

## Research Article

# Bulky Macroporous TiO<sub>2</sub> Photocatalyst with Cellular Structure via Facile Wood-Template Method

Qingfeng Sun,<sup>1,2</sup> Yun Lu,<sup>1</sup> Jinchun Tu,<sup>3</sup> Dongjiang Yang,<sup>4</sup> Jun Cao,<sup>2</sup> and Jian Li<sup>1</sup>

<sup>1</sup> Material Science and Engineering College, Northeast Forestry University, Harbin 150040, China

<sup>2</sup> College of Mechanical and Electrical Engineering, Northeast Forestry University, Harbin 150040, China

<sup>3</sup> Key Laboratory of Tropical Biological Resources of Ministry of Education, College of Materials and Chemical Engineering, Hainan University, Haikou 570228, China

<sup>4</sup> College of Chemistry, Chemical and Environmental Engineering, Qingdao University, Qingdao 266071, China

Correspondence should be addressed to Jun Cao; [zdhcj@126.com](mailto:zdhcj@126.com) and Jian Li; [jianli@nefu.edu.cn](mailto:jianli@nefu.edu.cn)

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We report a bulky macroporous TiO<sub>2</sub> particles with cellular structure prepared in the presence of wood slices as template. Firstly, TiO<sub>2</sub> sol was coated onto the wood slices by repeated dip-coating process. Then, after calcinations at 550°C, the wood template could be removed, and the bulky TiO<sub>2</sub> structure was obtained. The prepared samples were characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD), energy dispersive spectroscopy (EDS), and transmission electron microscope (TEM) techniques. XRD pattern confirmed the crystalline phase of the wood-templated TiO<sub>2</sub> is anatase phase. And interestingly, from the observation of SEM image, the wood-templated TiO<sub>2</sub> inherited the initial cellular structures of birch lumber (*B. albosinensis* Burk), and numerous macropores were observed in the sample. Meanwhile, the wood-templated TiO<sub>2</sub> presented a superior photocatalytic ability to decompose Rhodamine B (RhB) under ultraviolet irradiation.

## 1. Introduction

Template-directed synthesis is currently one of the most widely used methods for fabricating inorganic materials on account of its facility and versatility [1–4]. In addition to some artificial templates like latex particles [5], polymer foams [6], ion-exchange resins [7], and carbon fibers [8], many natural templates have been attracted to much attention for constructing a number of inorganic materials. The natural species utilized as templates for the formation of inorganic materials are available from bacterial superstructures [9], insect wings [10], wool and cotton/cloth textile [11], diatoms [12], dog and human hair [13], living cells [14], mushroom gills [13], plant leaves [10], paper [15], pollen grains [16], shell membrane [17], silk fiber [13], skeletal plates [18], spider silk [8, 10], and wood [19–24]. These materials from nature combine many inspiring properties, such as sophistication, miniaturization, hierarchical organizations, hybridization, resistance, and adaptability, after billions of years of stringent selection processes [4]. From the research results, using

natural species as template for building inorganic materials is an effective strategy to obtain morphology controllable materials with structural specialty, complexity, and related unique properties.

Among these natural templates, wood is a naturally grown composite material possessing complex hierarchical cellular and pit structures that are comprised principally of biopolymers, mainly cellulose, hemicellulose, and lignin [25, 26]. The wood vessels and fibers are hollow tubes, and the arrangement of these vessels and fibers varies considerably different with different wood species. The cellular structural hierarchy of wood ranges from the millimeter scale of the growth ring and longitudinal tracheid, the micrometer scale of the cell wall and diameter, to the nanometer scale of the molecular fiber and membrane structure of the cell wall. The open porosity is accessible for liquid or gaseous infiltration [3, 20]. These features of wood make it a perfect candidate as a template for generating porous inorganic materials with both structural and morphological complexities. Previously some efforts have been made

toward transforming wood cellular structures into inorganic SiC [27, 28], TiC [29], and macroporous zeolites [20]. By impregnating the wood templates with metal alkoxide solutions and applying standard sol-gel chemistry, various metal oxide porous materials, such as TiO<sub>2</sub> [30], Al<sub>2</sub>O<sub>3</sub> [31], Fe<sub>2</sub>O<sub>3</sub> [21], and Cr<sub>2</sub>O<sub>3</sub> [32], have been produced using this approach.

In these inorganic materials, TiO<sub>2</sub> is one of the most promising functional materials and extensively used in photocatalysis [33, 34], solar cells [35], and paints [36] because of its superior chemical stability and nontoxicity [37, 38]. TiO<sub>2</sub> exists mainly in four polymorphs in nature, anatase (tetragonal, space group  $I4_1/amd$ ), rutile (tetragonal, space group  $P4_2/mnm$ ), brookite (orthorhombic, space group  $Pbca$ ), and TiO<sub>2</sub>(B) (monoclinic, space group  $C2/m$ ) [39–42]. In photocatalytic study, anatase TiO<sub>2</sub> is generally considered to be more active than rutile crystalline [43]. Moreover, anatase TiO<sub>2</sub> with higher crystallinity is preferred for photocatalysis, since higher crystallinity means fewer defects for the recombination of photogenerated electrons and holes [36]. In the current research, different kinds of TiO<sub>2</sub> nanomaterials like nanoparticles, nanosheet, nanotube, or nanofiber have been fabricated using different method. However, TiO<sub>2</sub> with wood structures has been seldom reported.

To this purpose, we developed an efficient pathway to synthesize wood-templated TiO<sub>2</sub>. The morphology, chemical compositions, and crystalline phase of the prepared TiO<sub>2</sub> were characterized by SEM, EDS, HRTEM, and XRD, respectively. Furthermore, the photocatalytic activity of the wood-templated TiO<sub>2</sub> was measured under UV irradiation.

## 2. Materials and Method

**2.1. Raw Materials.** All the chemicals were supplied by Shanghai Boyle Chemical Co. Ltd. and used as received. The air-dried birch lumber (*B. albosinensis* Burk) was cut into slices with a size of 20 mm (tangential) × 20 mm (radial) × 2 mm (longitudinal). The wood slices were pretreated in a Soxhlet apparatus with 95% ethanol at 80°C for 6 h under reflux to remove the extracts like the fats and fatty acids in the wood. Then these slices were rinsed with deionized water. Finally, the treated wood slices were vacuumly dried at 60°C for over 24 hours.

**2.2. Preparation of TiO<sub>2</sub> Sol.** Tetrabutylorthotitanate (TBOT), diethanolamine (DEA), and absolute ethanol were used as starting materials. The 34 mL of TBOT and 11 mL of DEA were dissolved in 100 mL ethanol to prepare the precursor solution. After stirring for 2 h at room temperature, a mixed solution of 55 mL water and 9 mL ethanol was dropwise added following stirred another 3 h. Then TiO<sub>2</sub> sol was obtained.

**2.3. Fabrication of Wood-Templated TiO<sub>2</sub>.** Firstly, wood slices were coated by TiO<sub>2</sub> sol through a repeated dip-coating process. The pretreated wood slices were immersed into the as-prepared TiO<sub>2</sub> sol for 5 min. After that, these slices were

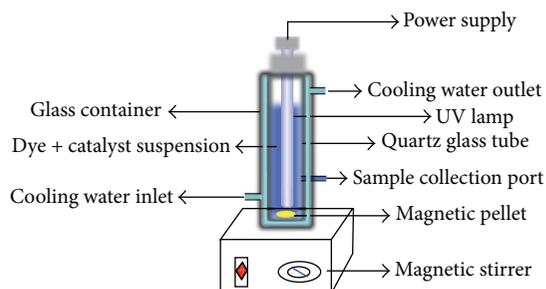


FIGURE 1: The schematic diagram of the photocatalytic reactor.

dried at 80°C for 3 h. This process was repeated for 5 times to ensure TiO<sub>2</sub> sol densely and evenly covered onto the wood slices. Then, calcination process was employed to remove the wood template. To get wood-templated TiO<sub>2</sub>, wood coated by TiO<sub>2</sub> sol was calcined in a muffle furnace at the temperature of 550°C for 3 h in air atmosphere and air cooled to room temperature. Finally, the calcined samples were taken out from the muffle furnace, and the wood-templated TiO<sub>2</sub> was obtained.

**2.4. Characterizations.** The morphology of the samples was observed by scanning electron microscopy (SEM, FEI, Quanta 200) and transmission electron microscopy (TEM, FEI Tecnai F20). The chemical compositions of the untreated and treated wood were measured by energy dispersive spectroscopy (EDS, attached to the SEM). The crystalline structures were identified by X-ray diffraction (XRD, Rigaku, D/MAX 2200) operating with Cu K $\alpha$  radiation ( $\lambda = 1.5418 \text{ \AA}$ ) at a scan rate ( $2\theta$ ) of 4°/min with an accelerating voltage of 40 kV and an applied current of 30 mA ranging from 10° to 70°.

**2.5. Evaluation of Photocatalytic Activity.** The photocatalytic activity of the samples was estimated through the decomposition of Rhodamine B (RhB) in a reactor with a volume of 100 mL. The irradiation source was an ultraviolet 100 W lamp (Shanghai Rongbo Co., China) with a wavelength of 254 nm. The photocatalytic activities of the prepared products were judged by measuring the loss of RhB in aqueous solution. A suspension involving 100 mg of catalyst and 100 mL RhB solution with an original concentration of 50 mg/L was magnetically stirred on the dark for 1 h without interrupting before irradiation. The change of solution concentration in every 30 min was measured to judge the absorbability of catalysts in the dark. At every interval during the process of 3 h, a certain sample of the mixture was collected, centrifuged, and then measured. The degradation rate for denoting the degradation efficiency of RhB was obtained by measuring the change in absorbance on a UV-Vis spectrophotometer (TU-190, Beijing Purkinje, China), and its maximum absorption wavelength of RhB was 554.5 nm. The schematic diagram of the photocatalytic reactor was represented in Figure 1.

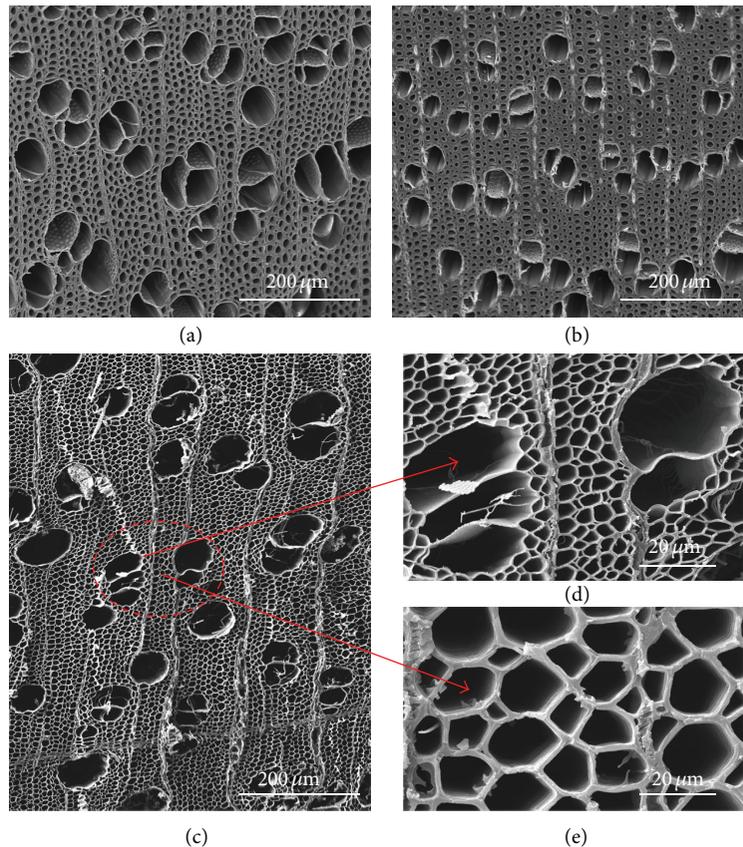


FIGURE 2: SEM images of the cross-section of (a) the original wood, (b) the wood coated by  $\text{TiO}_2$  sol, (c) wood-templated  $\text{TiO}_2$ , (d) the magnified calcined wood vessels, and (e) the magnified calcined wood fibers, respectively.

### 3. Results and Discussion

Figure 2 showed the morphologies of the cross-section of the original wood, wood coated by  $\text{TiO}_2$  sol, and wood-templated  $\text{TiO}_2$ , respectively. As shown in Figure 2(a), the cellular structures of the original wood could be clearly observed. The wood vessels with the diameter of about  $20\sim 50\ \mu\text{m}$  are irregular circular. The wood fibers with the diameter of about  $5\sim 20\ \mu\text{m}$  are uniformly arranged among the wood vessels. In Figure 2(b), the wood structure has not obviously changed after the dip-coating process by  $\text{TiO}_2$  sol. It can be clearly seen from the Figure 2(c) that the structures of the original wood were commendably remained after the calcined process. In the magnified SEM image (Figure 2(d)), calcined wood vessels with a little collapse could be obviously observed with the diameter of about  $35\ \mu\text{m}$ . Simultaneously, the calcined wood fibers shown in Figure 2(e) with a thinner cellular wall compared to the original wood were well arranged without any collapse. The diameters of these fibers are about  $15\ \mu\text{m}$ . In a word,  $\text{TiO}_2$  with the wood cellular structure was successfully prepared by dip-coating and calcination process. The microscopic structures of the original wood could not be destroyed with the previous process.

EDS spectra were also recorded for the original wood, wood coated by  $\text{TiO}_2$  sol, and wood-templated  $\text{TiO}_2$ , respectively. As shown in Figure 3(a), the carbon, oxygen, and

gold elements could be detected from the EDS spectrum of the original wood. The elements of carbon and oxygen are originated from the wood substrate, and the element of gold is from the coating layer used for SEM observation. Besides the signal of carbon and oxygen elements, titanium and nitrogen elements were displayed in the spectrum of  $\text{TiO}_2$  sol-coated wood slices (Figure 3(b)). The elements of titanium and nitrogen are stemmed from TBOT and DEA in the  $\text{TiO}_2$  sol, respectively. After calcination at  $550^\circ\text{C}$ , only oxygen and titanium elements could be probed from the EDS spectrum (Figure 3(c)), and no other elements were detected which confirmed that the components of the original wood and the  $\text{TiO}_2$  precursor were completely removed during the calcination process.

XRD patterns of the original wood, the wood coated by  $\text{TiO}_2$  sol, and wood-templated  $\text{TiO}_2$  were presented in Figure 4. Figure 4(a) revealed a typical XRD pattern of the original wood. The diffraction peaks at  $16^\circ$  and  $22.6^\circ$  were believed to represent the crystalline region of the cellulose in wood [25]. In Figure 4(b), no other new diffraction peaks were founded except for the diffractive characteristics of the initial wood, which might be due to that the crystal phase of  $\text{TiO}_2$  sol is consisted of amorphous  $\text{TiO}_2$  before the calcination. Apparently, in Figure 4(c), the diffraction peaks at  $16^\circ$  and  $22.6^\circ$  belonging to the original wood sample disappeared. New strong diffraction peaks were observed

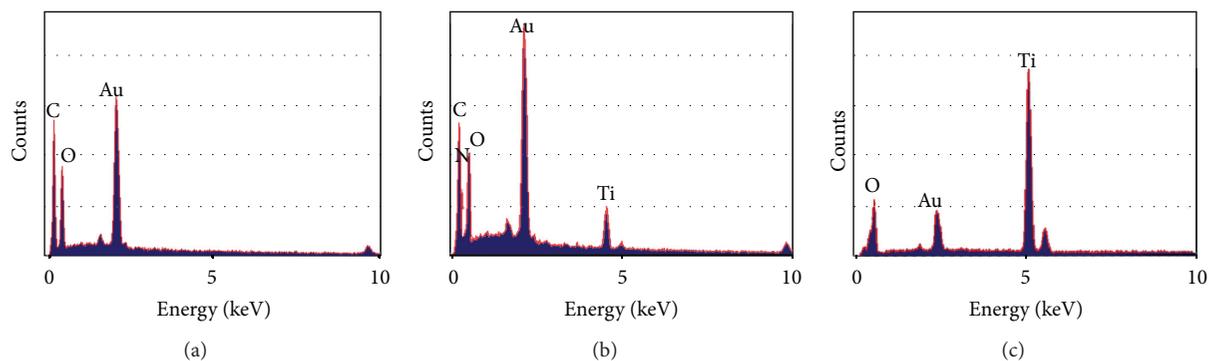


FIGURE 3: EDS spectra of (a) the original wood, (b) wood coated by  $\text{TiO}_2$  sol, and (c) wood-templated  $\text{TiO}_2$ .

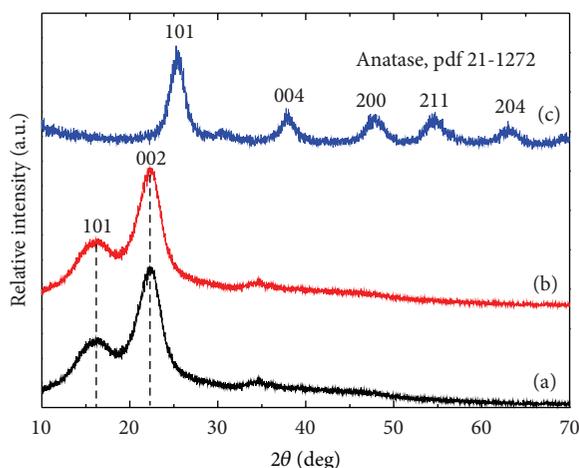


FIGURE 4: XRD patterns of (a) the original wood, (b) the wood coated by  $\text{TiO}_2$  sol, and (c) wood-templated  $\text{TiO}_2$ .

for the calcined sample, which indicated the formation of new crystal structures of the calcined wood sample. As shown in pattern of wood-templated  $\text{TiO}_2$ , the diffraction peaks at  $25^\circ$ ,  $38^\circ$ ,  $48^\circ$ , and  $54^\circ$  could be assigned to the diffractions of the (101), (004), (200), and (211) planes of anatase  $\text{TiO}_2$  (JCPDS number 21-1272) [36, 40], respectively. XRD pattern of the wood-templated  $\text{TiO}_2$  also confirmed that the components of the pristine wood were completely got rid of through the calcination process.

TEM images in Figure 5(a) revealed a much more fine structure of the obtained  $\text{TiO}_2$  sintered at  $550^\circ\text{C}$  for 3 h. Though  $\text{TiO}_2$  sample has been pulverized, the slices were found in the TEM image, just like the cellular framework of the original wood. Consequently, we could safely conclude that the cellular structure of the original wood has been preserved after the calcination process. The selected area electron diffraction (SAED) of the slice in Figure 5(b) illuminated the crystalline phase of the wood-templated  $\text{TiO}_2$  is polycrystalline with the coexistence of the diffraction rings and spots at the SAED pattern. The HRTEM image in Figure 5(c) revealed that the distance between the adjacent lattice fringes of (101) planes is about 0.352 nm which

is corresponding to the anatase  $\text{TiO}_2$  crystal ( $a = 0.325$  nm,  $c = 0.521$  nm).

The photocatalytic ability of the obtained cellular-structured  $\text{TiO}_2$  was investigated by decomposing RhB dye, a very difficult degradable and highly toxic organic pollutant. The photodegradation curves of RhB were exhibited in Figure 6. The wood-templated  $\text{TiO}_2$  could photodegrade the RhB under UV light irradiation. The original wood shown in Figure 6(a) had a little photocatalytic activity of about 4.6% on RhB. It was mainly ascribed to the physical absorption effect because of porous wood. In Figure 6(b), the wood coated by  $\text{TiO}_2$  sol showed a photocatalytic activity of about 8.21% on RhB. However, wood-templated  $\text{TiO}_2$  expressed a superior photocatalytic activity of about 92.5% on RhB (Figure 6(c)). The photocatalytic efficiency of the obtained  $\text{TiO}_2$  was greatly enhanced probably, since the prepared  $\text{TiO}_2$  remained the original wood cellular structures. The original wood porous structure could be affording a portable carrier for absorbing and degrading much organic pollutants.

Meanwhile, the photocatalytic activity of wood-templated  $\text{TiO}_2$  was compared to the commercially available  $\text{TiO}_2$  P25 (Degussa, Germany) that consist of both anatase (80%) and rutile (20%) phases of  $\text{TiO}_2$ . The same experimental conditions were selected for P25. From the comparison between catalysts wood-templated  $\text{TiO}_2$  and P25 (Figure 6(d)), it can be seen that RhB underwent decomposition of a little faster in the case of the latter catalyst. But the problem with the P25 powder is that the  $\text{TiO}_2$  disperses in water forming a white colloidal solution, and it is difficult to separate from the reaction mixture for reuse. The separation performance test showed that the wood-templated  $\text{TiO}_2$  has a good sedimentation ability and can decanted from the suspension in about 15 min, while the P25 do not decant after 45 min.

#### 4. Conclusions

In the present paper, the anatase wood-templated  $\text{TiO}_2$  was successfully fabricated by using a facile dip-coating and subsequent calcination process. The as-prepared  $\text{TiO}_2$  possessed the typical cellular structure with unbroken vessels and uniformly arranged wood fibers. Also, this wood-templated  $\text{TiO}_2$  presented a superior photocatalytic degradation of RhB.

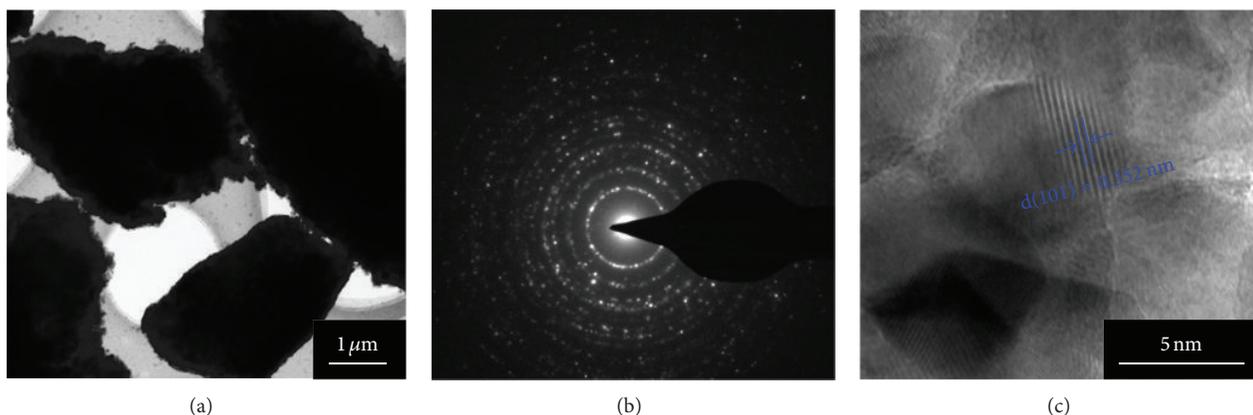


FIGURE 5: (a) TEM images, (b) the selected electron diffraction patterns, and (c) HRTEM images of the wood-templated  $\text{TiO}_2$ .

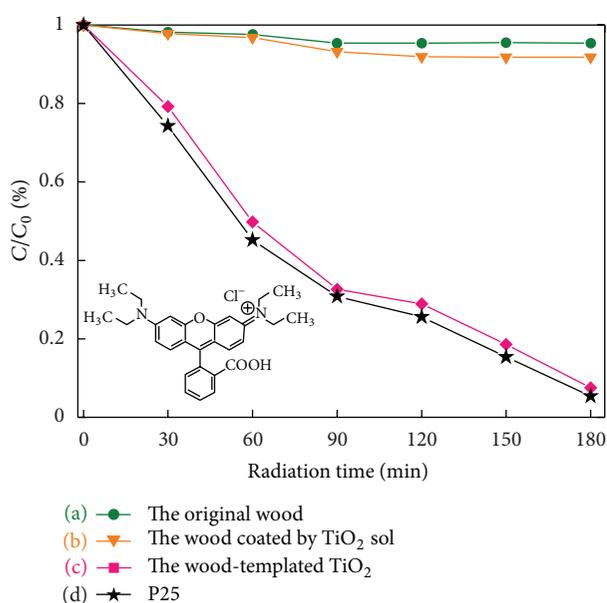


FIGURE 6: Photocatalytic activity of (a) the original wood, (b) the wood coated by  $\text{TiO}_2$  sol, (c) the wood-templated  $\text{TiO}_2$ , and (d) P25, respectively.

This paper probably provided another way for fabricating inorganic materials using the natural wood as template.

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