61 (20.1 H × 44.2 L × 21 W cm) with its surface coated by a Teflon film (BY-TAC Type AF-21) was used for this study. Illumination was provided by a 6 W UV lamp (Cole-Parmer) which emits a primary wavelength at 365 nm and its intensity was determined by a UV meter (Spectroline DRC-100X). The UV lamp was horizontally placed at the upper part of the reactor, 14 cm from both ends. UV intensity measured in all experiments was 750 μW/cm². The TiO₂ coated filter was supported by a Teflon film and fixed horizontally with a vertical distance of 5 cm between the UV lamp. Stainless steel sampling ports and Teflon tubing were used to connect the reactor and the analytical instruments.

A zero air generator (Thermo Environmental Inc. Model 111) was used to supply the air stream. Desired humidity of the flow was controlled by passing the zero air stream through a humidification chamber. The reactant stream and the zero air stream were connected to a mass flow calibrator (Advanced Pollution Instrumentation Inc. Model 700). The gas streams were mixed by a gas blender and the desired flow was controlled by a mass flow controller inside the calibrator. After the inlet and the outlet concentration achieved equilibrium (1 h), the UV lamp was turned on and initiated the reaction. The concentration of NO was continuously measured by a Chemiluminescence NO analyzer (Thermo Environmental Instruments Inc. Model 42c), which monitors NO, NO₂, and NO_x at a sampling rate of 0.7 l/min. Pre-cleaned Summa canisters were evacuated for BTEX sampling. Constant BTEX sampling time was achieved using a mass flow controller. Samples of BTEX were collected at designated times during the experiment. After collection, the canister sample was first concentrated by a Nutech Cryogenic Concentrator (Model 3550A), and the

trapped BTEX were separated and analyzed by Hewlett-Packard gas chromatograph (Model HP 6890) and quantified by a mass selective detector (Model HP5973). After analysis, the canister was sequentially evacuated and pressurized with humidified zero air until all compounds detected were smaller than 0.2 ppb. TO-14 (Toxi-Mat-14M Certified Standard (Matheson)) standard gas was analyzed using the GC/MS system seven times at 0.2 ppb to obtain the method detection limits [21]. Aldehydes samples were collected by an adsorbent cartridge (Waters) coated with 2,4-dinitrophenylhydrazine (2,4-DNPH) with an ozone scrubber at a flowrate of 1 l/min. Prior to the experiment, the background aldehydes samples were collected at the time when the pollutant was generated. After turning on the UV lamp, samples of the aldehydes were also collected. The cartridges were sequentially dissolved in acetonitrile and analyzed by a high performance liquid chromatography (HPLC) (Waters) according to the USEPA TO-11 method [24]. The concentrations of the aldehydes were calibrated by a standard purchased from Superco (CARB Method 1004 DNPH Mix 2). The reported aldehydes concentrations during the photodegradation process were subtracted from the background aldehydes concentration.

3. Results and discussion

3.1. Characterization of the catalyst and filter

The Brunauer–Emmett–Teller (BET) surface area of the activated carbon powder extracted from the activated carbon filter was determined by nitrogen adsorption–desorption isotherm measurements at 77 K on a Micromeritics ASAP 2000 nitrogen adsorption apparatus. The BET surface area of the TiO₂ powder and the activated carbon powder was 1115 and 46 m²/g, respectively. Scanning electron micrographs of the glass fiber filter, used as substrate, and the different views of fiber coated with TiO₂ were shown previously [23]. Fig. 1(a) shows the micrograph of the activated carbon which has a porous structure. The activated carbon filter consists multiple layers of glass fiber layer and activated carbon with a glass fiber layer at the top and bottom surface, as shown in Fig. 1(b). The glass fiber layer is so lean that the activated carbon is visible on its surface. Hence, the activated car-

bon filter presented a speckled appearance. After calcination, TiO₂ particles agglomerated and adhered on the activated carbon. On the utmost layer, the activated carbon and the glass fiber are coated with TiO₂. Fig. 1(c) clearly shows that TiO₂ particles are in contact with the activated carbon. Fig. 1(d) shows that the inner layer of the glass fiber and the activated carbon is also coated with TiO₂. The smaller size TiO₂ enable it to be penetrated and impinged inside the activated carbon filter.

3.2. Adsorption of NO and BTEX

Fig. 2(a) shows the adsorption of 200 ppb NO at a humidity level of 2100 ppmv under different residence time. Each experiment set was conducted four times and the average value was reported. Three kinds of filter were tested, namely TiO₂ filter, AC filter and TiO₂/AC filter. Under continuous flow at different residence times, no adsorption of NO was found for the TiO₂ filter within the experimental error. The adsorption capacity of the AC filter and TiO₂/AC filter was identical, owing to no adsorption capacity having been found on TiO₂. In addition, the BET surface area of the AC is 24 times higher than the TiO₂ (P-25) and the adsorption contributed by the TiO₂ is not significant. Matos and coworkers [25] also showed that no additive adsorption capacity was found when TiO₂ (P-25) was added to AC. The adsorption capacity of NO increased, with increasing residence time, for the AC filter and TiO₂/AC filter. The amount of NO adsorbed increased from 9 to 35% when the residence time increased from 0.6 to 3.7 min. With other parameters such as weight of carbon and inlet pollutant concentration fixed, the increased in residence time (decreased in volumetric flowrate) reduced the amount of pollutant exited the outlet stream [26].

Fig. 2(b) shows the adsorption of 20 ppb BTEX at a humidity level of 2100 ppmv under different residence time. The adsorption capacity of the AC filter was identical to that of TiO₂/AC filter. No adsorption of BTEX was found for TiO₂ within the experimental error. The four compounds have a very similar adsorption capacity, with only 1–2% difference. It is also reported [27] that the amount of benzene, toluene and *o*-xylene adsorbed on AC is similar.

Fig. 2(c) shows the adsorption of 200 ppb at a residence time of 1.2 min under different humidity levels.

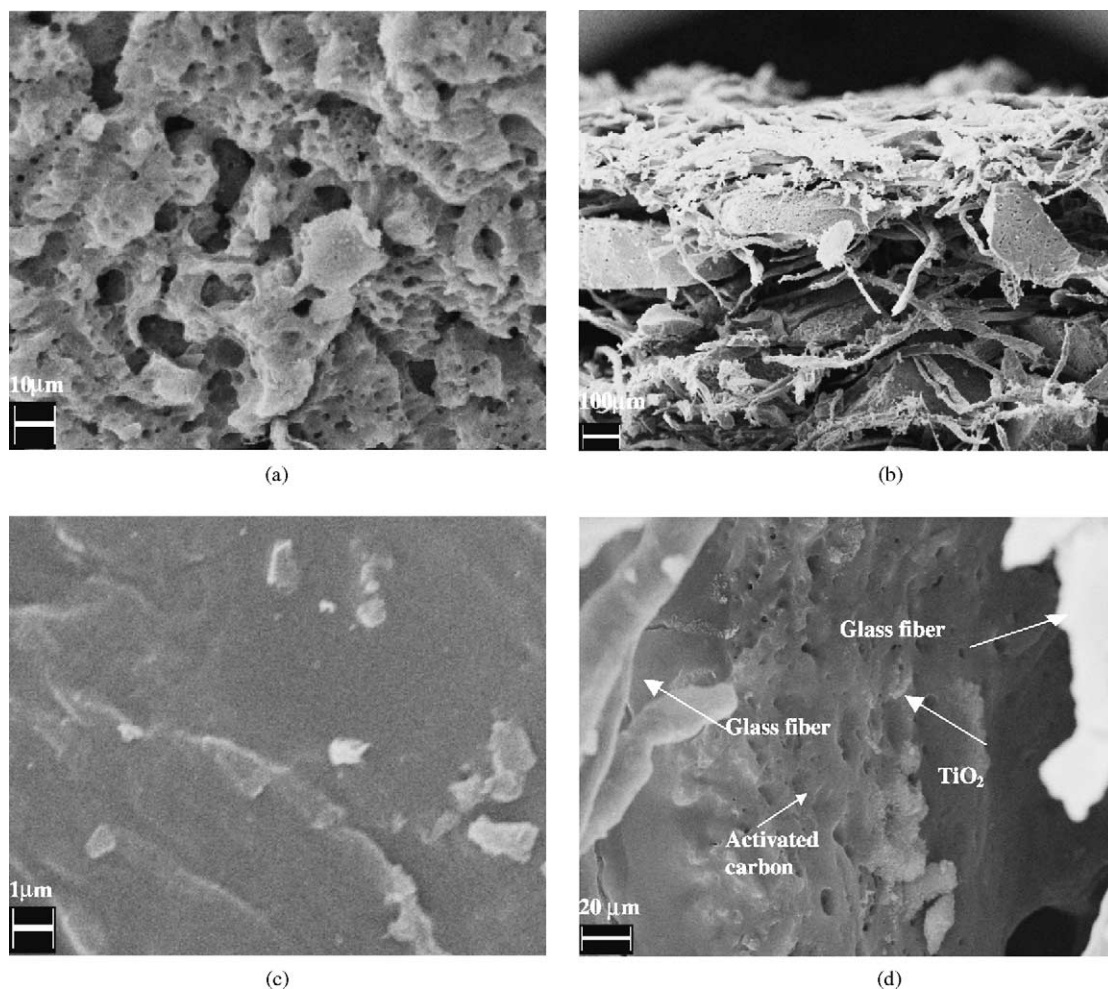


Fig. 1. Scanning electron micrographs of: (a) activated carbon; (b) glass fiber comphend with activated carbon; (c) intimate contact of TiO₂ and activated carbon; (d) inner layer of activated carbon coated with TiO₂.

The NO adsorption capacity decreased with increasing humidity levels. When the humidity level increased beyond 16,000 ppmv, the NO adsorption capacity significantly reduced. Richter and others also reported [28] that high levels of relative humidity inhibited the adsorption of NO on activated carbon. As seen in Fig. 2(d), the adsorption capacity of BTEX also shows a similar result. Studies found that the presence of water vapor significantly reduced the pollutant adsorption capacity on AC. At a relative humidity of 20,300 ppmv, capillary condensation of water vapor occurred inside the AC and blocked the adsorption

sites for the pollutant [29,30]. In this study, the adsorption capacity of NO and BTEX also significantly reduced at a similar humidity level. The above results suggested that the adsorption of pollutants primarily occurs on the activated carbon.

Blank tests were also conducted for the AC filter. The concentrations of NO and BTEX with the presence of illumination were monitored using an AC filter. No change in NO and BTEX concentrations were observed between the presence and absence of UV illumination. The result showed that only adsorption occurred on AC filter but no photolysis or photocatalysis

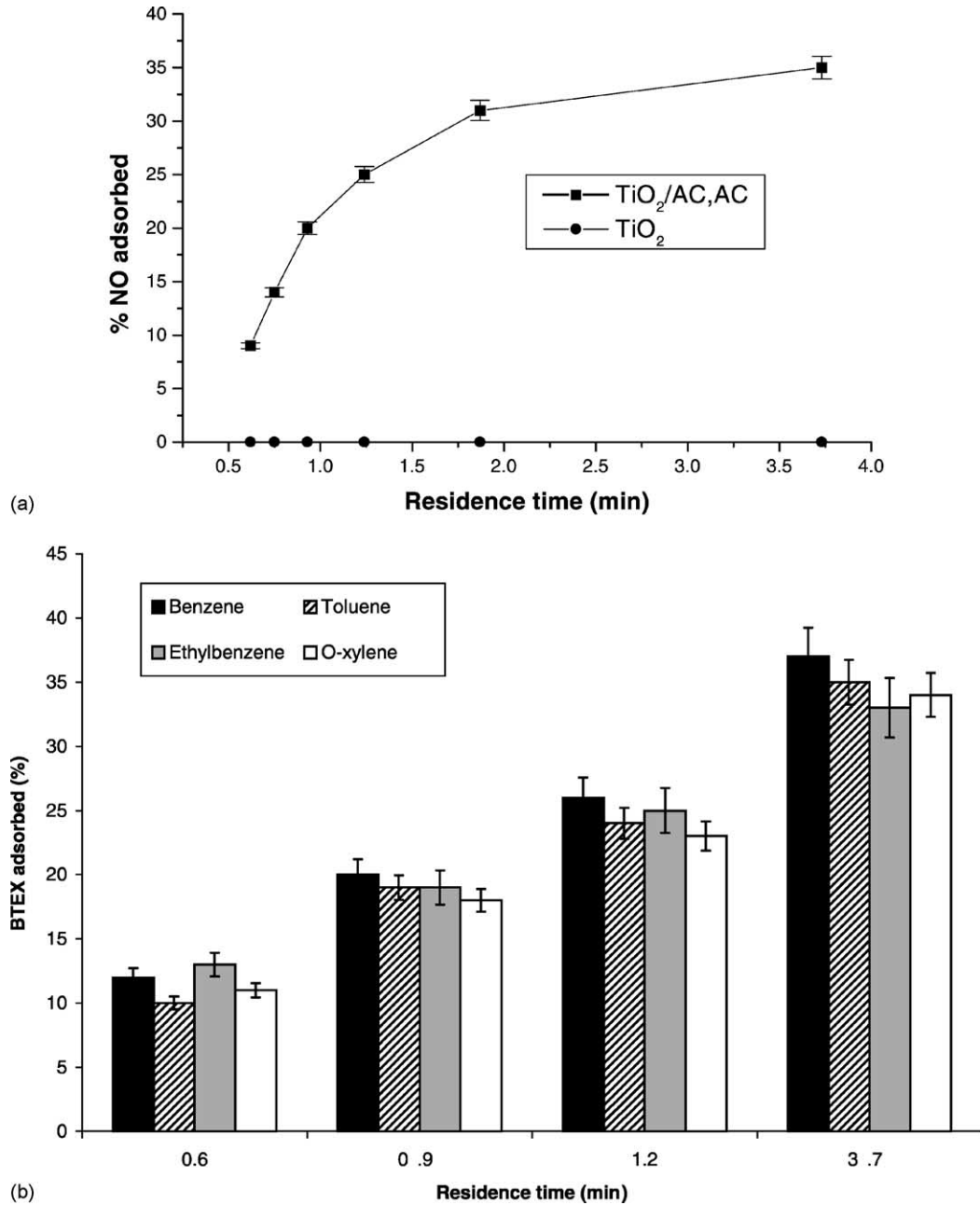


Fig. 2. (a) Amount of NO adsorbed in the dark under different residence time: humidity level 2100ppmv, 200ppb NO. (b) Amount of BTEX adsorbed in the dark under different residence times: humidity level 2100ppmv, 20ppb BTEX. (c) Amount of NO adsorbed in the dark under different humidity levels: residence time 1.2min, 200ppb NO. (d) Amount of BTEX adsorbed in the dark under different humidity levels: residence time 1.2min, 20ppb BTEX.

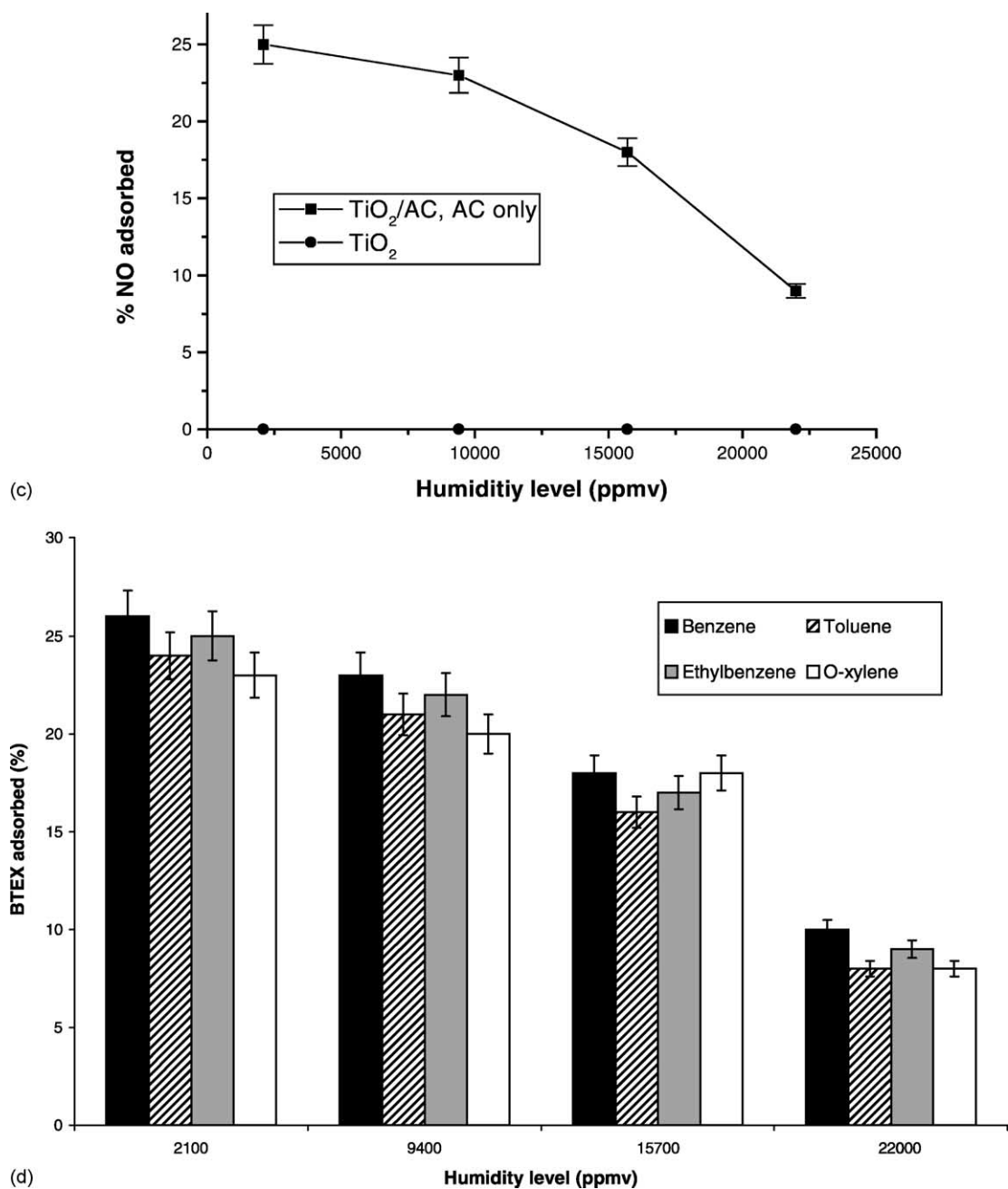


Fig. 2. (Continued).

was observed. For the TiO₂ glass fiber filter, no photodegradation of NO or BTEX occurred without the presence of UV illumination or the absence of TiO₂, as previously reported [23].

It is also reported that the adsorption of pollutants on activated carbon increased with increasing temperature [29,31]. All the experiments were conducted at a temperature of 25 ± 1 °C. This small temperature

variation on the pollutant adsorption capacities is insignificant compared to the effect of residence time and humidity levels. Matos and coworkers [25] also reported that the adsorption is not affected when the temperature increased by 1 °C.

3.3. Photodegradation of NO and BTEX by TiO₂ and TiO₂/AC under different residence time

The removals of benzene, toluene, ethylbenzene and *o*-xylene at a humidity level of 2100 ppmv under different residence times are shown in Fig. 3(a–d), respectively. The corresponding flowrates used in this study ranged from 5 to 30 l/min. The removal rate of the BTEX is calculated by Eq. (1)

$$\text{removal(\%)} = \frac{\text{initial concentration} - \text{final concentration}}{\text{initial concentration}} \times 100\% \quad (1)$$

where initial concentration is the inlet pollutant concentration and final concentration is the photosteady-state concentration at an irradiation time of 120 min [23]. The removals of BTEX using TiO₂ and TiO₂/AC increased with increasing residence time. At a longer residence time, a longer contact time between the pollutants and the hydroxyl radicals was achieved. At a residence time of 3.7 min, the removal rate between TiO₂ and TiO₂/AC is not significant. This is probably due to the fact that the pollutant diffusion rate from the gaseous phase to TiO₂ is similar to the pollutant diffusion rate from AC to TiO₂ under such a long residence time. In addition, under a low humidity environment where the competition effect between the water vapor and the pollutants for active adsorption sites is not significant, the pollutants are easily adsorbed on TiO₂ and photodegraded [23]. As the residence time decreased, however, the removals of BTEX between TiO₂ and TiO₂/AC increased significantly. Although the amount of BTEX adsorbed on the AC decreased from 35% (on average) to 10% (on average) when the residence time decreased from 3.7 to 0.6 min, a substantial amount of BTEX can still be adsorbed on the AC. The BTEX adsorbed on AC is then diffused to the nearby TiO₂ for photodegradation. It suggests that at a lower residence time, the collision rate of the pollutants from the gaseous phase decreased more sig-

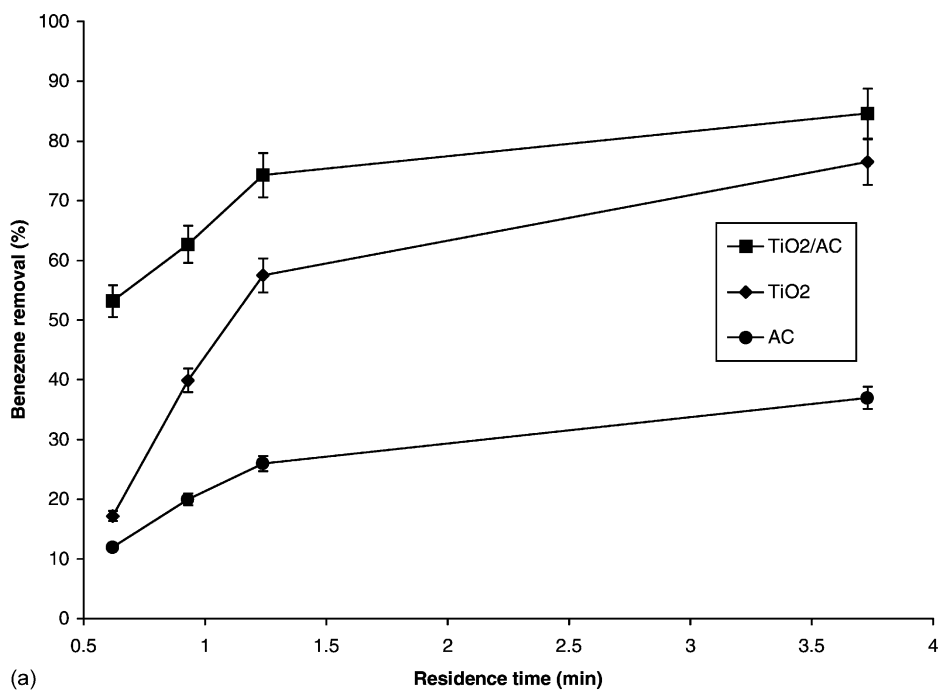
nificantly than the pollutants supplied from the AC. Studies [19,32] also showed that the addition of AC concentrated the pollutants around TiO₂ and increased the photodegradation rate. It is also noted that at the same residence time, the removal of *o*-xylene is higher than ethylbenzene, followed by toluene and benzene. The reasons were explained in elsewhere [23].

Fig. 3(e) shows the photodegradation rate of NO with the identical experimental conditions as shown in Fig. 3(a–d). Using TiO₂/AC, the NO removal is higher compared with using TiO₂. Similar to the adsorption of BTEX on the AC, the amount of NO adsorbed on AC decreased from 35 to 9% when the residence time decreased from 3.7 to 0.6 min. However, the NO_x removal rate between using TiO₂/AC and TiO₂ is not as significant as BTEX. Under low humidity in which the competition effect between water vapor and NO is not significant, the high NO removal rate under the experimental conditions might hinder the effect of AC.

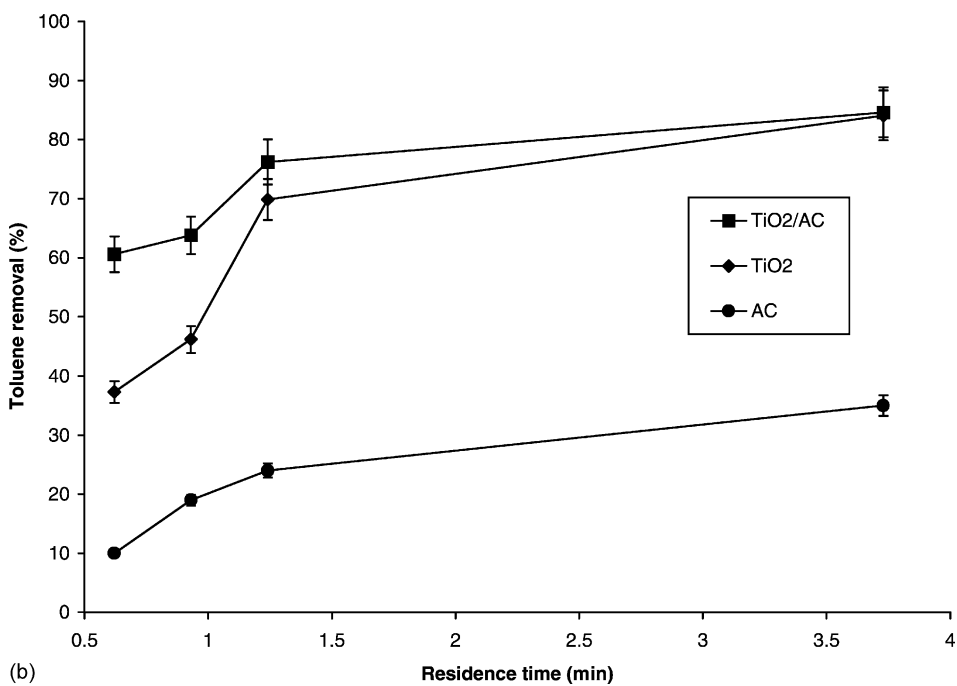
The removal of NO₂ is defined as the amount of NO₂ generated with respect to the initial concentration of NO. The generation of NO₂ increased with decreasing residence time. For instance, the generation of NO₂ increased from 1.6 to 9.2% when the residence time decreased from 3.7 to 0.6 min. The effect of TiO₂/AC on the generation of NO₂ also increased with decreasing residence time. NO₂ can be considered as an intermediate as shown in the following equations [33,34]



It is presumed that the NO₂ generated from the photodegradation of NO is assembled on the AC by its large adsorption capacity. The adsorbed NO₂ is further photodegraded to HNO₃, which reduced the amount of NO₂ exiting the system. The results clearly showed that the combination of TiO₂ and AC not only increased the target pollutants (NO and BTEX) removal but also reduced the amount of the intermediate (NO₂). It is also reported [32] that the use of TiO₂ loaded with zeolite also reduced the amount of intermediate for the photodegradation of propylamide in aqueous phase. Using TiO₂ or AC alone, the BTEX and NO removal rates are low. The combination of TiO₂ and AC has an enhancement effect on BTEX and NO removal rates.

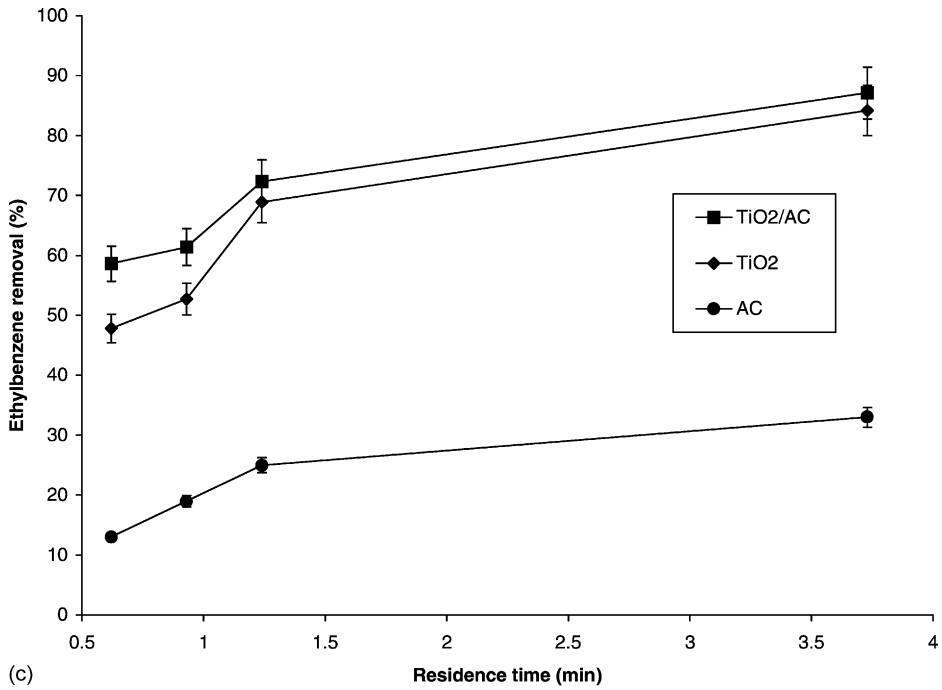


(a)

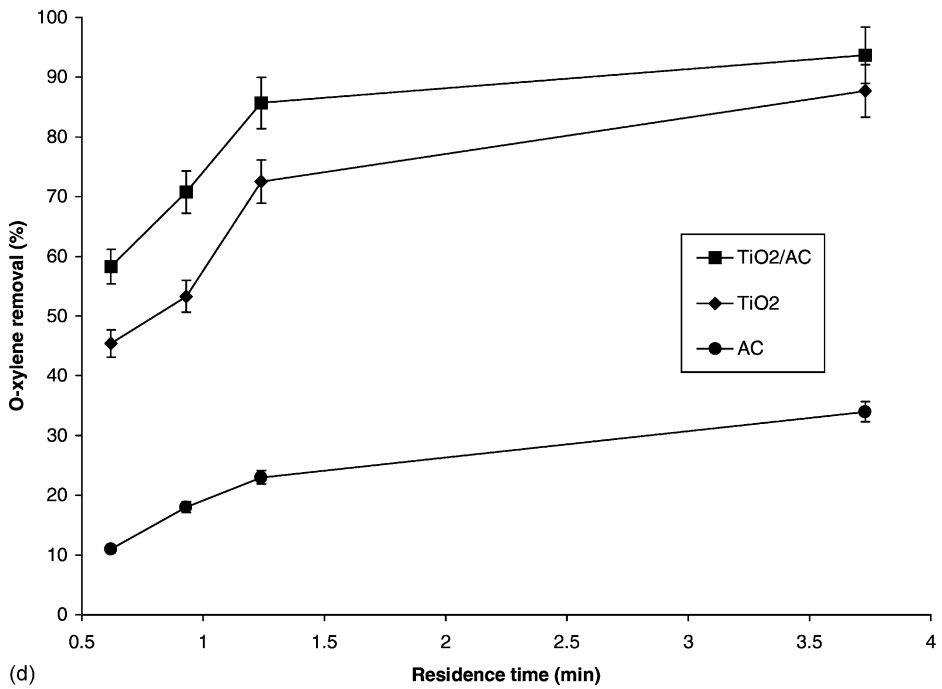


(b)

Fig. 3. (a) Benzene conversion under different residence time: humidity level 2100 ppmv, 20 ppb BTEX. (b) Toluene conversion under different residence times. Humidity level 2100 ppmv, 20 ppb BTEX. (c) Ethylbenzene conversion under different residence times. Humidity level 2100 ppmv, 20 ppb BTEX. (d) *o*-Xylene conversion under different residence time. Humidity level 2100 ppmv, 20 ppb BTEX. (e) NO and NO₂ conversion under different residence time: humidity level 2100 ppmv, 200 ppb NO.



(c)



(d)

Fig. 3. (Continued).

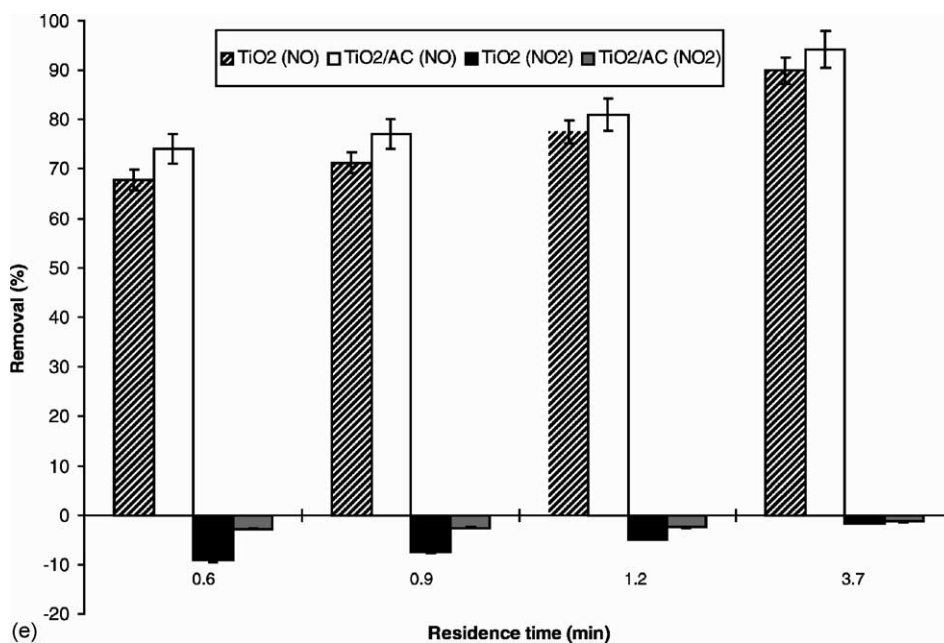


Fig. 3. (Continued).

3.4. Photodegradation of NO and BTEX by TiO₂ and TiO₂/AC under different humidity levels

The removals of benzene, toluene, ethylbenzene and *o*-xylene at a residence time of 1.2 min under different humidity levels are shown in Fig. 3(a–d), respectively. The removals of BTEX decreased significantly with increasing humidity levels owing to the competition effect between benzene and water vapor as we previously reported [23]. When TiO₂ was immobilized on the AC filter, the removal of BTEX only decreased by 14, 13, 9 and 18%, respectively when the humidity level was increased from 2100 to 22,000 ppmv. The removals of BTEX decreased by more than 50% when TiO₂ was immobilized on the glass fiber filter when the humidity level was increased from 2100 to 22,000 ppmv. The results clearly show that the combination of TiO₂ and AC increased the BTEX removals significantly. This is probably due to the amount of BTEX adsorbed on the AC under different humidity levels. Although at high humidity levels water competed with benzene for adsorption sites on TiO₂ and AC, the large adsorption capacity of AC still able to adsorb pollutants (Fig. 2(c–d)). At a humidity level

of 22,000 ppmv, 7.5% (on average) of BTEX was adsorbed on the AC. By immobilizing TiO₂ on AC filter, a portion of the TiO₂ is in contact with the AC which concentrated pollutants by adsorption. It is presumably that the difference in pollutant concentration between the TiO₂ adsorbed with water and the TiO₂ in contact with the AC contributes to the diffusion of pollutants from the AC to the TiO₂ for photodegradation. In essence, the AC acted as a local pollutant concentrator and supplier to the TiO₂ for photodegradation. Once the pollutant diffused from the AC to the TiO₂, AC adsorbed pollutants again from the gaseous stream and continued to diffuse to TiO₂. This pollutant transfer cycle from gaseous phase to AC to TiO₂ is the key factor for the improved pollutant removal. However, study [15] showed that the use of TiO₂ and AC did not greatly increase the photodegradation rate despite the AC concentrated the pollutants. This discrepancy is probably due to the method of TiO₂ immobilization on AC and experimental conditions being different. The work of these researchers is based on a mechanical mix of TiO₂ powder with AC powder, using ppm level NO as target pollutant. In this study, TiO₂ is immobilized on an AC filter and intimate

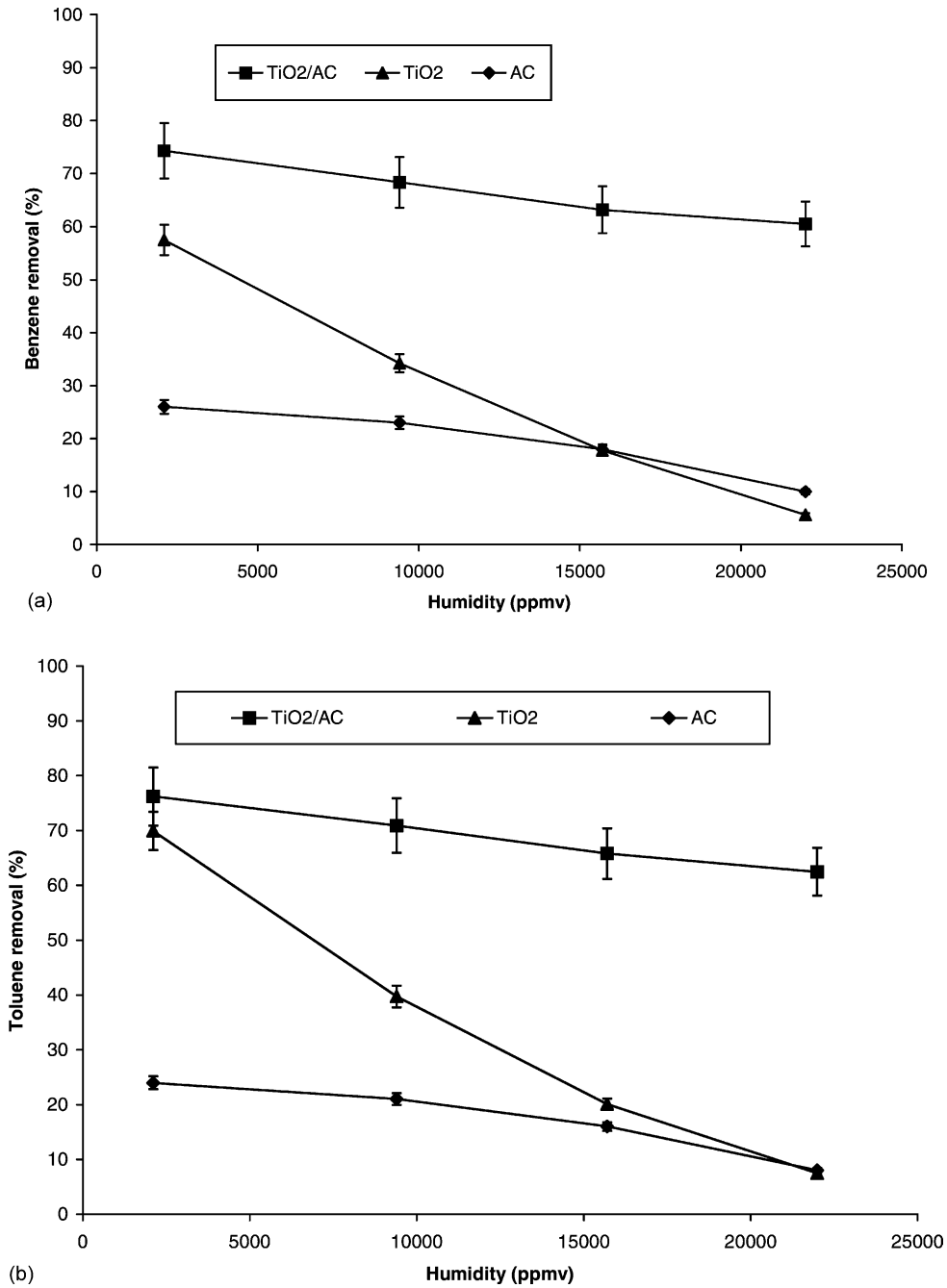
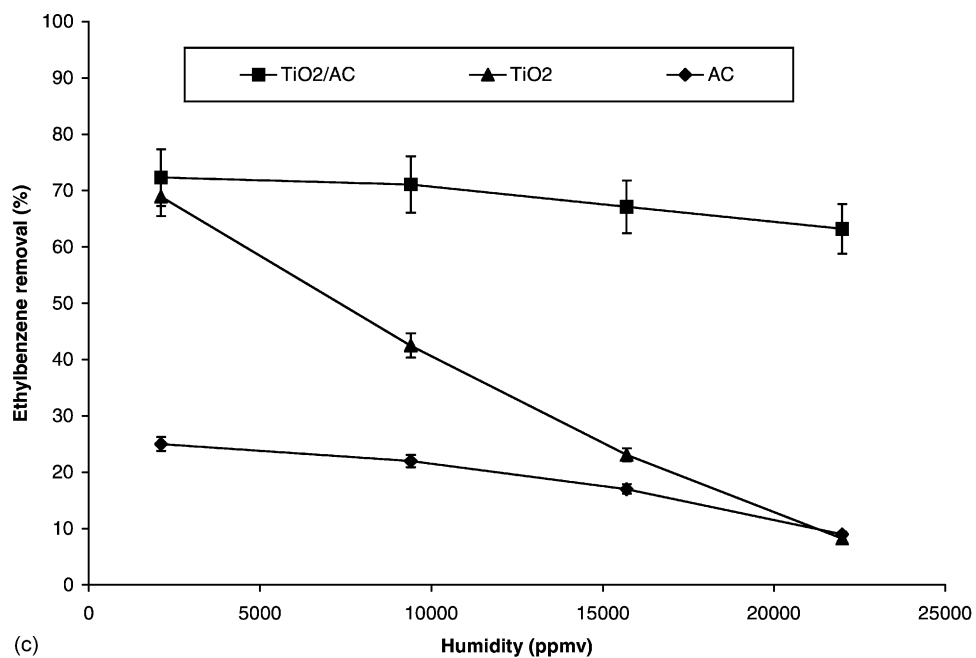
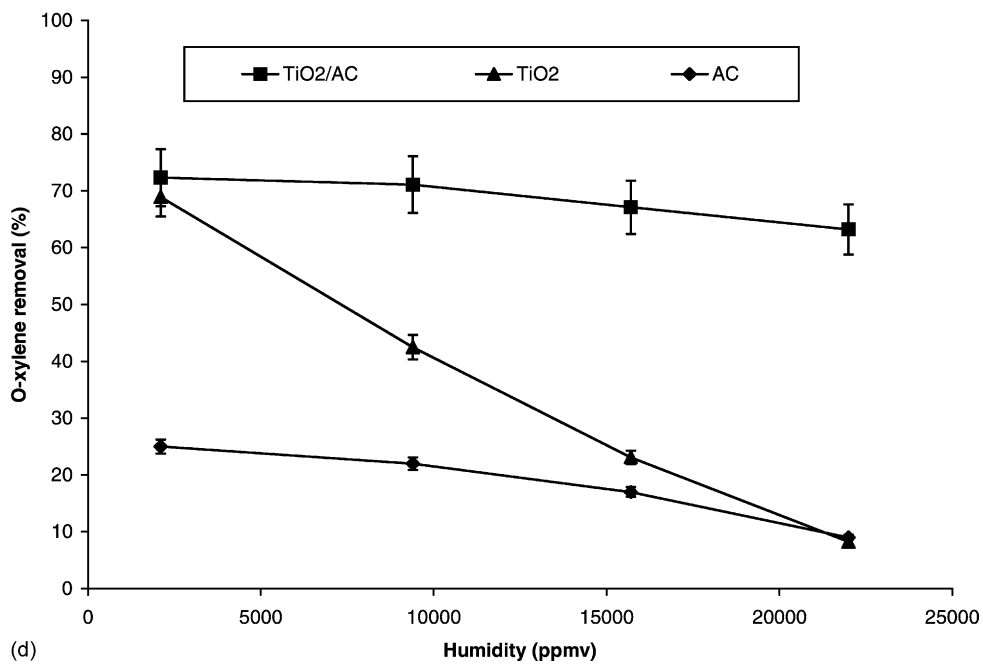


Fig. 4. (a) Benzene conversion under different humidity levels: residence time 1.2 min, 20 ppb BTEX. (b) Toluene conversion under different humidity levels: residence time 1.2 min, 20 ppb BTEX. (c) Ethylbenzene conversion under different humidity levels: residence time 1.2 min, 20 ppb BTEX. (d) *o*-Xylene conversion under different humidity levels: residence time 1.2 min, 20 ppb BTEX. (e) NO and NO₂ conversion under different humidity levels: residence time 1.2 min, 200 ppb NO.



(c)



(d)

Fig. 4. (Continued).

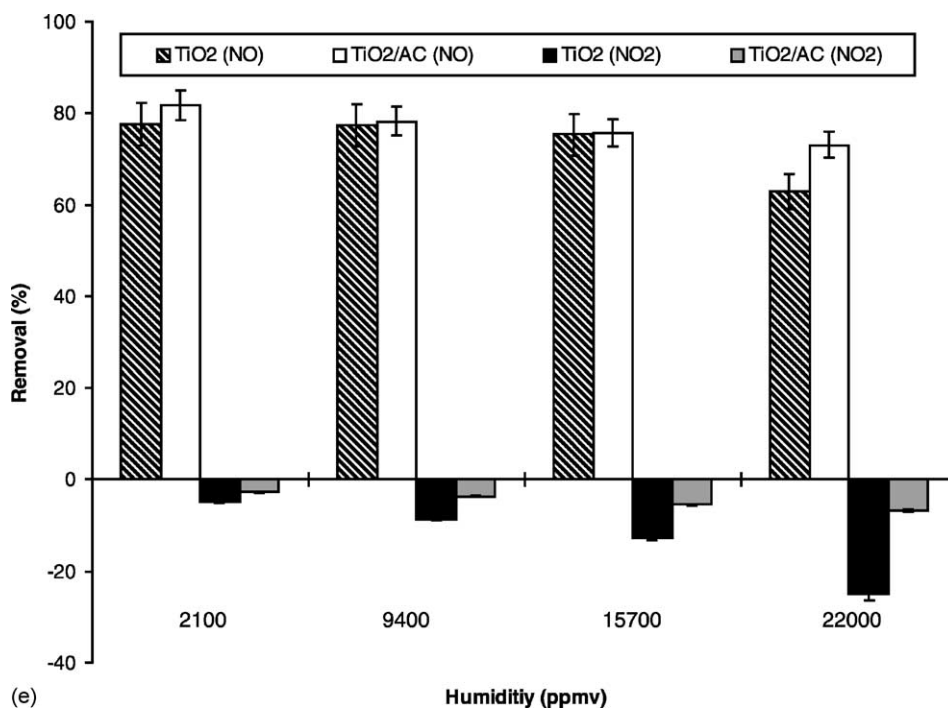


Fig. 4. (Continued).

contact is observed from the SEM micrographs. It is probably that the intimate contact between TiO₂ and AC is the key factor for pollutant diffusion and thus enhanced the photodegradation. It is also noted that the pollutant concentrations are lower in this study. At a ppb level concentration, the effect of adsorption and water vapor is more vital than ppm level concentration. Thus, the problem of competition for adsorption sites between water vapor and pollutants is successfully resolved by adsorbing pollutants on AC. From the above results, an enhancement effect of TiO₂ and AC is observed even under high humidity levels. The removal of TiO₂/AC is much higher than using TiO₂ or AC only. Previously we reported no aldehydes were detected for TiO₂ immobilized on a glass fiber filter. Aldehydes were also not detected using TiO₂/AC.

Fig. 4(e) shows the NO removal with the same experimental conditions as shown in Fig. 4(a–d). A higher NO removal was also achieved when TiO₂ is immobilized on an AC filter. The use of TiO₂/AC is less affected by the increasing humidity levels. As

can be seen clearly in the same figure that the use of TiO₂/AC decreased NO₂ generation. The NO₂ generation increased with increasing humidity levels owing to the water competition effect [23]. By immobilizing TiO₂ on the AC filter, the NO₂ generation from the photodegradation of NO is adsorbed and thereby further photodegraded to HNO₃ according to Eq. (3). Without the presence of AC, the NO₂ generated could not be adsorbed on TiO₂ and exiting the system.

3.5. Long term activity and deactivation

Previously we reported [23] that no deactivation of BTEX occurred when TiO₂ is immobilized on a glass fiber filter. Using an AC filter as the coating substrate, no deactivation was also observed for BTEX. The same TiO₂/AC was tested five times under the same conditions and the BTEX removals were within $\pm 5\%$ for each test.

Deactivation was found for NO when TiO₂ is immobilized on a glass fiber filter. From the practical point of view, it is important to have a long life filter and

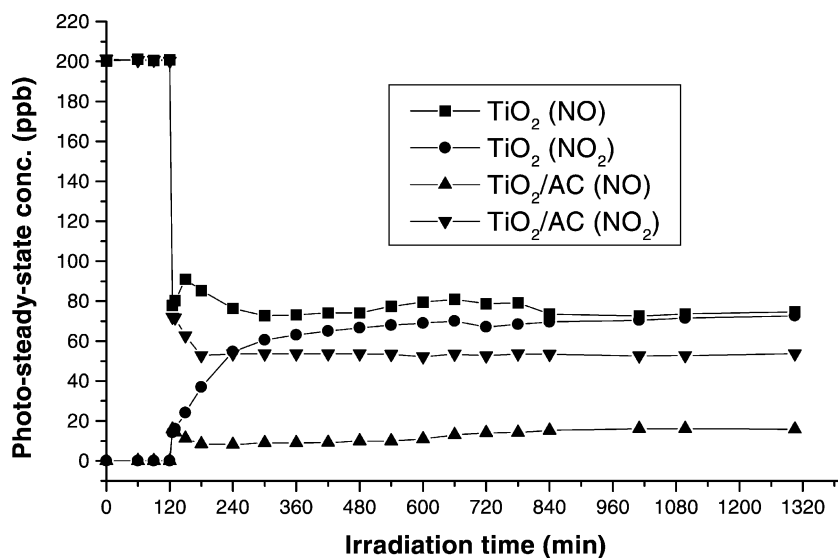


Fig. 5. Long term activity of TiO₂ and TiO₂/AC. Residence time 1.2 min, humidity level 22,000 ppmv, 200 ppb NO.

thus avoid the frequency of replacements. As shown in Fig. 5, the photosteady-state concentrations of NO and NO₂ using TiO₂ and TiO₂/AC at a residence time of 1.2 min and a humidity level of 22,000 ppmv. The use of the AC filter demonstrated clearly the suppression of NO₂ exiting the system. Deactivation was observed using TiO₂ only and the NO₂ concentration increased from 14.2 to 72.5 ppb, as shown in Fig. 5 from 5 to 1320 min. Using TiO₂/AC, no increase in NO₂ concentration was observed. The large adsorption capacity of AC successively decreased NO₂ exited to the system and increased the life time of the filter.

4. Conclusion

The removal of NO and BTEX at typical indoor air ppb levels by the use of TiO₂ immobilized on an activated carbon filter has significantly improved the pollutant removal. The improved removal using TiO₂/AC increased with decreasing residence time and increasing humidity levels compared to TiO₂. At a humidity level of 22,000 ppmv, only less than 10% of NO and BTEX are removed using AC only, whereas 40% and less than 10% of NO and BTEX respectively, are removed by TiO₂ only. When TiO₂ is immobi-

lized on activated carbon filter, the removal of NO increased to 66% and more than 60% for BTEX. The increased removal of using TiO₂/AC is owing to the large adsorption capacity of AC. Pollutants adsorbed on AC are diffused to the TiO₂ for photodegradation. The pollutant concentrations in AC are then reduced and adsorbed pollutants from the gaseous phase again and diffused to TiO₂. This adsorption and photodegradation cycle is suggested for the improved removal of TiO₂/AC. NO₂, as an intermediate from the photodegradation of NO, was also suppressed significantly by the use of TiO₂/AC. No deactivation was found for the photodegradation of BTEX. When using TiO₂/AC under prolonged testing, no deactivation was found for the NO which was found when using TiO₂ only. The combination of photocatalysis and adsorption is a promising direction for the photodegradation for indoor ppb levels pollutant.

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