

Effect of the Utilization of Electron Beam Irradiation on the Reactivity of Bamboo Dissolving Pulp

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Electron beam irradiation (EBI) was used to improve the reactivity of bamboo dissolving pulp. An EBI treatment with a dose lower than 10 kGy showed that the Fock reactivity of the dissolving pulp noticeably increased from 69.5% to 98.3% with negligible cellulose losses. However, when the irradiation dose was higher than 10 kGy, the Fock reactivity increased with an observable α -cellulose loss, which could result in the lower strength of end-products. The gradual increase of Fock reactivity has a good agreement with the reduction of the degree of polymerization (DP) of cellulose. This suggests that lowering the DP of cellulose could enhance cellulose reactivity. Later analyses confirmed that an EBI treatment creates fiber pores that facilitate a cellulose xanthation reaction. The EBI treatment could randomly destroy cellulose crystalline and amorphous regions. The results indicated that the reactivity improvement was due not only to the DP, but also due to the changes in the fiber morphology and cellulose structure caused by the EBI processing.

Keywords: EBI; Dissolving pulp; Reactivity; Fiber pores; DP

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INTRODUCTION

Cellulose is the main biopolymer in plant cell walls and one of the most abundant renewable resources in the world (Imai *et al.* 2014; Hu *et al.* 2015; Zhang *et al.* 2015). It has superior properties of being non-toxic, biodegradable, low-weight, inexpensive, and renewable. Cellulose has a wide array of applications in various fields such as paper, medicine, food, textiles, nano-materials, *etc.* (Ma *et al.* 2011; Abdel-Halim 2014; Jonoobi *et al.* 2015; Ramamoorthy *et al.* 2015). Moreover, due to the increasing concern of the environment, cellulose and its derivatives play an important role in living a low-carbon life.

A successful way to use cellulose applicably is by manufacturing dissolving pulp that has a high α -cellulose content, thus regenerated cellulose. From dissolving pulp to regenerating cellulose, many approaches have been investigated, such as the viscose, Lycocell, and Cellca processes. Among the three, the viscose process is widely preferred and used commercially. The process is composed of alkalization, aging, xanthation, and regeneration. Even though it is preferred, the viscose industry has faced many problems from high chemical costs of sodium hydroxide (NaOH) and carbon disulfide (CS₂) to environmental pollution from CS₂.

Chemicals are significantly consumed during the viscose process due to low accessibility and reactivity of the dissolving pulp. The cellulose molecular chains are strongly bonded by intramolecular and intermolecular hydrogen bonding. Thus, the

cellulose crystalline structure significantly limits the accessibility and reactivity of the cellulose in the dissolving pulp. In recent years, physical, chemical, and biological pretreatment methods have been investigated to improve the accessibility and reactivity of dissolving pulp (Iller *et al.* 2002; Dubey *et al.* 2004; Gehmayr *et al.* 2012; Miao *et al.* 2014; Tian *et al.* 2014). During physical methods, using electron beam irradiation (EBI) can be beneficial for modifying and dissolving pulp properties without needing chemical interference when the conditions are normal (temperature and pressure) (Alberti *et al.* 2005; Shin *et al.* 2012). Doing this process can make a positive contribution to reducing production costs and increasing environmental protection (Aaserud *et al.* 1990; Stepanik *et al.* 1998). In the EBI process, the electron energy from an electron accelerator is transferred to irradiated materials, which destroys the uniform cellulose structure by either oxidation and/or chain scission (Driscoll *et al.* 2014).

Bamboo pulp, a new dissolving pulp source, has recently attracted more attention. For this study, to promote the quality and value of bamboo dissolving pulp, EBI was employed as a potential and feasible treatment approach. First, the effect of EBI on the Fock reactivity, the degree of polymerization (DP), and α -cellulose content of bamboo dissolving pulp was investigated. As a result, the changes of the cellulose molecular weight and crystallinity from EBI were determined. The fiber morphologies and surface structure were also investigated.

EXPERIMENTAL

Materials

Bamboo dissolving pulp was provided by the Sichuan Bamboo Resource Development Group located in Sichuan, China. The moisture content of the bamboo dissolving pulp was 10.3%. The NaOH and CS₂ used in the experiments were of analytical grade, and deionized water was used in all of the experiments.

Methods

Electron beam irradiation treatment

Samples of bamboo dissolving pulp were treated by using a GRDZ-10/20 electron accelerator located in Shanghai, China from Textile Union Energy Technology Co., Ltd. The dose rate was 1.3 kGy/h. The irradiation doses were 0.5 kGy, 1 kGy, 2 kGy, 5 kGy, 10 kGy, 15 kGy, 20 kGy, and 35 kGy. The accelerator was operated at 6.0 KW and 7.0 A.

Analysis of alpha-cellulose content and fock reactivity

The α -cellulose content from the samples was determined according to the TAPPI T203 cm-99 standard (1999). The Fock reactivity of each sample was measured based on improved procedures where the sodium hydroxide concentration was 9%, the carbon disulfide dosage was 1.3 mL, the xanthation temperature was 19 °C, and the xanthation time was 3 h (Tian *et al.* 2013).

Analysis of cellulose degree of polymerization

The DP and intrinsic viscosity of the samples were investigated, according to the previous literature (Ma *et al.* 2013). The sample was dissolved in cupriethylenediamine solution at 0.5% cellulose concentration to determine the intrinsic viscosity and DP.

Scanning electron microscope (SEM) observation

The determination of surface morphologies was observed by using a Nova NanoSEM 230 device (FEI, Portland, OR, USA). Prior to the SEM characterization, all of the samples were coated in gold by using a E-1010 sputter (Hitachi, Tokyo, Japan).

Analysis of fiber morphology, pore volume, and specific surface area

The fiber morphology (fiber length and fines content) was tested by a Morfi Compact fiber analyzer (Techpap, Grenoble, France). The pore volume and specific surface area of the samples were determined by using a nitrogen adsorption method that included adsorption and desorption processes. The pressure range was $0.05 < p/p^\circ < 0.30$ for the monolayer covering.

Analysis of molecular weight distribution

The molecular weight distribution (MWD) of the dissolving pulp samples were determined by gel permeation chromatography (Waters, Milford, MA, USA) with waters at 400 E. The gel permeation chromatography (GPC) was equipped with a differential refractometer detector (Waters 2414) and two columns in series (Waters Styragel Column, Milford, MA, USA, HR4 DMF and HR5 DMF, 7.8*300 mm, 10- μ m particle size). Prior to the GPC analysis, an 8% LiCl/DMAc solution was used to dissolve the samples. The 0.5% LiCl/DMAc was used for the mobile phase at a flow rate of 1 mL/min. The solution was filtered with a 0.45 μ membrane and injected into the GPC system for analysis. The system ran at a column temperature of 50 °C, the injection volume was 50- μ L, with an operation time of 30 min.

Analysis of crystallinity index

The crystallinity of the control and the treatment of dissolving pulps were performed via X-ray diffraction (Philips, Amsterdam, Holland). The diffracted intensity of CuK α radiation, at 40 KV and 30 mA, was tested in a 2θ range between 5° and 90°. The crystallinity index was calculated using Segal's proposed crystallization index formula for natural cellulose fibers (Segal *et al.* 1959),

$$\text{Crystallinity Index (\%)} = 100 \times (I_{002} - I_{am}) / I_{002} \quad (1)$$

where $I_{(002)}$ is the maximum diffraction, k is the intensity of a 2θ angle at approximately 22.7° (representing crystalline areas), and $I_{(am)}$ is the peak intensity at a 2θ angle at approximately 16° (representing amorphous areas).

RESULTS AND DISCUSSION

Influence of EBI on Fock Reactivity, DP, and α -cellulose

The Fock reactivity from the EBI treatment is presented in Table 1, which shows that it could improve the Fock reactivity of bamboo dissolving pulp. For example, using 1 kGy in the EBI treatment resulted in an increase of Fock reactivity from 69.5% to 78.3%, while using 10 kGy showed a result of 98.3%. The results implied that the EBI treatment helped more cellulose molecules take part in xanthation reactions as a result of the exposure of more hydroxyl groups. Exposing more hydroxyl groups improved the accessibility of the cellulose (Iller *et al.* 2002; Dubey *et al.* 2004). The DP data shown in Table 1 confirmed

that EBI could cleavage the cellulose chain. When the DP was reduced, the Fock reactivity increased, revealing that lowering the DP in the cellulose could improve the dissolving pulp. The enhancement of the Fock reactivity may have been the result of the reduction in the DP (Engstrom *et al.* 2006). The α -cellulose content is one of the most important properties for dissolving pulp. In Table 1, the α -cellulose content of the dissolving pulp barely changed during the EBI treatment when the dose was lower than 10 kGy, while there was an evident decline that was seen when the dose exceeded 10 kGy. The α -cellulose content of the dissolving pulp decreased from 94.2% to 89.3% when the dose increased from 10 kGy to 35 kGy. This suggested that the cellulose with a low molecular weight could degrade when in an alkaline solution. As reported in a previous study, Yang *et al.* (2010) found that the α -cellulose decreased when using the EBI treatment of bamboo paper grade pulp to obtain good spinnability for a Lyocell process. To acquire higher α -cellulose content and yield, high Fock reactivity should be present, while having too low of DP or extensive degradation to mono-sugars will result in undesirable strength damage of end-product (Wang *et al.* 2015). Therefore, the EBI dose should be controlled within 10 kGy.

Table 1. Effects of EBI on Fock Reactivity, DP, and α -cellulose

EBI Dose (kGy)	0	0.5	1	2	5	10	15	20	35
Fock Reactivity (%)	69.5	73.7	78.3	89.6	95.6	98.3	99.6	99.9	100
DP	643	578	549	512	450	376	303	263	168
α -cellulose (%)	94.4	94.4	94.3	94.4	94.2	94.2	93.4	91.5	89.3

Influence of EBI on Molecular Weight of Bamboo Dissolving Pulp

The MWD values of the original dissolved pulp and the EBI-treated pulp with various doses are shown in Fig. 1. Clearly, increasing dosages in the EBI treatment shifted the MWD towards the left, especially those with a high molecular weight. The calculated data of molecular weight and polydispersity index (PDI) can be seen in Table 3. The original dissolved pulp had a M_w of 248,000 g/mol, a M_n of 55,000 g/mol, and a PDI of 4.52. It was apparent that the M_w declined drastically when the dose increased from 0 kGy to 35 kGy. The M_n decreased slightly when the dose increased from 0 kGy to 10 kGy, while it decreased when the dose exceeded to 10 kGy. Moreover, the difference in decrease rate between M_w and M_n was becoming apparent with increasing dose from 0 kGy to 10 kGy. This phenomenon may be attributed to the higher chance of degradation that occurred within long molecular chains of cellulose that had low EBI doses, which resulted in the reduction of molecular weight (Ma *et al.* 2014). After being treated with a dose of 35 kGy, the molecular weight dropped quickly with M_w 83,400 g/mol and M_n 29,100 g/mol due to the cleavage on the cellulose chains. The margin of decrease for M_w was 66.4% and M_n was 47.0%. There was a smaller difference in decrease rate between M_w and M_n when the EBI dose increased to 35 kGy. This indicated that the high and low molar components were both affected by higher doses of the EBI treatment. The authors have observed that the PDI decreased quickly from 4.52 to 2.95 due to the irradiation dose increasing to 10 kGy. A slight decline in the PDI occurred when the dose increased to 35 kGy. These results implied that a larger change of M_w occurred at 10 kGy, especially for the long-chain cellulose. Therefore, there was a favorable effect on the MWD of uniform cellulose. These observations were congruent with previous research involving the development of PDI, where the EBI treatment led to a substantial drop of the DP from 4.15 to 2.68 after 10 kGy, and to 2.12 when exposed to 50 kGy irradiation for Borregaard pulps (Iller *et al.* 2002).

Iller *et al.* reported that the reactivity of cellulose pulps improved after the EBI treatment and there was a reduction of the cellulose molecular weight.

The decrease of α -cellulose is apparent in Table 1. The results in Table 2 show that more and more fractions of low molecular weight could be soluble in alkaline solutions. The α -cellulose maintained the same level (94.4%) when the original dissolving pulp was used within 10 kGy. As for treatments up to 35 kGy, the reduction of α -cellulose was approximately 5%. Based on the changes of α -cellulose at various doses, it was found that approximately 1% to 5% of cellulose with molecular weight lower than 19,055 g/mol saw a decline from 94.4% when the EBI treatment doses ranged from 15 kGy to 35 kGy (Fig. 2.). For a similar reduction to occur between α -cellulose and molecular weight, the dissolution of cellulose with a molecular weight lower than 19,100 g/mol would decrease the α -cellulose content.

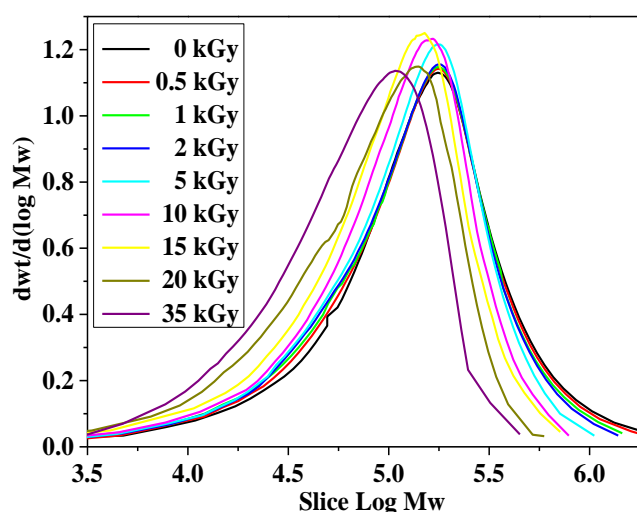


Fig. 1. The effect of EBI on molecular weight distribution of bamboo dissolving pulp

Table 2. Molecular Weight of the Bamboo Dissolving Pulp Subjected to Various EBI Dose (D)

EBI dose (kGy)	M_n (g.mol ⁻¹)	ΔM_n (%)	M_w (g.mol ⁻¹)	ΔM_w (%)	PDI
0	54798	-	247991	-	4.52
0.5	54783	0.03	222425	10.31	4.04
1	54362	0.80	207845	16.19	3.82
2	54085	1.30	188433	24.02	3.48
5	51878	5.33	165680	33.19	3.19
10	50273	8.26	148495	40.12	2.95
15	44268	19.22	125394	49.44	2.93
20	37410	31.73	109656	55.78	2.87
35	29059	46.97	83413	66.36	2.83

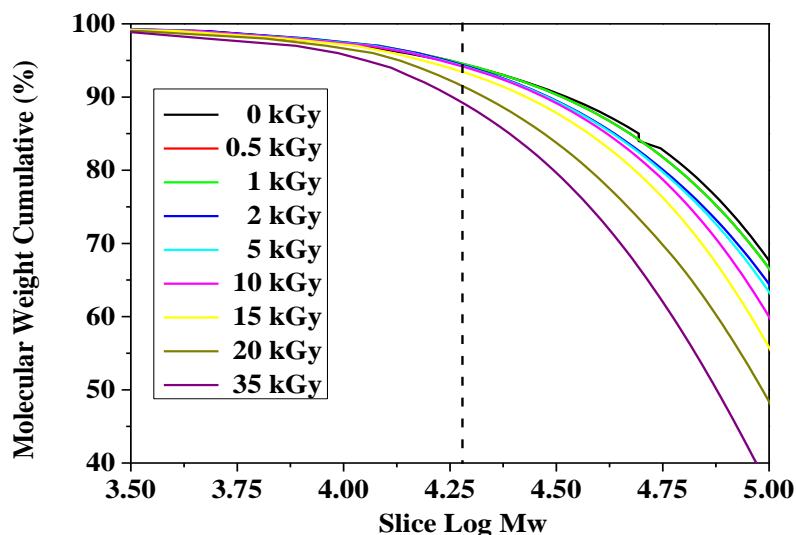


Fig. 2. Molecular weight cumulative curves of bamboo dissolving pulp subjected to various EBI dose (D)

Influence of EBI on Crystallinity Index of Bamboo Dissolving Pulp

Figure 3 shows the X-ray diffraction intensity of bamboo dissolving pulp as a function of a diffraction angle for the dissolving pulp samples and irradiated samples. The XRD spectra showed that a configuration transformation did not occur, while the XRD patterns for cellulose I were not noticeably changed. The degree of crystallinity for the bamboo cellulose decreased from 63.7% to 57.3% when the dose increased from 0 kGy to 35 kGy. These results suggested that cellulose degradation occurred in the crystalline and amorphous regions. The crystallinity index of microcrystalline cellulose (MCC) revealed a negative trend when there was an increase in the EBI dose (Alberti *et al.* 2005). Another study looked at the property changes of nanofibrillated cellulose prepared from cotton linter after the EBI treatment and found that the crystallinity index was decreased with an increased irradiation dose (Le *et al.* 2016). Decreasing the crystallinity in the cellulose made it more accessible to chemical reagents; thus making it easier to form transparent viscose (Stepanik *et al.* 1998, 2000; Kasprzyk *et al.* 2004)

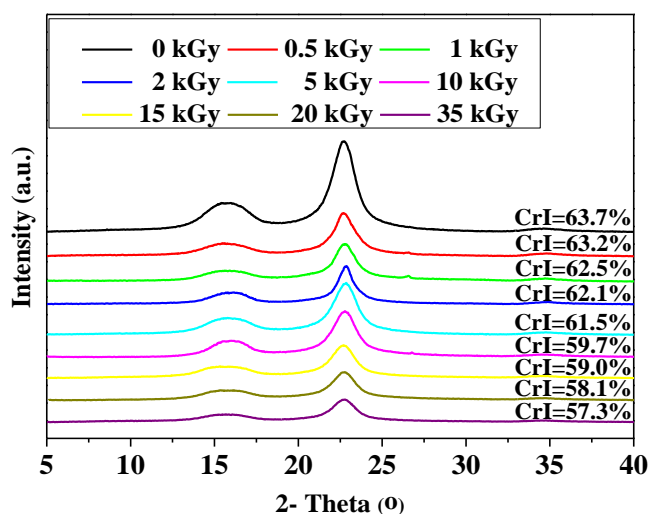


Fig. 3. XRD patterns and crystallinity index (CrI) of the bamboo dissolving pulp irradiated by a different dose

Influence of EBI on Fiber Morphology of Bamboo Dissolving Pulp

To regenerate cellulose production, the physical structure of the dissolving pulp was a decisive role in controlling the accessibility of cellulose to NaOH and CS₂ (Hubbe *et al.* 2007; Šaupperl and Stana-Kleinschek 2010; Miao *et al.* 2014).

Table 3. Fiber Morphologies of Pulps before and after the EBI

EBI Dose (kGy)	Pore Volume × 10 ⁻² (cm ³ /g)	Pore Diameter (nm)	Specific Surface Area (m ² /g)	Fines Content (%)
0	0.92	1.08	0.81	7.38
0.5	0.92	1.12	0.83	7.46
1	0.95	1.13	0.86	7.62
2	0.98	1.24	0.87	7.88
5	1.00	1.78	0.91	8.24
10	1.06	2.47	0.98	9.16
15	1.17	2.94	1.08	9.89
20	1.32	3.56	1.13	10.53
35	1.51	4.35	1.21	11.12

As noted in Table 3, the EBI treatment led to a notable increase in the total pore volume of the dissolving pulp, from 0.92 cm³/g in the original sample to 1.51 cm³/g of 35 kGy of the EBI sample. The expansion of the mean pore diameter was due to the EBI treatments of 1.08 nm to 2.47 nm with an irradiation dose of 10 kGy and the treatment of 4.35 nm with an irradiation dose of 35 kGy. The EBI treatment that increased the specific surface area from 0.81 m²/g to 1.21 m²/g with an irradiation dose of 35 kGy, increased the fines content from 7.38% to 11.12%.

The EBI treatment could increase the surface area of the MCC. Evidence of this was seen in Driscoll's study, where he observed the surface area increase from 274 m²/g to 318 m²/g when an irradiation dose of 1000 kGy was applied (Driscoll *et al.* 2009). In another study, Gehmayr and Sixta (2011) investigated the type of enzyme treatments that would be needed to upgrade paper-grade pulps to dissolving pulps. They found that the cellulosic fibers that came from the enzyme treatment exhibited improved porosity, increased pore volume and diameter, and increased the Fock reactivity (Gehmayr and Sixta 2011).

The fiber morphology of the sample after the EBI treatment was observed by SEM. Figure 4 shows that the EBI treatment improved the roughness of sample surface due to the peeling of the primary wall and exposed the S₁ layer. This was responsible for changes in the pore structure, the specific surface area, and fines content. Correspondingly, more cracks were exposed on the fiber surface during the EBI sample than when compared to the original sample.

It was concluded that the EBI treatment destroyed the fiber surface and this helped form a fuzzy fiber surface. The wrinkles and cracks reflected substantial structural changes that were caused by the irradiation (Takacs *et al.* 1999). It was concluded that the EBI treatment could noticeably modify the physical structure of the fiber and the morphologies, improve the pore volume and diameter, increase the specific surface area and surface roughness, and eventually promote the reactivity of bamboo dissolving pulp.

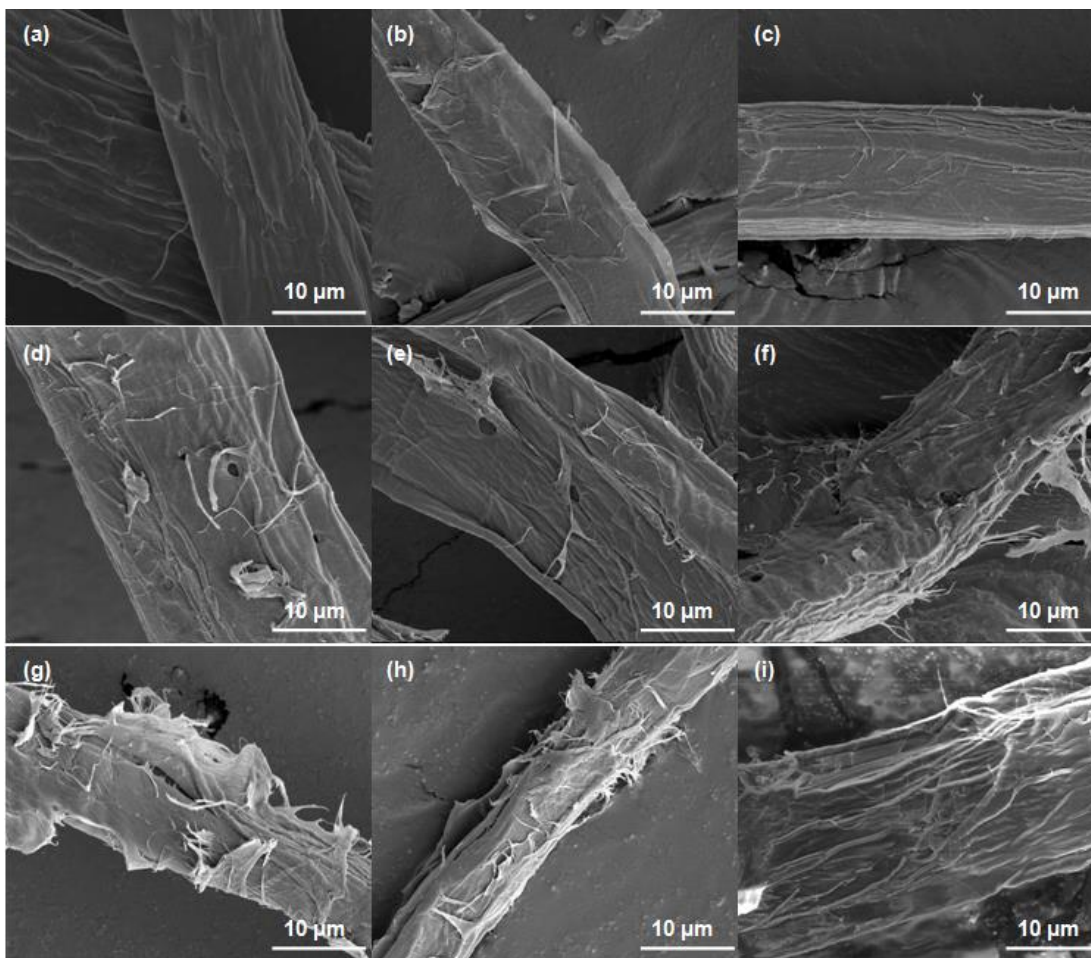


Fig. 4. Field-emission SEM micrographs of fibers; a: untreated dissolving pulp, b: treated with 0.5 kGy, c: treated with 1 kGy, d: treated with 2 kGy, e: treated with 5 kGy, f: treated with 10 kGy, g: treated with 15 kGy, h: treated with 20 kGy, i: treated with 35 kGy

CONCLUSIONS

1. Electron beam irradiation (EBI) was used to increase the reactivity of bamboo dissolving pulp. The optimal condition for the EBI treatment was an irradiation dose of 10 kGy because it resulted in the Fock reactivity increasing from 69.5% to 98.3%, with negligible cellulose loss.
2. The EBI treatment degraded and cut the cellulose molecular chains, which resulted in the drop of the molecular weight, DP, and the crystallinity index. This was favorable because it promoted the accessibility of the cellulose molecule to chemicals.
3. The EBI treatment positively affected the fiber physical structure and morphologies, improved the pore volume and diameter, and increased the specific surface area and surface roughness.

ACKNOWLEDGEMENTS

The authors acknowledge the support from National Natural Science Foundation of China, Grant No. 31500488. The authors are also grateful for the support of the Natural Science Foundation of Fujian Province, Grant No. 2016J01089.

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Article submitted: January 10, 2017; Peer review completed: May 2, 2017; Revised version received and accepted: May 17, 2017; Published: July 13, 2017.
DOI: 10.15376/biores.12.3.6251-6261