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Article

Aluminum sheet-based S-doped TiO₂ for photocatalytic decomposition of toxic organic vapors

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ABSTRACT

S-doped TiO₂ (S-TiO₂) films were immobilized on flexible low-cost aluminum sheets (S-TiO₂-AS) using a sol-gel dipping process and low post-processing temperatures. The photocatalytic degradation of toxic organic vapors using the prepared films was evaluated using a continuous-flow glass tube under visible light exposure. The surface properties of the S-TiO₂-AS and TiO₂-AS films were examined by scanning electron microscopy, energy-dispersive X-ray spectroscopy, X-ray diffraction, and ultraviolet-visible spectroscopy. The photolysis of benzene, toluene, ethyl benzene, and xylene (BTEX) did not occur on the bare AS. In contrast, the photocatalytic degradation efficiencies of the target pollutants using S-TiO₂-AS were higher than those obtained using reference TiO₂-AS photocatalyst. In particular, the average photocatalytic degradation efficiencies of BTEX using S-TiO₂-0.8-AS (S/Ti ratio = 0.8) over a 3-h process were 34%, 78%, 91%, and 94%, respectively, whereas those of TiO₂-AS were 2%, 11%, 21%, and 36%, respectively. The photocatalytic decomposition efficiencies of BTEX under visible irradiation using S-TiO₂-AS increased with increasing S/Ti ratios from 0.2 to 0.8, but decreased when the ratio further increased to 1.6. Thus, S-TiO₂-AS can be prepared using optimal S/Ti ratios. The degradation of BTEX over S-TiO₂-AS depended on the air flow rates and initial concentrations of the target chemical. Overall, under optimal conditions, S-TiO₂-AS can be effectively applied for the purification of toxic organic vapors.

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1. Introduction

TiO₂ n-type semiconductor consists of three polymorphs (anatase, rutile, and brookite), and is the most widely applied heterogeneous photocatalyst for environmental pollutant treatment owing to its oxidation capacity, chemical and thermal stability, and low cost [1–3]. For photocatalysis to proceed, energies greater than the band gap of TiO₂ are required to excite an electron from the valence band to the conduction band, producing an electron and positive hole pair. Ultraviolet (UV) is needed to effectively activate TiO₂ because the band gaps of anatase and rutile phases are 3.2 (≤ 387 nm) and 3.0 eV (≤ 413

nm), respectively, thereby limiting the use of TiO₂ in environmental applications. Therefore, many studies have been conducted to modify TiO₂ to extend the photocatalysis processes to the visible light range for environmental applications [4]. Modification techniques include non-metal impregnation [5,6], transition and noble metal impregnation [6,7], dye sensitization [8], and semiconductor coupling [9]. Particular attention has been paid to sulfur (S) impregnation in water and air purification applications because S absorbs visible light strongly, unlike other non-metal elements such as C and N [5,10,11]. Ohno et al. [5] reported that S-impregnated TiO₂ (S-TiO₂) exhibited strong visible light absorption and photocatalytic performance for the

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degradation of 2-propanol and methylene blue in aqueous media.

In environmental purification schemes, a support material for nanoparticles is necessary to avoid the post-separation process of the suspended nanoparticles from the treated water and to minimize their loss from the photocatalytic reactors with clean air during air purification [12,13]. A range of solid materials, such as aluminum sheets (AS) [14], glass fibers [15], stainless steel [16], glass tubes [11,17–20], and polymer materials [21], have been suggested as supports for nano-sized photocatalysts for the degradation of environmental contaminants. Specifically, continuous-flow glass tubes containing a photocatalyst via dip-coating of the inner wall in the photocatalyst solution have been widely used for the degradation of gaseous contaminants such as aromatic hydrocarbons, dimethyl sulfide, trichloroethylene, and perchloroethylene [11,17–20]. Dip-coating of the inner wall of the glass tubes with the photocatalyst requires a specifically designed apparatus that fits according to the geometry and size of the glass tube reactors, hence necessitating the use of simpler processes. This issue can be addressed by lining the inner wall of the glass tube with a flexible solid sheet coated with a photocatalyst film. AS are flexible and relatively cheap, and thus can be used as a support substrate for nano-sized photocatalysts [14]. Moreover, AS were proposed as a promising support for TiO_2 nanoparticles as they could easily fit according to the geometry and size of the glass tube reactors.

In this study, S- TiO_2 was immobilized onto a flexible AS using a simple sol-gel dipping process, and its photocatalytic degradation ability to decompose toxic organic vapors was evaluated using a continuous-flow glass tube under visible light irradiation. Low post-processing temperatures following photocatalyst coating are preferable to minimize loss of the photocatalytic activity resulting from interfacial interactions between the photocatalyst film and support substrate [22]. In contrast, the many conventional sol-gel dipping methods that have been used to prepare thin TiO_2 films on support materials require high post-processing temperatures ($\geq 300\text{ }^\circ\text{C}$) to crystallize the amorphous TiO_2 particles and burn out the residual organic substrates [23–25]. Therefore, in this study, the immobilization of S- TiO_2 onto AS (S- TiO_2 -AS) was conducted under low-temperature post-treatment conditions (room temperature). Four toxic organic vapors (benzene, toluene, ethyl benzene, and *o*-xylene—also known as BTEX) were selected because of their high detection occurrence in urban indoor and outdoor environments [26] and adverse health effects [27].

2. Experimental

2.1. Preparation of photocatalysts

The S- TiO_2 -AS photocatalysts were synthesized using a hydrolysis method followed by a dip-coating process and a low-temperature post-treatment process. Specifically, 0.25, 0.5, 2.0, and 4.0 g thiourea (99%, Sigma-Aldrich) were respectively mixed with 100 mL ethanol (99.9%, Sigma-Aldrich). The mixtures were stirred for 1 h, to which 2 g P25 TiO_2 powder (De-

gussa) was added, and the resulting mixtures were stirred for 4 h. The stirred solutions were then dried at $85\text{ }^\circ\text{C}$ for 20 h, after which the final products were ground and calcined at $450\text{ }^\circ\text{C}$ for 4 h at a heating rate of $2\text{ }^\circ\text{C}/\text{min}$ to obtain the white S- TiO_2 powder samples at varying S/Ti ratios of 0.2, 0.4, 0.8, and 1.6. To prepare the coating solution, 50 mL titanium (IV) isopropoxide (97%, Sigma-Aldrich) was mixed with 10 mL glacial acetic acid (99%, Sigma-Aldrich) with constant stirring. Then, the mixture was added dropwise to 1 L distilled water, after which 10 mL HNO_3 (98%, Sigma-Aldrich) was added and stirred for 30 min to produce a white precipitate. This product was heated to $80\text{ }^\circ\text{C}$ for 4 h in an oil bath to give a transparent sol and then further stirred for 2 h. The previously prepared S- TiO_2 powder (2 g) was added to this sol, and the mixture was sonicated for 1 h to obtain the final coating solution. For the dip-coating process, a previously cleaned and dried aluminum sheet (11 cm $\times$ 21 cm) was first dipped into the as-prepared coating solution (500 mL) in a glass container (12 cm $\times$ 23 cm $\times$ 2 cm) for 10 min. Then, the dip-coated sheet was slowly removed from the coating solution and dried at room temperature for 2 h. The dip-coating and low-temperature post-treatment processes were repeated twice to obtain the required level of coating. The S- TiO_2 -AS at S/Ti ratios of 0.2, 0.4, 0.8, and 1.6 are denoted as S- TiO_2 -0.2-AS, S- TiO_2 -0.4-AS, S- TiO_2 -0.8-AS, and S- TiO_2 -1.6-AS, respectively. In addition, a reference photocatalyst, TiO_2 -AS, was prepared using the same procedure as that used for the preparation of S- TiO_2 -AS, but in the absence of thiourea. The properties of S- TiO_2 -AS and TiO_2 -AS were examined by scanning electron microscopy (SEM, Hitachi S-4300), energy-dispersive X-ray spectroscopy (EDX, field-emission scanning microscope equipped with an EDX-350 unit), X-ray diffraction (XRD, Rigaku D/max-2500), X-ray photoelectron spectroscopy (XPS), transmission electron microscopy (TEM), and UV-visible spectroscopy (UV-Vis, Varian CARY 5G).

2.2. Photocatalytic degradation efficiency (PDE)

The photocatalytic degradation efficiencies (PDEs) of BTEX using S- TiO_2 -AS, TiO_2 -AS, and bare AS were determined. A glass tubing reactor (26.5 cm length and 3.8 cm inner diameter) with the inner wall lined up with S- TiO_2 -AS, TiO_2 -AS, or AS (Fig. 1) was used. A cylindrical daylight source (Youngwha Lamp Co., F8T5DL, 400–720 nm) was introduced into the reactor. Zero-grade air supplied by the air cylinder was allowed to flow through a hydrocarbon filter for additional purification. To prepare the standard gases, the purified air stream was passed through a humidification system consisting of water-containing

Fig. 1. Schematic diagram of the photocatalytic reactor.

impingers, after which the humidified air was directed to a glass mixing container and mixed with a standard BTEX solution that was injected using a syringe pump (KdScientific Lega-to 100). Finally, the standard gases flowed into the glass tubing reactor to test the photocatalytic performance of S-TiO₂-AS, TiO₂-AS, and AS.

The photocatalytic activity was investigated under a range of operating conditions by varying two major parameters: air flow rate (AFR) and input concentration (IC) of the target gas. The AFRs investigated were 1.0, 2.0, 3.0, and 4.0 L/min, and the ICs surveyed in this study were 0.1, 0.5, 0.7, and 0.9 ppm that are relevant to typical indoor air quality [26]. While testing a specific parameter, the other parameter was fixed to its representative value (AFR: 1.0 L/min or IC: 0.1 ppm). In addition, the relative humidity was fixed to 45%, which is within the human comfort range (40%–60%). The light intensity provided by the daylight lamp (8 W) was 2.8 mW/cm², which was measured at a distance from the lamp to the inner wall of the photocatalytic reactor using a Black-Ray radiometer (Model J-221). A visible light lamp was used to evaluate the application of the as-prepared photocatalysts for environmental applications under visible light exposure.

A time-series of air measurements were performed at the upstream and downstream ports of the Pyrex reactor before and after activating the lamp. After injecting the standard gases, but prior to light activation, air sampling was conducted using an evacuated Teflon bag. The adsorption equilibrium between the catalyst and BTEX occurred approximately 1 h after injection of the standard chemicals. This indicated that similar upstream and downstream air concentrations were achieved. Therefore, the light source was activated 1 h after injecting the standard chemicals, after which air samples were collected. The control test that was conducted on the bare glass tubing reactor while the light source was on showed no degradation of the target chemicals. The entire experimental run was conducted in triplicate to obtain reliable data and mean values are reported.

The gas-phase species were determined by gas chromatography mass spectrometry (GC-MS, PerkinElmer Clarus SQ 8); the unit was equipped with a thermal desorbing device (PerkinElmer ATD 350). The gaseous species were analyzed qualitatively based on both the retention times and mass spectra (Wiley 275 software library). Quantitative analysis was conducted using calibration equations determined from five standard concentrations of each compound. Laboratory blank and spiked standard samples were analyzed for quality control. The method measurement limits varied between 0.005 and 0.008 ppm, depending on the target compound. In addition, CO and CO₂ concentrations were determined via an online gas chromatograph/flame ionization detector (GC/FID)-CH₄ converting instrument (Synspec B.V. Model Alpha 12).

3. Results and discussion

3.1. Surface properties of as-prepared photocatalysts

The surface properties of a representative Al-based S-TiO₂ (i.e., S-TiO₂-0.8-AS) and reference photocatalysts (TiO₂-AS)

Fig. 2. SEM images of S-TiO₂-0.8-AS (a) and TiO₂-AS (b) samples.

were surveyed by SEM, EDX, XRD, and UV-Vis spectroscopy. Figure 2 presents the SEM images of S-TiO₂-0.8-AS and TiO₂-AS. The crystallite size of S-TiO₂-0.80-AS was smaller than that of TiO₂-AS. Liu et al. [28] also reported the smaller particle size of S-TiO₂, which was prepared using an acid-catalyzed hydrolysis process, relative to that of unmodified TiO₂. These results were ascribed to the incorporation of S into the TiO₂ crystal lattice that could promote dispersion of nanoparticles, thereby reducing the particle size of TiO₂.

As observed in Fig. 3, the EDX spectrum of S-TiO₂-0.8-AS displayed bands corresponding to Ti, O, and S elements, whereas that of TiO₂-AS only showed O and Ti characteristic peaks. The bands of the Ti and O elements were assigned to crystalline TiO₂, whereas the S-element band was attributed to S imbedded into the TiO₂ lattice. Liu et al. [28] reported the presence of S elements for S-TiO₂ prepared using the acid-catalyzed hydrolysis process via XPS. The XPS analysis also confirmed the presence of S, Ti, and O atoms in S-TiO₂-0.8-AS (Fig. 4). The non-designated peaks in the EDX patterns were

Fig. 3. EDX spectra of S-TiO₂-0.8-AS and TiO₂-AS samples.

Fig. 4. XPS spectrum of S-TiO₂-0.8-AS sample.

assigned to the Pt coating of the photocatalyst samples during the pretreatment process.

Figure 5 shows the XRD patterns of S-TiO₂-0.8-AS and TiO₂-AS. Both samples showed an anatase crystal structure with a major band at $2\theta = 25.2^\circ$ and a rutile crystal structure with a major band at $2\theta = 27.4^\circ$. These results were similar to those of pure P25 [29]. Thus all three photocatalysts (S-TiO₂-0.8-AS, TiO₂-AS, and P25), regardless of S-doping, featured similar band positions. This was attributed to the low amounts of S in the S-TiO₂ films. In addition, the XRD pattern of S-TiO₂-0.8-AS was consistent with those obtained from S-TiO₂ samples prepared using titanium tetrachloride and thiourea as Ti and S sources, respectively. In contrast, Ohno et al. [5] reported that S-TiO₂, which was prepared using titanium isopropoxide and thiourea as Ti and S sources, respectively, displayed an anatase crystal phase only.

Figure 6 shows the UV-Vis absorbance spectra of S-TiO₂-0.8-AS and TiO₂-AS. The reference TiO₂-AS showed a light absorption edge at $\lambda \approx 410$ nm, which is consistent with that observed by other studies on P25 [29,30]. In contrast, the absorption spectrum of S-TiO₂-0.8-AS considerably shifted towards the visible region, which are consistent with those obtained from other studies [5,28]. These red shifts were attributed to increased charge transfer rates between S and TiO₂ owing to the impregnated and/or substituting S atoms in the TiO₂ lattice that could generate impurity levels that could lower the band gap of TiO₂ [5,28]. This suggests that the prepared

Fig. 5. XRD patterns of S-TiO₂-0.8-AS and TiO₂-AS samples.

Fig. 6. UV-Vis spectra of S-TiO₂-0.8-AS and TiO₂-AS samples.

S-TiO₂-0.8-AS can function effectively under visible light irradiation. The calculated band gap energies of S-TiO₂-0.8-AS and TiO₂ were 2.75 and 3.02 eV, respectively.

3.2. Photocatalytic activity

The PDEs of BTEX using S-TiO₂-AS with different S/Ti ratios, TiO₂-AS, and AS were determined under visible light irradiation after a dark-adsorption process. Figure 7 shows the PDEs of BTEX over a 3-h photocatalytic process. The photocatalytic degradation of the target compounds would occur as a result of reactions involving hydroxyl radicals and/or super-oxide radical ions generated after initial formation of reactive electron and hole pairs when the photocatalysts were irradiated under light [2,4]. The control test involving the bare AS revealed negligible photolysis of BTEX. Additionally, the PDEs of the four target chemicals over the S-TiO₂-AS were higher than that determined for the reference TiO₂-AS. In particular, the average PDEs of BTEX over S-TiO₂-0.8-AS were 34%, 78%, 91%, and 94%, respectively, whereas those over TiO₂-AS were 2%, 11%, 21%, and 36%, respectively. This suggests that S-TiO₂-AS has superior photocatalytic activity over the reference photocatalyst for the degradation of toxic organic vapors under visible light irradiation. Ohno et al. [5] also reported that S-TiO₂ powder had higher activity than non-modified TiO₂ powder under visible light irradiation at wavelength longer than 420 nm. Bayati et al. [10] showed that the photocatalytic activity of S-TiO₂ nanoporous films was much higher than that of pure TiO₂ layers under visible light exposure. The higher photocatalytic activity of the S-TiO₂-AS samples was attributed to their effective visible light absorbance, owing to the substitution of Ti⁴⁺ by S⁴⁺ [5]. The shift in light absorption into the visible range for the prepared S-TiO₂-AS samples was demonstrated by UV-Vis spectroscopy, as discussed earlier (Fig. 6). In addition, the increased charge transfer rates between S and TiO₂ would decrease recombination rates of the electron-hole pairs, thereby enhancing the photocatalytic activity of S-TiO₂-AS [5,28]. Furthermore, the PDEs of BTEX over S-TiO₂-0.8-AS were higher than those obtained over glass-coated S-TiO₂, as reported in a previous study [31]. These were attributed to the different operating conditions employed between the two studies.

Fig. 7. Photocatalytic degradation efficiencies of benzene (a), toluene (b), ethyl benzene (c), and *o*-xylene (d) over the S-TiO₂-AS with different S/Ti ratios, TiO₂-AS, and AS under visible light irradiation.

Figure 7 also shows that the PDEs of BTEX over the S-TiO₂-AS photocatalysts under visible light irradiation increased with increasing S/Ti ratios from 0.2 to 0.8, but decreased when the ratio was increased further to 1.6. This suggests the existence of an optimum S/Ti ratio for the synthesis of S-TiO₂-AS with high photocatalytic activity. The decline in the PDEs for S-TiO₂-AS with a S/Ti ratio of 1.6 was ascribed to the presence of excess sulfur ions that might serve as centers for the recombination of the photon-produced charge carriers (electrons and holes) [10]. In addition, excess sulfur ions could absorb incident photons, thereby reducing light exposure onto the catalyst, consequently lowering the photocatalytic activity.

As shown in Fig. 8, the PDEs of all target compounds over S-TiO₂-0.8-AS decreased with increasing AFR. At the highest AFR (4.0 L/min), the PDEs of BTEX were 10%, 22%, 39%, and 56%, respectively, and at the lowest AFR (1.0 L/min), the PDEs were 30%, 75%, 90%, and 92%. Accordingly, Yu et al. [32] found that the PDE of NO over carbon-doped TiO₂ (C-TiO₂) under visible light irradiation decreased from 52% to 13% as the AFR increased from 1 to 5 L/min. Chun et al. [33] reported that the PDE of toluene over C-TiO₂ films under visible light irradiation decreased gradually from 89% to 0% with increasing AFR from 1 to 4 L/min. The low PDE values at high AFRs were attributed to insufficient reaction time of the photoca-

lytic process. The reaction times were calculated by dividing the volume of the photocatalytic reactor by the AFR and were 7.8, 3.9, 2.6, and 2.0 s at AFRs of 1.0, 2.0, 3.0, and 4.0 L/min, respectively. Under high AFR conditions, mass transport of BTEX from the air stream to the solid surface of the photocatalyst films may have decreased, owing to the high linear face velocity that reduces convection and diffusion in the photocatalytic reactor [34]. Consequently, this mass transfer effect would reduce the PDE values of BTEX under high AFR conditions.

The reaction rates were also determined using the following equation:

$$P_r = f(C_i - C_o)Q/A \quad (1)$$

where P_r is the photocatalytic reaction rate ($\mu\text{mol m}^{-2} \text{s}^{-1}$); C_i and C_o are the input and output BTEX concentrations (ppm), respectively; Q is the air flow rate (m^3/s); A is the coated area (m^2); and f is the conversion constant ($40.9 \mu\text{mol m}^{-3} \text{ppm}^{-1}$). The reaction rates of the target compounds increased as AFR increased. Specifically, the reaction rate of toluene increased from 0.43 to $0.86 \mu\text{mol m}^{-2} \text{s}^{-1}$ as AFR increased from 1.0 to 4.0 L/min. Yu et al. [35] have also reported an increasing trend in the photocatalytic reaction rates of studied volatile organic compounds with increasing AFR. These results were ascribed to the increased mass transfer rates from the air stream to the

Fig. 8. PDEs of benzene (a), toluene (b), ethyl benzene (c), and *o*-xylene (d) over S-TiO₂-0.8-AS at varying air flow rates.

Fig. 9. PDEs of benzene (a), toluene (b), ethyl benzene (c), and *o*-xylene (d) over S-TiO₂-0.8-AS at varying input concentrations of the target chemical.

photocatalyst surface.

Figure 9 shows the PDEs of BTEX over S-TiO₂-0.8-AS at varying ICs of the target gas. The PDEs displayed an increasing pattern with decreasing ICs. In particular, at the lowest IC, the PDEs ranged from 30% to 92%, whereas at the highest IC the PDEs ranged from 3% to 28%, depending on the target gas. Because PDEs depend strongly on the adsorption capacity of chemical species on the catalyst surface [36], the increasing pattern in PDEs with decreasing ICs was ascribed to competitive adsorption processes of the BTEX species onto the surface of S-TiO₂-0.1-AS. Under high IC conditions, only a small number of adsorption sites on the surface of S-TiO₂-0.1-AS would be available for pollutant adsorption before degradation. Yu et al. [32] reported a decreasing pattern in PDEs of NO with increasing ICs in the range of 0.1–1.0 ppm over C-TiO₂ under visible light irradiation. Ku et al. [37] examined a photocatalytic reactor with TiO₂-coated fibrous photocatalyst under UV irradiation and reported that the PDE of trichloroethylene (TCE) decreased from 80% to 55% as the IC of TCE increased from 240 to 640 ppm.

The mineralization efficiency (ME, %) of the target compounds was estimated using the following equation:

$$ME = [CO_2]_o \times 10^4 / ([10 \times [TC]_i \times PDE]) \quad (2)$$

where $[CO_2]_o$ represents the CO₂ output concentration and $[TC]_i$ represents target compound input concentration. The MEs of BTEX over S-TiO₂-0.8-AS were 18%, 33%, 41%, and 46%, respectively. The CO concentration measured at the outlet port of the photocatalytic reactor was less than 0.1 ppm, which is not a crucial contributor to indoor air quality deterioration.

4. Conclusions

The PDEs of S-TiO₂-AS photocatalysts for the purification of toxic organic vapors were examined under visible light exposure. S-TiO₂ powder was immobilized onto flexible low-cost aluminum sheets using a simple sol-gel dipping process and low post-processing temperatures. The S-TiO₂-AS samples revealed a shift in the absorbance spectrum towards the visible region, whereas TiO₂-AS showed major light absorption in the UV region, indicating that S-TiO₂-AS could function effectively

under visible light irradiation, and sulfur ions were embedded into the crystal lattice of TiO₂ in S-TiO₂-AS sample. S-TiO₂-AS displayed superior photocatalytic activity over the reference photocatalyst for the purification of toxic organic vapors under visible irradiation. In addition, the PDE results of BTEX over the S-TiO₂-AS photocatalysts suggested the existence of optimal S/Ti ratios for the synthesis of S-TiO₂-AS with high photocatalytic activity. Additionally, the results clearly demonstrated that the BTEX decomposition depended on the AFRs and ICs. Thus, S-TiO₂-AS photocatalysts can be used under optimal conditions for the highly efficient purification of toxic organic vapors.

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Graphical Abstract

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The photocatalytic decomposition efficiencies of benzene, toluene, ethyl benzene, and o-xylene (BTEX) over S-TiO₂-aluminum sheets at different S/Ti ratios were higher than those achieved over reference TiO₂-AS under visible light.

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