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In situ growth of silver nanoparticles on TEMPO-oxidized jute fibers by microwave heating

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ABSTRACT

Cellulose fibers deposited with metallic nanoparticles as one kind of renewable, biocompatible and antimicrobial nanomaterials evoke much interest because of their versatility in various applications. Herein, for the first time, a facile, simple and rapid method was developed to fabricate TEMPO (2,2,6,6-tetramethylpiperidine-1-oxyl radical) selectively oxidized jute fibers in situ deposited with silver nanoparticles in the absence of reducing reagents. The average size of silver nanoparticles deposited on the fibers is 50.0 ± 2.0 nm by microwave heating for 5 min and 90.0 ± 4.7 nm for 10 min heating sample, respectively. The versatile jute–silver nanoparticles nanocomposites with superior thermal stability and high crystallinity would be particularly useful for applications in the public health care and biomedical fields.

1. Introduction

Antimicrobial polymer-metallic nanocomposites have received considerable attention because of their excellent antimicrobial properties and potential applications in food packaging (Moura, Mattoso, & Zucolotto, 2012), water treatment, public health care, and biomedical field (Deng, Wang, Ding, et al., 2011a, 2011b; Kim, Kwon, & Ostler, 2009). Among them, silver based nanocomposites exhibit especially strong cytotoxicity toward a broad range of micro-organisms (Atiyeh, Costagliola, Hayek, & Dibo, 2007) and win more researchers' interest (Percival et al., 2011; Wu et al., 2008).

There are many ways of reducing silver salts to nanosilver particles such as electrochemical methods (Zhu, Liao, Zhao, & Chen, 2001), hydrothermal synthesis, and UV irradiation (Omrania & Taghaviniab, 2012; Pinto et al., 2009). However, most of the synthesis protocols pose major environmental problems and wasting of resources for using the reducing and stabilizing reagents such as hydroxylamine, ascorbic acid, polyols, monosaccharide (Garcia-Barrasa, Lopez-de-Luzuriaga, & Monge, 2011), N-vinypyrrolidone (Volkov, Klechkovskaya, Shtykova, et al., 2009), even poisonous hydrazine and sodium borohydride (Van Hyning, Klemperer, & Zukoski, 2001; Yang, Xie, Deng, Bian, & Hong, 2012). Whereas, it is known that the microwave heating, which is created by the interaction of the dipole moment of molecules with the high-frequency electromagnetic radiation, provides a promising method for the preparation of metallic nanoparticles, because of its characteristics of rapid volumetric heating, short reaction time, high reaction rate, enhanced reaction selectivity, and energy savings (Liu et al., 2003).

According to the actual eco friendly criteria, cellulose, as the most abundant and renewable bioresource, is widely used as one of the most promising raw materials for the fabrication of various high performance functional materials (Ding, Wang, Wang, Yu, & Sun, 2010; Praskalo-Milanovic, Kostic, Dimitrijevic-Brankovic, & Skundric, 2010; Wang, Ding, Yu, & Wang, 2011; Wang, Niu, Jia, et al., 2011). As a typical kind of cellulose fibers, jute fibers, which are mainly cultured in some Asian developing countries (Liu, Yu, Cheng, & Qu, 2009), have attracted greater attention in the field of high value textile end uses for its superior properties including perfect air permeability, high crystallinity, high strength and so on (Liu, Wang, Xia, Yu, & Cheng, 2010; Liu, Yu, Cheng, & Yang, 2009).

Meanwhile, cellulose with carboxyl functional groups was found to be an excellent eco-friendly reducing and stabilizing template for nanosilver particles (Hebeish, El-Rafie, Abdel-Mohdy, Abdel-Halim, & Emam, 2010). TEMPO (2,2,6,6-tetramethylpiperidine-1-oxyl radical) selective oxidization has been recently proposed as one of the most promising methods

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for cellulose functionalization, where carboxyl and aldehyde functional groups converted from hydroxyl groups at the C6 position of cellulose molecules could be effectively introduced into cellulose under aqueous and mild conditions in room temperature and atmospheric pressure at pH of 10–11 (Praskalo-Milanovic et al., 2010; Saito, Shibata, Isogai, Suguri, & Sumikwa, 2005). So far, most relevant research on the synthesis of silver nanoparticles/cellulose composites is based on carboxymethyl cellulose (Hebeish et al., 2010) and TEMPO selectively oxidized bacterial cellulose (Shinsuke, Manami, Minoru, Hiroyuki, & Hiroyuki, 2009). There is no report on the preparation of TEMPO oxidized jute fibers deposited with Ag nanoparticles via environmental-friendly microwave heating.

Herein, we introduced a rapid and facile method to prepare cellulose–silver nanocomposites using silver nitrate and TEMPO selectively oxidized jute fibers without any reducing reagents by microwave heating. The silver nanoparticles were formed in situ on the surface of jute cellulose fibers. The formation of silver nanoparticles was monitored via color, whereas their evaluation was made by making use of FE-SEM.

2. Experimental

2.1. Materials

Jute bast fibers used in this study were kindly supplied by Redbud Textile Tech. Inc., China. Sodium hydroxide (NaOH), 2,2,6,6-tetramethylpiperidine-1-oxyl radical (TEMPO), dimethylsulfoxide (DMSO), sodium bromide (NaBr), 12 wt% sodium hypochlorite (NaClO) solution, silver nitrate ($AgNO_3$), ethanol and other chemicals were of laboratory grade (Shanghai Aladdin Chemical Regent Inc., China) and used without further purification.

2.2. Preparation of TEMPO oxidized jute fibers

TEMPO-oxidized jute fibers were prepared according to the literature (Cao, Ding, Yu, & Al-Deyab, 2012; Ma, Burger, Hsiao, & Chu, 2011). 2 g jute fibers first soaked by 15 wt% NaOH at 60 °C for 4 h, followed by DMSO (liquor ratio 1:15) at 70 °C for another 4 h. After each treatment, jute fibers were fully washed with distilled water and dried. The aforementioned jute sample (2 g) was dispersed in the solution of distilled water (190 g), NaBr (0.20 g), and TEMPO (0.02 g). The reaction was started by the addition of 12 wt% NaClO solution (18 g) under stirring. The pH was kept at 10.5, monitored with a pH meter, by adjusting with 2 wt% NaOH aqueous solution. The reaction was quenched by adding 5 mL ethanol while no NaOH consumption, followed by stirring for another 20 min. The final product was washed with deionized water by successive centrifugations (5000 rpm for 10 min) until neutral. The sample was then dried at 70 °C overnight.

2.3. In situ growth of silver nanoparticles onto the jute fibers

The oxidized jute sample was immersed in 0.015 M $AgNO_3$ solution and kept in dark at room temperature overnight, followed by a thorough rinsing with deionized water. Finally, the suspension contained oxidized jute and $AgNO_3$ was put into the microwave oven (Galanz R6 series, Guangdong, China) for 5 and 10 min.

2.4. Field-emission scanning electron microraph (FE-SEM)

The morphology of jute fibers and jute fibers with silver nanoparticles were examined using a field emission scanning electron microscopy (FE-SEM) (S-4800, Hitachi Ltd., Japan).

2.5. Fourier transform infrared (FTIR) spectroscopy

The FTIR spectra of all samples were recorded on an attenuated total reflection Fourier transform infrared instrument (Nicolet Nexus 670, Thermo Fisher, USA) with smart iTR module in the range of 400–4000 cm^{-1} with a resolution of 4 cm^{-1}.

2.6. X-ray diffraction (XRD) measurement

In order to investigate the crystallinity of untreated jute, TEMPO oxidized jute, and TEMPO oxidized jute with silver nanoparticles, the milled sample powders were analyzed at ambient temperature by step scanning on a X-ray diffractometer (D/max-2550 PC, Rigaku, Japan) using a monochromatic Cu K_α radiation ($\lambda = 1.5406$ Å) in the range of $2\theta = 6–60°$ with a step size of 0.04° and a scanning rate of 5.0°/min. MDI Jade 5.0 software was used to calculate the crystallinity of each sample.

2.7. Thermal gravimetric analysis (TGA)

Thermogravimetric analyses were performed using a TA Instruments (TG209 F1, NETZSCH, Germany). In each typical experiment, about 5 mg of samples was placed in a clean platinum pan and heated from 20 to 600 °C under nitrogen atmosphere at a rate of 10 °C/min.

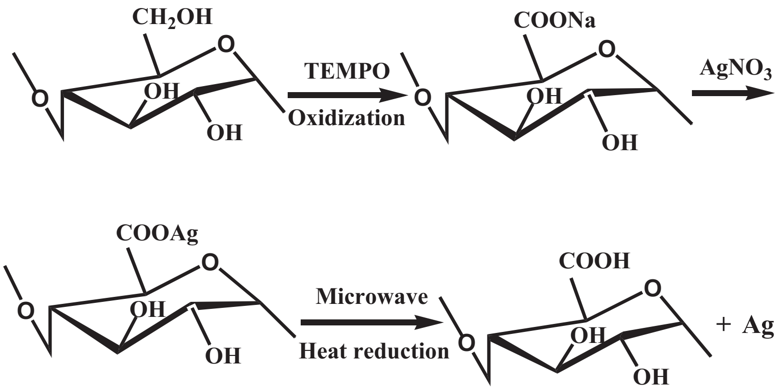

Fig. 1. Scheme for in situ growth of silver nanoparticles on jute fibers.

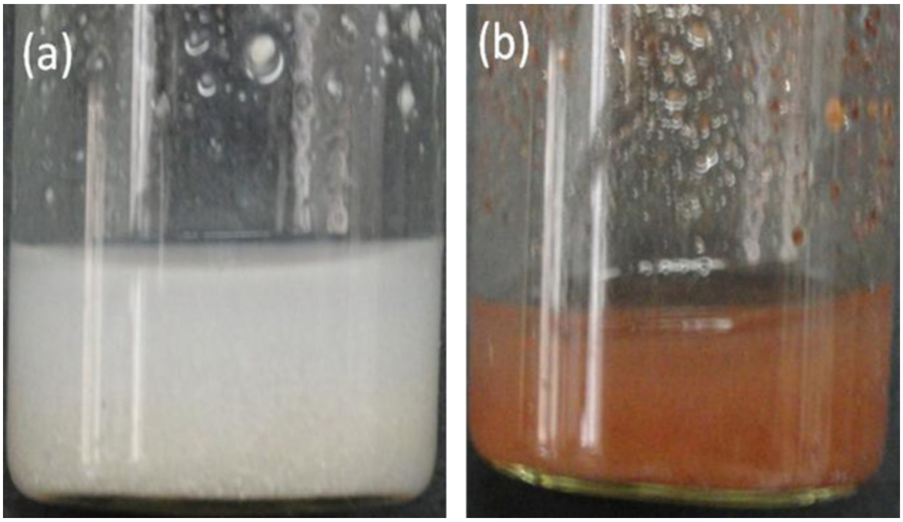

Fig. 2. Photograph of $AgNO_3$ mixed oxidized jute suspension before (a) and after (b) microwave heating for 10 min.

3. Results and discussion

3.1. Morphology and particle size distribution of silver nanopartilcles on jute fibers

Fig. 1 shows the scheme for in situ growth of Ag nanoparticles on the TEMPO oxidized jute fibers. The wet TEMPO oxidized jute fibers were immersed in the silver nitrate solution and left in dark overnight, then followed by microwave heating. The results showed that the Ag nanoparticles in situ grown onto the jute fibers after microwave heating, yielding a deep brown colored material. Fig. 2 shows a photograph of the jute cellulose suspension obtained after mixed with silver nitrate overnight and then microwave heated for 10 min (Fig. 2b) together with a photograph of jute cellulose suspension without microwave heating for contrast (Fig. 2a).

The morphology of the control jute, TEMPO oxidized jute fibers and TEMPO oxidized jute fibers deposited with silver nanoparticles were investigated by FE-SEM. Fig. 3 shows the FE-SEM micrographs of the control jute and its derivatives; and the size distributions of silver nanoparticles on the jute fibers with different microwave heating times. There are no significant differences in the morphology of jute control and TEMPO oxidized jute (Fig. 3a and b). As can be seen from the micrographs of the jute–silver nanocomposites (Fig. 3c and e), silver nanoparticles were homogeneously dispersed on the oxidized jute cellulose substrate. The size and size distributions of silver nanoparticles analyzed based on the FE-SEM images illustrated that the silver nanoparticles exhibited a small size and a narrow size distribution. As the microwave heating time increased from 5 to 10 min, the average diameter of silver nanoparticles increased from 50.0 ± 2.0 (Fig. 3d) to 90.0 ± 4.7 nm (Fig. 3f). This result confirmed that the TEMPO oxidized jute fibers were indeed a pattern of suitable template matrix for the synthesis of metal nanoparticles.

3.2. FTIR spectra of jute fibers and its derivative

FTIR–ATR spectra of jute fibers and its derivative were shown in Fig. 4. It could be seen that the original jute fibers have no obvious absorption band from 1500 to 2000 cm^{-1} (Fig. 4a). In Fig. 4b, an absorption band at 1594 cm^{-1} attributing to the carbonyl groups presented in response to the TEMPO oxidation of jute fibers, indicating that hydroxyl groups at the C6 position of jute cellulose molecules were converted to sodium carboxylate (Sbiai, Kaddami, Sautereau, Maazouz, & Fleury, 2011). After treatment with $AgNO_3$ aqueous solution, the C═O vibrations of the carboxylate anion were slightly shifted from 1594 to 1590 cm^{-1}, suggesting that sodium salt was changed to silver salt (Fig. 4c). Microwave heating of Cell-Ag^+ was carried out in a domestic microwave oven. The peak due to the C═O vibrations of carboxylate anion at 1590 cm^{-1} shift to 1604 cm^{-1} as shown in Fig. 4d, which indicates that silver ions of Cell-Ag^+ were reduced to zero valence metals during microwave treatment on jute fibers (Shinsuke et al., 2009). Thus, these spectra have indicated that the sodium salts were fully replaced by silver ions and the silver ions were completely reduced by thermal treatment in the microwave oven for 10 min.

3.3. XRD analysis

The phases and crystallinity of the jute fibers and the oxidized jute cellulose fibers deposited with silver nanoparticles were characterized by XRD. Fig. 5 shows the XRD patterns of the control jute and TEMPO-jute fibers. It could be seen that the XRD pattern of the control jute fibers exhibits a sharp high peak at $2\theta = 22.5^\circ$, and two overlapped weaker diffraction peaks respectively at $2\theta = 14.8^\circ$ and 16.4°, which are assigned to cellulose I. However, for the TEMPO oxidized treated jute, two additional diffraction peaks appear at $2\theta = 12.2^\circ$ and 20.2°, which are assigned to cellulose II. This lattice transformation is the result of 15 wt% NaOH treatment (Liu & Hu, 2008).

Fig. 6 showed the XRD patterns of the typical samples prepared using TEMPO-jute cellulose fibers and $AgNO_3$ by microwave heating for 5 and 10 min, respectively. The samples have also shown the diffraction peaks around $2\theta = 12.2^\circ$ and 20.2° corresponding to the typical diffraction patterns of crystalline cellulose type II. Additionally, we can seen that the diffraction peaks at $2\theta = 31.3^\circ$, 38.2° and 44.5° are attributed to the (1 1 1), (2 0 0) and (2 2 0) planes of the crystallized silver with a cubic structure, respectively, suggesting the successful reduction of silver by cellulose–Na. These results have indicated that all the samples are comprised of the mixed phase of the jute cellulose and silver. However, slight differences are observed among the XRD patterns. According to the JUDE calculating, the crystallinity decreased from 73.10% to 71.12% with the increasing microwave heating time from 5 to 10 min.

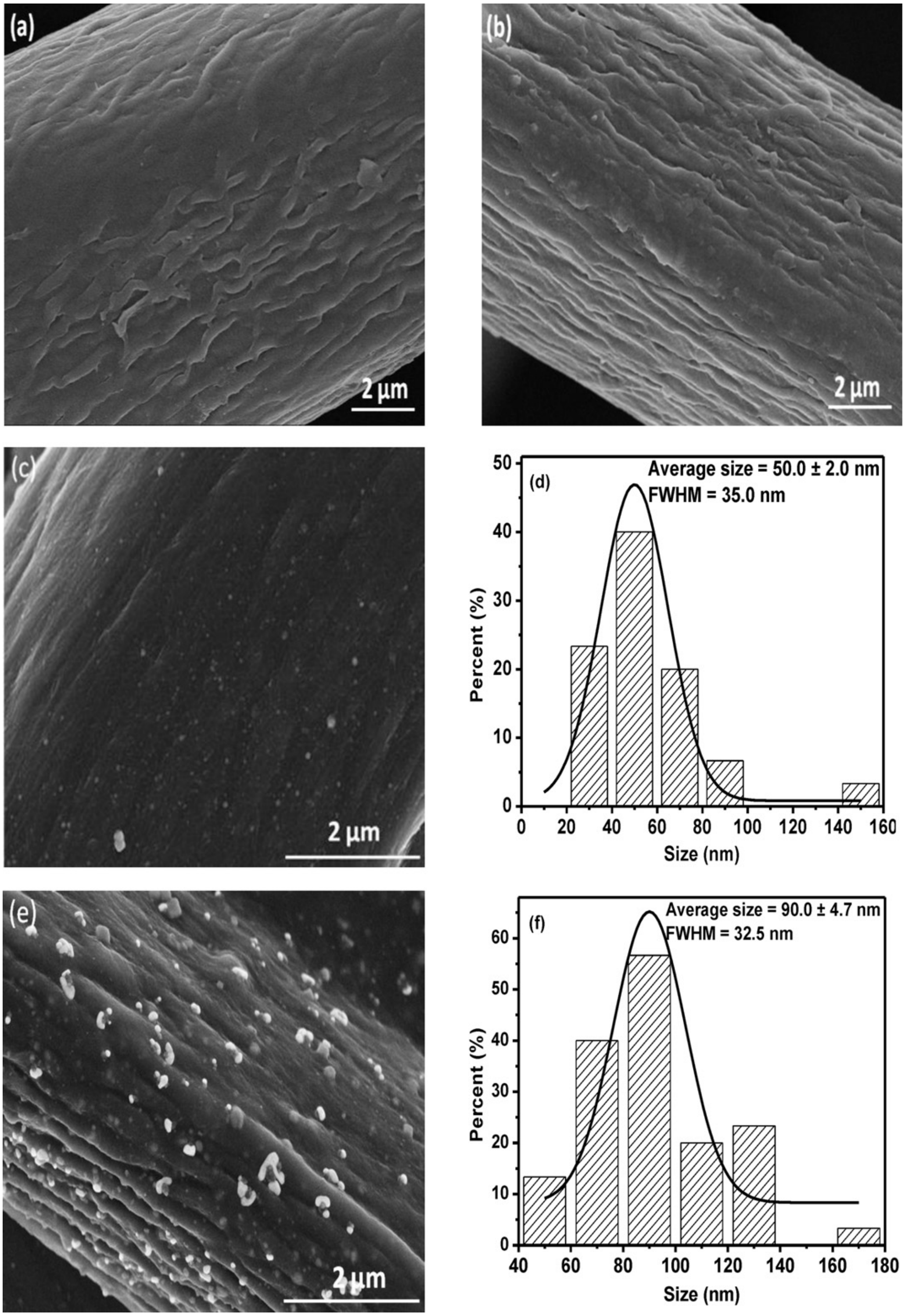

Fig. 3. FE-SEM images of control jute fibers (a), TEMPO oxidized jute fibers (b), jute fibers with $AgNO_3$ after microwave heating for 5 min (c) and 10 min (e); and particle size distributions of nanosilver on the jute fiber after microwave heating for 5 min (d) and 10 min (f), respectively.

3.4. Thermal stability

Thermal stability was determined from the TG and DTG curves (Fig. 7), showing the effects of chemical reagents and microwave heating time on the thermal behavior and interactions taking place in the jute fibers and its silver nanocomposites, as described below in a nitrogen atmosphere at a heating rate of 10°C/min. All the TG curves (Fig. 7A) showed an initial small drop between 50 and 150 °C, which corresponded to a weight loss of approximately 5% absorbed moisture on the surfaces of all samples including chemisorbed water and/or the intermolecularly H-bonded water. Thermal degradation of the TEMPO-oxidized jute fibers, and jute with silver nanoparticles samples started at approximately 200 °C in a N_2 atmosphere, while for the untreated jute, degradation began at approximately 270 °C. Thus, the formation of metallic carboxylate groups from the C6 primary hydroxyls of cellulose microfibril

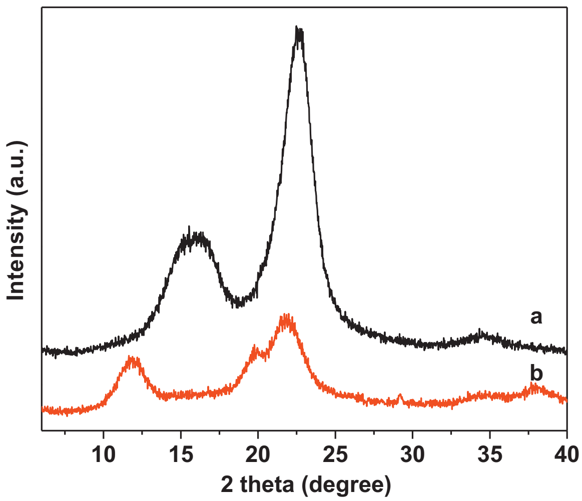

Fig. 4. XRD patterns of control jute fibers (a) and TEMPO oxidized jute fibers (b).

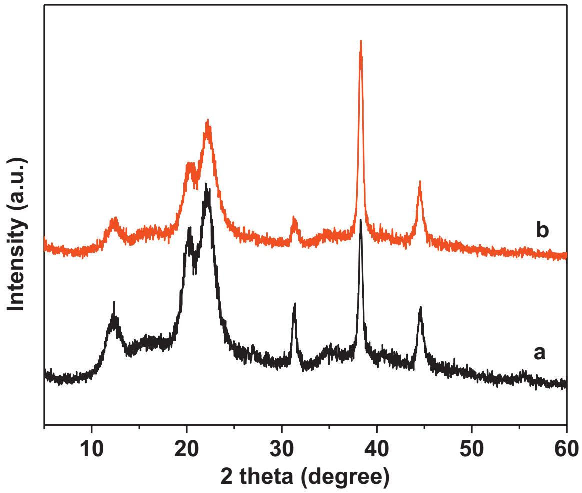

Fig. 5. XRD patterns of $AgNO_3$/TEMPO oxidized jute fibers after microwave heating for 5 min (a) and 10 min (b).

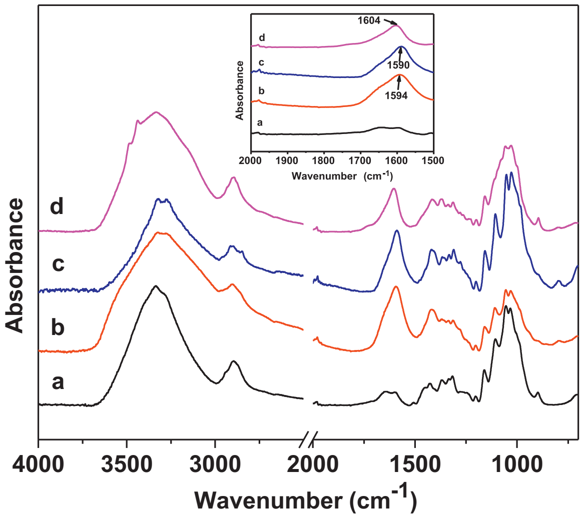

Fig. 6. FTIR spectra of jute control (a), TEMPO oxidized jute fibers (b), jute fibers with silver nitrate (c) and jute fibers with silver nanoparticles after microwave heating for10 min (d), respectively; inset is the enlarged spectra in the wave number range from 1500 to 2000 cm^{-1}.

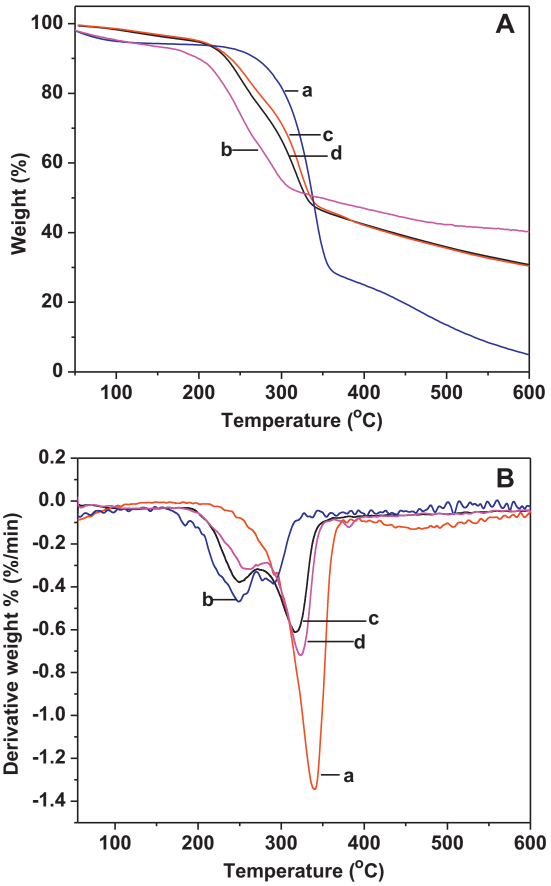

Fig. 7. TG and DTG curves of the jute control (a), TEMPO oxidized jute fibers (b), TEMPO oxidized jute fibers with 0.015 M $AgNO_3$ by different microwave heating times: 5 min (c) and 10 min (d), respectively.

surfaces by TEMPO-mediated oxidation leads to a significant decrease in the thermal degradation point (Fukuzumi, Saito, Okita, & Isogai, 2010).

One interesting thing to note is that the DTG curves of the TEMPO-oxidized jute and jute deposited with silver nanoparticles by microwave heating for 5 and 10 min (Fig. 7B) are broad, and seem to consist of two peaks (249 and 290 °C), (248 and 318 °C) and (255 and 320 °C), respectively. The former one was probably due to thermal degradation point of the metallic anhydroglucuronate units; the latter one was obviously lower than that of the control jute fibers (338.8 °C), indicating that the crystalline cellulose chains decreased in DTG peaks by the presence of thermally more unstable anhydroglucuronate units in the TEMPO-oxidized jute fibers and jute fibers deposited with silver samples.

4. Conclusions

In summary, silver nanoarticles in situ grows on the TEMPO selectively oxidized jute cellulose fibers template by microwave heating for several minutes in the absence of any reducing reagent. Additionally, the versatile jute cellulose fibers/silver nanoparticles composites with carboxylate functional groups, high crystallinity, good thermal stability and high surface areas, would be particularly useful for the applications such as green nanocomposites, ultrafiltration, antibacterial dressing, food package, water treatment, and biomedical field.

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