Original Article

Effect of pectin particles and cotton fibers on properties of thermoplastic cassava starch composites

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Abstract

Due to poor mechanical properties and high water uptake of thermoplastic starch (TPS), this research focused on property improvement of thermoplastic cassava starch (TPCS) using natural compatible polymers, i.e. pectin particles and cotton fibers. Different TPCS composites, reinforced by the pectin particles and/or the cotton fibers, were compounded and shaped using an internal mixer and a compression molding machine, respectively. It was found from infrared (IR) spectra that the peak position of O-H stretching of the TPCS polymer clearly shifted to lower wavenumber by the addition of the pectin particles and/or the cotton fibers. Moreover, the significant increase of stress at maximum load and Young's modulus of the TPCS/pectin particle and the TPCS/cotton fiber composites was observed. The drop of water uptake was also found when the cotton fibers were incorporated into the TPCS matrix with/without pectin particles. In addition, X-Ray Diffraction, Scanning Electron Microscopy and Thermogravimetric Analysis were used to characterize different TPCS composites.

Keywords: biodegradable polymer, pectin, composite, thermoplastic starch, fibers

1. Introduction

In the last decade, biopolymers based on starch have been of interest as a replacement for synthetic thermoplastics due to its availability, low costs and renewability. Starch can be extracted from many botanical sources including cassava, rice, corn, potato and wheat. Starch commonly exists in a granular structure and the granules are mainly composed of two different glucose polymers; amylose and amylopectin molecules (Stepto, 2004; Bemiller *et al.,* 2009). Amylose is a linear polymer of anhydroglucose units linked through a-1-4 glucosidic bonds. Amylopectin is a branched polymer constituted by a backbone structure linked amylose with 4-5% of its units bearing β-1-6 glucosidic linkages generating amylose-like branches (Stepto, 2004; Bemiller *et al.,* 2009).

Starch, itself, cannot be processed with traditional thermoplastic processing technologies because of its large size and stiffness of the molecules and the resulting high glass transition temperature. Because of strong hydrogen bonds developing among the starch molecules, native starch cannot be molten and processed. Plasticization partially resolves these problems and the plasticized starch or thermoplastic starch (TPS) can, then, be processed under high temperature, shear and pressure by synthetic thermoplastic processing machines machines (Stepto, 2004; Ma *et al.,* 2005; Martins *et al.,* 2009; Muller *et al.,* 2009; Brejnhoil *et al.,* 2010; Prachayawarakorn *et al.,* 2011; Kaewtatip *et al.,* 2012; Prachayawarakorn *et al.,* 2012; Lopez *et al.,* 2013; Prachaya-warakorn *et al.,* 2013; Ibrahim *et al.,* 2014; Muller *et al.,* 2014). However, TPS polymers still have disadvantages, i.e. poor mechanical properties and high water sensitivity. In order to overcome these drawbacks, TPS polymers can be modified by blending with natural polymer based on polysaccharide such as pectin.

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Stepto, 2004; Ma *et al.,* 2005; Martins *et al.,* 2009; Muller *et al.,* 2009; Brejnhoil *et al.,* 2010; Prachayawarakorn *et al.,* 2011; Kaewtatip *et al.,* 2012; Prachayawarakorn *et al.,* 2012; Lopez *et al.,* 2013; Prachayawarakorn *et al.,* 2013; Ibrahim *et al.,* 2014; Muller *et al.,* 2014).

Pectin, one of the natural gelling agents, is a hetero-polysaccharide that contains in the primary cell walls of terrestrial plants. The characteristic structure of pectin is a linear chain of α-(1, 4) linked D-galacturonic acid that forms the pectin backbone, a homogalacturonan. It is produced commercially as a white to light brown powder, mainly extracted from citrus fruits and is used in food as a gelling agent and a thickening agent (Brejnhoil *et al.,* 2010).

Pectin and maize starch was reported to be produced into a biodegradable film (Fishman *et al.,* 2000). It was shown that the phase-compatible pectin-starch film have a large-glass-transition temperature at about -50°C and the storage modulus at room temperature was above 10^3 MPa (Fishman *et al.,* 2000). Pectin was also prepared as biodegradable film with soy protein ((Giancone *et al.,* 2009; Piazza *et al.,* 2009). Moreover, ternary films of casted chitosan/poly(vinyl alcohol)/pectin film (Tripathi *et al.,* 2010) were reported to be crystalline and the film could be used as an antimicrobial food-packaging film since it showed the positive antimicrobial activity against pathogenic bacteria, i.e. *E. coli* and *S. aureus.*

Another interesting method for improving properties of TPS materials is to reinforce by natural cellulose fibers (Ma *et al.,* 2005; Martins *et al.,* 2009; Muller *et al.,* 2009; Prachayawarakorn *et al.,* 2011; Kaewtatip *et al.,* 2012; Prachayawarakorn *et al.,* 2012; Lopez *et al.,* 2013; Prachayawarakorn *et al.,* 2013; Ibrahim *et al.,* 2014; Muller *et al.,* 2014). It was reported that winceyette fibers reinforced thermoplastic corn starch showed the improvement of tensile properties and water uptake (Ma *et al.,* 2005). In addition, enhancement of thermal stability of TPS/cellulose fiber composites was also observed (Ma *et al.,* 2005; Prachayawarakorn *et al.,* 2013; Ibrahim *et al.,* 2014). Similar results was also found in many reports for TPS polymers reinforced by different types of cellulose fibers such as newspaper (Lopez *et al.,* 2013), wood fibers (Muller *et al.,* 2014), flax fiber (Ibrahim *et al.,* 2014), kapok fibers (Prachayawarakorn *et al.,* 2013) or cotton fibers (Prachayawarakorn *et al.,* 2011).

Cotton is a soft, fluffy staple fiber that grows in a boll, around the seeds of cotton plants of the genus *Gossypium.* Cotton is used to make a number of products such as clothes and other products, like towels, carpets or sheets. The fiber is almost pure cellulose. The cotton fibers contain mainly cellulose and very low contents of hemicellulose, lignin, pectin and wax. The cellulose and hemicellulose are very hydrophilic; whereas, the lignin is totally amorphous and hydrophilic in nature (Morton *et al.,* 1997; Mather *et al.,* 2011).

Even though there are many research works regarding the utilization of cellulose fibers as reinforcement in polysaccharide matrices, the publication on TPS matrix reinforced by using either pectin particles or both pectin particles and cotton fibers has not been found. In this work, biocomposites based on thermoplastic cassava starch (TPCS) have been studied. The TPCS matrix was reinforced with the pectin particles and/or the cotton fibers by melt processing technique. The biocomposites were, then, characterized through IR, XRD, SEM and TGA techniques and also tested for mechanical properties.

2. Experimental Details

2.1 Materials

Cassava starch (11.5%-13.0% moisture) was purchased from Thaitam (Chonburi, Thailand), containing 20±1 %wt amylose and 75±2 %wt amylopectin. Glycerol (plasticizer) was obtained from Lab System Co. Ltd. (Thailand). Pectin (food grade) with 72% degree of methylation was purchased from Ruam Chemie 1986, Co. Ltd. (Bangkok, Thailand). Cotton fibers with the L/D ratio of approximately 200/1 were obtained locally and used as-received.

2.2 Sample preparation

Firstly, cassava starch was firstly pre-mixed with glycerol in polyethylene bags. The weight ratio of cassava starch and glycerol was maintained at 65:35. Blending was carried out using an internal mixer (Brabender, PL 2000/PL 2001) at the temperature of 140°C with the rotor speed of 40 rpm for 5 min. in order to obtain a homogeneous material. The processed sample was, then, compressed using a compression molding machine (LabTech Engineering, Thailand) at the temperature of 140°C into 2 mm thick plates. The property modification of the resulting TPCS polymer was carried out using the pectin particles and/or the cotton fibers. The TPCS/pectin particle composites (TPCS/P) were prepared using different contents of pectin, i.e. 0%, 2%, 4%, 6%, 8% and 10% by the TPCS weight. The TPCS/cotton fiber composite (TPCS/F) was also prepared by using 10% weight of the cotton fibers by the TPCS weight. Finally, the TPCS composite reinforced by the combination of using both the pectin particles and the cotton fibers (TPCS/P/F) was also examined. It should be noted that the number presented in each formulation was the used weight of the modifying agent.

2.3 IR spectroscopic study

FTIR spectrum of a sample was recorded on a Spectrum 2000 GX spectrometer (Perkin Elmer, USA) using KBr disk technique. A resolution of 4 cm^{-1} in a spectral range of 600-4000 cm^{-1} with 16 scans was operated.

2.4 X-ray diffraction

Wide angle X-ray diffraction measurements were conducted using a D8 Advance X-ray diffractometer (Bruker, Madison, USA), with CuK$_a$ radiation (wavelength 0.1542 nm)

operating at 40 kV and 35 mA. The scattering angle (2q) covered the range from 5° to 60° (q is the Bragg angle) with a step size of 0.02° and 10s sampling interval. A newly prepared sample was tested under relative humidity of 60± 5%.

2.5 Morphology

A LEO 1455 VP scanning electron microscope (Oberkochen, Germany) was used to study morphology of a sample. A fractured sample under liquid nitrogen was sputter-coated with a thin layer of gold to prevent electrical charge during observation.

2.6 Mechanical properties

Mechanical tests were conducted according to ASTM D-638 at the temperature of 23±1°C and relative humidity of 60±5%. A mechanical measurement of each dumbbell-shaped specimen was performed using a Universal Testing Machine (Lloyd Instrument, LR 5K, West Sussex, UK) with a 100 N load; the crosshead speed was fixed at 40 mm/min. The mechanical property results of different composites were obtained by averaging from ten independent specimens.

2.7 Water absorption

Newly prepared sample was dried at 105°C for 3 h and then stored at 50% relative humidity at a temperature of 30± 2°C prior to water absorption evaluation. The 50% relative humidity was obtained using a saturated salt solution of $CaCl_2$ in a closed vessel. The amount of water absorbed by a sample was determined until the constant weight was obtained. The percentage of water absorption was calculated as followed:

$$\text{Water absorption} = (W_2\text{-}W_1)/W_1 \times 100$$

where W_2 and W_1 are the wet weight and the dried weight of a sample, respectively.

2.8 Thermal properties

TGA and DTG thermograms of a sample were recorded by a thermogravimetric analyzer (Pyris 1, Perkin Elmer, Massachusetts, USA). The sample was tested under nitrogen atmosphere within a temperature range of 50-700°C at a heating rate of 10°C/min. Thermal degradation temperature (T_d) was reported by the onset degradation temperature where the weight loss started to occur.

3. Results and Discussion

3.1 IR spectroscopic study

IR spectroscopy is a useful technique for identifying and characterizing polymers. IR spectra of different TPCS composites are shown in Figure 1. It can be seen that all spectra showed similar absorption peak positions regarding to polysaccharide structure of starch. The peak positions at 3,200-3,500 cm^{-1}, 2,900-3,000 cm^{-1} and 1,400-1,450 cm^{-1} were based on O-H stretching, C-H stretching and O-H bending, respectively. The wavenumber at 1600 cm^{-1} was due to bounded water in starch. In addition, the bands in the range of 1,100-1,300 cm^{-1} and 800-850 cm^{-1} was belonged to C-O stretching and C-H bending, respectively (Bower et al., 1989).

It should be noted that the incorporation of the pectin particles into the TPCS matrix (TPCS/P10) (Figure 1) caused the O-H stretching and O-H bending peaks shifted to lower wavenumber. This was claimed to be due to the new formation of hydrogen bonds between the TPCS and the pectin components since both components were based on polysaccharide chemical structures. The peak shift was also reported by Pawlak et al. (2003). Moreover, the new peak position at approximately 1,720 cm^{-1} was detected by the use of pectin particles and this was assigned for the C=O stretching from the carboxylic groups in the pectin chemical structure (Bower et al., 1989).

Similar to the TPCS polymer modified by the pectin particles, the TPCS polymer modified by the cotton fibers (TPCS/F10) (Figure 1) also presented the peak shift of the O-H stretching and O-H bending peaks since the cotton fibers are also composed of cellulose structure that is compatible with the polysaccharide structure of starch. By adding both the pectin particles and the cotton fibers into the TPCS matrix (TPCS/P10/F10), the IR spectrum in Figure 1 represented the similar peak shift of the O-H stretching and O-H bending peaks. The results implies that new hydrogen bonds can form in the TPCS matrix modified by the pectin particles, cotton fibers or both pectin particles and cotton fibers. As expected, new IR absorption peaks was observed at 1720 cm^{-1} due to the presence of the pectin particles in the TPCS/P10/F10 composite. Similar IR peak shifts results were also reported for TPCS/kapok fiber (Prachayawarakorn et al., 2013) and TPS/luffa fiber composites (Kaewtatip et al., 2012).

3.2 X-ray diffraction

X-ray diffraction patterns of the TPCS polymer and its corresponding composites are shown in Figure 2. For the TPCS polymer (Figure 2), the majority of starch granules have been gelatinized and, hence, the glucosidic chains with B-type crystal structure are retrograded into the V-type (Hartel, 2001). The retrograded TPCS polymer gives V-type diffraction pattern with the high intense peak at 2θ of 19.6° and the low intense peak at 2θ of 18.2°.

Figure 2 also presents the diffraction patterns of the TPCS/P10 composite and the main peaks are located approximately at 2θ of 18.2° and 19.6°, corresponded to the diffraction patterns from both the TPCS and the pectin components. Normally, the crystal structure of pectin, based on β-helix structure, shows diffracted peaks mainly at 2θ of 18.6°, 20.6° and 28.4° (Hartel, 2001). In addition, the TPCS/F10 composite
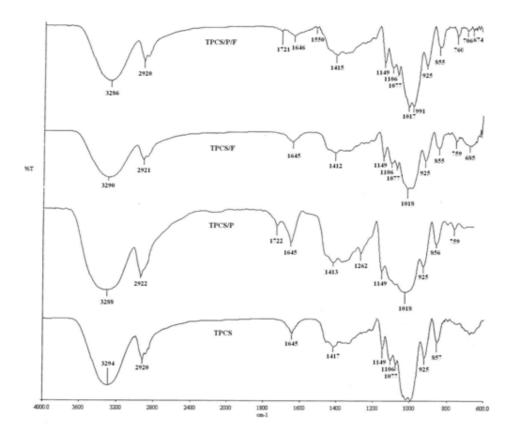
Figure 1. FT-IR spectra of the different TPCS composites reinforced by the pectin particles and/or the cotton fibers
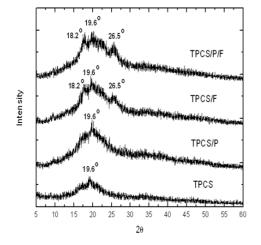

Figure 2. X-ray diffraction patterns of different TPCS composites reinforced by the pectin particles and/or the cotton fibers.

(Figure 2) shows the main diffraction peaks at 2θ of 18.2°, 19.6° and 26.5°, related to the diffraction patterns from both the TPCS and the cotton fiber components. The peak at 2 of 26.5° was diffracted from the cotton fibers. Generally, native cotton fibers show the main XRD peaks at 2θ of 17.7° and 26.5°, corresponding to cellulose I crystal structure (Morton et al., 1997)

Moreover, the TPCS/P10/F10 composite showed the main diffraction peaks approximately at 2θ of 18.2°, 19.6° and

26.5°. It should be mentioned that the addition of the pectin particles, the cotton fibers or both components into the TPCS matrix, clearly caused the increase of XRD intensity and diffracted area under the thermograms, presenting the increase of crystallinity of different composites since area under diffracted peaks represents crystallinity in polymers. Similar observation was found for the thermoplastic rice starch/cotton fiber composite (Prachayawarakorn et al., 2011) and the thermoplastic corn starch/bacterial cellulose composite (Martins et al., 2009)

3.3 Morphology

Morphology of different composites can be examined using SEM technique. SEM micrographs of the fractured surfaces of different TPCS composites are shown in Figure 3. It can be seen in Figure 3(a) that the TPCS polymer presented continuous surface with some starch granules. Moreover, good interfacial boundary between the pectin particles and the TPCS matrix was observed in Figure 3(b) due to the similarity of both polysaccharide chemical structures. No sign of void was found at the interfacial boundary between both phases.

The incorporation of the cotton fibers into the TPCS matrix led to the morphology as seen in Figure 3(c). It can be seen that the cotton fibers were distributed within the TPCS matrix and the surface wetting was observed, showing good
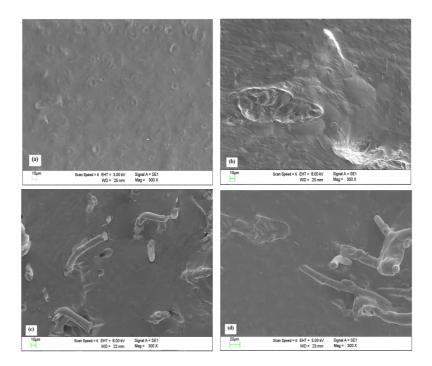
Figure 3. SEM micrographs of different composites (a) TPCS (b) TPCS/P10 (c) TPCS/F10 and (d) TPCS/P10/F10 composites.

phase compatibility between the TPCS matrix and the cotton fiber reinforcement. For the use of both the pectin particles and the cotton fibers, good phase compatibility was also observed as shown in Figure 3(d).

3.4 Water absorption

Water absorption is a very important factor for many applications of TPS products. The results of water absorption experiments, performed at the temperature of 30°C and 50% relative humidity, are shown in Figure 4. It can be seen that the TPCS polymer showed the increase of water absorption at the first stage. After that, the percentage water absorption tended to increase slowly and reached its maximum within 15 days

Similar relationship was observed for different TPCS composites. The TPCS/P10 composite presented higher percentage water absorption than the TPCS polymer since the pectin particles can absorb more water than starch (Bemiller et al., 2009; Brejnhoil, 2010). Nevertheless, the addition of the cotton fibers into the TPCS matrix clearly caused the decrease of the water absorption, as presented in Figure 4. The result is caused by different absorption characteristics between the TPCS and the cotton fiber. Generally, moisture absorption of cotton fiber is approximately 7.0-8.0%; whereas, it is in the range of 11.5-13.0% for starch (Morton et al., 1997; Bemiller et al., 2009; Mather et al., 2011). In addition, the drop of water absorption may also be attributed to the establishment of hydrogen bonding between the fiber and the hydroxyl functional group of the TPCS matrix (as shown by IR spectra in Figure 1), causing the decrease of free

hydroxyl groups. The drop of water absorption for TPS polymer added by cellulose fibers was also reported by many researchers (Ma et al., 2005; Prachayawarakorn et al., 2011; Kaewtatip et al., 2012; Prachayawarakorn et al., 2013).

Moreover, the TPCS/P10/F10 composite showed slightly higher water absorption than that of the TPCS/F10 composite but slightly lower than that of the TPCS polymer. The results indicate that the utilization of both the pectin particles and the cotton fibers can also cause a drop of water absorption of the TPCS composite. In this study, the lowest water absorption was obtained from the TPCS matrix with the reinforcement of the cotton fibers.
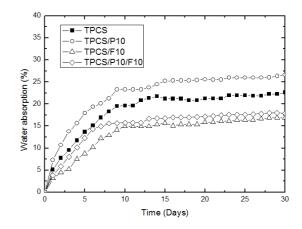

Figure 4. Water absorption of different TPCS composites reinforced by the pectin particles and/or the cotton fibers determined at 30°C and 50% RH.

3.5 Mechanical properties

Mechanical properties are essential for various TPS utilizations. Mechanical properties of different TPCS/pectin particle composites are represented in Figure 5. It was found from Figure 5 that the stress at maximum load and Young's modulus of the TPCS matrix clearly increased by the addition of the pectin particles. Moreover, the increasing contents of the pectin particles resulted in the significant improvement of the stress at maximum load and Young's modulus of the TPCS matrix. This was in agreement with the results observed from the new hydrogen bond (from IR peak shift in Figure 1), the increase in the XRD crystallinity (from XRD diffractograms in Figure 2) as well as the fiber surface wetting (from SEM micrographs in Figure 3).

Nevertheless, the strain at the maximum load of different TPCS/pectin particle composites decreased notably with the incorporation of the pectin particles (Figure 4) due to the more stiffness of the pectin particles, leading to the difficulty to elongate.

Figure 6 shows the mechanical properties of the TPCS composites reinforced by the pectin particles and/or the cotton fibers. It was found that the stress at maximum load and Young's modulus of the TPCS matrix increased significantly with the addition of the pectin particles, the cotton fibers or both components. The stress at maximum load and Young's modulus of the TPCS/F10 composite were higher than those of the TPCS/P10 composite; however, the highest stress at maximum load and Young's modulus was found for the TPCS/P10/F10 composite (Figure 6). The strain at maximum load clearly dropped when the pectin particles or the cotton fibers were added into the TPCS matrix. The TPCS polymer showed the highest strain at maximum load as shown in Figure 5. The increase of the stress at maximum load and Young's modulus values for approximately 36% and 1700% were found for the TPCS/P10/F10 composite.

3.6 Thermal properties

TGA is an accurate technique to examine the decomposition temperature and thermal stability of materials. Figure 7 and Table 1 present TGA and DTG thermograms and also

thermal degradation temperatures of different TPCS composites. All thermograms showed the first weight loss at around 100-150°C. This was due to the evaporation of water and glycerol (Ma et al., 2005). The temperature at which maximum weight loss occurred approximately at 280°C was due to the decomposition of the starch (Ma et al., 2005).

The addition of the pectin particles into the TPCS matrix (TPCS/P10 composite) caused the new onset degradation step at approximately 203°C which is due to the decomposition of the pectin particles. The additional degradation step at about 390°C was arisen from the degradation of the cotton fiber in the TPCS/F10 composite thermogram. The
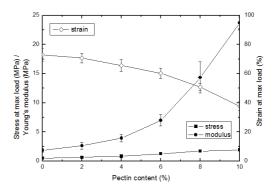

Figure 5. Mechanical properties of the TPCS/pectin particle composites with different pectin particle contents.
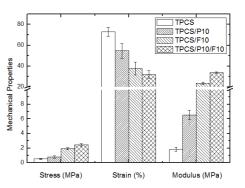

Figure 6. Mechanical properties of different TPCS composites reinforced by the pectin particles and/or the cotton fibers.
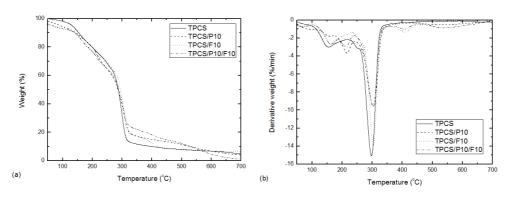

Figure 7. (a) TGA and (b) DTG thermograms of different TPCS composites reinforced by the pectin particles and/or the cotton fibers.

Table 1. Onset degradation temperatures of different TPCS composites reinforced by the pectin particles and/or the cotton fibers.

Composites	Onset degradation temperature (°C)			
	Step 1 (Glycerol)	Step 2 (Pectin particles)	Step 3 (Starch)	Step 4 (Cotton fibers)
TPCS	139.7	-	281.3	-
TPCS/P10	140.4	203.6	282.5	-
TPCS/F10	141.3	-	285.0	391.4
TPCS/P10/F10	139.1	203.3	283.9	391.6

presence of both degradation steps at 202°C and 390°C was, therefore, due to the decomposition of both pectin particles and cotton fibers in the TPCS/P10/F10 composite.

For different TPCS composites, it can be seen that the onset thermal temperatures apparently shifted to higher temperature as shown in Figure 7 and Table 1. This could be due to the phase compatibility for different compositions of the polysaccharide-cellulose based composites. It should be mentioned that the TPCS/F10 composite presented the highest onset thermal degradation temperature. Moreover, the TPCS/P10/F10 composite showed higher onset thermal degradation temperature than that of the TPCS/P10 composite. The results imply that the cotton fibers can improve thermal property of the TPCS polymer more effective than the pectin particles.

4. Conclusions

Different TPCS composites reinforced by the pectin particles and/or cotton fibers were successfully prepared by thermal technique. The O-H stretching and O-H bending IR peak shifts were found, indicating of the new hydrogen bond formation when the TPCS matrix was reinforced by the pectin particles and/or the cotton fibers. From XRD technique, the incorporation of the pectin particles and/or cotton fibers caused the increase in crystallinity of the TPCS matrix. Morphology from SEM revealed the good phase compatibility between the TPCS matrix with the reinforcements. Furthermore, the water absorption of the TPCS matrix was clearly reduced by using the cotton fibers including the use of both the pectin particles and the cotton fibers. The highest stress at maximum load and Young's modulus was found for the TPCS/P10/F10 composite. In addition, the increase of the thermal decomposition temperature was also observed when the pectin particles and/or cotton fibers were introduced into the TPCS matrix.

Acknowledgements

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