IMPROVING PHOTOSTABILITY AND ANTIFUNGAL PERFORMANCE OF BAMBOO WITH NANOSTRUCTURED ZINC OXIDE

*Yan Yu**
Associate Professor

Zehui Jiang
Professor

Genlin Tian
Assistant Professor

Hankun Wang
PhD Candidate
Division of Biomaterials
International Center for Bamboo and Rattan
100102 Beijing, China

Ye Song
Masters Candidate
School of Materials
Central South Forestry Science and Technology University
410004 Chang Sha, China

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Abstract. We report on the formation of zinc oxide (ZnO) films with various morphologies on bamboo to simultaneously furnish it with excellent photostability and antifungal properties. A simple two-step process was adopted, consisting of generation of ZnO seeds on the bamboo surface followed by solution treatment to promote crystal growth. Effect of reaction conditions on film morphologies was systematically investigated. Results indicate morphologies of ZnO films can be tailored from nanoparticles to nanostructured networks and irregular aggregates at the micron scale with different crystallinities through specific combinations of reaction conditions. The photostability and antifungal performances of coated bamboo were greatly improved and highly dependent on both crystallinity and morphologies of ZnO films.

Keywords: Bamboo, nanostructured ZnO, morphology, photostability, antifungal performance.

INTRODUCTION

Zinc oxide (ZnO) is a wide band gap (3.37 eV) semiconductor with a large exciton binding energy (60 meV). It forms a variety of nanostructures including nanorods, nanowires, nanobelts, nanosprings, nanocombs, and nanorings (Wang 2004). Nanostructured ZnO has great potential for many practical applications, such as dye sensitized solar cells, piezoelectric transducers, UV-light emitters, and transparent conductive coating (Özgür et al 2005). It also exhibits intensive UV absorption and can potentially be used as UV-shielding materials and antibacterial agents (Kim and Osterloh 2005). Fabrication of nanostructured ZnO generally requires high temperatures or economically prohibitive devices. Recently, increasing efforts have been devoted to the fabrication route of low-temperature aqueous solutions (Yamabi and Imai 2002). This approach

* Corresponding author: yuyan@icbr.ac.cn

can decrease reaction temperature to lower than 95°C and no expensive equipment is needed, therefore it is very suitable for heat-sensitive natural biomaterials. Wang et al (2004) recently demonstrated that oriented hexagonal ZnO nanorods could be grown on cotton fabrics using low-temperature aqueous solutions. Lu et al (2006) further proposed a novel approach to fabricate ZnO/polystyrene nanohybrid coatings on cotton fabrics, which gave treated fabrics ultrahigh UV protection properties and superior washing color fastness. Paper could also be given excellent antibacterial properties when coated with ZnO nanoparticles (Ghule et al 2006). Although these successes demonstrate the feasibility and potential of functionalizing biomaterials with nanostructured ZnO, controlling morphologies of the formed ZnO films is still a significant challenge.

In a previous study, we demonstrated that highly oriented ZnO nanorod arrays could be grown on softwood surfaces using similar approaches (Yu et al 2010). Morphology of ZnO nanofilm was dependent on immersion time in ZnO nanosol. Greatly enhanced photostability was achieved with treated wood. In this work, much more systematic research was conducted on bamboo. The effect of reaction conditions on morphologies and crystallinity of ZnO film was emphasized. Bamboo is one of the most important nontimber forest resources in the world because it grows faster than almost all trees on earth. Bamboo is being widely used as an alternative resource for wood because of its rapid growth rate, great strength and hardness, and superior flexibility (Liese 1987). However, bamboo is very susceptible to fungi and insect attack because of its high sugar and starch content compared with wood, which results in degraded performance, shortened service life, and decreased value (Liese and Kumar 1998). Also, bamboo is very sensitive to UV irradiation during outdoor service (Wang and Ren 2008, 2009). Our objective was to explore if photostability and antifungal properties of bamboo can be simultaneously improved by being coated with ZnO films and how morphologies and crystallinities of films affect resulting protective performances.

MATERIALS AND METHODS

Sample Preparation

Formation of zinc oxide crystal seeds on bamboo. Bamboo blocks 20 mm long, 20 mm tangential, and 5.8 mm radius were rinsed ultrasonically in deionized water for 20 min and oven-dried at 60°C for 3-6 h. All chemicals were used as received without further purification. ZnO films were grown on bamboo using a modified simple two-step process consisting of seed coating in ZnO nanosol and crystal growth in a zinc salt aqueous solution. ZnO nanosol was prepared using a method proposed by Pacholski (Pacholski et al 2002; Greene et al 2003). Zinc acetate dihydrate ($ZnAc•2H_2O$) and sodium hydroxide (NaOH) were added slowly into methanol with vigorous stirring at about 60°C and stirred for 3 h to form a transparent homogeneous solution. ZnO nanosols with two different pH values were prepared by changing the mole ratio between ZnAc and NaOH (Table 1). Bamboo samples were then immersed in the sol at room temperature for different periods (0.5, 1, 2, and 4 h) followed by heat treatment in air at 100°C for 3 h. This procedure was repeated three times to form ZnO nanocrystal seeds on bamboo, which acted as crystal nuclei to facilitate growth of ZnO in the next treatment.

Growth of zinc oxide films on bamboo. Seed-coated samples were then immersed in aqueous growth solution containing equal mol zinc nitrate six hydrate ($ZnNO_3•6\ H_2O$) and methenamine ($[CH_2]_6N_4$). Solution temperature was kept at 90°C, and the time ranged from 0.5-9 h. Mol concentration of zinc salt solution was set at

Table 1. Formula for zinc oxide (ZnO) nanosol with different pH values.

	ZnO nanosol			
NO	$ZnAc•2H_2O$ (mol)	NaOH (mol)	Methanol (L)	pH value
1	0.01	0.03	1	11.45
2	0.01	0.015	1	9.37

Table 2. Formula used for producing zinc oxide (ZnO) films with different morphologies on bamboo.

Morphologies of ZnO films	Nanosol (pH value, treatment time)	Growth solution (mol concentration and treatment time)	Weight gain (%)
Nanoparticles	11.45; 1 h	0.025 mol/L; 6 h	1.20
Micron balls with network structure on surface	9.37; 4 h	0.020 mol/L;6 h	2.39
Nanostructured networks	11.45; 2 h	0.020 mol/L; 6 h	1.43
Irregular layered aggregates at micron scale	11.45; 4 h	0.020 mol/L; 6 h	1.50

0.015, 0.020, and 0.025 M, respectively. Finally, samples were rinsed with deionized water and dried at 60°C for 3 h.

The appearance and texture of treated bamboo was unchanged after treatment. No attempts were made to measure thickness of ZnO films because the rough and uneven surface of bamboo makes it almost impossible to obtain reliable thickness data. Weight gain after treatment is shown in Table 2. This value was calculated by taking into account weight loss of bamboo extractives during immersion, which was measured by extracting bamboo samples in solutions with similar pH value, temperature, and time. This simulation procedure was also used for preparing control samples.

Structural Characterization

For each treatment condition, three samples were randomly selected for SEM and X-ray diffraction characterization. A field-emission scanning electron microscope (XL30-FEG-SEM; FEI, Hillsboro, OR) combined with energy-dispersive analysis X-ray (EDAX) was used to examine morphologies and elemental compositions of films formed on bamboo. A Philips (Andover, MA) X'pert Pro diffractometer was used at $2\theta/\theta$ scanning mode to investigate effects of various reaction conditions on crystallization behavior of films.

Performance Evaluation

Photostability. An accelerated weathering test chamber (Atlas, Germany) was used to accelerate photo discoloration of bamboo samples. Radiation intensity was set at 42 W/m^2, and chamber temperature was 40°C. Samples were fixed in stainless steel holders and rotated around the fixed xenon light source at 65% RH for 0-120 h. Changes in surface color of bamboo with irradiation time were determined with a color meter (BYK-6834; BYK Co., Germany). CIELAB L*, a*, b*, and E* parameters were measured at five locations of each specimen, and average value was calculated. Five specimens were measured for each treatment condition. In the CIELAB system, L* axis represents lightness and a* and b* are chromaticity coordinates. L*, a*, and b* values are used to calculate overall color changes, ΔE*, using the following equation:

$$\Delta E^* = \sqrt{(\Delta a^*)^2 + (\Delta b^*)^2 + (\Delta L^*)^2} \quad (1)$$

where Δa*, Δb*, and ΔL* are differences between initial and final values of a*, b*, and L*, respectively. Lower ΔE* value corresponds to lower color changes and indicates better photostability.

Antifungal performance. A very convenient and quick method for evaluating bamboo antifungal performance was developed based on the fact that molds grow much more quickly on bamboo than on wood under a high moisture environment. This makes fungal inoculation to bamboo unnecessary, whereas it is usually required for wood. Both control and treated samples (five samples for each treatment condition) were water-saturated and then placed in a sealed vessel with 95 ± 2% RH and a temperature of 23-25°C. The vessel was placed in a common laboratory with normal illumination in the daytime and dark break at night for 35 da. Pictures and qualitative description were taken everyday during the initial stage and at intervals of 3-5 da in the final stage.

RESULTS AND DISCUSSION

Dependence of Film Morphologies on Reaction Conditions

Immersion time in zinc oxide nano sol. Figure 1 shows the effect of immersion time in ZnO nano sol on final morphologies of ZnO films on bamboo. Mol concentration of zinc salt in growth solution was 0.025 mol/L, and growth time was 6 h. Samples pretreated in ZnO nanosol from 0.5-1 h were all coated with films consisting of nanoparticles ranging from 10-30 nm in diameter (Fig 1a-b). However, if immersion time was set at 2 h, nanoparticle films were replaced by nanostructured networks that were composed of randomly oriented irregular sheets with wall thickness normally less than 50 nm or randomly arranged nanorods/nanowires occurring with lower probability (see insets in Fig 1c-d). Growth of both ZnO nanoparticles and nanostructured network films on bamboo are thought to be based on heterogeneous nucleation and subsequent crystal growth, respectively, involved in the two-step process of seed coating in ZnO nanosol and crystal growth in zinc nitrate aqueous solution. A similar approach has been successfully used for fabrication of oriented ZnO nanorod arrays or nanowires on various inorganic and synthetic polymer substrates (Choy et al 2003; Vayssieres 2003). However, when the immersion time was further increased to 4 h, many irregular aggregates at the micron scale were observed. They were approximately round or hexagonal in shape. Furthermore, these aggregates usually precipitated randomly on the top of the underlying nanostructured networks on bamboo (Fig 1d), which supported the opinion that they were not likely to have developed

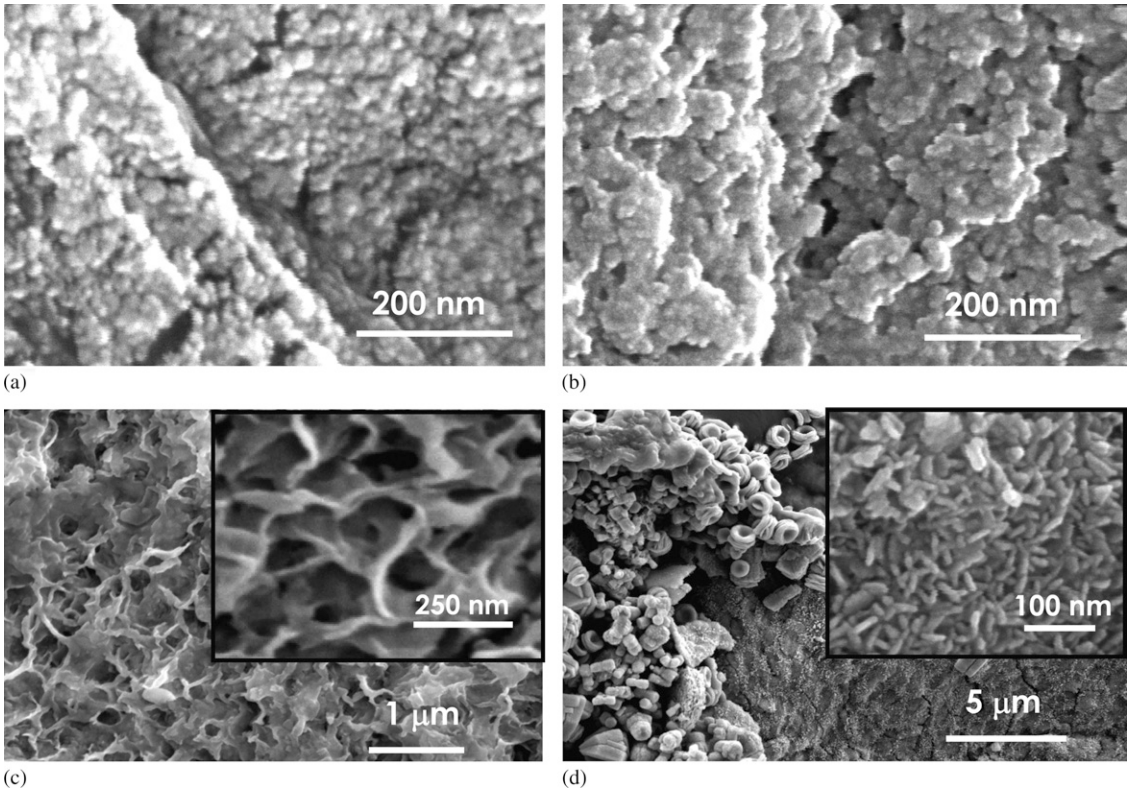
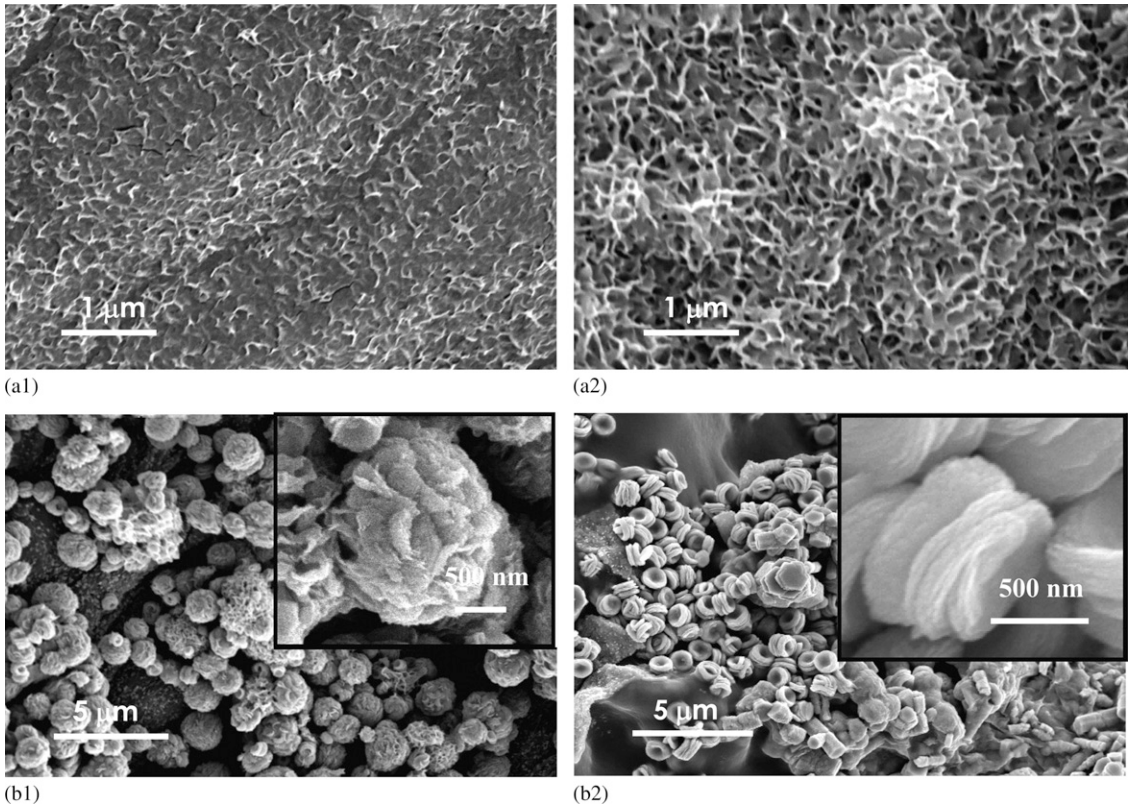


Figure 1. Effect of immersion time in zinc oxide nanosol (pH value 11.45) on morphologies of films formed on bamboo. (a, 0.5 h; b, 1 h; c, 2 h; d, 4 h). Mol concentration of zinc salt in growth solution is 0.025 mol/L, and growth time is 6 h.

from nanoparticles or networks on the surface of bamboo but arose directly from the bulk solution. Although no precise measurements have been conducted as yet to evaluate adhesion between ZnO films and bamboo substrate, continuous water flushing on bamboo surfaces resulted in almost no change in nanostructured networks, whereas distribution of irregular aggregates was decreased, which indicated that no strong interaction existed between irregular aggregates and bamboo. We believe a longer period of seed coating will form and release more ZnO seeds into the solution during growth and significantly accelerate growth and aggregation of ZnO in the bulk solution. These aggregates finally precipitated on bamboo because of gravity. These results indicate that final morphologies of ZnO films on bamboo were significantly dependent on pretreatment time in ZnO nanosol.

pH in zinc oxide nano sol. Figure 2 compares final morphologies of films that were formed by immersion in ZnO nanosol with different pH for 1 and 4 h, respectively, and then in the same growth solution for 6 h. It was observed that increased pH tended to result in better developed nanostructured networks (Fig 2a1-a2). This was caused by improved crystallinity of ZnO crystal seeds under higher pH. Better matching of the crystal lattice between ZnO crystal seeds and the end products favors growth of crystals (Li et al 2005; Tak et al 2006). Furthermore, pH value also affects nucleation



(a1) (a2)

(b1) (b2)

Figure 2. Effect of pH value in zinc oxide (ZnO) nanosol on final morphologies of films formed on bamboo (1, pH 9.37; 2, pH 11.45). Immersion time in ZnO nanosol is 1 h for a and 4 h for b. Mol concentration of zinc nitrate in growth solution is 0.020 mol/L, and growth time is 6 h.

and growth in the zinc nitrate bulk solution. Figure 2b shows many micron balls with nanostructured networks on their surfaces precipitated on the bamboo surface (see inset in Fig 2b1). This novel structure has not been described before as far as we know. We speculate that it was ZnO seeds with lower crystallinity or more defects that dispersed into growth solution and resulted in this unique structure. However, at pH 11.45, many short disks with variable diameters ranging from several hundred nanometers to several microns were observed (Fig 2b2). These disks were approximately round or hexagonal in shape and were characterized by their layered structures (see inset in Fig 2b2). The subsequent section will reveal that layered micron disks had significantly higher crystallinity than that of micron balls and provided better preservative performance for bamboo. Based on the previous observations, it was concluded that pH value of ZnO nanosol was the key factor to control quality and crystallinity of the ZnO seed layer on bamboo.

Zinc nitrate mole concentration in growth solution. Figure 3 shows the effect of zinc nitrate mole concentration on final morphologies of films on bamboo. Importance of immersion time in ZnO nanosol for the final morphologies of films was highlighted again. The predominant growth mode of ZnO on bamboo was heterogeneous nucleation when immersion time in ZnO nanosol was 1 h, because no superimposed precipitates were observed. Furthermore, high concentrations tended to prohibit formation of networks, because only nanoparticle films were formed in 0.025 mol/L, whereas a nanostructured network was grown in both 0.20 and 0.15 mol/L (Fig 3a1-a3). We postulated that a high concentration of zinc nitrate in growth solution would promote ZnO growth in the bulk solution and result in suppressed heterogeneous nucleation on the bamboo surface. This assumption was also supported by the observation that a higher concentration of zinc nitrate tended to produce more micron-scale aggregates when samples were immersed in ZnO nanosol for 4 h before growth (Fig 3b1-b3). These irregular

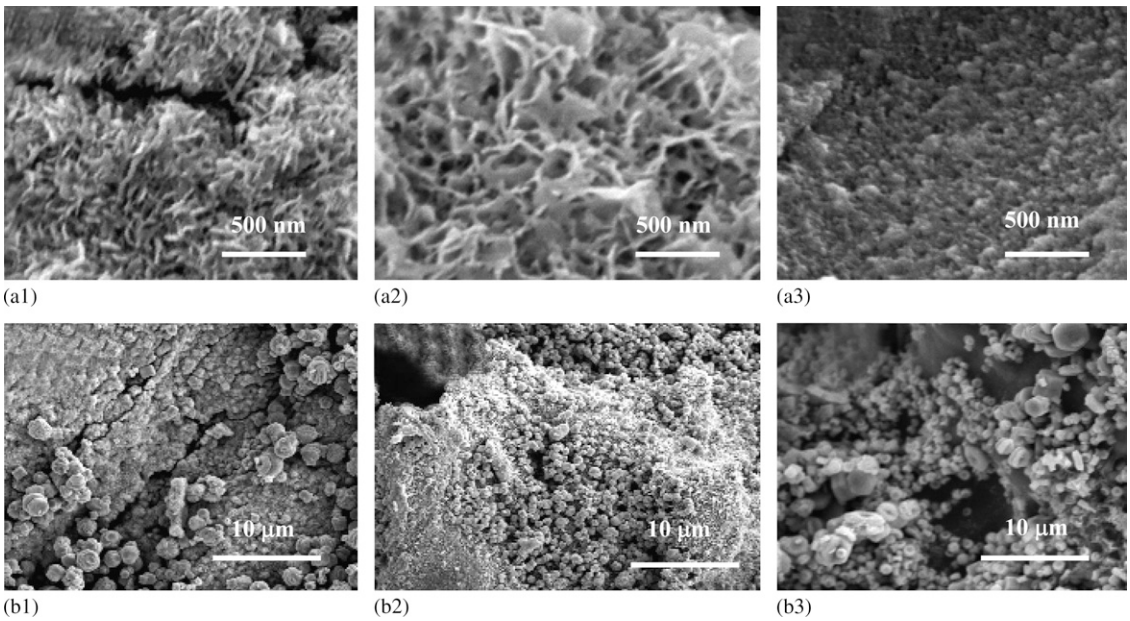


Figure 3. Effect of concentration of zinc nitrate on morphologies of films formed on bamboo (1, 0.015 mol/L; 2, 0.020 mol/L; 3, 0.025 mol/L). Immersion time in zinc oxide nanosol (pH 11.45) is 1 h for a and 4 h for b. Growth time in zinc nitrate solution is 6 h.

precipitates also came from the growth and aggregation induced by ZnO seeds released from bamboo into the bulk growth solution.

Growth time in growth solution. For this reaction system, nanostructured networks of films tended to be more packed as growth time increased from 0.5-9.5 h (Fig 4a1-a5). This implies that heterogeneous nucleation and growth on bamboo were predominant during the growth period for samples pretreated in ZnO nanosol for 2 h. However, if samples were pretreated in ZnO nansol for 4 h before growth, nucleation and growth in the bulk solution became much more

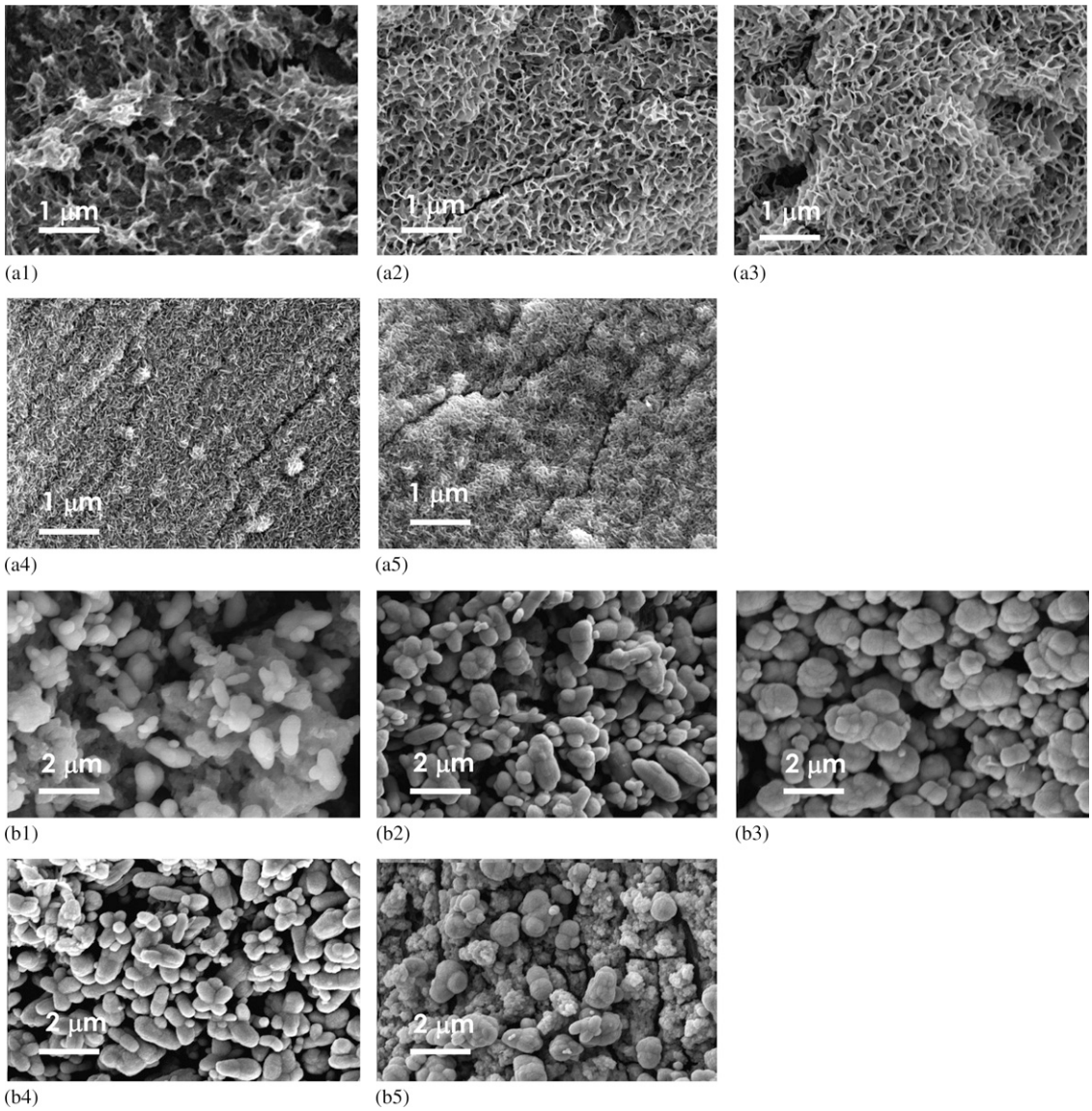


Figure 4. Effect of growth time in zinc nitrate solution on morphologies of films formed on bamboo (1, 0.5 h; 2, 1 h; 3, 3.5 h; 4, 6 h; 5, 9.5 h). Immersion time in zinc oxide nanosol (pH 11.45) is 2 h for a and 4 h for b.

significant because many irregular aggregates at the micron scale started to precipitate, although growth time was only 0.5 h. Furthermore, distribution density or sizes of these aggregates tended to increase from 0.5-6 h because of crystal growth (Fig 4b1-b4). The looser distribution of aggregates in Fig 4b5 can be explained by redissolution of solid ZnO accelerated by decreased concentration of zinc cations in the solution. Increasing amounts of white precipitate assumed to be ZnO were observed on the bottom of vessels with increasing growth time, which could have consumed considerable amounts of zinc cations in the solution.

Chemical Compositions and Crystalline Structure of Films

Growth of ZnO films on bamboo is much more complicated than that on smooth inorganic or chemically stable synthetic polymer substrates. There were as many as five typical morphologies obtained at different reaction conditions, including nanoparticles, micron balls, nanostructured networks composed of nanosheets or nanorods/nanowires, and irregular layered aggregates at micron scale (Fig 5a1-e1). Nanoparticles and nanostructured networks were believed to form based on a mechanism of heterogeneous nucleation and growth on the bamboo surface, whereas micron balls and irregular aggregates came from homogeneous nucleation and growth in the bulk growth solution. It should be pointed out that morphology of densely packed ZnO array of nanorods was never observed on bamboo, whereas this morphology can be formed on softwood surfaces with similar process (Yu et al 2010). The relatively smooth inner wall in the cell cavity of softwood tracheids could have predominantly contributed to successful growth of highly oriented ZnO nanorod arrays. Another explanation might be higher extractive content from bamboo, resulting in more significant change of pH in growth solutions.

Corresponding EDAX spectra of the five morphologies are presented in Fig 5a2-e2. Zinc, oxygen, carbon, and platinum were detected. Platinum probably came from the conductive layer on the surface of samples for SEM observation. Carbon signals and some of the oxygen signals are believed to have originated from the bamboo substrate underneath. The relative signal intensity of carbon and oxygen to zinc can be an indicator of thickness or density of films. The relatively higher signal intensity of carbon and oxygen to zinc in Figs 5a2 and 5c2 might be attributed to the thinner or looser structure of the films, which permits more areas of bamboo to be hit by accelerated electrons and more X-ray signals to be emitted.

EDAX can tell us what elements exist in films, but it cannot tell us if there is $Zn(OH)_2$ mixed with ZnO or how well the film is crystallized. This can be revealed by X-ray diffraction. X-ray patterns in Fig 5a3-e3 show characteristic peaks of ZnO with different intensity, which means the extent of crystallization of these films was different. Samples coated with irregular aggregates had the sharpest diffraction peaks, representing the best crystallization behavior followed by nanostructured networks consisting of nanosheets or nanowires/nanorods. Micron balls and nanoparticles were found to have the lowest crystallinity. No characteristic peak for other possible impurities such as $Zn(OH)_2$ was detectable, which means no or only extremely minor crystallized impurities could have existed in the formed films. The cellulose characteristic peak at 22.4° from bamboo substrates can be observed clearly for all samples.

Photostability of Zinc Oxide-Coated Bamboo

Figure 6 shows the effect of photoirradiation on color changes of control samples and samples coated with different morphologies of ZnO films. A positive ΔL^* indicates a tendency of bamboo to white, whereas negative values indicate a tendency to black. For Δa^*, positive values indicate a tendency to red, whereas negative values mean a tendency to green. Positive values of Δb^* indicate a tendency to yellow, and negative values indicate a tendency to blue.

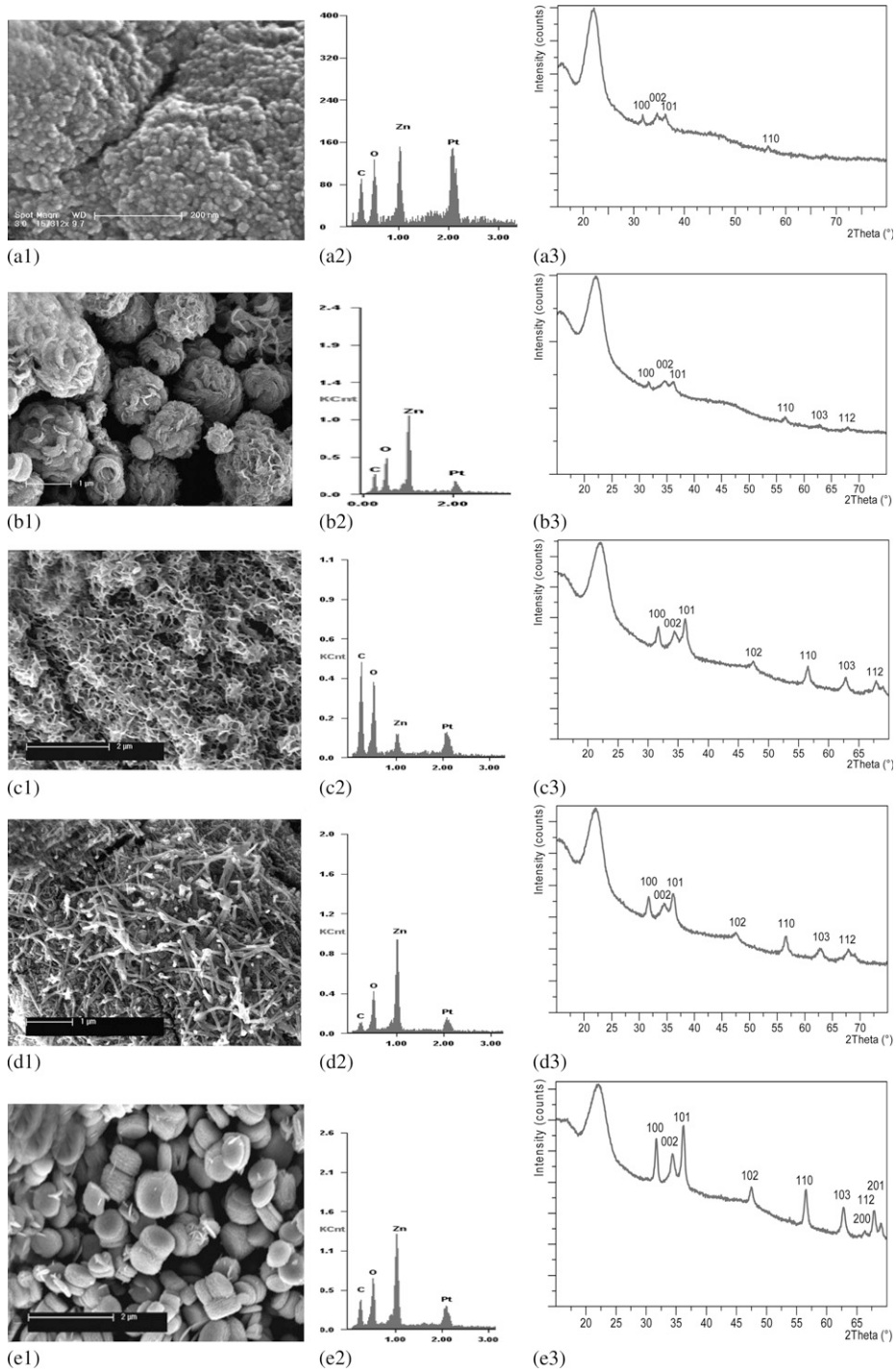


Figure 5. Five typical morphologies of zinc oxide films formed on bamboo. a, Nanoparticle films; b, micron balls with network structure on surface; c, networks consisting of nano sheets; d, networks consisting of nanowires; e, irregular aggregates at micron scale. 1, SEM images; 2, energy-dispersive analysis X-ray spectra; 3, X-ray diffraction patterns.

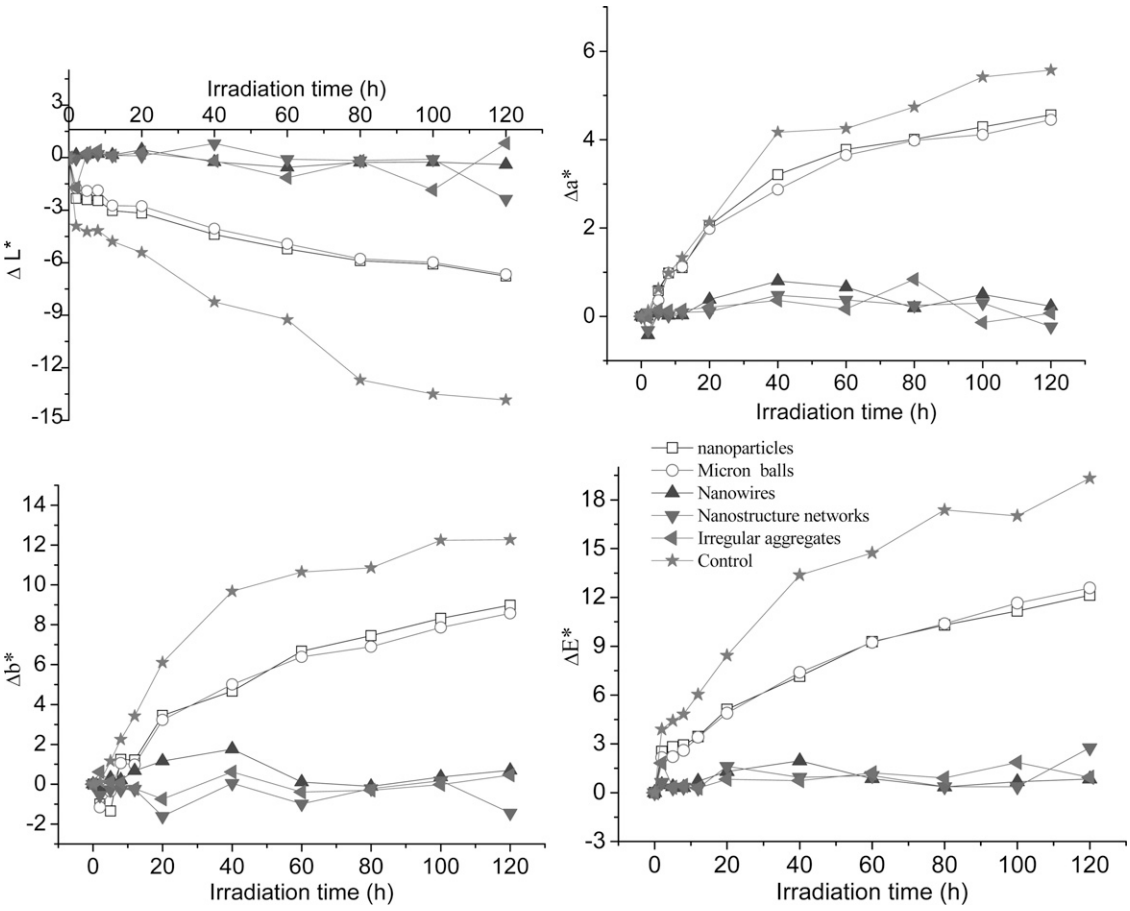


Figure 6. Variation of CIELAB parameters ΔL*, Δa*, Δb*, and ΔE* at different irradiation time for bamboo coated with different morphologies of zinc oxide films.

Generally, color changes of all samples coated with ZnO films were significantly less than those of controls after 120 h irradiation, which indicated ZnO films effectively improved photostability of bamboo. The smallest total color difference ΔE* observed was less than 1/9 of the control samples. Furthermore, morphologies of the films affected efficiency of color protection. Samples coated with nanostructured networks, nanowires, and irregular aggregates showed similar photostability, but all were much superior to samples coated with micron balls and nanoparticles. This could be attributed to lower crystallinity of micron balls and nanoparticles. Although irregular aggregates had nearly the same resistance to UV light as that of nanostructured networks, UV blocking efficiency of the latter was superior to the former if film thickness is accounted for. Our results indicate that UV blocking efficiency was dependent on both crystallinity and characteristic size of ZnO films. ZnO nanostructured networks have stronger capability in UV shielding, which can be explained by their higher separation efficiency of electron and hole pairs because of nanosized effects.

Antifungal Performance of Zinc Oxide-Coated Bamboo

Mold fungi are universally detected in water-damaged wood constructions (Andersson et al

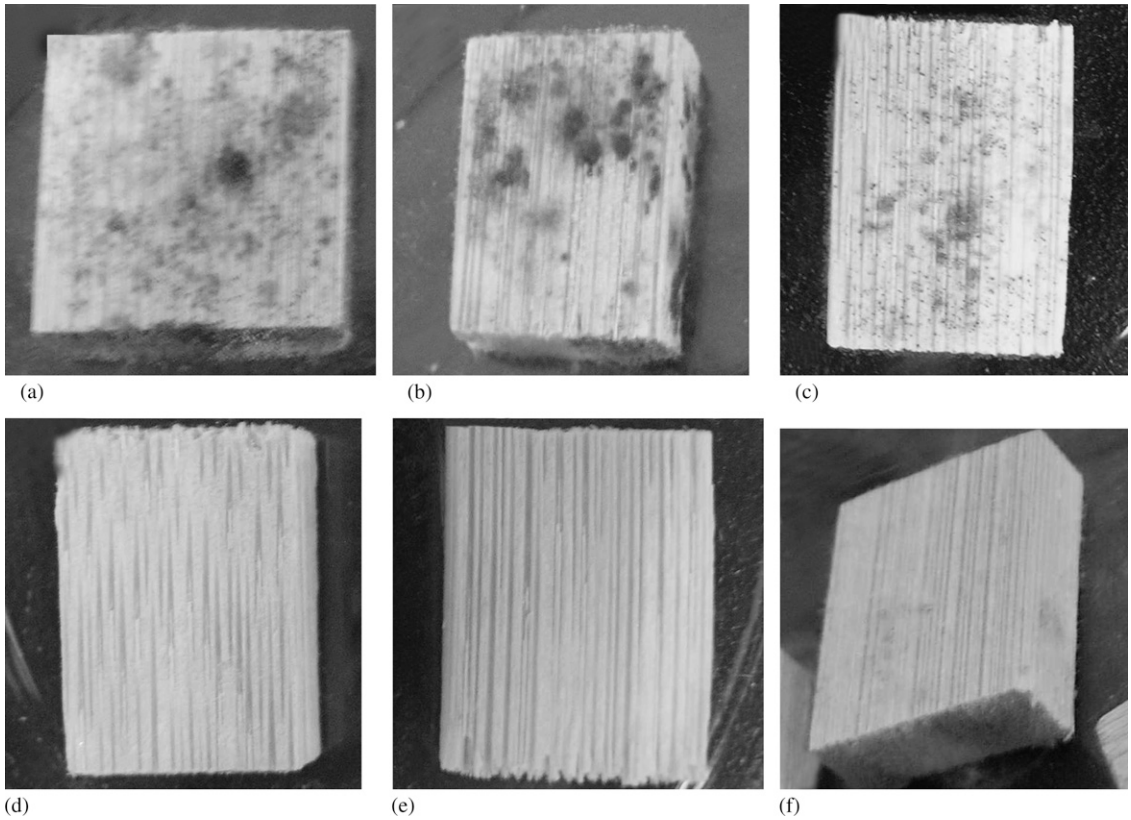


Figure 7. Mold growth on bamboo coated with zinc oxide films. These pictures were taken on 35th day. a, Control; b, nanoparticle films; c, micron balls with network structure on surface; d, networks consisting of nanosheets; e, networks consisting of nanowires; f, irregular layered aggregates at the micron scale.

1997. This problem becomes much more serious with bamboo resources (Okahisa et al 2006). Only photographs taken on the 35th day are presented here. For control samples, mold appeared in 24 h and quickly covered the whole surface, normally in less than 7 da (Fig 7a). Samples with nanoparticle films were stained on the fourth day but exhibited nearly the same appearance as that of the control on the 35th day (Fig 7b). Although fungal contamination of samples coated with ZnO micron balls was only slightly less severe than that of the control (Fig 7c), the first occurrence of mold was postponed for more than 20 da. There was almost no visible mold growth on the surface of samples coated with nanostructured networks and nanowires (Fig 7d-e). In addition, samples coated with irregular layered aggregates were only slightly contaminated. These results indicated that ZnO nanostructured networks possessed the best antifungal capacity followed by irregular aggregates and micron balls. ZnO nanoparticle ranked last with almost no inhibitory effect on mold growth. This sequence is almost the same as that in crystallinity, indicating crystallinity was the key factor of ZnO antifungal activity. However, the role of nanostructure was also appreciable because ZnO irregular aggregates showed weaker antifungal activity compared with nanostructured networks, although the former was thicker and higher in crystallization than the latter.

CONCLUSIONS

An aqueous solution technique to grow ZnO films with controllable morphologies on bamboo

substrates at low temperature was reported. Five typical morphologies have been formed, among which nanostructured networks and irregular aggregates at the micron scale were observed most frequently. Nanostructured networks are composed of randomly oriented nanosheets and sometimes of nanaowires/nanorods formed through heterogeneous nucleation on the bamboo surface, whereas irregular aggregates grow in bulk growth solution and then precipitate on bamboo because of gravity.

Morphologies of ZnO films coated on bamboo were correlated to reaction conditions and were significantly dependent on immersion time in ZnO nanosol. Longer immersion time tended to promote production of irregular aggregates. In this study, 4 h was found to be the critical value. Higher pH values in ZnO nanosol, longer growth times, and lower concentrations of zinc nitrate all facilitated development of ZnO nanostructured networks when immersion time in ZnO nanosol was less than 4 h.

All ZnO films were capable of simultaneously improving photostability and antifungal performances of bamboo. Nanostructured networks showed the best comprehensive performances. Improvement in photostability and antifungal performance was highly dependent on both crystallinity and morphologies of ZnO films.

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