

High Photocatalytic Properties of Zinc Oxide Nanoparticles with Amidoximated Bacterial Cellulose Nanofibers as Templates^{*}

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Abstract The freshly prepared water-wet amidoximated bacterial cellulose (Am-BC) serves as an effective nanoreactor to synthesis zinc oxide nanoparticles by *in situ* polyol method. The obtained ZnO/Am-BC nanocomposites have been characterized by field emission scanning electron microscopy (FE-SEM), X-ray diffraction (XRD), Fourier transformed infrared spectroscopy (FTIR) and thermogravimetric analysis (TGA). The influence of the zinc acetate concentration on the morphologies and size of ZnO nanoparticles and the possible formation mechanism were discussed. The results indicated that uniform ZnO nanoparticles were homogeneously anchored on the Am-BC nanofibers through strong interaction between the hydroxyl and amino groups of Am-BC and ZnO nanoparticles. The loading content of ZnO nanoparticles is higher using Am-BC as a template than using the unmodified bacterial cellulose. The resultant nanocomposite synthesized at 0.05 wt% shows a high photocatalytic activity (92%) in the degradation of methyl orange.

Keywords: Amidoximated bacterial cellulose; Nanoreactor; ZnO nanoparticles; High photocatalytic activity.

INTRODUCTION

Nanostructured ZnO material with high surface area and quantum confinement effect is currently attracting considerable interest due to its extensive applications in photo-catalysis, anti-bacterial materials, sensors and dye sensitized solar cells^[1–10]. Various techniques have been employed to synthesize ZnO nanoparticles, including sol-gel method, vapor deposition, precipitation, thermal decomposition, hydrothermal synthesis and spray pyrolysis^[11–16]. However, it is generally difficult to disperse nanoparticles in a reaction system, as nanoparticles tend to aggregate due to their high surface energy. Therefore, addition of a surfactant or surface modification is usually required to stabilize metallic nanoparticles^[17–19]. Another effective approach is the immobilization of metallic nanoparticles through hybridization with an template to obtain great performance with good dispersion and stabilization. Recently, several research have been published about the preparation of nanocomposites with organic templates such as polyimide, kraft fiber, natural cellulose and its derivatives, *etc*^[20–23]. However, immobilization on substrates is still a major challenge in the field of metal nanoparticles, because the immobilization process is sensitive to balance between nucleation and crystal growth. Besides, the current environmental issues show a pressing need for innovative, sustainable and recyclable synthesis methods.

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Bacterial cellulose (BC), biosynthesized cellulose, has gained more and more attention these years for its favorable properties. BC is generated as an ultrafine nano-sized three-dimensional (3D) fibrous network comprised of the ribbon-shaped nanofibers with diameters of 10–100 nm^[24]. In addition, with unique properties and structures such as high crystallinity, good biocompatibility^[25], high specific surface area with a great deal of hydroxyl groups and excellent mechanical strength, BC therefore could be an excellent candidate for organic support to stably fabricate metallic nanoparticles with a high surface-to-volume ratio, high mechanical toughness and low thermal expansion properties. Taking advantage of these unique properties, BC can serve as a “nanoreactor” to synthesize nanomaterials with specific shape and size through its three dimensional and porous networks and certain nano-pore size distribution^[26]. Recently, many investigations regarding the application of BC as a nanoreactor to obtain nanomaterials such as noble metal, CdS and CdSe nanoparticles and PANI nanosheets were reported^[26–30]. However, the properties of the obtained composites are unsatisfactory with a low yield and sparse distribution of nanoparticles due to the relatively weak electrostatic interactions between the metallic ions and the hydroxyl groups on the surface of BC fibers^[31–32]. In order to control the formation of nanomaterials, the BC fibers as nanoreactor should be chemically functionalized to endow them with new functionalities, and thus to produce BC nanocomposites with tailored characteristics. Several approaches for the modification of BC substrates have been reported during the last decades^[25, 31, 33, 34]. Our group has successfully prepared amidoximated BC (Am-BC) by introducing amidoxime groups into bacterial cellulose^[35–36]. Advantages of the application of amidoximated BC nanofibers are mainly as follows: (1) Amidoxime groups on the surface of BC nanofibers could greatly absorb and stably anchor plenty of metallic ions from aqueous solutions; (2) Am-BC prevents the ZnO nanoparticles from aggregating during the *in situ* polyol method.

In this paper, we reported the *in situ* synthesis of well-controlled ZnO nanoparticles using Am-BC hydrogels as an effective nanoreactor. Am-BC as host matrix can stably anchor numerous zinc ions and then as nanoreactors to fabricate ZnO nanoparticles by hydrolysis of zinc acetate in a polyol medium. Thermogravimetric analysis (TGA) suggested that the residues referred to the content of the ZnO nanoparticles and the photocatalytic activity are much higher compared with the ZnO nanoparticles using unmodified bacterial cellulose as templates^[37], which proves the Am-BC is a highly effective nanoreactor for the synthesis of nanoparticles in a high yield.

EXPERIMENTAL

Materials

Purified BC mats obtained from acetobacter xylinum cultures were prepared in our laboratory and they were cut into approximately 4 cm × 5 cm × 0.5 cm pieces, and they were pretreated with 1 wt% NaOH aqueous solution for 2 h to increase the reactivity before use. Acrylonitrile (AN), diethylene glycol isopropanol, acetone and hydroxylamine were purchased from Sinopharm Chemical Reagent. Zinc acetate dihydrate and methyl orange (MO) solution were purchased from Shanghai Chemical Company. All reagents are of analytical grade and were used without further purification. High purity water with resistivity of 18.2 MΩ cm was used in the whole experimental process.

Field emission scanning electron microscope (FE-SEM) images of the films and nanoparticles were taken on an S-4800 FE-SEM. X-ray diffractometer (XRD) patterns were obtained on a Rigaku D/max-2550 PC X-ray diffractometer with the Cu K α radiation ranging from 2° to 80° (2 θ). The FT-infrared (FTIR) spectra of samples were recorded on a Nicolet NEXUS-670 FTIR. Each sample was grounded with dried potassium bromide (KBr) powder and compressed into a disc, and then was subjected to analysis. Thermal gravimetric measurement was performed under nitrogen atmosphere using a Netzsch TG 209 F1 with a heating rate of 20 K/min. Prior to analysis, the samples were cut into small pieces from the freeze-dried samples.

Preparation of Amidoximated BC

The Am-BC was prepared in our laboratory according to the reported method^[35–38]. The purified BC mats were immersed in 10 wt% tetramethylammonium chloride aqueous solution for 2–3 min. Then the mats, 70 mL of AN

and 2 mL of 10 wt% NaOH are mixed in a round bottom flask with stirring for about 1 h at room temperature. Subsequently, 30 mL of isopropanol was added into the resultant aqueous solution and the pH of the solution was adjusted to be neutral by adding acetic acid. After rinsing with distilled water until neutral and then washing with acetone for 2 times, cyanoethyl bacterial cellulose (CE-BC) was prepared as an intermediate product. The obtained CE-BC and 40 mL 10 wt% hydroxylamine hydrochloride aqueous solution, whose pH was adjusted to 9–10 by sodium carbonate, were added into round bottom flask with stirring vigorously for 3 h at 80 °C. After that, the amidoximated BC was finally prepared.

In situ Synthesis of ZnO Nanoparticles on the Amidoximated BC Nanofibers

Water-wet freshly prepared Am-BC mats were dipped in 0.05 wt%, 0.1 wt% and 1 wt% zinc acetate aqueous solution at room temperature for 2 h. After being rinsed with distilled water for three times, the Am-BC mats, 5 mL zinc acetate aqueous solution, 50 mL diethylene glycol were mixed in a round bottom flask and stirred vigorously in an oil bath at 170 °C for 10 min. The final composite mats were washed with distilled water and ethanol, then freeze-dried.

Measurement of Photocatalytic Activity

The photocatalytic activities of the ZnO/Am-BC nanocomposites were evaluated by the photocatalytic degradation of methyl orange (MO) solution. 2 g Freeze-dried nanocomposites were first cut into small pieces and then added to 100 mL of MO solution with a concentration of 10 mg/L with magnetical stirring for 1 h to reach the adsorption equilibrium of MO with the catalyst. Then the system was stirred under ultraviolet light. After a certain time, 10 mL suspension solution was made to stand to remove the photocatalyst, and the absorption value (A_t) of the solution at 460 nm (the maximum absorption wavelength of MO) was determined. The degradation efficiency (D_t) of MO solution was calculated as follows: $D_t(\%) = (A_0 - A_t)/A_0 \times 100\%$, where A_0 is absorption value of 10 mg/L methyl orange solution. The photocatalytic activities of these samples were evaluated by changing of D_t with the reaction time (t). The photocatalytic activities of ZnO/Am-BC nanocomposites at different reaction conditions were measured for five times respectively to obtain the standard deviation.

RESULTS AND DISCUSSION

The Formation Mechanism of ZnO Nanoparticles on Am-BC Nanofibres

Am-BC was prepared by a simple two-step synthetic route as shown in Fig. 1. First, BC mats was added into AN under alkaline conditions to prepare cyanoethyl bacterial cellulose (CE-BC). Secondly, CE-BC was treated by an aqueous solution of hydroxyl amine hydrochloride at a known concentration to synthesize the Am-BC. Elemental analysis shows the nitrogen content of cyanoethyl bacterial cellulose is 3.36% and Am-BC 5.78%, respectively. According to the relations reported by Saliba *et al.*^[40] the value of the degree of substitution in amidoxime groups obtained from the elemental nitrogen analysis was about 0.401 and the amino-group content was found to be 1.723 mmol per gram cellulose. Although the content corresponds, on average, to one amino group per 3.5 anhydro glucose units according to the relations reported by Ifuku *et al.*^[31], amidoxime groups are mainly introduced into BC nanofibers surface. The amidoxime group content under the reaction condition seems to be high-density on BC surface. Both of the nitrogen atoms in amino groups and the oxygen atoms in original BC fibers have lone pair electrons that have strong interaction with metal ions to form stable chelate compound by chelation, electrostatic attraction and precipitation effect to anchor metal ions on the surface of BC fibers^[30]. Compared with BC, the Am-BC mats possesses an exquisite three dimensional network structure which could provide a good tunnel for adsorption of Zn^{2+} , and the plenty of amidoxime groups on the Am-BC surface behave a strong interaction between guest metal ions and host cellulose fibers, which leads to the homogeneous distribution of Zn^{2+} on the nanofibers^[35].

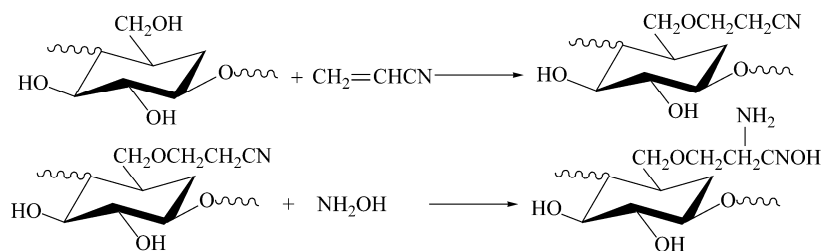


Fig. 1 Two-step synthetic route of Am-BC

A synthetic route for ZnO nanoparticles with template of Am-BC in the presence of water through a polyol mediated synthesis is shown in Fig. 2. During the synthesis process, zinc ions are incorporated and anchored onto the stable absorption sites by the strong interaction between Zn^{2+} and amidoxime groups of Am-BC. Then a sufficiently rinsing step was taken to remove unanchored Zn^{2+} . When water was injected into hot precursor solution maintained at 180 °C, a short burst of homogeneous nucleation happened: First, the pre-anchored $\text{Zn}(\text{CH}_3\text{COO})_2$ can be hydrolyzed, forming $\text{Zn}(\text{OH})(\text{CH}_3\text{COO})$ as an intermediate in the presence of water. Secondly, condensation between the hydrolyzed $\text{Zn}(\text{CH}_3\text{COO})(\text{OH})$ species can occur to yield a $\text{Zn}-\text{O}-\text{Zn}$ bond by leaving behind either H_2O or CH_3COOH . ZnO is nucleated *via* successive hydrolysis and condensation reactions followed by fast particle growth on the surface of growing particles^[41]. With the help of Am-BC, the ZnO nanoparticles have the tendency to grow into relatively spherical particles and to be anchored at the ex-absorbed sites, preventing the nanoparticles from aggregating.

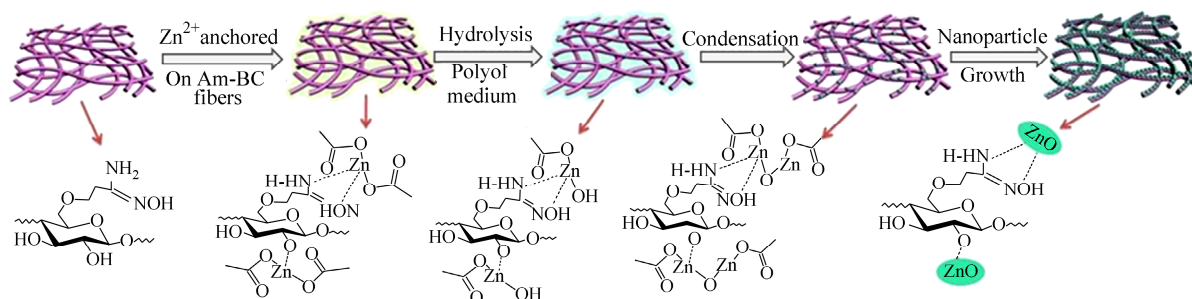


Fig. 2 Schematic diagram of the formation of ZnO nanoparticles onto nanofibers of Am-BC mats

FE-SEM Analysis

The FE-SEM images of the ZnO nanoparticles synthesized under different concentrations of zinc ions are shown in Fig. 3. The result indicated that the size and distribution of the particles are greatly affected by the concentration of zinc ions. The increasing concentration of zinc ions can offer greater opportunities for Am-BC to anchor more Zn^{2+} , resulting in larger amount of ZnO nuclei and higher loading of ZnO particles onto Am-BC nanofibers. But at the same time, the unstable particles on fibers may have the tendency of aggregation to form irregular large particles at high concentration of zinc ions^[42]. When the concentration of zinc ions is 0.05 wt%,

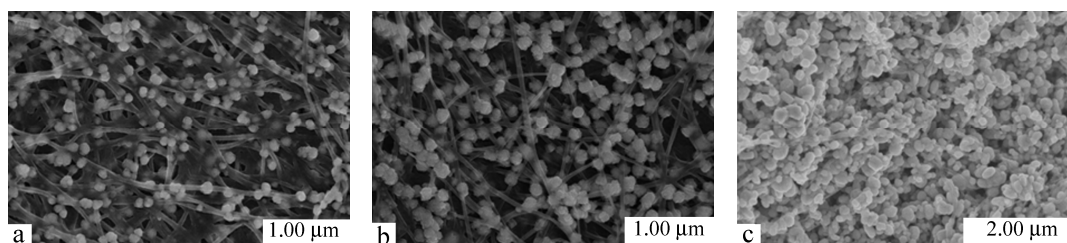


Fig. 3 FE-SEM images of ZnO nanoparticles synthesized at different concentrations of zinc acetate: (a) 0.05 wt%, (b) 0.1 wt% and (c) 1 wt%

the obtained ZnO nanoparticles with a uniform distribution and an average diameter of 100 nm are anchored discretely onto the Am-BC nanofibers.

XRD Analysis

The crystallographic natures of the nanocomposites were characterized by XRD to confirm the formation of ZnO phase. Figure 4 shows the X-ray diffraction patterns of the ZnO/Am-BC nanocomposites synthesized at different Zn^{2+} concentrations. The broad diffraction peaks at 14.6° , 16.8° and 22.8° are assigned to the nature of Am-BC^[26, 30]. The other characteristic major peaks at 31.7° , 34.4° , 36.2° , 47.5° and 56.5° correspond to the (100), (002), (101), (102) and (110) crystal planes of wurtzite structure ZnO (JCPDS, 36-1451), respectively. When the concentration of zinc ions increases from 0.05 wt% to 1 wt%, the diffraction of ZnO becomes more and more strong, which indicates that the crystal gradually tend to be perfect. No peaks from any other impurities such as $\text{Zn}(\text{OH})_2$ are observed which indicates the high purity and crystallinity of the obtained samples. The average crystal sizes of ZnO nanoparticles obtained with different concentrations of zinc ions at 0.05 wt%, 0.1 wt% and 1 wt% are estimated to be about 19.1, 20.4 and 30.5 nm by applying the Debye–Sherrer formula at (100) crystal plane.

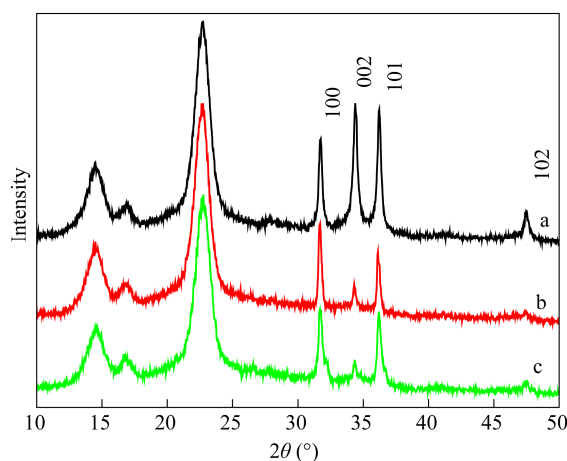


Fig. 4 XRD patterns of ZnO/Am-BC synthesized at different concentrations of zinc acetate: (a) 1 wt%, (b) 0.1 wt% and (c) 0.05 wt%

FTIR Analysis

To reveal the reactive site during hydrolytic process and evaluate the interaction between ZnO nanoparticles and Am-BC matrix, the FTIR spectra of BC, Am-BC and ZnO/Am-BC nanocomposite samples were analyzed as shown in Fig. 5. It can be seen that after the formation of ZnO nanoparticles, the peaks at 928 cm^{-1} and 1657 cm^{-1} corresponding to the stretching vibration of $\text{C}=\text{N}$ and $\text{N}-\text{OH}$ bonds are weakened and move slightly to lower wavenumbers of 917 cm^{-1} and 1630 cm^{-1} , respectively. The stretching vibrations of hydroxyl groups of Am-BC at 3400 cm^{-1} are shifted to 3425 cm^{-1} in the spectrum of the nanocomposite. This phenomenon suggested that there is probably a strong interaction between the hydroxyl and amino groups of Am-BC and ZnO nanoparticles^[27, 33, 35, 39].

Thermal Properties

The thermal properties of Am-BC and ZnO/Am-BC nanocomposites synthesized under different concentrations of Zn^{2+} are illustrated in Fig. 6. The TG curve of ZnO/Am-BC nanocomposite is almost similar to that of Am-BC mats. But the introduction of ZnO nanoparticles undermined the thermal stability of Am-BC, which also indicated the interaction between Am-BC and ZnO nanoparticles. The residues at 700°C could be referred to the content of ZnO nanoparticles which are about 20.8%, 21.6%, and 22.9% for Am-BC nanocomposites synthesized with the concentration of Zn^{2+} at 0.05 wt%, 0.1 wt% and 1 wt%, respectively. The content of the ZnO nanoparticles is much higher in ZnO/Am-BC compared with that of the ZnO nanoparticles in composites

with template of the unmodified bacterial cellulose under the same condition, indicating that the Am-BC is a highly effective nanoreactor for the synthesis of nanoparticles in a high yield^[37, 39].

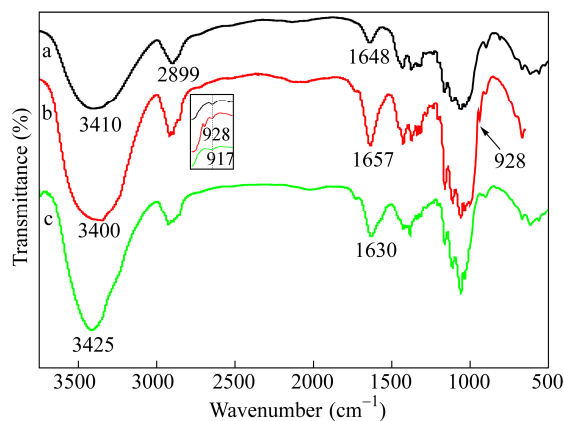


Fig. 5 The FTIR spectra of (a) BC, (b) Am-BC and (c) ZnO/Am-BC nanocomposite

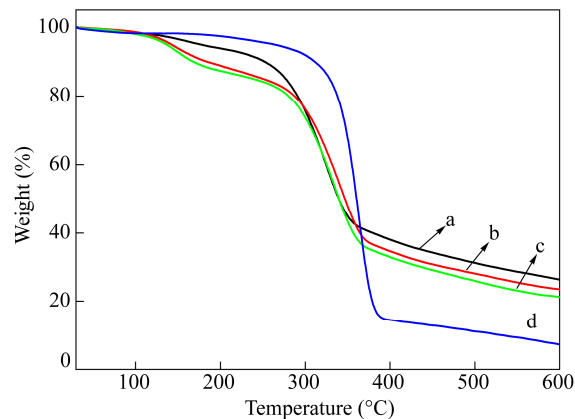


Fig. 6 TG curves of ZnO/Am-BC nanocomposites synthesized with the concentration of Zn^{2+} at (a) 1 wt%, (b) 0.1 wt%, (c) 0.05 wt% and (d) Am-BC

Photocatalytic Activity

The photocatalytic properties of ZnO/Am-BC nanocomposites obtained at different concentrations of Zn^{2+} are shown in Fig. 7 through photocatalytic degradation of organic dye MO. The nanocomposites synthesized at 0.05 wt% Zn^{2+} displays a high decolonization efficiency of 92% at 120 min, followed by that of nanocomposites synthesized at 0.1 wt% and 1 wt% Zn^{2+} . It has been demonstrated that there is no obvious difference between the composited content of ZnO nanoparticles in the Am-BC matrix under different concentrations of Zn^{2+} from TGA. The higher photocatalytic efficiency of the nanocomposites synthesized at 0.05 wt% Zn^{2+} may be attributed to the larger specific surface areas and smaller crystallite size of ZnO nanoparticles than those at 0.1 wt% and 1 wt% Zn^{2+} , which strengthen the photo asorption of solid catalyst due to increase in specific surface areas, thus greatly improving the photocatalytic efficiency. Herein, Lower Zn^{2+} concentration resulted in a positive impact on the photocatalytic properties^[39].

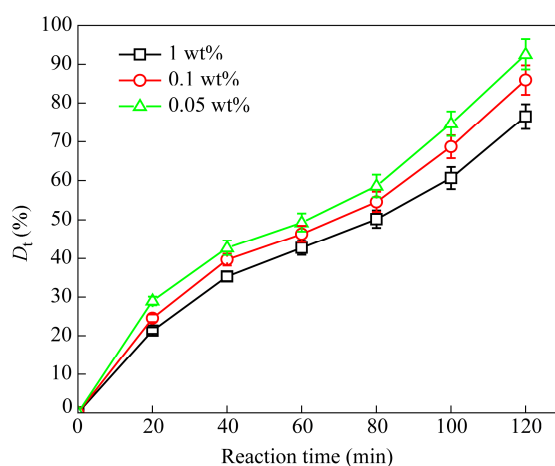


Fig. 7 Photocatalytic properties of ZnO/Am-BC nanocomposites synthesized with different concentrations of Zn^{2+}

CONCLUSIONS

In conclusion, we have successfully fabricated nanocrystalline ZnO particles with a spherical morphology and sizes around 100 nm through a simple *in situ* polyol method by using template of the prepared Am-BC. The ultrafine network architecture and the hydroxyl and amidoxime groups of Am-BC act as reactive sites for the formation of zinc oxide nanoparticles. The results suggest that Am-BC as an effective nanoreactor plays an important role in synthesis of nanoparticles in a high yield compared with that by the unmodified bacterial cellulose and prevents the ZnO nanoparticles from aggregating to guarantee the monodispersed and nanosized ZnO nanoparticles at the same time. In addition, under optimized conditions, well-dispersed and regular ZnO nanoparticles can be *in situ* assembled into Am-BC network and high photocatalytic properties can be obtained. This simple method could also be used as a facile and effective approach to prepare various metal nanoparticles.

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