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Graft copolymerization of methyl methacrylate onto bamboo cellulose under microwave irradiation

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ABSTRACT

Grafting of methyl methacrylate (MMA) onto bamboo cellulose using ceric ammonium nitrate (CAN) as an initiator was carried out under microwave irradiation. The graft copolymers were characterized by Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), thermogravimetric analysis (TGA), and scanning electron microscopy (SEM). The effects of varying the microwave power, exposure time and concentration of initiator of graft polymerization were studied by determining the grafting parameters, such as grafting percentage and grafting efficiency. The experimental results showed that the optimal conditions for grafting were: [CAN] = 0.004 mol/L, which was acidified with 0.1 mol/L nitric acid; exposure time, 9 min; and the microwave power, 160 W. Moreover, the moisture absorption ability of the graft copolymers decreased significantly with the increase in grafting percentage.

1. Introduction

Bamboo is a natural composite and is common in Asia such as China, Thailand and Vietnam. Bamboo sticks are strong, lightweight, flexible, economical and handy to build. It is widely used in the building and construction industry for repairing, decoration, sign erection and slope maintenance works (Lo, Cui, Tang, & Leung, 2008). With the continuous growth in the construction and building industry, a large quantity of construction wastes is generated each year. More than 50,000 tonnes of bamboo scaffolding is disposed to the landfills as construction waste each year (Chan, Cheung, Allen, & McKay, 2009; Choy, Barford, & McKay, 2005). Therefore, recycling and reusing bamboo waste are the important environmental tasks that the world is facing, to reduce environmental loading and promote the most effective use of resources. In this paper, bamboo cellulose was made from bamboo waste. The cellulose is obtained from bamboo waste via the acidification process using hydrochloric acid or nitric acid, and then digested with sodium hydroxide (Mo, Zhao, Chen, Niu, & Shi, 2009). In this way, the environment was protected and the waste was avoided.

Natural cellulose has numerous advantages, such as low cost, low density, high toughness, biodegradability, and nontoxicity. It may be used as a packaging material, natural cellulose-reinforced composites, and other industrial applications (Kaith & Kalia, 2008). However, the cellulose use in thermoplastic matrices has been limited by the incompatibility between the cellulose and the matrix. It is known that the hydrophilic nature of cellulose prevents adhesion to hydrophobic thermoplastic matrices. Recently there has been a growing interest in chemical modification of cellulose to improve its biocompatibility and widen its applications (Dong et al., 2008; Lee & Wang, 2006). Graft copolymerization is among the widely used chemical modification methods for cellulose in order to improve its properties. This may be achieved by treating cellulose with an ester agent, and other agent, or grafting various monomers onto cellulose. In recent years, a number of monomers grafted onto cellulose by copolymerization are as follows: ethyl acrylate (Princi et al., 2006); styrene (Hassanpour, 1999); allyl-dimethylhydantion (Liu & Sun, 2008).

Heating at certain temperature is required during the graft copolymerization, which usually takes a long time and consumes much energy using the conventional thermal method. Microwave (MW) heating is a potentially attractive technique as it provides a volumetric heating process at improved heating efficiencies as compared with conventional techniques (Appleton, Colder, Kingman, Lowndes, & Read, 2005). Currently the graft copolymerization has been successfully conducted under microwave radiation. Grafting of polyaniline onto gum acacia (Tiwari & Singh, 2008) and grafting of acrylic acid onto chitosan (Ge, Wan, & Luo, 2006) have involved the use of MW irradiation. Polyacrylamide was graft copolymerized onto chitosan using MWR and maximum grafting 169% was observed in 1.16 min, under optimum reaction conditions (Singh, Tiwari, Tripathi, & Sanghi, 2006).

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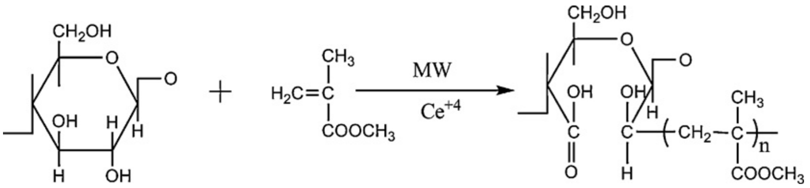

Scheme 1. Synthesis procedure of cellulose graft copolymers.

In this study, the graft polymerization of MMA onto bamboo cellulose using CAN as initiator under microwave irradiation has been investigated. The effects of change in reaction variables on grafting percentage and grafting efficiency have been studied. The graft copolymers are characterized by FTIR, XRD, TGA and SEM. Moreover, we have investigated the moisture absorption ability of the graft copolymers.

2. Experimental

2.1. Materials

Bamboo cellulose (BC) with a degree of polymerization (DP) of 2000, α-cellulose content: >85% and the size of 74 μm, was provided by Research Institute of Comprehensive Utilization of Biomaterials, Huazhong Agricultural University, China. It was purified through soxhlet extraction in acetone for 48 h, was dried at 120 °C for 5 h; methyl methacrylate (Yongda Co., Ltd., Tianjin, China) was freed from inhibitor, dried over anhydrous $CaCl_2$, and stored in a refrigerator. Ceric ammonium nitrate (CAN) (Kermel Co., Ltd., Tianjin, China) was dried at 120 °C for 5 h. The CAN solution was acidified with 0.1 mol/L HNO_3. All other chemicals and solvents were of analytical grade and used without further purification.

2.2. Graft copolymerization

The BC-g-MMA copolymer was prepared using methyl methacrylate (MMA) as a monomer and ceric ammonium nitrate as an initiator under microwave irradiation as shown in Scheme 1. The microwave-assisted graft copolymerization was carried out in a 2.45-GHz microwave oven (Galanz, Model No: WP800PS, 800 W). The typical procedure was described as follows: bamboo cellulose (1 g) was placed in a 250 mL triangular flask. Distilled water (50 mL) was added and stirred to disperse the cellulose. A predetermined amount of CAN was added to the reaction mixture, followed by the monomer with constant stirring. To remove the dissolved oxygen from the solution, nitrogen purging was carried out for 10 min. The triangular flask was sealed with preservative film. Thereafter the triangular flask was immersed into an water bath (100 mL water was placed in a 1000 mL glass beaker) which was then placed on the turntable of the microwave oven, and heated by the microwave irradiation for 3 min and then the triangular flask was cooled in an ice bath to stop the reaction for 3 min; after that, the above was repeated for several times. The total heating time was calculated as 3 × *N* min, *N* being the number of repetitions. After the stipulated time period, the reaction mixture was immediately filtered, washed by distilled water. The residue was dried in vacuum oven at 70 °C to constant weight. Then the dry residue was refluxed in a soxhlet apparatus using acetone as a solvent for 20 h. Finally, the residue was dried in a vacuum oven at 70 °C to constant weight.

The percentage and efficiency of grafting were calculated according to Kojima et al. as (Singh, Tiwari, Tripathi, & Sanghi, 2004):

$$\%\text{Grafting}\,(\%\text{G}) = \frac{(W_1 - W_0)}{W_0} \times 100 \qquad (1)$$

$$\%\text{Grafting Efficiency}\,(\%\text{GE}) = \frac{(W_1 - W_0)}{W_2} \times 100 \qquad (2)$$

where W_1, W_0 and W_2 denote, respectively, the weight of the grafted bamboo cellulose, the weight of original bamboo cellulose and weight of the monomer used.

2.3. Characterizations

2.3.1. Infrared (IR) spectral analysis

The IR spectra of the bamboo cellulose and the graft copolymers were recorded in the range of 400–4000 cm^{-1} using KBr pellets with a Nicolet (USA) Nexus 470 FTIR spectrometer for investigation of intermolecular interaction. Spectra were signal with a resolution of 4 cm^{-1} at room temperature.

2.3.2. X-ray diffraction studies

The X-ray diffraction (XRD) curves of the bamboo cellulose and graft copolymers were recorded with a Rigaku (Japan) D/max-RB X-ray diffractometer, and a Cu Kα target was used at 40 kV and 50 mA. The diffraction angle ranged from 65° to 5°. The crystallinities of the samples were calculated by Xc (%) = (Fc/(Fc+Fa)) × 100, where Fc and Fa were the areas of the crystal and non-crystalline regions, respectively.

2.3.3. Thermogravimetric analysis

Thermogravimetric analysis of the bamboo cellulose and graft copolymers was performed using a TG209 (NETZSCH, Germany) at a heating rate of 20 °C/min under nitrogen atmosphere. The temperature ranged from 30 to 600 °C.

2.3.4. Scanning electron microscopy (SEM)

The surface morphology of the bamboo cellulose and the graft copolymer was analyzed using JSM-6390/LV (NTC, Japan) scanning electron microscope.

2.4. Measurement of moisture absorption properties

The bamboo cellulose and the graft copolymers were dried in an oven at 110 °C for 4 h. Samples (1 g) were placed in the desiccators that contained water saturated with NaCl (75% relative humidity, RH) at room temperature. The moisture absorption ability (Ma) was evaluated by the percentage increase of weight:

$$\text{Ma}\,(\%) = \frac{W_i - W_0}{W_0} \times 100 \qquad (3)$$

where W_0 and W_i were the weight of samples before and after they were placed in the desiccators, respectively. Samples were consecutively tested at different time points.

3. Results and discussion

3.1. FTIR spectroscopy

Fig. 1 shows the infrared spectra (FTIR) for bamboo cellulose (A), the graft copolymers with different grafting content (B–D)

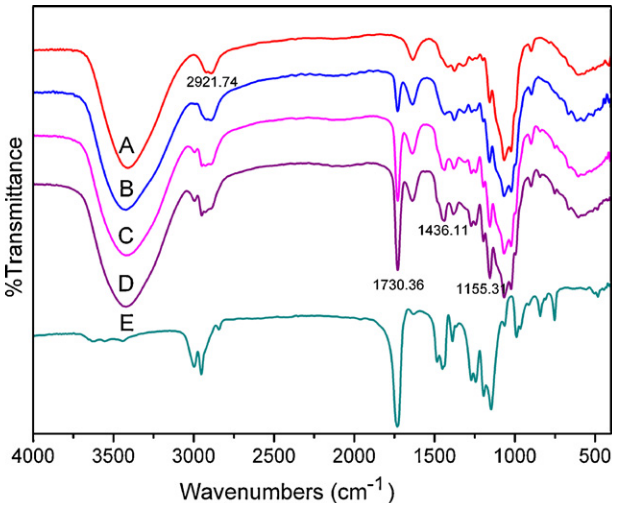

Fig. 1. Infrared spectra of (A) bamboo cellulose, (B) BC-g-MMA (%G = 15.14%), (C) BC-g-MMA (%G = 29.88%), (D) BC-g-MMA (%G = 45.46%) and (E) PMMA.

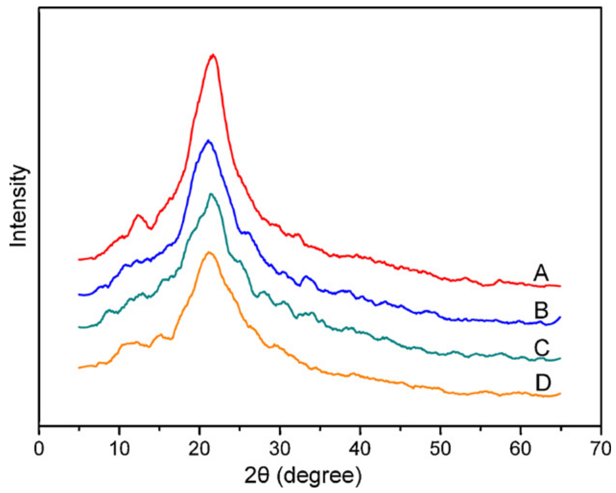

Fig. 2. XRD pattern of (A) bamboo cellulose, (B) BC-g-MMA (%G = 15.14%), (C) BC-g-MMA (%G = 29.88%) and (D) BC-g-MMA (%G = 45.46%).

and PMMA (E). Bamboo cellulose spectrum showed that absorption peak at 3390.6 cm^{-1} was due to –OH stretching. The peak at 2921.7 cm^{-1} was due to C–H stretching and the absorption bands at 1436.1 and 1155.7 cm^{-1} were attributed to C–C and C–O stretching, respectively.

Compared with the spectra of bamboo cellulose, the BC-g-MMA graft copolymers exhibited the strong absorption bands at 1436 and 1155 cm^{-1}. Moreover, the new characteristic band in the spectra of bamboo cellulose grafted MMA had a strong band at 1730 cm^{-1} corresponding to the stretch vibration of carbonyl group. PMMA showed a characterized band at 1731 cm^{-1} for carbonyl group of ester stretching vibration. It could also be clearly seen that with the increase of grafting percentage, the intensity of these absorption bonds increases. This effect could be due to the amount of PMMA on the graft copolymers. These results indicated that the surface of bamboo cellulose was grafted successfully with MMA copolymer.

3.2. *XRD studies*

Fig. 2 exhibits the XRD pattern of bamboo cellulose and the graft copolymers. Compared to the diffraction patterns of original bamboo cellulose, the diffraction patterns of the grafted cellulose show a decrease of intensity and a broadening of peak at around $2\theta = 21.2^\circ$. Meanwhile, the crystallinity was found to decrease in the grafted cellulose (15.14% grafting, Xc = 0.4856; 29.88% grafting, Xc = 0.4550; 45.46% grafting, Xc = 0.4260), as compared to bamboo cellulose (Xc = 0.5006). It may be explained by the fact that the randomness of the amorphous phase in the graft copolymers was enhanced by the grafting with MMA, which gave rise to a perturbation of long-ranged spacing between the chains (Kim, Choi, Park, & Kim, 2008; Wang et al., 2009).

3.3. *Thermogravimetric behavior*

The TGA and DTG curves shown in Fig. 3 demonstrate the thermal stability of the bamboo cellulose, BC-g-MMA, and PMMA, and the results are summarized in Table 1. The DTG curve of the bamboo cellulose exhibited a peak, indicating single decomposition stage. The DTG curve of the PMMA also showed a single wider peak, indicating a single decomposition stage, as shown in Fig. 3. The initial mass loss temperature (T_i) of the PMMA was 264 °C, which was higher than that of the bamboo cellulose (208 °C), while the maximum decomposition temperature ($T_{\max1}$) of the PMMA was 403 °C, which was higher than that of the bamboo cellulose (331 °C), as shown in Table 1. The degradation behavior of the grafted cellulose was located in between that of the bamboo cellulose and the PMMA. The grafted cellulose (Table 1 and Fig. 3) exhibited a two-stage decomposition behavior. They may be attributed to bamboo cellulose at low temperature range and grafted MMA

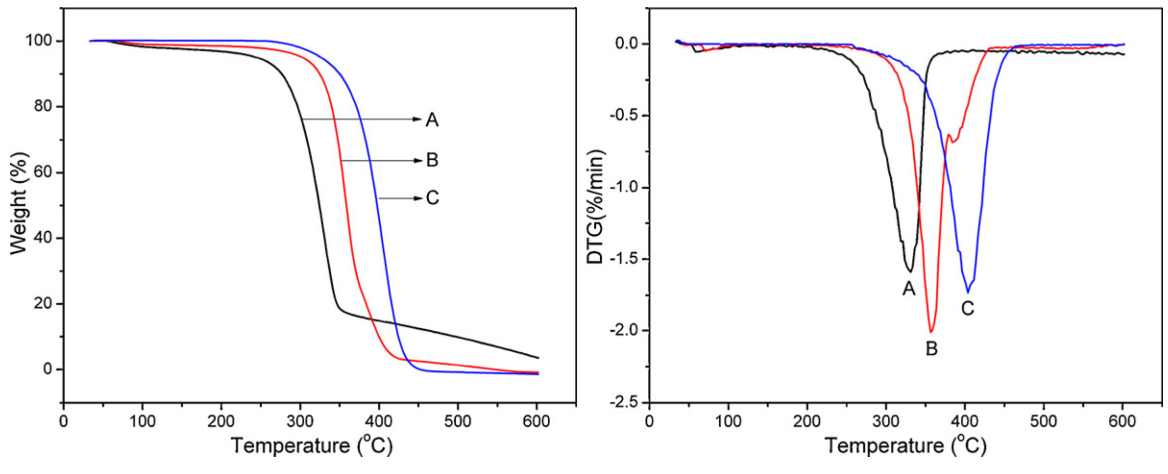

Fig. 3. TGA and DTG curves of (A) bamboo cellulose, (B) BC-g-MMA (%G = 29.88%) and (C) PMMA.

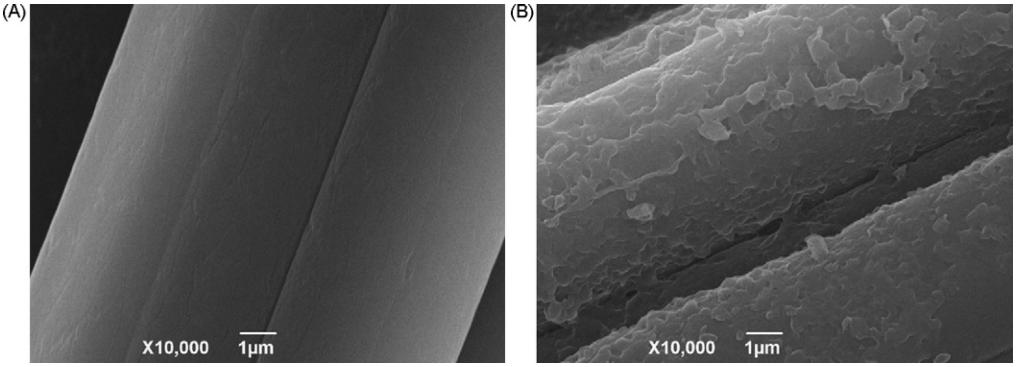

Fig. 4. SEM photographs of (A) bamboo cellulose and (B) BC-g-MMA (%G = 29.88%).

decomposition at high temperatures, because these two stages occurred at the similar temperature ranges for cellulose and PMMA, respectively.

From Table 1, it could be seen that the 20%-weight-loss temperature ($T_{20\%}$) and 50%-weight-loss temperature ($T_{50\%}$) as well as the maximum decomposition temperature ($T_{\max1}$) of BC-g-MMA were higher than those of bamboo cellulose. The possible reason was that the stereo structure of BC-g-MMA was more incompact than that of bamboo cellulose and the carbonization was less (Wang, Tan, Dong, & Shao, 2006).

3.4. SEM studies

Morphological studies of bamboo cellulose and grafted bamboo cellulose were performed by scanning electron microscopy, as shown in Fig. 4. The smoothness and evenness of the cellulose surface were observed in the micrograph of original cellulose. The change in surface morphology after grafting with MMA was clearly revealed by the SEM micrograph as grafting affected the surface of bamboo cellulose. The unevenness of surface resulted from deposition of polymer, which formed during graft copolymerization with MMA.

3.5. Grafting optimization

The effects of initiator concentration, microwave power and exposure time on the graft copolymerization reactions were estimated and the optimum conditions obtained for graft copolymerization were mentioned and discussed in subsequent sections.

3.5.1. Effect of initiator concentration

The effect of the initiator concentration on the graft reaction was studied. Graft copolymerization was carried out at various initiator concentrations ranging from 0.001 to 0.005 mol/L, keeping the other reaction condition constant: methyl methacrylate, 1 mL; microwave power, 160 W; reaction time, 9 min. As shown in Fig. 5a, with the increase of the initiator concentration, %G and %GE had an increase at first, and then decreased. The maximum grafting ratio was achieved at around 0.004 mol/L of the initiator. The initial increase of the %G and %GE at a low CAN concentration can be attributed to the formation of more reaction sites on the cellulose backbone followed by the graft copolymerization of monomers (Sang & Xiao, 2009). The decrease in %G and %GE may be rationalized as follows: (i) the termination of the growing grafted chains; (ii) the initiation of homopolymerization by excess ceric ions (Athawale & Lele, 1998).

3.5.2. Effect of microwave power

Graft copolymerization was studied at various microwave powers ranging from 80 to 400 W, keeping the other reaction condition constant: methyl methacrylate, 1 mL; initiator concentration, 0.004 mol/L; reaction time, 9 min. Fig. 5b exhibits the effect of microwave power on the grafting of MMA onto bamboo cellulose. %G and %GE both increased initially with the increasing microwave power up to 160 W. This behavior could be explained on the basis that as the power was increased, more macro radicals were generated, which resulted in the increase of grafting percentage (Kaith, Jindal, Jana, & Maiti, 2009). At microwave power more than 160 W, decrease in %G and %GE may be attributed to more homopolymerization at high microwave power or to some decomposition of the graft copolymer.

3.5.3. Effect of microwave exposure time

Graft copolymerization was studied at various reaction times ranging from 3 to 15 min, keeping the other reaction condition constant: initiator concentration, 0.004 mol/L; methyl methacrylate, 1 mL; microwave power, 160 W. Fig. 5c shows the effect of microwave power on %G and %GE. With the increase of the exposure time, %G and %GE had an increase at first, reached maximum value in 9 min, and then decreased. Increase in %G and %GE with time could be explained on the basis of increased interactions between MW, initiator, monomer and bamboo cellulose backbone resulted in more generation of free radical sites on the backbone as well as MMA chains. However, the decrease in the %G and %GE with time could be attributed to the depletion in concentration of initiator and monomer as well as shortage of the available grafting sites (Trivedi, Kalia, Patel, & Trivedi, 2005).

3.6. Measurement of moisture absorption properties

The moisture absorption properties of bamboo cellulose and the graft copolymers are shown in Fig. 6. The weight of moisture absorbed increased gradually at first, and then became constant.

Table 1
Data of thermal decomposition of the bamboo cellulose, the graft copolymer (%G = 29.88%) and PMMA.

Sample	T_i (°C)	$T_{20\%}$ (°C)	$T_{50\%}$ (°C)	$T_{\max1}$ (°C)	$T_{\max2}$ (°C)
Bamboo cellulose	208	296	323	331	–
The graft copolymer	251	336	356	353	384
PMMA	264	365	393	403	–

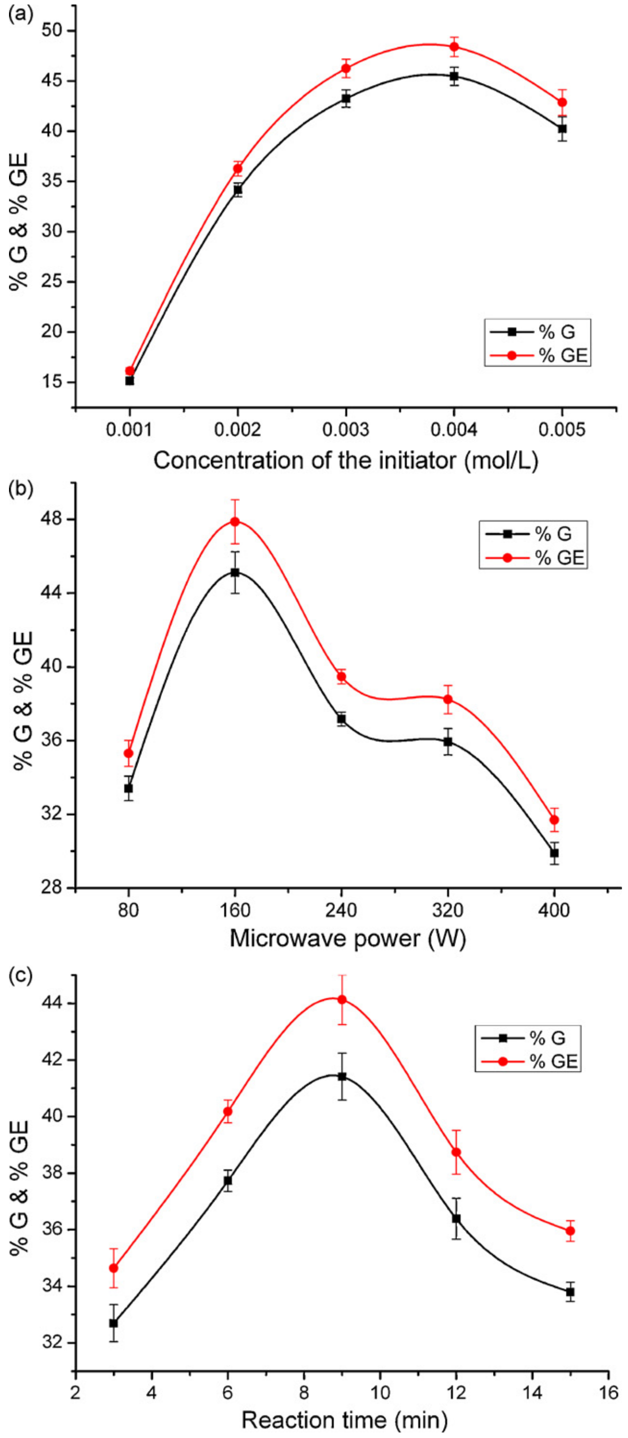

Fig. 5. (a) Effect of initiator (ceric ammonium nitrate) concentration on the grafting percentage (%G) and grafting efficiency (%GE). Reaction conditions: methyl methacrylate, 1 mL; microwave power, 160 W; reaction time, 9 min. (b) Effect of microwave power on grafting percentage (%G) and grafting efficiency (%GE). Reaction conditions: methyl methacrylate, 1 mL; initiator concentration, 0.004 mol/L; reaction time, 9 min. (c) Effect of reaction time on the grafting percentage (%G) and grafting efficiency (%GE). Reaction conditions: initiator concentration, 0.004 mol/L; methyl methacrylate, 1 mL; microwave power, 160 W.

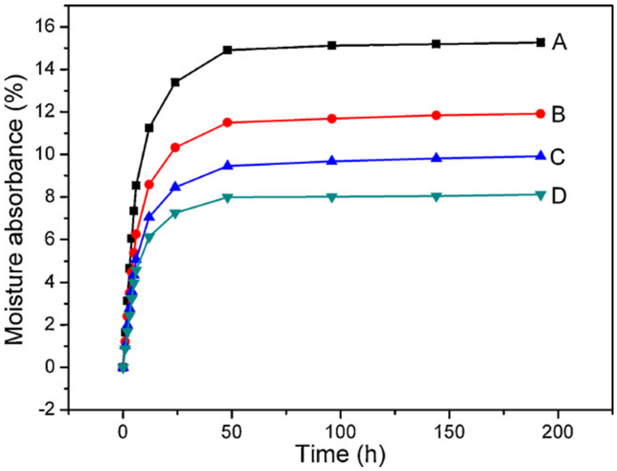

Fig. 6. Moisture absorption properties of (A) bamboo cellulose, (B) BC-g-MMA (%G = 15.14%), (C) BC-g-MMA (%G = 29.88%), (D) BC-g-MMA (%G = 45.46%).

This behavior could be explained on the basis that the water molecules were accessible to the surface of the cellulose and readily developed hydrogen-bond interactions with the cellulose molecular chains, and the moisture content approached equilibrium. The moisture absorption ability of BC-g-MMA decreased with the increase of %G. The decreasing moisture absorption may be attributed to barrier effect of the presence of hydrophobic MMA chains on grafting.

4. Conclusion

The graft copolymerization of MMA onto bamboo cellulose was successfully conducted by microwave irradiation. The %G and %GE could be adjusted by varying the reaction parameters. The optimum conditions worked out on the basis of present work were as follows: [CAN] = 0.004 mol/L; exposure time, 9 min; the microwave power, 160 W. The graft copolymer was characterized by FTIR, SEM, which elucidated the structure changes in comparison with bamboo cellulose. The crystallinity of the graft copolymers decreased in comparison with that of bamboo cellulose, and TGA revealed that copolymer had more thermal stability. Furthermore, the moisture absorption capacity of the graft copolymers decreased significantly with the increase in grafting percentage.

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