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Preparation of Sol-Gel Derived Nanocrystalline TiO₂ Particles in Titania-Silica Composite for Photocatalytic Applications

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Three different titania-silica nanocomposite systems (8/2, 5/5, 2/8 molar ratio of TiO₂/SiO₂) were prepared by a modified hydrolysis-condensation reaction. The synthesized specimens had two structural phases; one is crystalline TiO₂ in anatase form and the other is glass phase SiO₂. The sizes of the nanocomposites ranged from 10 to 11 nm. All the resulting nanocomposites were found to be excellent as photocatalytic materials.

Keywords: photocatalyst; titania-silica nanocomposite; sol-gel process

INTRODUCTION

Recently much attention has been paid to the preparation of titanate nanoparticles with various morphology, size, and surface property. Because titanium dioxide can serve as a key material in the fields of photocatalysts, catalyst supports, inorganic additives, microelectronics, electrooptics, (photo)-electrochromics, and photovoltaics.^[1,2] Specifically, considerable research activity has been concentrated in developing heterogeneous photocatalysts employing nanocrystalline semiconductors.^[3] Among various semiconductors, titanium dioxide is the most suitable for many practical applications due to its strong oxidizing power, high chemical durability, and nontoxicity.

In this paper, we describe the synthesis of nanocrystalline TiO_2 particles in titania-silica nanocomposite system with three different compositions (8/2, 5/5, 2/8 molar ratio of $\text{TiO}_2/\text{SiO}_2$) by sol-gel technique. In addition, the photocatalytic activity for the degradation of methylene blue on the titania-silica nanocomposite thin films is discussed.

EXPERIMENTAL

The titania-silica nanocomposite system has been prepared by a modified hydrolysis-condensation reaction according to the following procedures; 15.2 mL of $\text{Ti}(\text{OCH}(\text{CH}_3)_2)_4$ (aldrich, 97%) and 10 mL of isopropanol were slowly added to 90 mL deionized water under vigorous stirring. One or two drops of conc. HNO_3 were added to the solution to enhance the hydrolysis process. Then the solution was heated to 80 °C for 8 h and cooled down to room temperature (solution A). A silica polymeric sol (solution B) was prepared by

mixing $\text{Si}(\text{OC}_2\text{H}_5)_4$, H_2O , HNO_3 , and ethanol with a ratio of 14.0 mL/9.0 mL/0.2 mL/13.0 mL at room temperature. Finally the solution A and solution B were intermixed to get desired molar ratio of $\text{TiO}_2/\text{SiO}_2 = 8/2, 5/5$, and $2/5$. The mixtures were stirred for another 2 hours at room temperature and used for coating solution.

Preparation of the titania-silica nanocomposite thin film onto glass was done by dip coating process, withdrawing the substrates from the solution at a constant rate of 15 cm/min. The coating samples were dried at 80 °C for 2 h. The thickness of the nanocomposite films is typically 0.08-0.15 μm .

The photocatalytic activity of various nanocomposite thin films was evaluated by measuring the absorption spectra at 580 nm from the aqueous phase. All photoreactions were carried out in a glass reactor containing 25 mL of methylene blue aqueous solution (initial concentration = 0.25×10^{-3} wt%) which was 12 cm from the lamp. A 15 W germicidal lamp (254 nm) was used as the light source. The solution was stirred continuously and was exposed to air during the reaction.

RESULTS AND DISCUSSION

Figure 1 shows the x-ray diffraction patterns for the air-dried $\text{TiO}_2\text{-SiO}_2$ nanocomposite. These patterns are well consistent with crystalline TiO_2 with anatase structure in glassy SiO_2 matrix, indicating that the modified sol-gel process is suitable for the preparation of crystalline $\text{TiO}_2\text{-SiO}_2$ nanocomposite under extremely mild processing temperature (80 °C). As the content of SiO_2 increases, the crystallinity of the resulting TiO_2 particles becomes lower. Assuming that the x-ray line broadening is essentially due to the size effect the calculated particle sizes fall into the range 10-11 nm from Debye-Scherrer formula.

The photocatalytic activity of the immobilized titania-silica nanocomposite has been tested by the photodegradation of methylene blue (Figure 2 (a)) solution. The results are shown in Figure 2 (b). When the light source is turned on, the percent of decomposition increases rapidly up to about 30 min and then remains constant. Also, as the content of SiO₂ increase, the photocatalytic activity becomes lower.

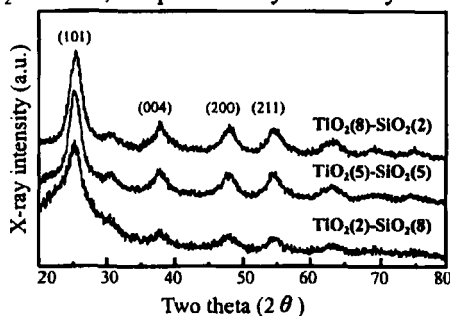
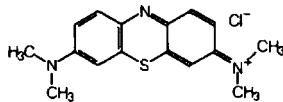
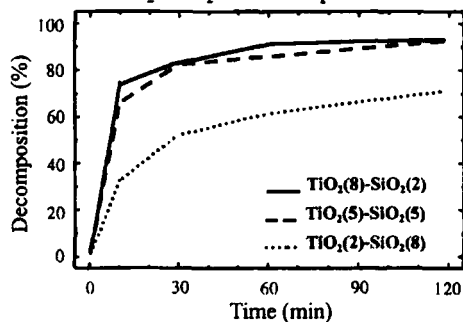


FIGURE 1. XRD patterns of TiO₂-SiO₂ nanocomposite.



(a)



(b)

FIGURE 2. (a) Structure of methylene blue, (b) Photocatalytic activity of TiO₂-SiO₂ nanocomposite.

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