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Improvement of TiO_2/LDPE Composite Films for Photocatalytic Oxidation of Acetone

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Abstract. Titanium dioxide with coupling agent (ETES) was applied as a photocatalyst for a synthesis of the TiO_2/LDPE composite film. The physical properties of TiO_2/LDPE composite film were analyzed by a Scanning Electron Microscope (SEM). TiO_2 particles were impregnated into the polymer matrix film as a LDPE composite film. The results from the X-ray Diffraction (XRD) technique revealed that the structure of TiO_2/LDPE composite film were anatase crystalline. The chemical structure of the TiO_2/ LDPE composite films were analyzed by an ATR- Fourier transforms infrared (ATR-FTIR) spectrometer. Wavenumber of FTIR spectra at 719 cm^{-1} indicated the Ti-O-Ti bond. Band gap energies of the films ranged from 3.19-3.29 eV. The photocatalytic activity of the film was tested for removal of gaseous acetone in a closed chamber. Experimental conditions were set as follows: a UV light intensity of approximately 2.7 $mW.cm^{-2}$, flow rate of 2 $L.min^{-1}$, and an initial acetone concentration of about 435±20 ppm. While the catalyst dosage was varied from 3% to 15% (wt. cat/wt. film).The degradation rate of acetone increased when increasing dosage of TiO_2 from 3% to 10%, then decreased a little bit when increasing the dosage to 15%. The TiO_2/LDPE composite film at the dosage of 10% yielded the highest removal efficiency of 75%, followed by the film at the dosage of 15%, 5%, and 3%, respectively.

Introduction

Indoor air pollution is one of the environmental risks to public health [1]. Indoor air pollutants can be classified as biological contaminants (bio aerosol), chemical contaminants, and particulate. Among them, volatile organic compound (VOCs) is one of the major pollutants [2].Several technologies have been applied for indoor air purification. Among these technologies, adsorption has been the most commonly used method to treat indoor air contaminants. However, recently Photocatalytic Oxidation (PCO) has been gaining attention as a possible alternative method for indoor air purification because it promises to clean air more efficiently and effectively. PCO uses UV light to activate the catalyst to generate hydroxyl radicals ($OH^{\bullet}$), which then react with organic contaminants. The contaminants are then degraded or transformed into less harmful substances. Titanium dioxide (TiO_2) is one of the most interesting photocatalyst in the PCO processes [3]. However, the technique to obtain well uniformly dispersion of TiO_2 impregnated into the film is crucial. There are some recommended techniques such as a sol-gel technique, a spray or a dipping technique, and immobilization of TiO_2 in polymer matrix. Among these techniques, the Blown film preparation technique has advantages as compared to others due to its economy and simplicity of preparation. The nano TiO_2/LDPE composite film was previous introduced for photocatalytic degradation of gaseous dichloromethane. The highest removal efficiency obtained was slightly lower than 80% [4]. Therefore, the improvement of the TiO_2/LDPE composite film was the main objective of this study. The enhancement of TiO_2 dispersion into the film was investigated by adding coupling agent during the film preparation step. Because coupling agents are organosilicon compounds that are generally used to bond organic materials (LDPE) to inorganic materials (TiO_2). Therefore, the agglomeration of TiO_2 could be minimized [5]. The physical properties of the film were analyzed using the X-ray diffraction (XRD), the UV-VIS spectrophotometer, the scanning electron microscope (SEM), and the Fourier transform infrared spectroscopy with ATR mode (ATR-FTIR). The removal efficiency of the film using treatment of gaseous acetone in a small closed chamber was also included in this work.

Materials and Methods

Photocatalyst TiO_2/LDPE composite film preparation

The commercial grade of low density polyethylene (LDPE) was supplied by the PFTLIN .The TiO_2 powder with crystalline size ca. 45 nm from the Wilson, China, was used at varying contents, e.g. 0, 3,5,10, and 15 %(wt. cat./wt. film). Ethyl triethoxysilane (ETES) 5 %(wt. cat./wt. film) with purity 96.0% (Aldrich) was used as coupling agent to improve the uniformly dispersion of TiO_2 into the film. First, TiO_2 powder, LDPE, and ETES were mixed well in the mixer. Then the mixture was introduced into the twin screw extruder. The TiO_2/LDPE composite films with the thickness of about 30 micrometers were obtained from the blown film extrusion technique using the operation temperature in a range of 150 ^{0}C to 180 ^{0}C [4,6].

Characterization of the TiO_2/LDPE composite film

(1) Physical property: The surface morphology of the TiO_2/LDPE composite film was examined using the Scanning Electron Microscope (SEM, Hitachi S-3400N). In order to prevent the charge build-up during SEM observation, TiO_2/LDPE composite films were coated with gold which was used for ion sputter target. The sputter rate and time were set for 10 nm.min^{-1} and 3 min, respectively. The gold film thickness was approximately 30 nm [6].

(2) Optical property and band gap energy analysis: Ultraviolet-visible spectra of the TiO_2/ LDPE composite films were recorded using the UV-VIS spectrophotometer (Lamda 950, Perkin Elmer instrument) equipped with an integrating sphere. All spectra were monitored in the absorbance mode and acquired under ambient conditions. The spectra were measured in the range from 200 to 700 nm. The band gap energy of TiO_2/LDPE composite films can be calculated from absorbance results [6, 7].

(3)Attenuated total reflectance-Fourier Transform Infrared (ATR-FTIR) Spectroscopy analysis: Chemistry functional groups of the TiO_2/LDPE composite films were studied the by Fourier transform infrared (FTIR) spectroscopy with ATR mode. ATR-FTIR spectra were obtained using a Model Perkin Elmer System 2000. TiO_2/LDPE composite films were dried in an oven at 120 °C for 6 h, and then KBr pellets were prepared with the TiO_2/LDPE composite films. The spectra were collected in the range from 4000 to 650 cm^{-1} [7,8].

(4) X-ray Diffraction (XRD) analysis: The crystalline structures of TiO_2/ LDPE composite films were determined by X-Ray Diffraction (XRD) technique. XRD patterns were obtained on the X-Ray Diffract meter (Shimadzu,XRD-600) using Cu Kα and radiation (λ=1.5418 A°). The scan ranges from 20 ° to 60 ° with a scan rate of 2 ° min^{-1} [7].

(5) Photocatalytic oxidation removal of acetone: An annular photoreactor made of a stainless steel was consisted of the TiO_2/LDPE composite film and an ultraviolet lamp type C at 254 nm wavelength as shown in Fig. 1. The electrical power of a UV lamp was 9 watts corresponding to the light intensity about 2.7 mW.cm^{-2}. Total volume of the photoreactor was about 700 mL. The TiO_2/LDPE composite film was placed against the wall of the photoreactor. The photoreactor was connected with an air compressor equipped with a rotameter used to maintain the recirculation flow rate of 2 L.min^{-1}. The relative humidity and temperature inside the photoreactor were measured continuously using the Thermo-hygrometer. The photodegradation of acetone was conducted to investigate the removal efficiency of the film at different TiO_2 dosages (3% (wt. cat./wt. film), 5% (wt. cat./wt. film), 10% (wt. cat./wt. film), and 15% wt. cat/wt. film). An initial concentration of acetone was about 435±20 ppm. The residence time was set at 150 min (or until steady state was reached). The acetone gas sample applied for all experiments was from a compressed air tube. After the acetone initial concentration was at steady state, the UV lamp was turned on. This step is necessary in order for the acetone concentration to reach the adsorption equilibrium. The

quantitative analysis of acetone concentration was made with the Gas chromatography (GC) equipped with a Flame Ionization Detector (FID, Model GC 2010, Shimadzu) [4,6].

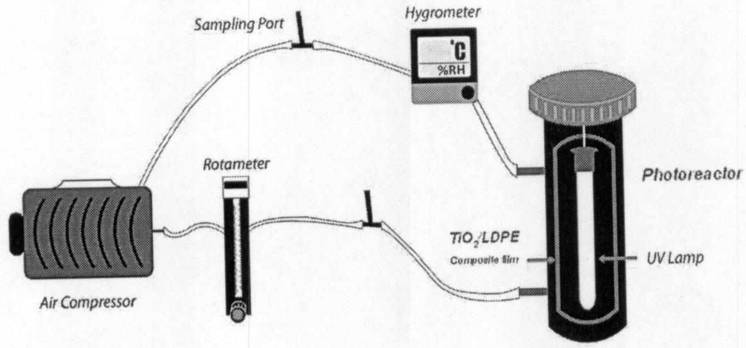

Fig. 1 Schematic of the photocatalytic reactor

Results and Discussion

Characterization of TiO_2/LDPE composite film

Scanning electron microscopy (SEM) images of the TiO_2/LDPE composite film at difference dosages are displayed in Fig.2. As seen from the figure, TiO_2 was well dispersed onto the inner surface of the film. The better results were obtained in this work as compared to the previous [4]. However, a few clumps of TiO_2 particles onto the inner surface of the film at the high dosages of TiO_2 are also noticed. This may be caused by aggregation of TiO_2 particles during the heat of blown film process.

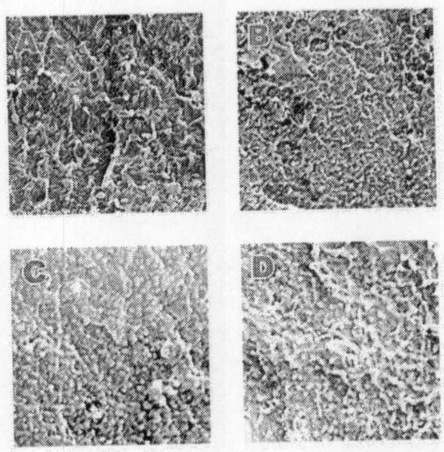

Fig. 2 The SEM images of (A) 3% (wt. cat./wt. film) TiO_2/LDPE composite film; (B) 5% (wt. cat./wt. film) TiO_2/LDPE composite film; and (C)10% (wt. cat./wt. film) TiO_2/LDPE composite film; and (D) 15% (wt. cat./wt. film) TiO_2/LDPE composite film (10,000X)

The band gap refers to the energy difference between the valence bands to conductive band. The results from the UV-Vis spectroscopy showed that TiO_2/LDPE composite film absorbed appreciably at wavelength less than 400 nm. The band gap energy results are shown in Table 1. The results obtained in this study agree well with other works [5,6].

Table 1 Calculated Results from the absorbance of TiO_2/LDPE composite film at various Dosages of TiO_2.

No.	TiO_2/LDPE composite film	Band Gap Energy (E_g, eV)
1	3% wt. cat/wt. film	3.19
2	5% wt. cat/wt. film	3.29
3	10% wt. cat/wt. film	3.27
4	15% wt. cat/wt. film	3.24

Functional groups of the TiO_2/LDPE composite films were determined by FTIR. The same results were obtained for all samples. Example of the FTIR results is illustrated in Fig. 3. From the figure, the characteristic bands corresponding to the vinyl groups (–CH=CH_2) were those at 1,463, 1,378, and 1,044 cm^{-1}. Moreover, alkyl groups were formed at the peaks of 2,915 and 2,848 cm^{-1}. A broad band at 719 cm^{-1} should be due to the envelope of the bands of Ti-O-Ti [8,9].

Fig. 4 displays the XRD spectra of the TiO_2/LDPE composite film at various loading of TiO_2. The XRD spectra of 3, 5, 10, and 15% (wt. cat./wt. film). TiO_2/LDPE composite films show the clear sharp peaks indicating the anatase crystalline structure. The XRD spectra also reveal that the anatase peaks (101) obviously increased with the amount of TiO_2 [10]. Moreover from Fig.4 (B-D), diffraction peaks corresponding to the LDPE were observed as well.

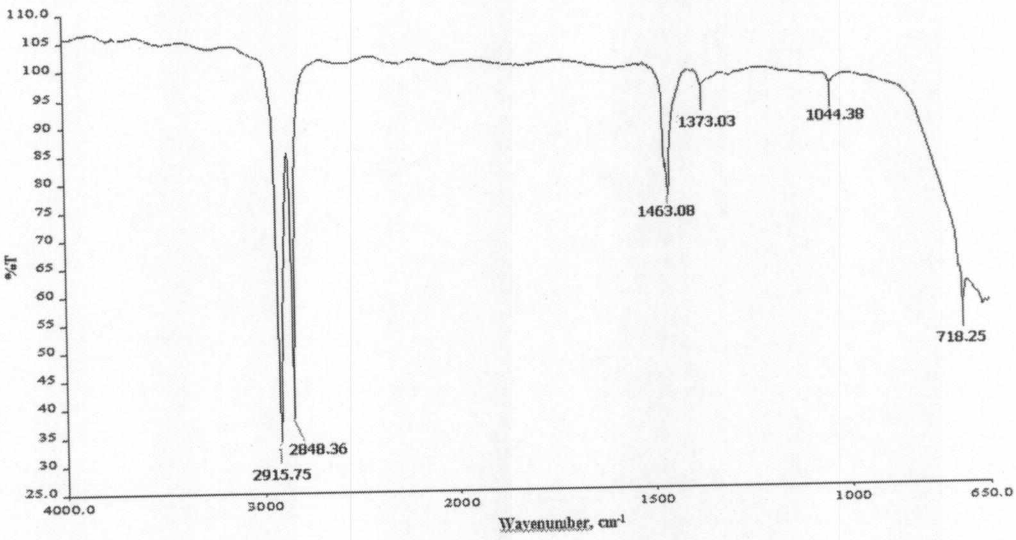

Fig. 3 FTIR spectra of the 10% (wt. cat./wt. film) TiO_2/LDPE composite film

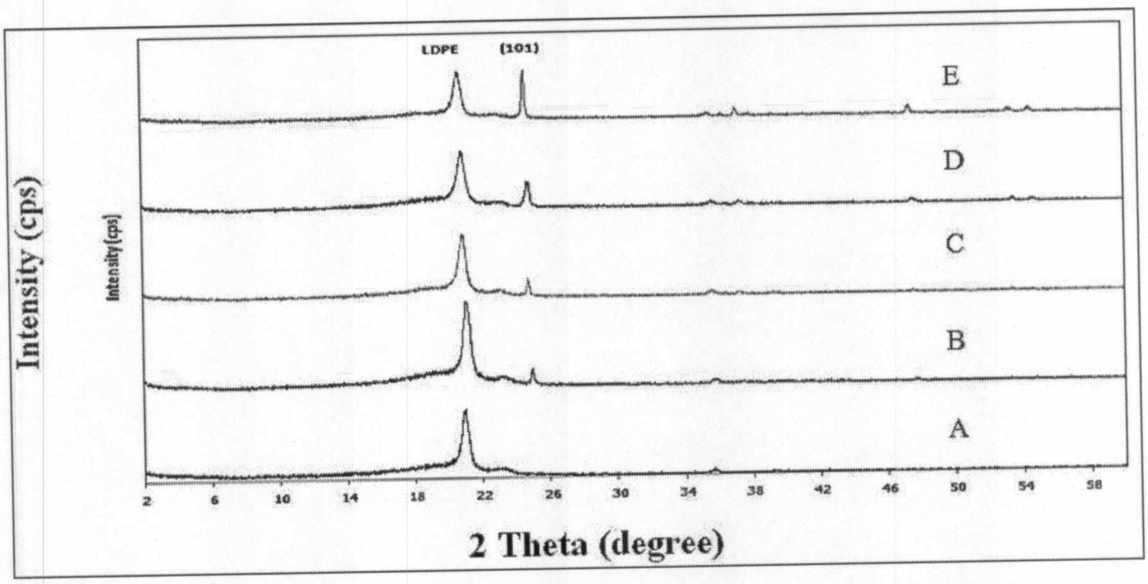

Fig. 4 XRD spectra (A) virgin LDPE film (B) 3 %(wt. cat./wt. film) TiO_2/LDPE composite film (C) 5 %(wt. cat./wt. film) TiO_2/LDPE composite film (D) 10 %(wt. cat./wt. film) TiO_2/LDPE composite film (E) 15 %(wt. cat./wt. film) TiO_2/LDPE composite film

Photocatalytic Oxidation of Gaseous Acetone

Photocatalytic oxidation of acetone using the TiO_2/LDPE composite film at different dosages of TiO_2 was investigated. The results are shown in Fig. 5. From the figure, it is seen that the film at the TiO_2 dosage of 10% (wt. cat./wt. film) yielded the highest removal efficiency (75%), followed by the films at 15, 3, and 5% with the removal efficiencies of 69 %, 68%, and 58%, respectively. Removal efficiency increased with increases of TiO_2 dosage from 3% to 10%. Then it was slightly decrease as an increase of TiO_2 dosage from 10% to 15% (wt. cat./wt. film). This may be due to the higher TiO_2 dosage, the higher hydroxyl radicals to react with acetone resulting in the higher removal efficiency of the film. However, above a certain dosage of TiO_2, the degradation rate was started to decline caused by the mass transfer and light penetration limitation [11]. The highest removal efficiency obtained from this work was comparable to that from the previous work [4]. However, the treatment time of this study was much shorter than that of the previous research.

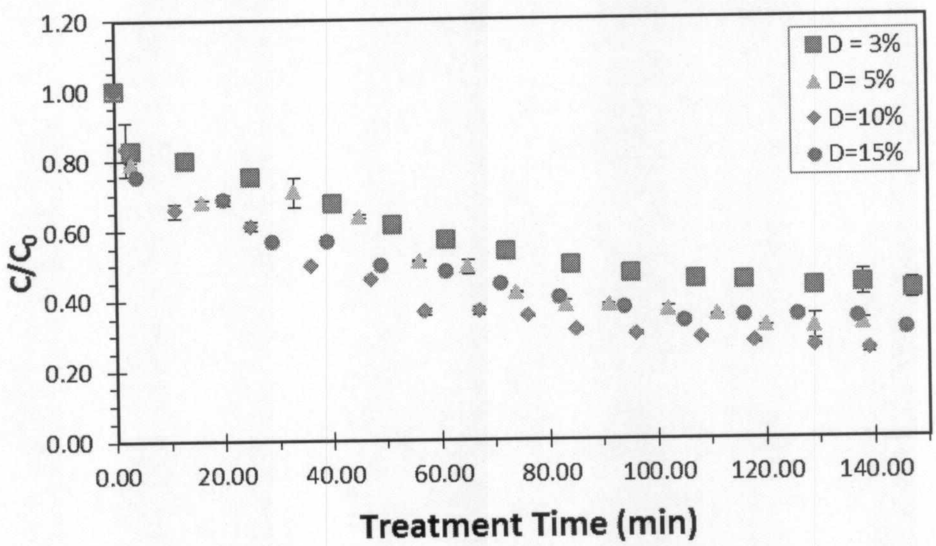

Fig. 5 Photocatalytic oxidation of gaseous acetone at the different TiO_2 dosages (initial acetone concentrations of 420 to 465 ppm, UV light intensity of 2.7 mW.cm^{-2}, flow rate of 2 L.min^{-1})

Conclusion

This studied has demonstrated that the improvement of TiO_2/LDPE composite film preparation can be achieved using the ETES as a coupling agent. The SEM results revealed the better uniform dispersion of TiO_2 onto the inner surface of the film. The band gap energy of TiO_2/LDPE composite films ranged from 3.19 to 3.29 eV. The results of XRD examination showed that the TiO_2 on the LDPE composite film had the major structure in anatase form. The acetone removal efficiency increased with dosages of TiO_2.

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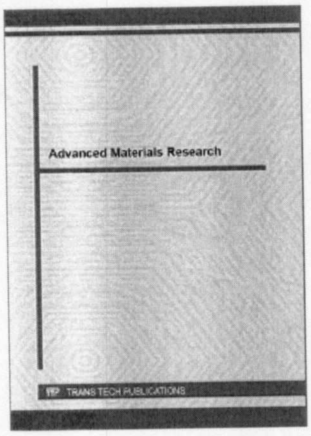

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