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Synthesis of TiO_2 with Cotton as Template and Its Applications as Photocatalysis

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Spherical titanium dioxide photocatalytic material was prepared by the combined method of template and sol-gel method with cotton as the template. The sample surface morphology, composition and crystal structure were characterized by scanning electron microscopy, X-ray diffraction. The photodegradation experiment was also investigated with phenol as the substrate. The results showed that the prepared TiO_2 distributed in three-dimensional spherical structure of cotton, the grain size of TiO_2 was around 25 nm and the degradation rate was 92 % under the ultraviolet light irradiating for 4 h.

Keywords: Titanium dioxide, Cotton, Stereo, Photocatalysis, Template.

INTRODUCTION

In recent years, the photocatalytic technology has been involved in rapid development, TiO_2 is the most promising and most studied photocatalyst with many advantages such as high photocatalytic activity, chemically stable, non-toxic corrosion-resistant and low cost, *etc*[1-3]. Photocatalyst was used mainly in two forms, namely direct use of TiO_2 powder suspension system and loading on the base for catalyzed reactions. Since the suspension system has many disadvantages such as low light efficiency, difficult to recovery the TiO_2 particles after water treatment, complex and costly process and easy to get the catalyst poisoning, so the TiO_2 powder suspension system almost has no application value. Therefore the curable loading photocatalyst technology has become an inevitable trend[4]. There are some common loading bases such as glass, metal, adsorbents, but these carriers have poor adhesion of TiO_2 because their smooth surface. So the adsorbent with a large surface area for TiO_2 loading and three-dimensional structure of prepared TiO_2 have been becoming a new hotspot The three-dimensional structure of TiO_2 mostly prepared by template method, such as biological templates[5], organic material template, *etc.*[6,7] and their structure are mainly concentrated in the micro tube, hollow microspheres and core-shell structure, *etc*. The hollow micro nano-structure have important applications in many technical fields such as micro reactors, drug delivery carriers, photonic crystals, catalyst and energy storages, because the hollow part can accommodate the object of large or small size, which can produce micro "package" effect similar to human cells "micro- environment"[8-10].

Reticulated TiO_2 were prepared by Li[11] using leaves as the template. The leave tissue are rich in cellulose, which involving a large number of hydroxy groups. Hydrogen bonds are formed between hydroxy groups and TiO_2 particles which inhibit the growth of TiO_2 grains and small size of TiO_2 nanoparticles are prepared. Therefore, the use of cellulose-rich materials shows excellent load performance, such as improving stability and controlling agglomeration of nano-particles. Cellulose-TiO_2 composite membrane with good catalytic performance was prepared by Zheng *et al.*[12] using the cotton pulp as sources of cellulose.

Cotton is a good natural template for preparing nano TiO_2 particles because it contains a lot of cellulose which have spherical three-dimensional distribution. Nano TiO_2 particles of 25 nm were prepared using cotton as biological template, which have many advantages such as simple preparation, recycling mixture and good photocatalytic performance.

EXPERIMENTAL

All of the chemical reagents such as tetrabutyl titanate, anhydrous ethanol and nitric acid used in this study were of analytical grade. Scanning electron microscopy, X-ray diffraction, infrared spectroscopy and TU-1901 spectrophotometer were used for structural determination.

Preparation of nano TiO_2 particles: 5 mL of tetrabutyl titanate was added into 30 mL of absolute ethanol in a flask with constant stirring at 30 °C. After 0.5 h, a colourless transparent solution A was obtained. Meanwhile, 20 mL of anhydrous ethanol was mixed with 20 mL of nitric acid, the solution A was slowly added dropwise into this mixed solution under the condition of fast stirring for 0.5 h. Thus, a pale yellow solution B was formed. The prepared cotton (washed with deionized water, stored at the dark place) was put into the solution B for 8 h and then was filtered, washed with ethanol, dried at room temperature for 24 h and at 80 °C for 2 h and finally annealed at 600 °C for 2 h. The sample 1 of spherical white titanium dioxide particles was obtained.

5 mL of tetrabutyl titanate was added into 30 mL of absolute ethanol in a flask under the condition of stirring at 30 °C. After 0.5 h, a colourless transparent solution A was obtained. Meanwhile, 20 mL of anhydrous ethanol was mixed with 20 mL of nitric acid, the solution A was slowly added dropwise into this mixed solution under the condition of fast stirring for half an hour. Thus, a pale yellow solution B was formed. The prepared solution B was dried at room temperature for 24 h and at 80 °C for 2 h and finally annealed at 600 °C for 2 h. The sample 2 of titanium dioxide particles was obtained.

Photocatalytic experiment: 20 mL of phenol solution (100 mg/L) was added into 3 quartz beakers, respectively and 0.01 g of sample was added into No. 2 and No. 3 beaker. The samples were placed under UV lamp irradiation for 2 h at room temperature. The absorbance of phenol was determined by UV spectrophotometer and the degradation rate was calculated.

RESULTS AND DISCUSSION

Formation mechanism of spherical titanium dioxide: Cotton contains a lot of cellulose which have spherical three-dimensional distribution, which provide a natural biological template for titanium dioxide preparation. The cotton was soaked in a mixture of tetrabutyl titanate and nitric acid, the stratum corneum of the epidermis was damaged by oxidation of nitric acid. So a large number of tetrabutyl titanate was immersed into three-dimensional structure of cotton tissue and hydrolysis reaction of the immersed tetrabutyl titanate ester occurs:

$$nTi(OBu)_4 + 4nH_2O \rightarrow Ti(OH)_4 + 4nBuOH \quad (1)$$

After calcination, TiO_2 was distributed in the three-dimensional structure of cotton cellulose.

$$nTi(OH)_4 \rightarrow nTiO_2 + 2nH_2O \quad (2)$$

Scanning electron microscopy: Fig. 1 shows the scanning electron micrographs of sample 1 and 2. From A and B of Fig. 1, it can be seen that the resulting distribution of the sample was layered, laminated retained the original shape of cotton and the sample relatively loose, spherical TiO_2 of different sizes were distributed in the layer of fibrous tissue According to a further enlarge and calculation of XRD diffraction, TiO_2 grains size are around 25 nm (Fig. 1c), while the sample 2 was regular blocks.

X-ray diffraction: Fig. 2a shows the XRD determination of the sample 1, the main peak of 25.28° is anatase (101) plane peak according to standard card, the second peak of 27.44° is rutile (101) plane peak according to standard card. Fig. 2b shows the XRD patterns of the sample 2, the main peak of 27.44 ° is rutile (101) plane peaks.

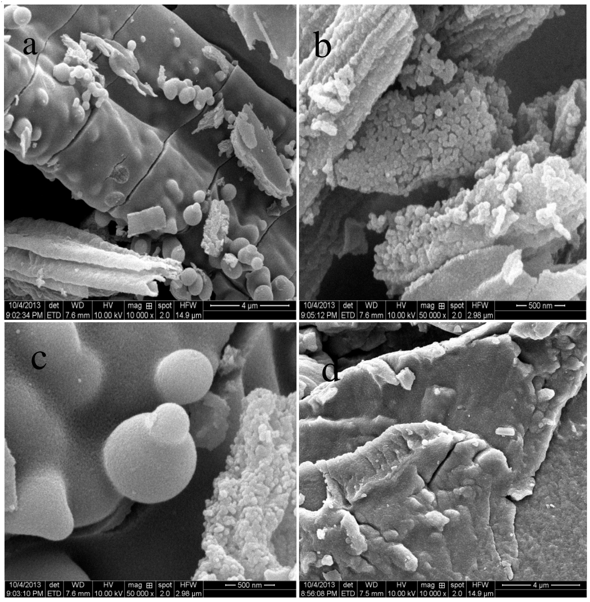

Fig. 1. Scanning electron micrographs of sample 1 and 2

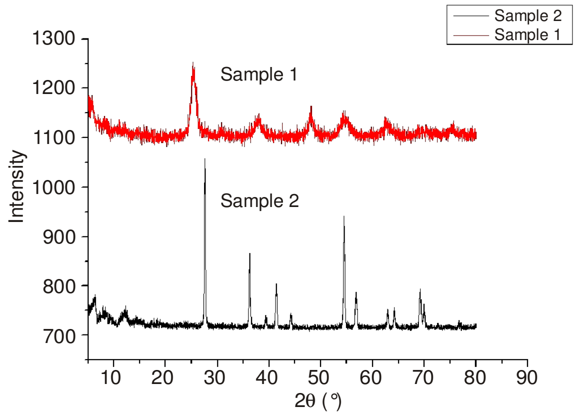

Fig. 2. Determination results of X-ray diffraction (XRD)

Photocatalytic performance of samples: Catalytic activity of the samples was evaluated according to the degradation rate. Fig. 3 shows the UV spectrum of photocatalytic degradation of samples 1 and 2. The degradation rate of samples 1 was 66 %. Fig. 4 shows the photocatalytic degradation rate with different time of sample 1.

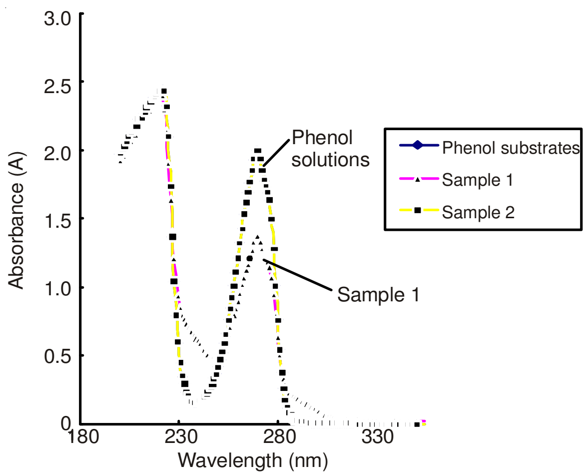

Fig. 3. UV spectrum of photocatalytic degradation

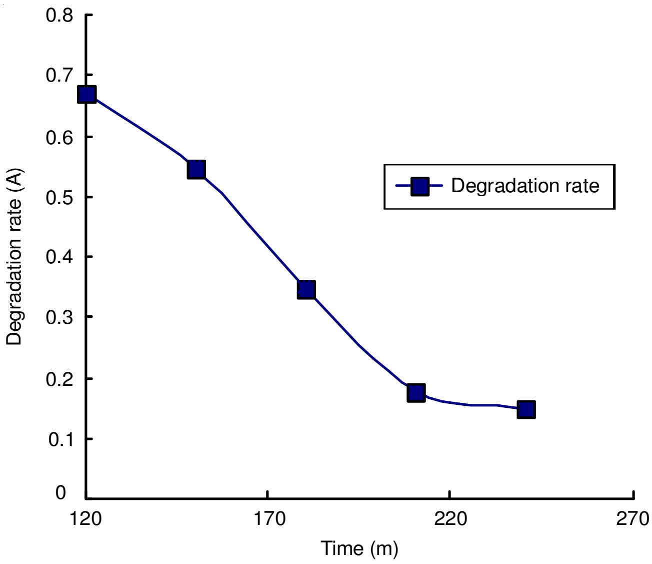

Fig. 4. Photocatalytic degradation rate with different time of sample 1

Conclusions

• Anatase TiO_2 grains was prepared by the combined method of template and sol-gel method with cotton as the template, the grain size of TiO_2 was around 25 nm.

• Through the photocatalytic degradation of phenol experiment, the degradation rate was 92 % under the ultraviolet light irradiating for 4 h.

• A simple method for prepared TiO_2 distributed in three-dimensional spherical structure with ideal degradation effect was investigated.

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