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# Facile process to greatly improve the photocatalytic activity of the $TiO_2$ thin film on window glass for the photodegradation of acetone and benzene

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#### HIGHLIGHTS

- TiO<sub>2</sub> thin film was fabricated on window glass via PVP modified sol-gel method.
- Photocatalytic activity was improved by 4.37 times for decomposing acetone.
- Photocatalytic activity was improved by 4.62 times for decomposing benzene.
- TiO<sub>2</sub> thin film has high transmittance and excellent photocatalytic stability.
- TiO<sub>2</sub> thin film demonstrates high photocatalytic activity under sunlight.

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#### ABSTRACT

TiO<sub>2</sub> thin film fabricated on window glass (soda-lime glass) demonstrates various promising applications, such as for the photodegradation of air pollutants like VOCs. However, the photocatalytic activity of the TiO<sub>2</sub> thin film fabricated on window glass through the currently reported traditional sol–gel method is relatively low, because usually it has poor crystallinity, which hinders its practical application. In this research, we have developed a facile and novel method to greatly improve the crystallinity and the photocatalytic activity of the TiO<sub>2</sub> thin film fabricated on window glass, that is the PVP (polyvinylpyrrolidone) modified sol–gel method. By simply adding 3%, 6% and 9% (weight percentage) PVP into titanium sol, the photocatalytic activities of the obtained TiO<sub>2</sub> thin films are 3.01, 4.37 and 3.33 times higher than that of the TiO<sub>2</sub> thin film fabricated without PVP for the photodegradation of acetone. As for the photodegradation of benzene, the photocatalytic activities of them are improved by 2.80, 4.62 and 3.07 times, respectively. Additionally, the obtained TiO<sub>2</sub> thin film also exhibits excellent photocatalytic stability, high visible light transmittance, firm adhesion and high photocatalytic activity under natural sunlight. These advantages demonstrate that it has the prospect of being large-scale applied on window glass for air purification under sunlight.

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#### 1. Introduction

For the past decades, numerous research interests have been focused on the fabrication of  $TiO_2$  thin film [1–4], because it has wide potential applications, such as for air and water purifications [5–7], antibacteria [8,9] and self cleaning [1,10], etc. Volatile organic compounds (VOCs) such as formaldehyde, acetone and benzene are air pollutants that widely exist in both indoor and outdoor environments [7,11,12]. Long-term exposure to these toxic

VOCs would greatly elevate the risk for human beings to suffer from allergy, respiratory illness, leukemia and cancer [13–15]. TiO<sub>2</sub> is a well known photocatalyst which demonstrates high capability of mineralizing VOCs into nontoxic CO<sub>2</sub>, H<sub>2</sub>O and mineral acids under UV light [16]. TiO<sub>2</sub> thin film fabricated on window glass (soda-lime glass) has a promising application prospect for the purification of indoor and outdoor air pollutants, because of the cheap price and the wide use of window glass.

Many techniques have been developed to fabricate  $TiO_2$  thin film, including magnetic sputtering deposition, chemical vapor deposition, liquid phase deposition and sol-gel process [1,3,4], etc. Among them, sol-gel process is the most prevailing one because it's operated at mild conditions and can be easily scaled





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up to fabricate large-sized TiO<sub>2</sub> thin film [17]. However, it has been reported that the TiO<sub>2</sub> thin film fabricated on window glass via the traditional sol-gel method had very low photocatalytic activity [3,18–20], hindering its practical application. Because usually the obtained TiO<sub>2</sub> thin film on window glass has poor crystallinity, which is caused by the thermal diffusion of sodium ions from glass substrate into TiO<sub>2</sub> thin film during calcination [18,19]. Several methods have been proposed to solve this problem, most of them focused on preventing the thermal diffusion of sodium ions, such as inserting a  $SiO_2$  or  $SiN_x$  barrier layer between window glass and TiO<sub>2</sub> thin film [20], and using borosilicate or quartz glass which contains few sodium ions as substrate [3,21]. However, there are some disadvantages of these methods, the former increases the complexity of fabrication process which raises up total cost, the latter employs more expensive borosilicate or quartz glass instead of cheap and widely applied soda-lime glass (window glass), which also greatly increases total cost. Consequently, it is crucial to fabricate well crystallized TiO<sub>2</sub> thin film on window glass (soda-lime glass) through a facile and cost-effective method, in order to improve its photocatalytic activity and to achieve the goal of large-scale practical application.

Polyvinylpyrrolidone (PVP) is a nontoxic polymer which is widely used in the industries of medicines, beverages and cosmetics, etc. It has been reported that PVP can be used to fabricate the hierarchically porous TiO<sub>2</sub> sphere [22,23], the bowl/ring/inkbottle like TiO<sub>2</sub> clusters [24], the truncated tetragonal bipyramidal  $TiO_2$  nanocrystalline with exposed (001) facet and  $TiO_2$  tube/fiber [25-27]. In this research, PVP was introduced into titania sol to fabricate transparent TiO<sub>2</sub> thin film with high crystallinity and high photocatalytic activity on window glass (soda-lime glass). Combining the FESEM, HRTEM, XRD and fluorescence measurements, the influences of PVP content on the photocatalytic activity and the crystallographic properties (crystallinity and crystallite size) of TiO<sub>2</sub> thin film were systematically investigated. In addition, the FTIR and XPS characterizations were also used to explore the mechanisms of the influences of PVP content on the crystallographic properties of TiO<sub>2</sub> thin film. Additionally, the optical transmittance. the photocatalytic stability and the adhesive property of the obtained TiO<sub>2</sub> thin film were also studied. Besides, the performances of the obtained TiO<sub>2</sub> thin film for the photodegradation of acetone and benzene under natural sunlight were also investigated.

#### 2. Experimental section

#### 2.1. Preparations

All chemicals used in this work were in analytical grade, and they were used as received without any further treatment. For the preparation of titania sols, 0.2 M tetrabutyl titanate (TBOT) and 0.1 M diethanolamine (DEA) were dissolved in ethanol, after homogenization with magnetic stirring, a certain amount of PVP K30 ( $M_w$  = 58,000, with various amounts of 0, 3, 6 and 9 wt%) was added and dissolved. Then the mixture of distilled water (0.4 M) and ethanol was added dropwise into the former solution while violently stirred. The obtained sols were aged for seven days before use. TiO<sub>2</sub> thin films were fabricated on window glass (soda-lime glass, 8.5 cm × 8.0 cm × 1.0 mm, Wuhan Changli Glass Co. Ltd, China) via the dip-coating technique with different withdrawal speeds varying from 7.5 to 14 mm/s. The glass substrates were ultrasonic cleaned in distilled water and ethanol before dip-coating. After being coated, the obtained thin films were calcined at 450 °C for 1 h.

#### 2.2. Measurements of photocatalytic activity and stability

Acetone and benzene were selected as the representations of VOCs to evaluate the photocatalytic activities of  $TiO_2$  thin films.

The liquids of acetone and benzene were injected into the photocatalytic reactor using a microsyringe with amounts of 0.9 and 0.3 µL, respectively. After evaporation, the detected concentrations of them were  $500 \pm 40$  and  $110 \pm 10$  ppm, respectively. The concentrations of gaseous acetone, benzene and evolved CO<sub>2</sub> were measured with a gas chromatograph (Huaai, GC9560), which was equipped with a flame ionization detector (FID), a methane converter, a porapak R column, and PEG20 M column [28]. A xenon lamp (Trusttech, CHF-XM-500 W) was used as the light source. A homemade gastight stainless steel cylinder container with a guartz window possessing a volume of 415 mL was used as the photocatalytic reactor (reaction cell). The distance between xenon lamp and reactor was kept at ca. 16 cm. The intensity of UV light within the reactor was 11.02 mW/cm<sup>2</sup>, which was measured with an UV radiometer (Beijing normal university Factory, UV-A) using a 365 nm detector, which could detect UV light within 320-400 nm. The photocatalytic stability of TiO<sub>2</sub> thin film was measured with the photodegradation of acetone by repeating the photodegradation process under same conditions, and no additional treatment was applied to TiO<sub>2</sub> thin film.

The photodegradation of acetone (also  $0.9 \,\mu$ L) under natural sunlight using the obtained TiO<sub>2</sub> thin film was carried out from 11 to 12:30 am on 3 August of 2015. In the case of benzene (also 0.3  $\mu$ L), the experiment was carried out from 10:30 to 13:30 am on 4 August of 2015. Both of these two days were sunny and cloudless. The change of the intensity of UV light of sunlight was recorded using the UV radiometer. The results are shown in Fig. S1 (see Supplementary material). The calculated average intensities of UV light of sunlight are 3.78 and 3.75 mW/cm<sup>2</sup> during the photodegradation of acetone and benzene, respectively.

#### 2.3. Measurements of fluorescence spectra

 $TiO_2$  thin films on soda-lime glass were cut into small pieces (0.9 cm × 3.0 cm × 1.0 mm), and they were placed in a quartz cuvette (1.0 cm × 1.0 cm × 4.0 cm) containing 2 mL aqueous solution, which was consisted of 20 mM NaOH and 6 mM terephthalic acid (TA) [29]. Then the cuvette was illuminated with the xenon lamp for 20 min, the distance between them were also kept at ca. 16 cm. After illumination,  $TiO_2$  thin films were removed from the cuvette, then the obtained solutions were tested with a fluorescence spectrophotometer (Shimadzu, RF-5301) to measure the fluorescence intensity of generated 2-hydroterephthalic acid (TAOH), and the wavelength of excitation light was at 320 nm.

#### 2.4. Characterizations

Surface and cross sectional morphologies were examined with a field emission scanning electron microscope (FESEM, Hitachi, S-4800) operated at 5 kV. High resolution images were taken with a transmission electron microscope (TEM, JEOL, JEM2100F), which was operated at 200 kV. XRD patterns were obtained by using the grazing incidence X-ray diffractometer (XRD, PANalytical, Empyrean), which was operated at 40 kV and 40 mA using Cu  $K_{\alpha}$  radiation, the incident angle was fixed at 0.3°, and the scanning speed was kept at 1.82° per minute. Surface elemental compositions were investigated by an X-ray photoelectron spectrometer (XPS, Thermo Scientific, VG Multilab 2000) using Mg  $K_{\alpha}$  radiation. The detected spectra were calibrated by referencing the binding energy of adventitious carbon with a value of 284.6 eV of the  $C_{1s}$  electrons. Titania sols containing various amounts of PVP were dried at 70 °C for 24 h, the obtained xerogel powders were grinded and mixed with KBr, then they were pressed into transparent pellets and examined with a Fourier transform infrared spectroscopy (FTIR, Thermo Electron, Nicolet 6700). Optical transmittances were detected with an UV-Vis spectrophotometer (Shimazu, UV-3600).

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