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CTMP-based cellulose fibers modified with core–shell latex for reinforcing biocomposites

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ABSTRACT

The toughening of cellulose fiber reinforced polypropylene (PP) was performed via adsorbing the cationic latex with core–shell structure onto chemithermomechanical pulp (CTMP) fibers as reinforcements, which is a novel approach for rendering the surface of cellulose fibers elastomeric. The mechanical, morphological and thermal properties of the resulting biocomposites, containing 40% (wt) of the modified fibers, were investigated. The results showed that with the increasing of the latex dosage up to 2% (wt on dry CTMP fibers), the impact, tensile and flexural strengths of the modified CTMP/PP biocomposites were significantly increased. The toughening mechanism was discussed based on the retarding of crack propagation and the promoting of crystallization of PP matrix (as revealed by DSC characterization). The overall performance of the biocomposite demonstrated that cationic latex-modified CTMP fiber is very effective in reinforcing thermoplastic-based biocomposites along with the synergetic effect on enhancing crystallinity of polymer matrix.

1. Introduction

In the past decades, polymer composites are being prepared combined with various reinforcing agents in order to improve the mechanical properties and obtain the characteristics demanded in practical application (Chatterjee, Mitchell, Hadjiev, & Krishnamoorti, 2007; Chen, Lawton, Thompson, & Liu, 2012; Diez-Pascual, Naffakh, Marco, Ellis, & Gomez-Fatou, 2012; Jin, Ko, Yang, & Bae, 2010; Zhu, Bakis, & Adair, 2012). With the exhaustion of petroleum resources, polymer industry is under pressure to utilize more cost-effective or green based raw materials. One of the key approaches to address this problem is to use low-cost filler, including fibers, for polymer. Recently extensive studies have been performed on exploring the ways to use cellulose-based fibers in place of synthetic fibers as reinforcements for synthetic polymer such as PP as composites (Arrakhiz et al., 2013; Faruk, Bledzki, Fink, & Sain, 2012; Liu, Zhou, Qian, Shen, & An, 2013; Spoljaric, Genovese, & Shanks, 2009). These cellulose-based materials are abundant, inexpensive and easily obtained from renewable natural resources. They also possess other advantages, such as relatively low density, high specific strength and stiffness that allow the production of low-density composites with higher filler content. In addition, plant fibers are flexible and therefore less susceptible to fracture during processing (Frone et al., 2011). Most importantly, they are biodegradable in comparison to manmade or synthetic fibers such as glass fibers (Javadi et al., 2010; Liu, Zhong, Chang, Li, & Wu, 2010; Siqueira et al., 2013; Xiao, Li, Chanklin, Zeng, & Xiao, 2011). Therefore, plant fibers obtained from wood or non-wood have recently attracted the attention of researchers both from academia and industry. And the growth of composites using cellulose fibers as reinforcement as sustainable materials is generating a steadily increasing impact in various industrial and consumer sectors. This trend has been prompted mainly due to various environmental, economic and performance issues (Gershon, Bruck, Xu, Sutton, & Tiwari, 2010; Khiari, Marrakchi, Belgacem, Mauret, & Mhenni, 2011; Liu, Wu, & Zhang, 2009).

Cellulose, the most abundant biopolymer on earth, is the main constituent of wood and non-wood. It is located predominantly in the secondary wall of the wood fiber. Approximately 45–50% of extractive-free dry substance in most plant species is cellulose, and it is the most single important component in the fiber cell wall in terms of its volume and effect on the characteristics of wood. Fiber surface accompanies with a great number of hydroxyl groups (Li, Xiao, Zheng, & Xiao, 2011), which the fiber with a strong polarity and high water absorption. However, most of the thermoplastic polymeric matrices used for composites such as polyethylene (PE), polypropylene (PP) are nonpolar, therefore, the interfacial compatibility is poor between enhanced phase and

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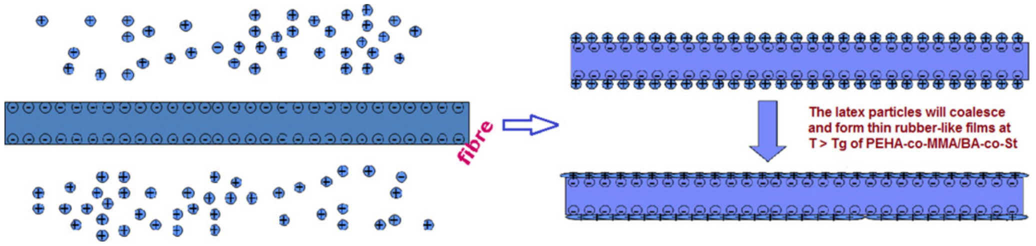

Fig. 1. A schematic of adsorption and filming of nano-sized latex on the surface of cellulose fiber.

matrix phase. Unmodified fiber added into the composites makes a significant reduction in impact strength: one is because fiber is used as a filler of high rigidity around the base of their emotional bonds with the role of the binding layer to increase rigidity; the other is that fiber wrapped in the matrix material has played a role of body of stress concentration. The weak bonding between fiber and matrix, connecting points between fiber to each other, as well as structural defects of the fiber itself are all stress concentration points, and the fiber itself elongation at break is very low too, which increases the brittleness of the material. The phase morphologies of the blends are coarse, and their mechanical properties are poor for most of these blends are highly immiscible (Koning, van Duin, Pagnoulle, & Jerome, 1998). Maleic anhydride wax was proven to be effective of improving the interfacial adhesion for PP composites. Maleic anhydride grafted polypropylene was also used to achieve good wetting of natural fiber surface (Ashori & Nourbakhsh, 2010; Morandim-Giannetti et al., 2011).

An elastomer is usually employed to improve impact resistance of PP; whereas the filler or fiber is often added to increase stiffness. Structure can be quite complicated in such multicomponent materials. Two boundary structures may form in them: the two components, i.e. the elastomer and the filler, can be distributed separately from each other in the polymer matrix, which will jeopardize the mechanical properties. In contrast, the elastomeric material modified cellulose fiber could address the disadvantage, i.e., chemical treating the surface of fiber could overcome the wetting problems of wood fiber, which will then endow the good compatibility with polymer matrix.

In this work, polymer latex bearing flexible core and cationic shell was used for the surface treatment of CTMP fibers. Positive-charged poly(2-ethylhexylacrylate -co-methyl methacrylate/butyl acrylate-co-styrene) [PEHA-co-MMA/BA-co-St] latex with core–shell structure was synthesized with cationic surfactant and cationic initiator. The resultant cationic groups (i.e., quaternary ammonium salt) on the surface of PEHA-co-MMA/BA-co-St latex promote their adsorption on the surfaces of cellulose fibers which are often negative-charged. The repulsion between cationic particles prevents their homocoagulation and drives the particles to form monolayer of discrete particles on the natural fiber surface. The latex particles tend to coalesce and form thin rubber-like films (at $T > T_g$ of PEHA-co-MMA/BA-co-St), which further improved the toughness of the PP composites. Such a process is schematically illustrated in Fig. 1. The objectives of this work were to investigate the adsorption of cationic PEHA-co-MMA/BA-co-St polymer on the CTMP fiber surface; and to identify the effects of the resulting fibers as reinforcements on the mechanical properties of the polypropylene composites.

2. Material and methods

2.1. Materials

The chem-thermal mechanical pulp (CTMP) fiber was obtained from Weyerhaeuser USA. All chemicals were used as received without further purification unless otherwise specified. The novel cationic P(BA-co-MMA/EHA-co-St) latex with core–shell structure was prepared via a two-stage seeded semi-continuous emulsion polymerization using hexadecyl-trimethyl ammonium bromide (HTAB) as an emulsifier (Pan, Xiao, & Song, 2013).

2.2. Adsorption of cationic P(EHA-co-MMA/BA-co-St) latex on fibers for surface treatment

To quantify the amount of adsorption of the latex on CTMP fiber or determine the adsorption isotherms, the supernatant of each suspension containing CP(EHA-co-MMA/BA-co-St) at various dosages and pre-mixed with the fixed amount of CTMP fibers was collected by a syringe and filtered to exclude fibers. Turbidity of the sample was measured, and the concentration of the latex in the supernatant was determined using an established calibration curve which represents the linear relationship between turbidity and concentration. The difference between the amounts of latex added and that found in the supernatant is attributed to the amount deposited or adsorbed on the fibers.

2.3. Preparation and characterization of biocomposites

2.3.1. Material compounding and specimen preparation

Prior to compounding, the CTMP fibers were dried in an oven at 50 °C for 24 hours. The next step was to compound the fiber and PP in a K-mixer (manufactured by Werner and Pfleiderer Gelimat) at 190–200 °C. The K-mixer applied high shear force which produced sufficient heat to melt the matrix and to mix the fibers and the matrix. Fibers were K-mixed for about 1 min and then polypropylene and coupling agent was added. After the temperature of the mixture reached 190 °C, the compound was discharged automatically. The fiber content in the composites was fixed at 40% by weight in the current work.

The discharged compound was cooled to room temperature and then granulated using a C. W. Brabender Granulator (Model S-10-9). An Engel injection molder (Model ES-28 was used to form standard tensile and impact test bars as specified in ASTM D-258 and D-638. Specimens were conditioned at room temperature (RH = 70%) for 48 h prior to the testing of mechanical properties.

2.3.2. Impact strength

Impact testing was performed in accordance with ASTM D256. The specimens made from injection molding were notched with a purpose-built router. Before testing, the thickness and width of specimen was measured, and the notch tip was sharpened with a sharp blade. The test was conducted in a TINIUS-OLSEN Plastic Impact Tester (Model 892). For each sample, at least six specimens were tested.

2.3.3. Tensile and flexural properties

Tensile and flexural properties were measured on a standard computerized testing machine (Instron Model 20). Tensile properties were measured in accordance with the ASTM D-638 procedure

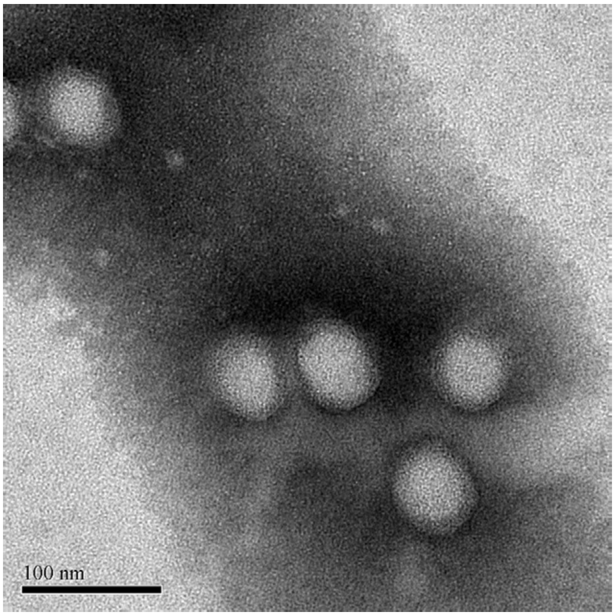

Fig. 2. TEM image corresponding to core–shell particles.

using 6 ASTM type 1 specimens at a cross head rate of 12.5 mm/min. Flexural properties were measured on 6 specimens in accordance with the procedure in ASTM D790 using the aforementioned machine in the three-point loading model at a cross head speed of 12.5 mm/min and a span width of 50.8 mm.

2.4. Morphological observation of latex and modified fiber

The morphology of latex particle was observed using a transmission electron microscope (TEM, JEM-100CX-II, Hitachi, Japan) operated at 200 KV. The samples were diluted and stained with 2% phosphotungstic acid; mounted on 400-mesh carbon coated copper grids and then dried prior to observation. TEM images were recorded using a Gatan 4kx4k digital camera. The surface morphology of the cellulose fiber treated with the latex was revealed using a scanning electron microscope (model Joel 6400 SEM). The specimens were mounted on aluminum stub using double side carbon tape and then coated with gold using a S150A sputter coater.

2.5. Determination of thermal characteristics of the biocomposites

Thermogravimetric analyses (TGA) were collected with a Thermoanalyzer (TG 209, NETZCH Co.). Measurements were taken under nitrogen atmosphere and using a ramping temperature profile of 20 °C/min, within a temperature range 50–660 °C. DSC scans were carried out under nitrogen atmosphere on approximately 10 mg of material using a Mettler Toledo 840 differential scanning calorimeter. The runs were performed from 60 °C to 210 °C (10 °C/min) and, after a 3 min isothermal, back down to 60 °C (10 °C/min) in order to observe the crystallization behaviors of the biocomposite and to determine the T_c.

3. Results and discussion

3.1. Observation on the morphology of latex particles

TEM was used to visualize the morphology of latex. The image is presented in Fig. 2, which is a representative image of the core–shell latex particles addressed above. As can be seen, the core–shell structure of latex was clearly revealed by TEM observation. The relatively rigid shell, the dark edges of the latex in TEM image, consists of the copolymer of BA and St whereas the cationic surfactant on peripheral surfaces renders the latex cationic. The cationic surface was verified based on zeta-potential measurements; similar to those reported elsewhere (Pan et al., 2013). The morphology maintained and cationic surface ensure the strong adsorption or high retention of the latex on fiber prior to blending. The core of the latex, the copolymer of EHA and MMA, has low Tg and promotes the formation of thin elastomer layer for enhancing the compatibility with PP matrix during blending and toughening the biocomposites.

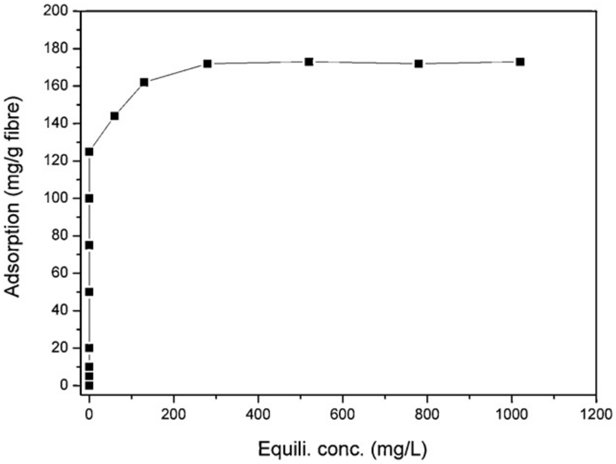

Fig. 3. Adsorption isotherm of latex CPEHA-co-MMA/BA-co-St adsorption on fiber surface. Latex sample: EHA:MMA:BA:St = 1:1:1:1 (mol/mol). [Fiber] = 10.0 g/L.

3.2. Characterization of modified CTMP

3.2.1. Surface-modification of CTMP via cationic latex adsorption or deposition

Fig. 3 presents the adsorption isotherms of the latex on CTMP surface. Clearly, the saturated adsorption was reached at approximately 173 mg/g fiber. From Fig. 3, it also can be seen that latex adsorbed onto the fiber surfaces completely in the addition range from 0.5– 12.5 wt% on fiber, i.e., 50–1250 mg/L in Fig. 3), implying that the strong adsorption of cationic latex on fibers due to the electrostatic association between cationic latex and negative-charged CTMP cellulose fibers.

3.2.2. Thermal analysis of modified CTMP

The adsorption of cationic latex on the surfaces of fiber can be further confirmed by thermal analysis TGA. As shown in Fig. 4(a), there is only one decomposed temperature for untreated CTMP fiber. In contrast, two peaks appear in the modified fiber (see Fig. 4(b)), one corresponding to the fiber at 335 °C, and another to the polymer film formed on the surfaces of fibers at 442 °C. The amount of adsorption quantified from the TGA results is close to the design value.

3.2.3. Surface morphology of modified CTMP

Fig. 5 presents the SEM images of the CTMP fiber treated with latex (see Fig. 5). As can be seen, the polymer film from the latex seems to cover the fiber surface, creating smoother surface. Even though the modified fiber was nonheat-treated, the surface of modified fiber is still similar to that heat-treated. No individual latex particles are visible on the surface, which is probably attributed to very low Tg of the latex and the filming of adsorbed latex during the sample preparation for SEM observation.

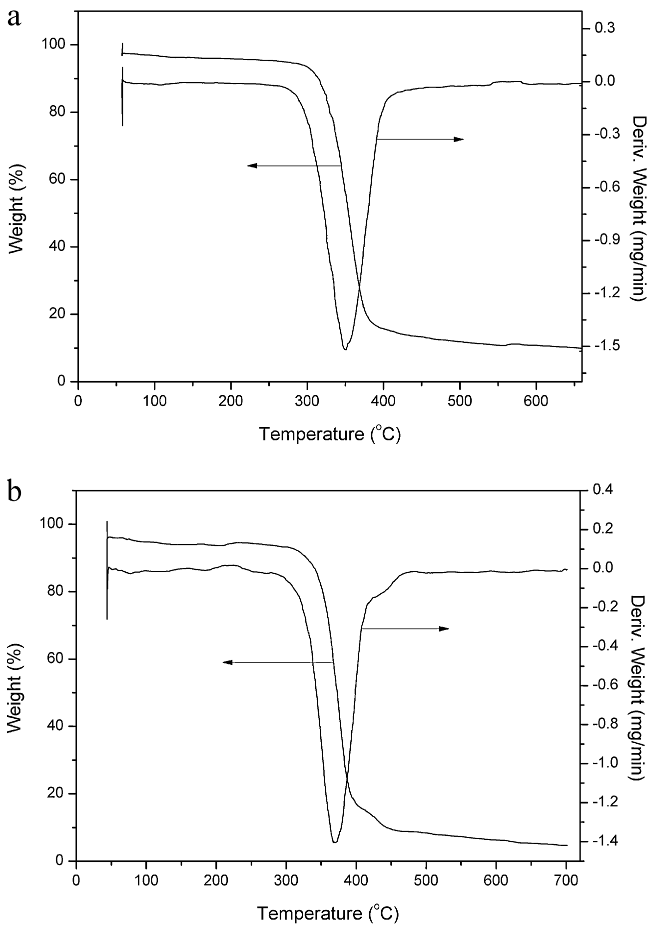

Fig. 4. Thermal analysis of (a) untreated fiber and (b) CPEHA-co-MMA/BA-co-St (1:1:1:1, mol/mol) modified fiber (latex dosage rate of 4%).

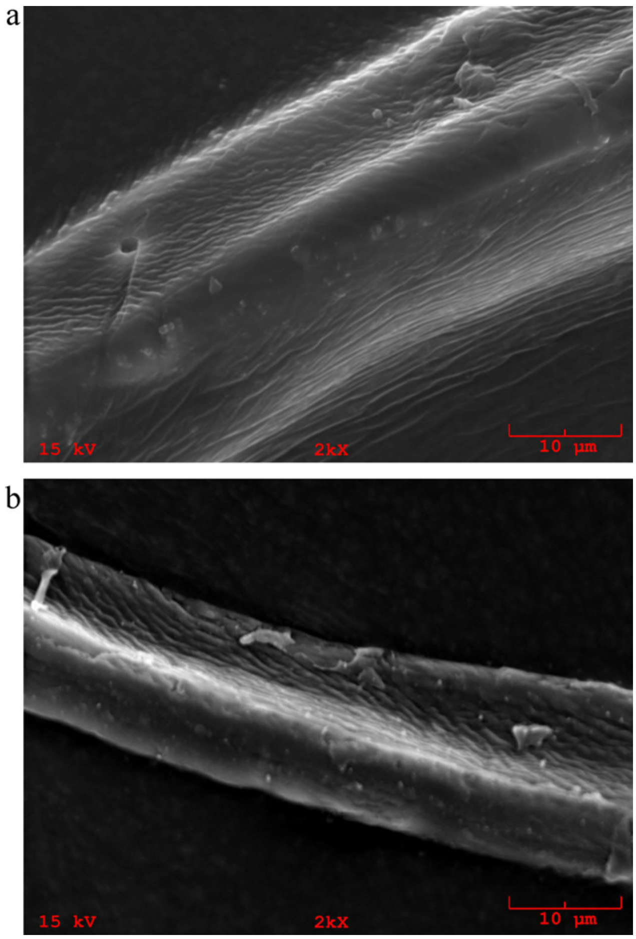

Fig. 5. SEM images of surface of (a) untreated CTMP and (b) CPEHA-co-MMA/BA-co-St (1:1:1:1, mol/mol) modified CTMP.

3.3. Mechanical properties of PEHA-co-MMA/BA-co-St latex modified CTMP fiber polypropylene biocomposites

The notched impact (NI) and unnotched impact strengths (UNI) are the key mechanical properties for characterizing the toughenss of the PP-based biocomposites reinforced by the CTMP fibers modified with latex; and were measured in this work. Fig. 6 presents the influence of the dosage of the latex used for CTMP fiber modification on both impact strengths. As can be seen, in the presence of 2.0% of P(EHA-co-MMA/BA-co-St) (weight on fibers), the maximum NI strength was achieved. Meanwhile, the unnotched impact strength was also improved significantly at the same dosage. However, further increase in P(EHA-co-MMA/BA-co-St) content in treated fibers lowered the UNI value slightly.

The similar trend was observed for the tensile strength (see Fig. 7). The strength was increased when 2.0% of the latex was used and then dropped evidently with further increase of the latex dosage. Meanwhile, the flexural modulus also reached its maximum value at the same dosage (i.e., 2% latex wt on fiber).

Overall, the tensile and unnotched impact strengths of the composites were increased significantly in the presence of 40% of CTMP fibers treated with P(EHA-co-MMA/BA-co-St) latex even at a low dosage range (0.8–2.0% on CTMP). This is due to the strong elastomeric characteristic of the latex film after adsorbing on fiber surfaces. This novel approach distinguishes from the traditional one to enhance the toughness of PP via blending elastomer or rubber with PP. In the current systems, we aimed at using CTMP coated with the latex as impact energy absorbents and meanwhile improving the interfacial bonding between the fibers and PP matrix. It should be addressed that the fiber/interface/matrix interactions could be complex due to porous and fibrillated surfaces of fibers. Based on the toughening mechanism, the notched impact energy is a measure of crack propagation; but the unnotched impact energy involves both crack initiation and propagation. The positive results on enhancing the unnotched Izod impact strength obtained from this work suggest the effectiveness of the latex-treated fiber in

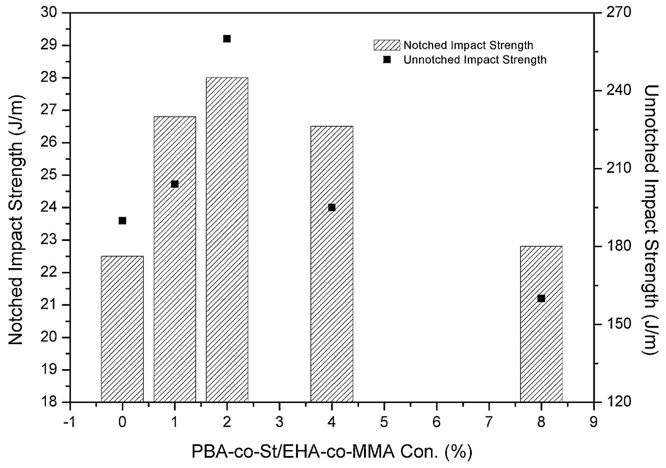

Fig. 6. Notched and unnotched impact strengths of the biocomposites at various dosages of the latex for CTMP treatment (the amount of fibers in PP-based biocomposites was fixed at 40 wt%).

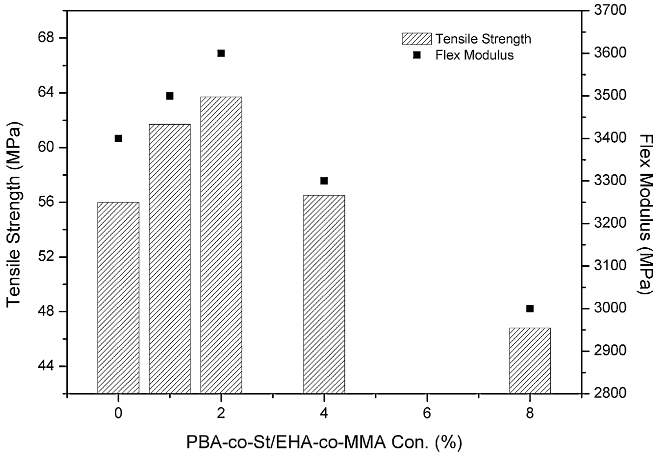

Fig. 7. Comparison of P(EHA-co-MMA/BA-co-St) content with tensile strength and flexural modulus of at various dosages of the latex for CTMP treatment (the amount of fibers in PP-based biocomposites was fixed at 40 wt%).

retarding the crack initiation. On the other hand, the CTMP fibers could also act as stress concentrators. After modified by the latex, the fiber either improved itself strength or its adhesion to the matrix. The presence of latex-treated CTMP as a reinforcement might cause the significant change of crack initiation energy. As a result, the failure of fibers is likely accompanied with the tearing of matrix.

Apart from the strength measurements, the observation on the morphology of the fractured surface of the biocomposite, which could be visualized using SEM, also provides the evidence for the improved adhesion between the modified CTMP fibers and PP matrix. For the latex-modified fibers, the fibers are integrated with the PP matrix and broken together with PP when the biocomposite is fractured (often conducted in liquid nitrogen). The interface between the modified fiber surface and the PP matrix appears to be much dimmer, compared with the unmodified one, which demonstrates the improved compatibility between cellulose fibers and hydrophobic polymer matrix.

3.3.1. *Effect of cellulose fibers on crystallization behavior of PP in the biocomposites*

The crystallization of PP has direct impact on the physical properties of the biocomposite. When polypropylene is cooled below its melting temperature, nucleation and crystallization occur (Yi, Wen, Niu, & Dong, 2013). To decrease the induction period of crystallization, the addition of nucleating agent has been a conventional approach to identify whether the modified fiber can act as a nucleating agent (apart from a reinforcement). DSC scans were conducted on pure PP sample, unmodified CTMP/PP biocomposites and CP(EHA-co-MMA/BA-co-St) modified CTMP (latex:fiber = 5:100)/PP biocomposites.

The results are presented in Fig. 8. Clearly, the shift of T_c, which represents the temperature at the maximum crystallization rate, was observed for the biocomposite. The crystallization temperatures were 114, 120 and 125 °C for neat PP, unmodified CTMP/PP, and CP(EHA-co-MMA/BA-co-St) modified CTMP/PP biocomposites, respectively. Such crystallization behavior suggested that the heterogeneous nucleation was induced by cellulose fibers in the biocomposite, regardless of the latex modification. Moreover, the presence of cellulose fiber could potentially increase the degree of crystallization of PP, which could be quantified approximately based on the ratio of the heat of fusion of modified PP, measured by DSC, to that for the 100% crystalline PP. In addition, the crystallization induction period of the modified PP composites was also shortened, which accelerated the nucleation rate and allowed the nucleation to occur at a higher temperature or higher T_c.

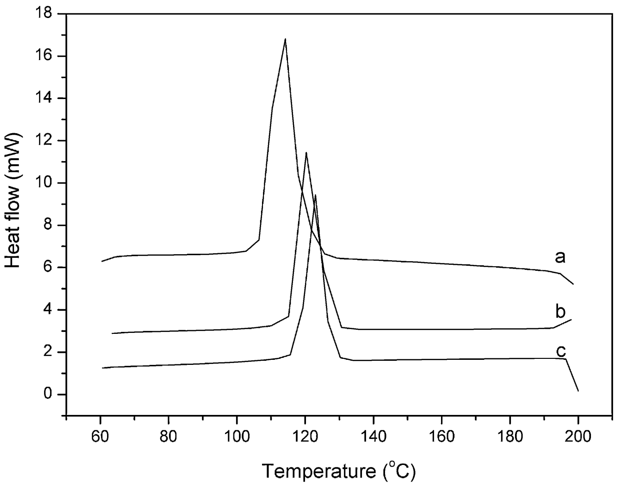

Fig. 8. Three DSC cooling crystallization diagrams at the rate of 10 °C/min for PP (a), unmodified CTMP/PP (b) and CPEHA-co-MMA/BA-co-St (1:1:1:1, mol/mol) modified CTMP/PP (c).

In other words, DSC results demonstrate that both untreated CTMP fiber and the one treated with P(EHA-co-MMA/BA-co-St) acted as a nucleation agent. The crystallization induction period of the modified PP biocomposites were all shortened, which accelerated the nucleation rate and allowed the nucleation to occur at a higher temperature (i.e., higher T_c).

In conjunction with the toughening effect contributed by P(EHA-co-MMA/BA-co-St) latex on fiber surfaces, the synergy significantly enhanced the impact strength and tensile properties of PP biocomposites.

4. Conclusions

CTMP fibers treated with cationic P(EHA-co-MMA/BA-co-St) latex are promising for improving the mechanical properties of the fiber-reinforced PP biocomposites. In the presence of 40% of the fibers treated with the core–shell latex (2% wt on dry fibers), the unnotched Izod impact strength of the biocomposites containing was increased significantly; and meanwhile, the tensile strength and flexural modulus were also enhanced. However, increasing latex content for fiber surface modification did not show the further improvement; and the corresponding impact strength of the biocomposites was only increased marginally. The latex particles adsorbed on fiber surface nearly completely due to electrostatic association, leading to the formation of interfacial elastic thin film which not only improves the compatibility between hydrophilic cellulose fibers (as reinforcements) and hydrophobic PP matrix, but also terminates the crack propagation. Moreover, the latex-treated fibers could act as nucleating agents, thus promoting the crystallization of PP and enhancing the toughness of the biocomposite thereafter.

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