

Preliminary studies of multi-micro/nanomaterials immobilized on the bamboo timber surface

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Abstract Bamboo, a kind of forest resources only less important than wood, is especially easy to be degraded during exposure to outdoor environments. Nowadays, researchers endeavor to synthesize the multi-micro/nanomaterials on the bamboo timber surface to improve its intrinsic properties and impart excellent performances to bamboo products. In this study, multi-micro/nanomaterials including ZnO, TiO₂, CaCO₃, MnO₂, SiO₂, Ag, λ-Fe₂O₃, and graphite/ZnO, have been successfully immobilized on the bamboo timber surface using different methods including hydrothermal method, silver mirror reaction, and co-precipitation process. The geometric microstructures and crystalline structures of the materials on the bamboo timber surface were studied by scanning electron microscopy (SEM) and X-ray diffraction (XRD), respectively. SEM observations showed that the surface of the bamboo timber was densely covered with the uniform micro/nanomaterials, which surrounded the entire surface of the bamboo timber. XRD patterns confirmed that only high purity micro/nanomaterials were formed on the surface of bamboo timber. The mechanical stability of the coating on the bamboo timber surface was also characterized by a 3M tape test. In the process of manufacturing of these micro/nanomaterials, it is exceedingly important to

offer a potential opportunity to accelerate the large-scale production of woody functional materials for new industrial applications.

Keywords Bamboo timber · Micro/nanomaterials · Hydrothermal method · Silver mirror reaction · Co-precipitation process

Introduction

Bamboo has long played a vital role in the social, economic and environmental development of human history. Because of the increasing demand for the limited forest resources in various applications, has led to the shortage in wood supply. Thus, there is an urgent need to look for new materials as alternatives of wood. Bamboo is the fastest growing woody plants in the world, which grows to its maximum height in about 3 months and reaches maturity in 3–4 years (Scurlock et al. 2000). Due to its natural aesthetic beauty, incredible strength, and an advantage as a sustainable and eco-friendly substitute for increasingly depleted wood recourses, bamboo has been taken as notable economical and versatile raw material extensively used in construction and decoration fields for centuries (Onozawa et al. 2009). However, compared with wood products, bamboo timber (Li et al. 2015a) is susceptible to environmental degradation without protective treatment. When exposed in an outdoor environment, bamboo shows bad decay resistance, which would be attacked by fungi and insects, or degrade caused by moisture, air, acid rain, and sunlight, and thus shortens its service life (Chung et al. 2008). In order to overcome this problem and enhance the economic value of bamboo products, various approaches have been carried out.

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In previous studies, Yu et al. (2012) have demonstrated that bamboo timber can be simultaneously functionalized with photostability and antifungal and antibacterial performances by being coated with nanostructured ZnO using hydrothermal method. Sun et al. (2012) have proved that bamboo timber treated with chitosan-copper complex had better resistance against mould fungi. In our early reports, Ag nanoparticles (NPs) were successfully in situ deposited onto the surface of the bamboo timber through a simple silver mirror reaction, which made the intrinsic insulating bamboo timber conductive (Jin et al. 2015). Jin et al. (2014) prepared a portable catalyst of cross-linked ZnO nano-walls/bamboo composites for the photocatalytic application under UV light irradiation. We have reported a simple and facile method for fabricating an anatase TiO₂ film with superhydrophobicity, acid rain resistance and flame retardancy based on the bamboo timber surfaces (Li et al. 2015a). Furthermore, inspired from the biomineralization of nacre in seawater, a novel continuous plate-like CaCO₃ coating with hierarchical nano- and microstructures self-assembled on the surface of bamboo timber was also prepared (Li et al. 2015b). Over the past decade, metal nanomaterials have received much attention because of their unique physical and chemical properties, and their important applications in the areas of photography, catalysis, photonics, optoelectronics, biomedicine and surface-enhanced Raman scattering. Nano-sized particles of metal, metal oxides and semiconductor nanomaterials, including Ag, TiO₂, ZnO, SiO₂, MnO₂, Fe₂O₃, CeO₂, CaCO₃, CuO, etc., are extensively adopted due to their outstanding properties and potential applications (Chen et al. 2006). According to recent reports, the nanomaterials modified cellulose-based materials, such as wood, paper, cotton textiles, etc., display superior flame retardancy, acid rain resistance, UV stability, antimicrobial performance, and water-resistant properties (Chen et al. 2009; El-Rafie et al. 2010; Lu et al. 2014; Gao et al. 2015). Therefore, the bamboo timber modified using nanomaterials with multiple functions are of significance to improve performance of products and desirable in modern society.

Primary objective of present work was to investigate the growth behavior of eight different kinds of micro/nanomaterials on the surface of bamboo timber. Bamboo timber with enhanced functionalities, such as UV protection, mould-resistance, acid rain resistance, and antimicrobial performance, were greatly appreciated by a more discerning and demanding consumer market for high-value-added products. Herein, the final purpose was to explore whether bamboo timber can be imparted the multiple functions through coating with multi-micro/nanomaterials.

Materials and methods

Materials

All chemicals were supplied by Shanghai Boyle Chemical Company Limited and used as received. Moso bamboo slices (L × T × R) of 10 mm × 10 mm × 5 mm were ultrasonically rinsed in deionized water and then washed with acetone for 30 min, and then they were completely dried in the oven at 60 °C for 24 h.

Immobilization of the ZnO microsheets on the bamboo timber surface via a hydrothermal method

In a typical synthesis process, a zinc acetate dihydrate (Zn(CH₃COO)₂·2H₂O, ZnAc) solution in methanol (0.75 M) was added slowly to a solution of monoethanolamine (NH₂CH₂CH₂OH, MEA) with a volume ratio of 1:1 at room temperature. The resulting mixture solutions were then stirred for 30 min at 60 °C using a magnetic stirrer until a homogeneous and stable ZnO colloid solution were formed. Next, bamboo slices were immersed into the ZnO colloid solution for 5 min, then the slices were dried at 80 °C for 5 h, and the dip-coating process was repeated for five times to obtain the multi-layer films. Subsequently, the reaction solution was prepared by the following procedure: equimolar aqueous solutions (0.05 M) of zinc nitrate hexahydrate (Zn(NO₃)₂·6H₂O) and hexamethylenetetramine (C₆H₁₂N₄, HMTA) were prepared in a vessel under constant stirring, and then, 1.0 mL PEG-400 was added. The mixed solution was vigorously stirred for 30 min until the clear solution was formed, then was transferred into a Teflon-lined autoclave. The samples were collected and rinsed with distilled water several times after the hydrothermal treatment was performed at 95 °C for 1 h. Finally, the samples were dried at 50 °C for 48 h.

Fabrication of TiO₂ nanoparticles (NPs) on the surface of bamboo timber

In a typical synthesis process, ammonium fluorotitanate (4.0 g) and boric acid (3.7 g) were dissolved in distilled water in a 100-mL glass container at room temperature under vigorous magnetic stirring. Then, 1.0% hydrochloric acid aqueous solution was added drop by drop until the pH reached approximately 3. Then, 75 mL of the adjusted solution was transferred into a 100 mL Teflon container. Bamboo specimens were subsequently placed into the above reaction solution. The autoclave was sealed and maintained at 85 °C for 5 h, then cooled to room

temperature naturally. Finally, the prepared samples were removed from the solution, ultrasonically rinsed with anhydrous ethanol for 15 min, and dried at 50 °C for more than 48 h in vacuum oven.

Growth of CaCO₃ coating on the surface of bamboo timber

In a typical synthesis process, several bamboo timber specimens adhered to the wall of a beaker containing 250 mL of Na₂CO₃ aqueous solutions (0.3 mol/L) under vigorous magnetic stirring. Then 250 mL of 0.3 mol/L CaCl₂ aqueous solutions were quickly added into the Na₂CO₃ aqueous solutions, and the obtained solutions were kept under vigorous stirring at 60 °C for 30 min. The bamboo timber specimens covered with CaCO₃ seed layers were washed with deionized water, and then dried at 50 °C for 12 h. Reaction solution was prepared by consecutively adding 62 mmol/L NaCl, 4 mmol/L NaHCO₃, 8 mmol/L NaSO₄, 2.4 mmol/L KCl, 2.5 mmol/L CaCl₂, 1.6 mmol/L MgCl₂·6H₂O and 500 mL deionized water. The pH value of the mixed solution was 8.1 and then transferred into a Teflon-lined autoclave. Several bamboo timber specimens with CaCO₃ seed layers were subsequently placed into the above reaction solution. The growth of CaCO₃ coating was carried out at 50 °C for 8 h using a hydrothermal mineralization method. After hydrothermal treatment, the samples were collected and rinsed with distilled water for several times. Finally, the samples were dried at 50 °C for 48 h.

Immobilization of the MnO₂ microparticles on the bamboo timber surface

In a typical procedure, 1 mmol KMnO₄ and 1 mmol NH₄Cl were dissolved in 50 mL distilled water to form a clear solution under magnetic stirring, then this solution was transferred into a Teflon-lined autoclave of 75 mL capacity. Bamboo specimens were subsequently placed into the above reaction solution. The autoclave was sealed and maintained at 140 °C for 3 h, then cooled to room temperature naturally. The resulting products were collected by filtration and rinsed with distilled water and absolute alcohol several times, and then dried at 50 °C for 48 h.

Deposition of SiO₂ microsphere on the bamboo timber surface

In this study, we prepared SiO₂ microspheres following the Stöber method (Stöber et al. 1968). Briefly, 5 mL of tetraethyl orthosilicate (TEOS) was added dropwise, under magnetic stirring, to a flask containing 5 mL deionized

water and 45 mL of ethanol. Bamboo specimens were subsequently placed into the above reaction solution and continue mixing for 15 min at 60 °C. Then, 5 mL of ammonia solution (28.0%, catalyst) was added dropwise to the above reaction solution. The stirring was continued for 2 h. The samples were then washed by ethanol five times, and then dried at 50 °C for 48 h.

In situ formation of Ag NPs on the surface of bamboo timber by silver mirror reaction

Bamboo timber samples were treated with 10 wt% aqueous NaOH solution at room temperature for 5 min followed by rinsing with copious amount of distilled water. Aqua ammonia (10 wt%) was added drop by drop into a 0.5 M AgNO₃ aqueous solution with stirring until a transparent colorless [Ag(NH₃)₂]⁺ solution was formed. The alkali treated bamboo timber samples were dipped into the [Ag(NH₃)₂]⁺ solution for 1 h, then transferred into a 0.2 M glucose stock solution. After 5 min, the residual [Ag(NH₃)₂]⁺ solution was also poured into the glucose solution. The reaction kept on for 30 min. At last, the bamboo timber samples were rinsed with water and dried at 50 °C for 48 h.

Fabrication of magnetic γ-Fe₂O₃/bamboo timber composites

The magnetic γ-Fe₂O₃ NPs on surface of bamboo timber were synthesized by a co-precipitation method. Typically, at room temperature, bamboo slices were immersed into a 100-mL mixture solution containing 0.6 M FeCl₃·6H₂O and 0.3M FeCl₂·4H₂O for 5 h and followed by vacuum-impregnation for 2 h. Then ammonia solution was added dropwise to adjust the pH value to 10.5 and continuously stirring for 2 h. After this treatment, the bamboo samples were removed from the solutions and ultrasonic rinsed in deionized water for 10 min, and subsequently vacuum-dried at 50 °C for 48 h.

Fabrication of graphite/ZnO/bamboo timber composites

The method for fabricating graphite/ZnO/bamboo timber composites is similar to the “[Immobilization of the ZnO microspheres on the bamboo timber surface via a hydrothermal method](#)” section. The only difference is as following: The graphite powder was well mixed with the ZnO colloid solution and then magnetically stirred at room temperature for 30 min. The weight percentage of the graphite/ZnO colloid solution was set at 1:50. The bamboo slices obtained were immersed into the above mixed solutions for 10 min. Next, the bamboo slices were dried at

80 °C for 5 h using a drying oven. The procedures from dip-coating to drying were repeated three times to obtain multilayer films.

Characterization

The surface morphologies of the samples were characterized by scanning electron microscopy (SEM, FEI, Quanta 200). The crystalline structures of the samples were identified by X-ray diffraction technique (XRD, Rigaku, D/MAX 2200) using Cu $K\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$) at a scan rate (2θ) of 4° min^{-1} , 40 kV, 40 mA, ranging from 5° to 80° .

Results and discussion

Figure 1 shows the SEM images of untreated and treated bamboo timber surfaces at low and high magnification, respectively. Figure 1a shows the microstructure of a

longitudinal section of original bamboo timber. Vascular bundles are embedded in the matrix of ground parenchyma. The original bamboo timber displayed clean and smooth surface. Figure 1b showed the SEM images of ZnO microsheets on the bamboo timber surface at low magnification. From the SEM image, it could be clearly seen that thickly ZnO microsheets had been immobilized onto the bamboo timber surface after the hydrothermal reaction. In the magnified SEM image (inset), the morphology of ZnO microsheets could be clearly observed. These cross-linked ZnO microsheets were randomly oriented due to the unevenness of the bamboo timber surface, leading to a most frequent observation of the cross-linked ZnO microsheets. In Fig. 1c, the pristine bamboo timber surface was covered by a dense and even film of TiO_2 particles. Figure 1c inset presented the SEM image of TiO_2 -treated bamboo under higher magnification. Clearly, the surface of bamboo timber was covered by plentiful irregularly shaped TiO_2 aggregations with diameters of about 0.5–3.5 μm .

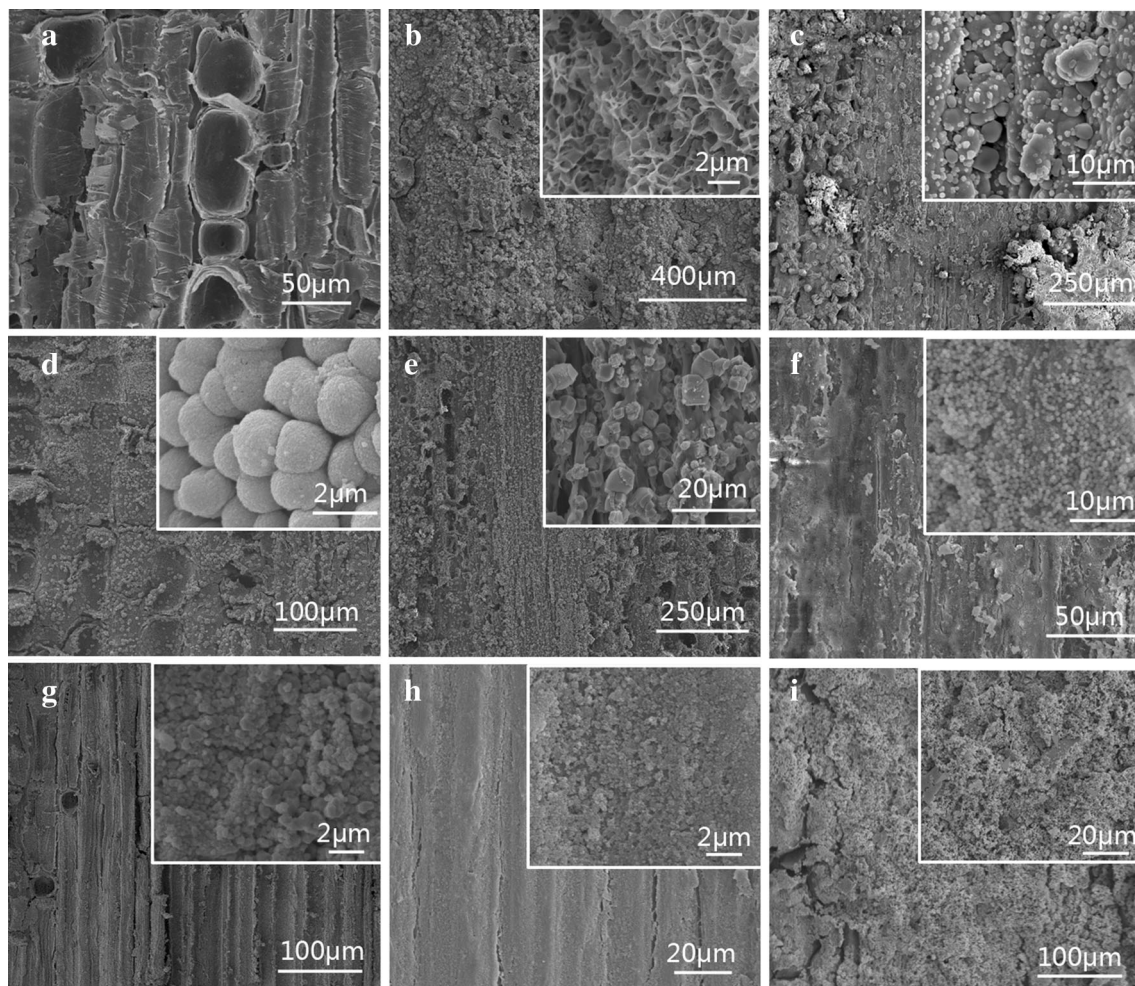


Fig. 1 SEM images of original bamboo timber (a) and multi-micro/nanomaterials including ZnO (b), TiO_2 (c), CaCO_3 (d), MnO_2 (e), SiO_2 (f), Ag (g), $\lambda\text{-Fe}_2\text{O}_3$ (h), and graphite/ZnO (i), on the

bamboo timber surfaces, respectively. The insets in (b–i) are the corresponding amplifying SEM images

Figure 1d presented that the CaCO_3 particles with irregular structures were formed on the surface of bamboo timber after hydrothermal mineralization at 50 °C for 8 h. The higher magnification image in Fig. 1d inset showed that the particles with the sizes ranging from 2.0 to 2.5 μm were clearly visible on the surface of bamboo timber. In Fig. 1e, it was obvious that the surface of bamboo timber was densely covered by MnO_2 microparticles. The inset in Fig. 1e indicated that the particle size of the MnO_2 microparticles was about 1.0–5.0 μm . It could be observed in Fig. 1f that the obtained material SiO_2 presented as a uniform-sized microsphere on the bamboo timber surface, which was further proved by the characterization of SEM (Fig. 1f, inset). The distribution of the particle size was centered at about 1.0 μm . As shown in Fig. 1g, after silver mirror reaction, the surface of bamboo timber displayed a compact and uniform covering of Ag NPs. The higher magnification image of the coated bamboo timber in Fig. 1g inset showed that the particles with the sizes ranging from 100 to 300 nm were clearly visible. In our previous research, the compact coating of Ag NPs also imparted the metallic feature to the surface rendering the bamboo timber conductive. In Fig. 1h, after the co-precipitation process, a densely packed layer of $\gamma\text{-Fe}_2\text{O}_3$ NPs covered the surface of the bamboo timber, masking the vascular bundles and other details. The size of $\gamma\text{-Fe}_2\text{O}_3$ NPs was 10–20 nm (Fig. 1h inset). In Fig. 1i, it was clear that the graphite/ZnO particles were successfully covered on the bamboo timber surface using a hydrothermal method. From Fig. 1i inset, we found that the graphite (the red arrows) and ZnO particles were cross-linked each other on the bamboo timber surface. The sizes of graphite and ZnO particles were about 20 μm and 1–2 μm , respectively.

To identify the purity of the multi-micro/nanomaterials coated on the surface of bamboo timber, the XRD technique was performed. Figure 2a showed the XRD pattern of the original bamboo timber. Apparently, the original bamboo timber displayed a typical cellulose I crystalline structure, exhibiting (100), (002), and (040) peaks at around 15.9°, 22.1°, and 34.8°. After the hydrothermal process, all of the diffraction peaks except for the characteristic peaks of the original bamboo timber could be indexed to wurtzite-type ZnO (JCPDS card No. 36-1451). No characteristic peaks from other phases of ZnO were found (Fig. 2b), suggesting that the ZnO microsheet had a high phase purity and was completely composed of wurtzite crystals. According to the Fig. 2c, except for the cellulose diffraction peaks from bamboo timber, some new strong diffraction peaks at 25.3°, 37.8°, 48.0°, 54.2°, 55.9°, and 62.5° correspond to (101), (004), (200), (105), (211), and (204) planes of anatase TiO_2 crystal (JCPDS cards: 21-1272), respectively. Moreover, no characteristic peaks were observed from the other impurities indicating purity of the anatase TiO_2 coating. As shown in Fig. 2d, all

diffraction peaks could be indexed to pure calcite CaCO_3 (JCPDS 47-1743) except the characteristic peaks of the bamboo timber. No characteristic peaks of other phases of CaCO_3 were found, suggesting that the CaCO_3 coating had a high phase purity and was totally composed of calcite crystals. From Fig. 2e, the diffraction peaks at about 12.5°, 25.2° and 37° from the as-prepared MnO_2 microparticles matched the standard XRD pattern of birnessite-type manganese oxide crystal (JCPDS 80-1098). No other peaks that would be associated with another phase or impurity were observed. In Fig. 2f, no sharp diffraction peaks were observed in SiO_2 /bamboo timber sample. The XRD pattern of SiO_2 /bamboo timber showed a single broad peak centered at about 23°, which were characteristic of amorphous SiO_2 . As shown in Fig. 2g, three obvious peaks at 38°, 44°, and 64° corresponded to (1 1 1), (2 0 0), and (2 2 0) planes of silver crystal (JCPDS cards: 04-0783), respectively. No characteristic peaks were observed for the other impurities such as Ag_2O . Thus, the coating on the surface of bamboo timber was only composed of silver crystals and no other compounds existed, which imparted high conductivity to the bamboo timber. For $\gamma\text{-Fe}_2\text{O}_3$ /bamboo timber composites (Fig. 2h), new sharp peaks at 2θ of 30.1°, 35.4°, 43.1°, 53.4°, 57.1° and 62.6° can be ascribed to (220), (311), (400), (422), (511), and (440) crystal planes, which could be attributed to the spinel phases of maghemite $\gamma\text{-Fe}_2\text{O}_3$ (JCPDS 39-1346). Additionally, the diffraction peaks of the original bamboo timber were not observed, demonstrating that a relatively dense $\gamma\text{-Fe}_2\text{O}_3$ film was formed at the bamboo timber surface. Figure 2i and j displayed the XRD patterns of graphite/ZnO/bamboo timber and pure graphite samples. By comparing the Fig. 2i with j, it was confirmed that the new diffraction peaks could be assigned to wurtzite-type ZnO. These observations indicate that the graphite/ZnO coating has been successfully deposited at the surface of bamboo timber.

One of the limitations of the nanomaterials coated bamboo timber to apply as commercial products is lack of adhesion. For the purpose of evaluating the adhesion between the nanomaterials and bamboo timber substrates, Ag nanomaterial was selected as the research target, which could be made as a direct-viewing scenario. The adhesion between the Ag NPs and bamboo timber substrate has been measured by a 3M tape test. As shown in Fig. 3, after three times cycle tests, the conductivity of the obtained bamboo timber did not show significant change and could still light a bulb with the dazzling light, which indicated the Ag layer on the surface of bamboo timber was not destroyed. However, after five times cycle tests, only a small number of Ag-coated bamboo timber surface could light a bulb. The results indicated that the multi-micro/nanomaterials coated on the bamboo timber surface could be easily damaged after cycle of mechanical fracture. In the future, new ways to enhance the mechanical property of

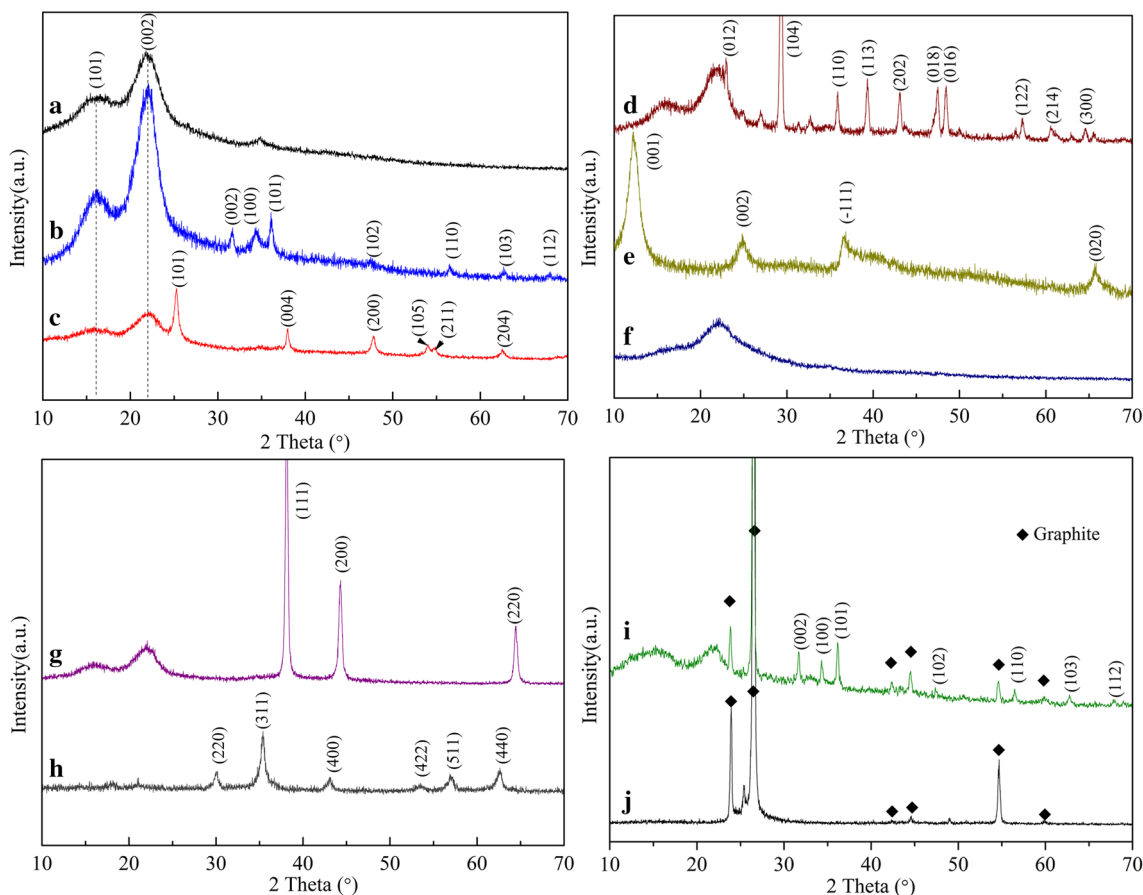
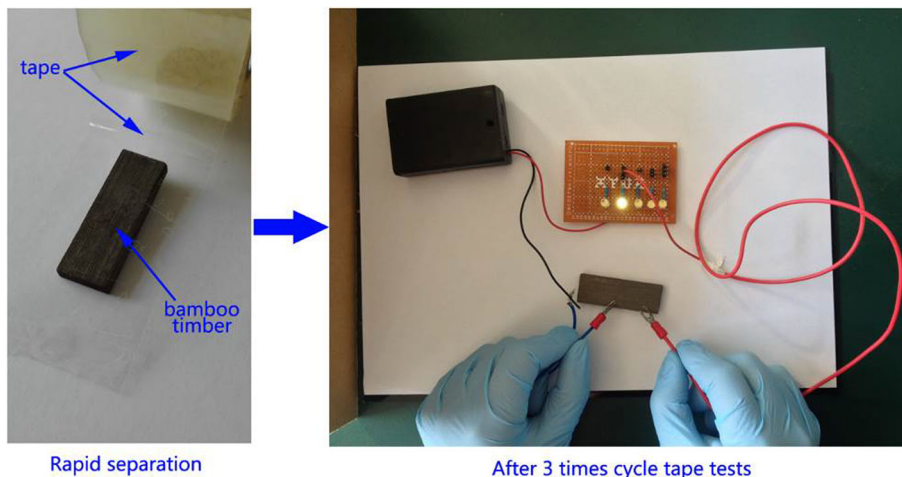


Fig. 2 XRD patterns of pristine bamboo timber (a) and multi-micro/nanomaterials including ZnO (b), TiO₂ (c), CaCO₃ (d), MnO₂ (e), SiO₂ (f), Ag (g), λ -Fe₂O₃ (h), and graphite/ZnO (i), on the bamboo timber surfaces. Diamond indicates the crystal phase of graphite (j)

Fig. 3 Conductive image of Ag NPs coated bamboo timber after three times cycle tests by a 3M tape test



micro/nanomaterials modified bamboo timber surface are to be found, which offered a potential opportunity to accelerate the large-scale production of multifunctional bamboo timber material for new industrial applications.

A possible mechanism for the formation of multi-micro/nanomaterials on the surface of bamboo timber is suggested as follows (Fig. 4). The whole course consists of

three stages (Thanh et al. 2014): the first stage is that metallic ions are sequestered by functional groups such as hydroxyls present on the surface of bamboo timber, which may help in the generation of the crystal nucleus on the surface (second stage). The third stage is a growth process; when the crystal nuclei forms on the bamboo timber substrate, the micro/nanomaterials gradually start to grow

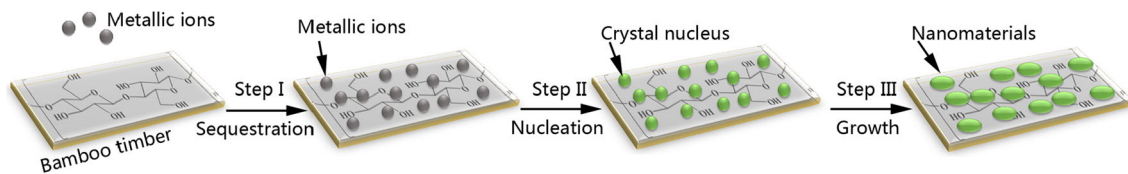


Fig. 4 Mechanism for the nucleation and growth of the multi-micro/nanoparticles on the surface of bamboo timber

surrounding the crystal nucleus on the bamboo timber surface. During this process, nanocrystals from smaller nanoparticles transport to larger structures by surface diffusion along the bamboo timber substrate, different shapes of micro/nanomaterials such as ZnO microspheres, TiO₂ particles, SiO₂ microspheres, and Ag nanoparticles, and so on, can be formed on the surface of bamboo timber by adjusting concentration of metallic ions, reaction temperature and reaction time. The detailed mechanism is worthy of further exploration for investigations on the interactions between metallic micro/nanomaterials and bamboo timber in an attempt to create novel composites.

Conclusions

In this work, multi-micro/nanomaterials including ZnO, TiO₂, CaCO₃, MnO₂, SiO₂, Ag, λ-Fe₂O₃, and graphite/ZnO, have been successfully immobilized on the bamboo timber surface using different methods like the hydrothermal method, silver mirror reaction, and co-precipitation process. The multi-micro/nanomaterials are not only surrounded the entire surface of the bamboo timber, but also formed highly pure materials. This study may be extended for the immobilization of other metal/metal oxide micro/nanoparticles onto bamboo timber. Furthermore, present work could provide a platform for further investigation of the growth mechanism of micro/nanomaterials on the bamboo timber surface. Further research is required to assess the improvements in the properties of bamboo timber through depositing multi-micro/nanomaterials on its surface.

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