

# Effect of Cotton Fiber Contents and Lengths on Properties of Thermoplastic Starch Composites Prepared from Rice and Waxy Rice Starches

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**Abstract** Biodegradable polymer was prepared as thermoplastic starch (TPS) using rice and waxy rice starches. In order to increase mechanical properties and reduce water absorption of the TPS, cotton fiber was incorporated as the fiber reinforcement into the TPS matrix. The effect of cotton fiber contents and lengths on properties of the TPS was examined. Internal mixer and compression molding machine were used to mix and shape the samples. It was found that the thermoplastic rice starch (TPRS) showed higher stress at maximum load and Young's modulus but lower strain at maximum load than the thermoplastic waxy rice starch (TPWRS). In addition, stress at maximum load and Young's modulus of both TPRS and TPWRS increased significantly with the addition of the cotton fiber. Cotton fiber contents and lengths also affected mechanical properties of the TPRS and TPWRS composites. Moreover, water absorption of the TPRS and TPWRS composites decreased by the use of the cotton fibers. FT-IR and XRD techniques were used to study a change in functional group and crystallinity of the thermoplastic starch composites. Morphological, thermal and biodegradable properties of different thermoplastic starch composites were also investigated.

**Keywords** Biodegradable polymer · Cellulose fiber · Composite · Thermoplastic starch

## Introduction

Starch can be extracted from many botanical sources including rice, corn, cassava, 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 [1, 2]. Amylose is a linear polymer of anhydroglucose units linked through  $\alpha$ -1-4 glucosidic bonds. Amylopectin is a branched polymer constituted by a backbone structure link amylose with 4–5% of its units bearing  $\beta$ -1-6 glucosidic linkages generating amylose-like branches [1, 2].

Generally, starch contains approximately 25% amylose and 75% amylopectin but waxy genotypes contain considerably higher amounts of amylopectin [1, 2]. However, the proportions of amylose and amylopectin polymers in native starches vary appreciably in starch from different species and even within a given vegetable [2]. As for rice starch (*Oryza sativa*), the non-waxy indica rice starch contains about 17–22% amylose, the non-waxy japonica rice starch contains about 14–18% amylose, but the waxy rice starch contains only small content of amylose (5–7% amylose) [3].

The development of new starch-based materials has gained much interest in recent years due to their biodegradability, low cost and wide availability. Thermoplastic starch (TPS) is one of the starch-based polymers that have been widely investigated [4–14]. Starch can be transformed into TPS through disruption of its granular structure and plasticization in the presence of plasticizer. Under the action of high temperature and shear, the plasticized starch can be turned into TPS and processed using conventionally thermoplastic processing machines used for most synthetic polymers such as extrusion, compression or injection moldings [4–14].

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Nevertheless, TPS has two main limitations, i.e. poor mechanical properties and high water absorption [4, 5, 8, 11–14]. One interesting method to overcome these drawbacks is the use of natural fibers as reinforcement for TPS [4, 5, 11–14]. When natural fibers are mixed with TPS, their mechanical properties are obviously improved due to the chemical similarity of starch and plant fibers [4, 5, 11–14]. It was found that the blending of distinct natural fibers such as eucalyptus, wood pulp, flax, jute and others in starch/cellulose derivatives blends yielded the increase of tensile strength and Young's modulus as well as the decrease in elongation when the fiber content increased [4, 5, 11–14]. A significant improvement in water resistance was also obtained by the addition of cellulose fibers [4, 11–14]. This behavior is related to more hydrophobic characteristic of natural fibers in comparison to starch hydrophilic property. Moreover, thermal stability of TPS was reported to improve by higher thermal resistance of cellulose fibers [4, 5, 13, 14].

There was a report on properties of TPS prepared from rice starch (*Oryza sativa indica*) (TPRS) [14], however, TPS prepared from waxy rice starch (TPWRS), containing lower amylose and higher amylopectin contents, has never been reported. In general, rice starch is friable and brittle; whereas, waxy rice is sticky and elongated. It was expected that different characteristics and contents of amylose and amylopectin in rice and waxy rice starches would lead to various properties of the TPS. In this research, the TPRS and TPWRS sample without cotton fiber reinforcement were prepared using internal mixer and compression molding. As mentioned that properties of TPS could be improved by reinforcing with cellulose fibers; therefore, different TPRS and TPWRS composites were reinforced by cotton fibers in order to improve properties. The effect of cotton fiber contents and lengths on properties of different TPRS and TPWRS composites was also investigated.

## Materials and Methods

### Materials

Rice starch (11.5–13.0% moisture) was obtained from Bangkok Interfood (Bangkok, Thailand), containing  $20 \pm 1\%$ wt amylose and  $75 \pm 2\%$ wt amylopectin. Waxy rice starch (11.0–13.0% moisture) was also obtained from Bangkok Interfood (Bangkok, Thailand), containing  $5 \pm 1\%$ wt amylose and  $95 \pm 1\%$ wt amylopectin. Glycerol (plasticizer) was purchased from Lab System Co. Ltd. (Thailand). Cotton fibers were obtained locally and used as-received.

### Sample Preparation

Either rice or waxy rice starch was pre-mixed with glycerol in polyethylene bags. The weight ratio of rice starch and glycerol was maintained at 2:1. Blending was carried out using a high-speed mixer (Lab-Tech Engineering, Thailand) at the temperature of 160 °C at the rotor speed of 40 rpm for 5 min in order to obtain a homogeneous material. The processed samples were then compressed at the temperature of 160 °C into 2 mm thick plates. The property modification of the TPRS and TPWRS composites was carried out using cotton fibers. Different contents of cotton fiber, i.e. 5, 10 and 15% were added into the TPRS and TPWRS matrix and two averaged different lengths of cotton fibers were used, i.e. 2.11 and 5.27 mm, corresponding to the aspect ratios of 167:1 and 417:1, respectively.

### IR Spectroscopic Study

FTIR spectra of different TPRS and TPWRS composites were recorded on a Spectrum 2000 GX spectrometer (Perkin Elmer, USA) using KBr disk technique with a resolution of  $4 \text{ cm}^{-1}$  in a spectral range of 4,000–600  $\text{cm}^{-1}$  using 16 scans per sample.

### X-Ray Diffraction

Wide angle X-ray diffraction measurements were carried out using a D8 Advance X-ray diffractor (Bruker, Madison, USA),  $\text{CuK}_\alpha$  radiation (wavelength 0.1542 nm) operating at 40 kV and 35 mA. The scattering angle ( $2\theta$ ) covered the range from 3° to 60° ( $\theta$  is the Bragg angle) with a step size of 0.02° and a sampling interval of 10 s.

### Morphology

A LEO 1455 VP scanning electron microscope (Oberkochen, Germany) was employed to study the morphology of the TPRS and TPWRS composites with and without the incorporated cotton fiber. Each tested sample was sputter-coated with a thin layer of gold to prevent electrical charge during observation and a sample was immersed into liquid nitrogen before being fractured.

### Mechanical Properties

Mechanical tests were conducted according to ASTM D-638 at the temperature of  $23 \pm 1$  °C and relative humidity of  $60 \pm 5\%$ . A mechanical measurement of each dumbbell-shaped specimen was carried out using a Universal Testing Machine (Lloyd Instrument, LR 5 K, West Sussex, UK) with a 100 N load; the crosshead speed was

maintained at 40 mm/min. It should be noted that the mechanical property results of the TPRS and TPWRS composites were obtained by averaging the measurement results of ten independent specimens.

### Water Absorption

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

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

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

### Thermal Properties

TG and DTG thermograms of a sample were recorded by a thermogravimetric analyzer (Perkin Elmer, Pyris 1, Massachusetts, USA). A TPS sample without or with cotton fiber reinforcement was tested under nitrogen atmosphere within a temperature range of 50–600 °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. Percentage weight loss was obtained from the onset decomposition temperature determined only for the starch decomposition.

### Biodegradation by Soil Burial Test

A composite sample with the dimensions of 20 × 50 mm was buried at approximately 10 cm under the soil surface. The pH and temperature of the soil were maintained at 7 and  $32 \pm 2$  °C, respectively. The water content of the soil was in the range of 30–40% and the weight change was recorded daily for 22 days. Averaged percentage weight change was recorded from three independently tested samples.

## Results and Discussion

### IR Spectroscopic Study

IR spectra of both TPRS and TPWRS composites without/with cotton fiber reinforcement display the typical profile of polysaccharide and are shown in Fig. 1a and b, respectively. The peak in the range of 1,020–1,030 cm<sup>-1</sup>

and 1,075–1,155 cm<sup>-1</sup> are attributed to C–O stretching of C–O–C group in the anhydroglucose ring and C–O stretching of C–O–H group, respectively. The wavenumbers in the range of 1,400–1,450 cm<sup>-1</sup> are assigned for O–H bonding. The peak position approximately at 1,646 cm<sup>-1</sup> is due to the bound water present in the starch. The band at 2,840–3,000 cm<sup>-1</sup> is associated with C–H stretching. A broad band due to hydrogen bonded hydroxyl group (O–H) appeared at 3,300–3,500 cm<sup>-1</sup> and is attributed to the complex vibrational stretching, associated with free, inter and intra molecular bound hydroxyl groups.

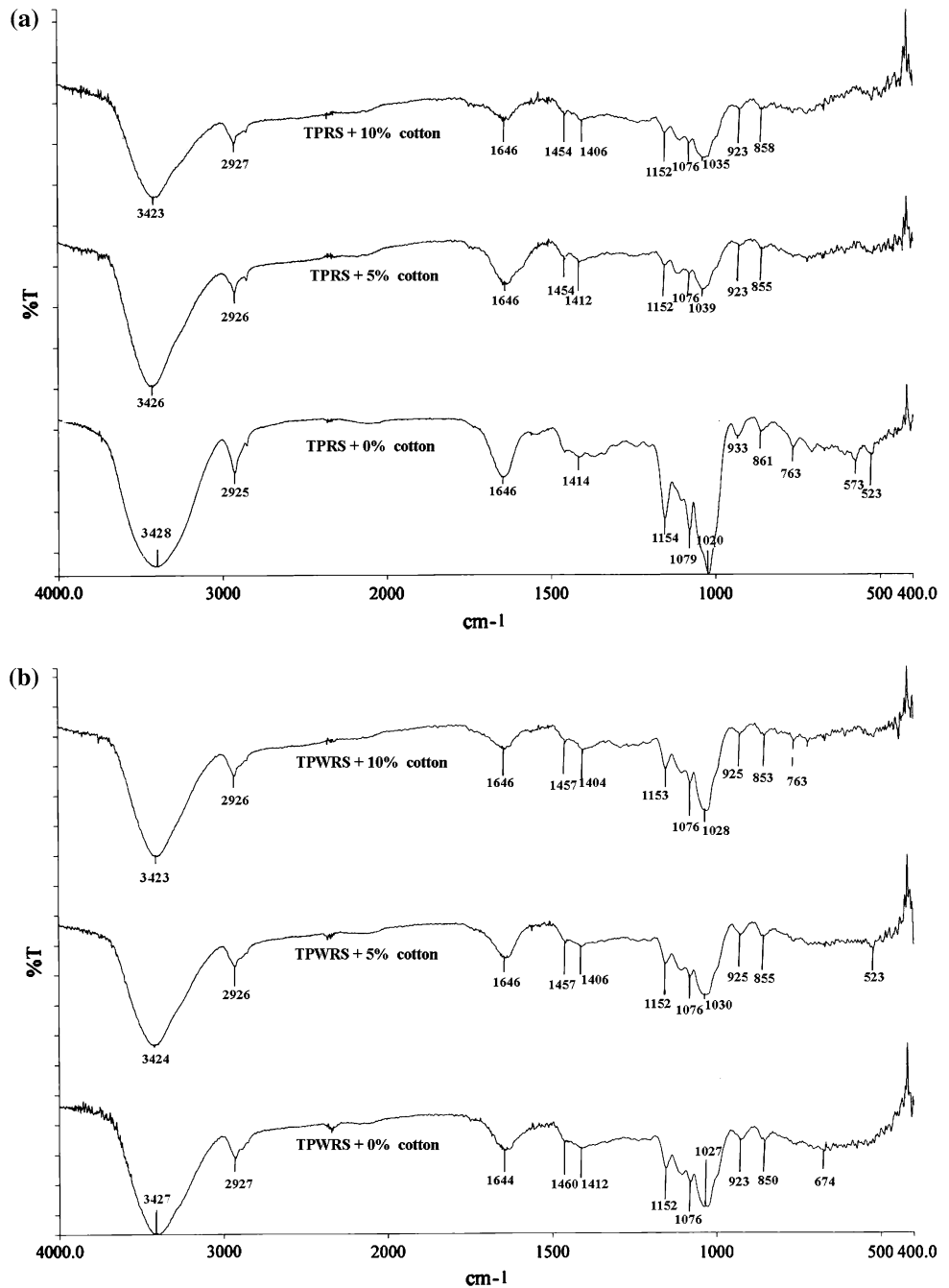
For the TPRS and TPWRS composites reinforced with cotton fibers, it can be observed that the broad band at 3,300–3,500 cm<sup>-1</sup> slightly shifted to lower wavenumber by the addition of cotton fiber. The peak position shift indicates the increasing of intermolecular hydrogen bonding by the addition of cotton fiber. It was known that, if two polymers were compatible, a distinct interaction (hydrogen bonding or dipolar interaction) existed between the chains of one polymer and those of the other, causing the IR spectra of the composites to change (e.g. band shifts, broadening) [15].

### X-Ray Diffraction

X-ray diffraction technique is used to identify crystal structure and regular molecular arrangement presented in native and processed starches. The X-ray diffraction patterns of native rice starch, TPRS, cotton fiber and its corresponding composite are shown in Fig. 2. Native rice starch (Fig. 2a) showed a typical A-type X-ray diffraction pattern, typical of cereal crystalline structure, with strong peaks at  $2\theta$  of 14.9°, 16.9° and 22.9° [16]. For the TPRS without cotton fiber (Fig. 2b), the majority of starch granules have been gelatinized and hence the glucosidic chains are retrograded into the V form. The retrograded TPRS sample without cotton fiber reinforcement gives V-type diffraction pattern with the main peaks at  $2\theta$  of 13.1° and 20.0° [17]. In cotton fiber (Fig. 2c), the main peaks were observed at  $2\theta$  of 18.0°, 22.5° and 25.7°, which are typical of cellulose I structure [18]. Figure 2d shows the diffraction pattern of the TPRS composite and the main peaks are located approximately at  $2\theta$  of 13.1°, 18.0°, 20.0°, 22.5° and 25.7°, corresponded to the diffraction patterns from both cotton fiber and TPRS components. Similar result was observed in the thermoplastic corn starch/bacterial cellulose composite [13]. The results indicate that the cotton fiber caused the increase in crystallinity in the TPRS composite.

The X-ray diffraction patterns of native waxy rice starch, TPWRS, cotton fiber and TPWRS composite are shown in Fig. 3. Native waxy rice starch (Fig. 3a) also showed a typical A-type X-ray diffraction pattern with

**Fig. 1** FT-IR spectra of the **a** TPRS and **b** TPWRS composites with different cotton fiber contents



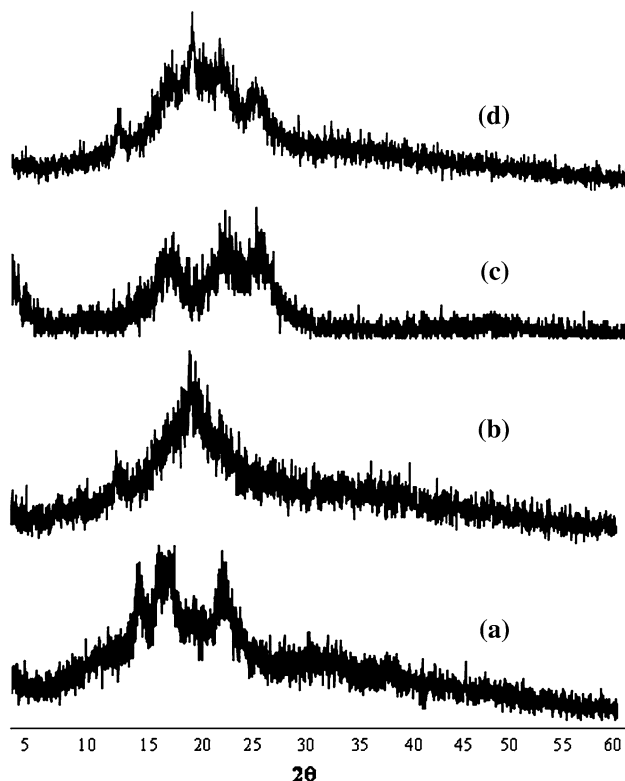
strong peaks at  $2\theta$  of  $15.1^\circ$ ,  $17.1^\circ$  and  $23.1^\circ$  [16]. The TPWRS sample without cotton fiber reinforcement (Fig. 3b) showed only main peak at  $2\theta$  of  $20.2^\circ$ . Similar to the TPRS composite, the TPWRS composite (Fig. 3d) showed the diffraction peaks from both cotton fiber reinforcement and the TPWRS matrix.

#### Morphology

Morphological structure of polymer is a very important characteristic because it determines property of polymer.

SEM micrographs of the fractured surfaces of the TPRS, TPWRS and their composites are shown in Fig. 4. Rough surfaces of the TPRS and TPWRS sample without cotton fiber reinforcement could be observed as presented in Fig. 4a and d, respectively.

For the TPRS composite reinforced by cotton fiber with 2 mm length, the SEM micrograph in Fig. 4b showed the long cotton fiber embedded into the rice starch matrix and the fiber surface was wetted by rice starch matrix, indicating of the phase compatibility. Similar morphology was found in the TPWRS composite as presented in Fig. 4e. It



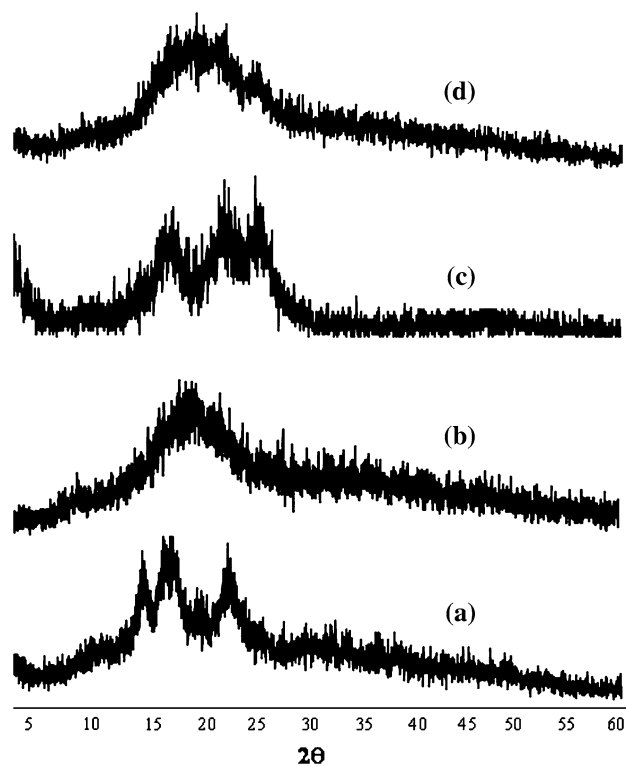
**Fig. 2** X-ray diffraction pattern of **a** native rice starch, **b** TPRS, **c** cotton fiber and **d** TPWS reinforced by cotton fiber (10%–2 mm)

should be noted that more voids could be observed in the TPWS composite. The TPRS and TPWS composites with the addition of 5 mm length cotton fiber (Fig. 4c–f) show the similar morphology as observed in the TPRS and TPWS composites with the addition of 2 mm cotton fiber (Fig. 4b–e). However, more cracks and voids can be noticed with the use of 5 mm cotton fiber.

#### Mechanical Properties

Mechanical properties of different TPRS and TPWS composites are shown in Fig. 5. It can be seen in Fig. 5 that, without cotton fiber reinforcement, the TPRS sample shows higher stress at maximum load and Young's modulus but lower strain at maximum load than those of the TPWS sample due to the greater amylose content of rice starch causing the stiffness of the starch sample. The results confirmed the earlier report by P. Myllärinen et al. [19] that amylose film showed higher stress but lower strain than amylopectin film. Similarly, the TPRS composite reinforced by cotton fiber also show higher stress at maximum load and Young's modulus but lower strain at maximum load than those of the TPWS composite.

Figure 5 also shows the relationship between cotton fiber content on the mechanical properties of different TPRS and TPWS composites. Incorporation of 5% and

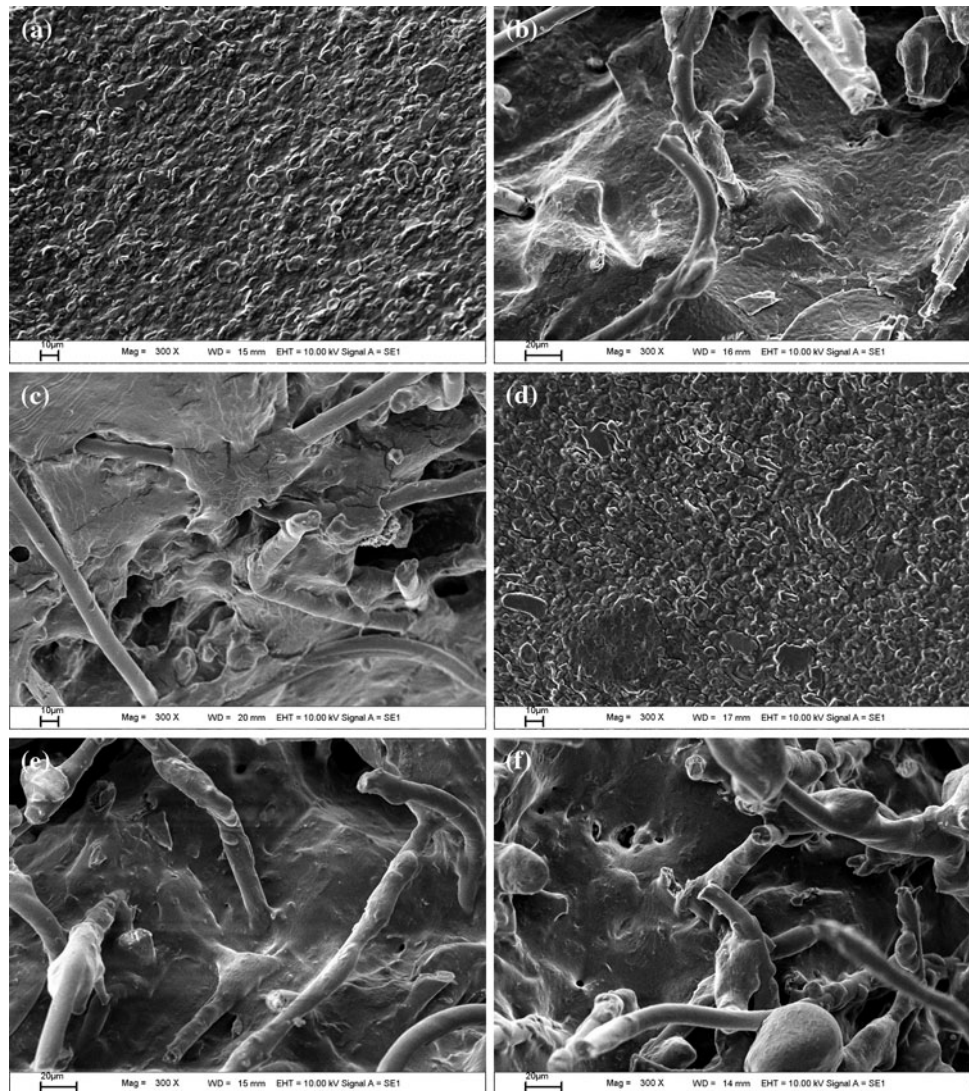


**Fig. 3** X-ray diffraction pattern of **a** native waxy rice starch, **b** TPWRS, **c** cotton fiber and **d** TPWRS reinforced by cotton fiber (10%–2 mm)

10% cotton fiber into either TPRS or TPWRS matrix caused a significant increase in the stress at maximum load (Fig. 5a) and Young's modulus (Fig. 5b). The results suggest that cotton fiber can reinforce the TPRS and TPWRS matrix due to the similar chemical structure of the fiber and the starch, the increase of hydrogen bond as shown by IR spectra in Fig. 1 and the fiber surface wetting as presented by SEM micrograph in Fig. 4. However, at 15% cotton fiber, stress at maximum load and Young's modulus tended to be maintained. Strain at maximum load also continued to decrease with the increase in the cotton fiber contents. Similar trends have been reported for corn starch or cassava starch/cellulose fibers [7, 8, 13, 14].

When different lengths of cotton fiber is considered, it was found from Fig. 5 that the addition of the 2 mm length cotton fiber into the TPRS and TPWRS composites led to greater stress at maximum load and Young's modulus than those of the 5 mm length cotton fiber due to the higher surface area of the low aspect ratio of the cotton fiber. In addition, the use of 10 mm cotton fiber caused more fiber agglomeration as shown in Fig. 4c–f, resulting in the lower stress at maximum load and Young's modulus of the TPRS and TPWRS composites. On the contrary, the addition of the 2 mm length cotton fiber into the TPRS and TPWRS

**Fig. 4** SEM micrographs of **a** TPRS, **b** TPRS with cotton fibers (10%-2 mm), **c** TPRS with cotton fibers (10%-5 mm), **d** TPWRS, **e** TPWRS with cotton fibers (10%-2 mm), and **f** TPWRS with cotton fibers (10%-5 mm)



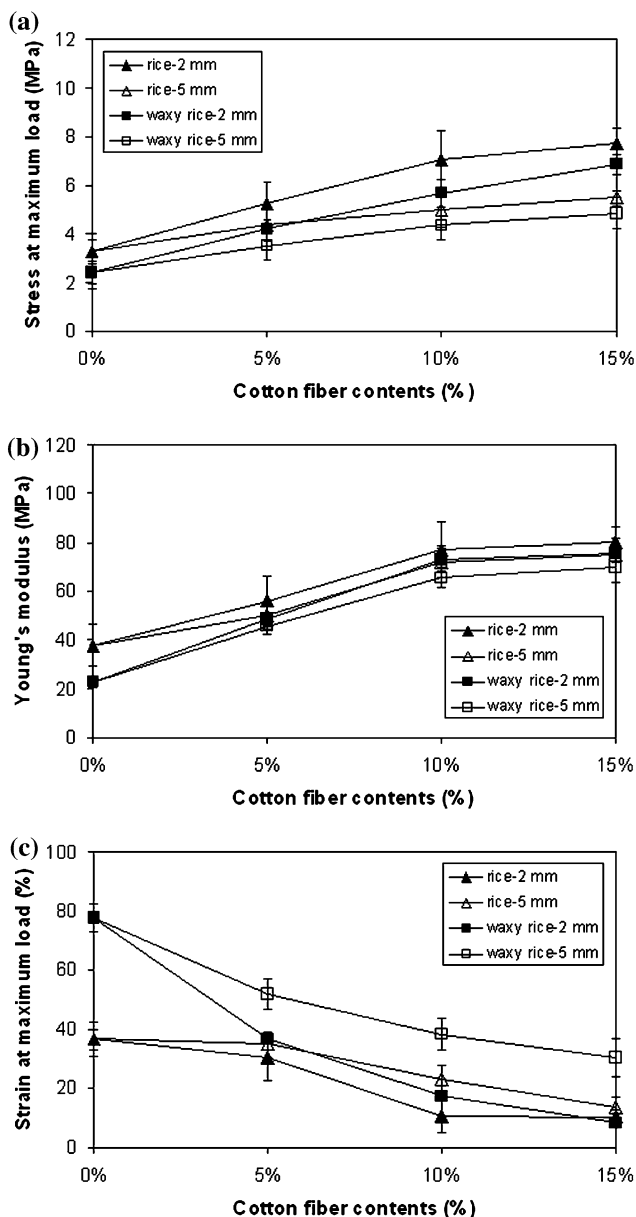
matrices led to lower strain at maximum load than those of the 5 mm length cotton fiber.

The maximum stress at maximum load and Young's modulus was found for both TPRS and TPWRS with the addition of 10 and 15% cotton fiber; however, strain at maximum load was higher in the TPRS and TPWRS with 10% cotton fiber. In this study, the maximum mechanical properties could be obtained by the use of TPRS and TPWRS matrix reinforced by 10% and 2 mm cotton fiber. The stress at maximum load increased approximately 116 and 82% and the Young's modulus increased approximately 103 and 222% for the TPRS and TPWRS composites reinforced by 10% and 2 mm cotton fibers, respectively. Nevertheless, the strain at maximum load decreased approximately 70.9 and 77.9% for the TPRS and TPWRS composites, respectively.

### Water Absorption

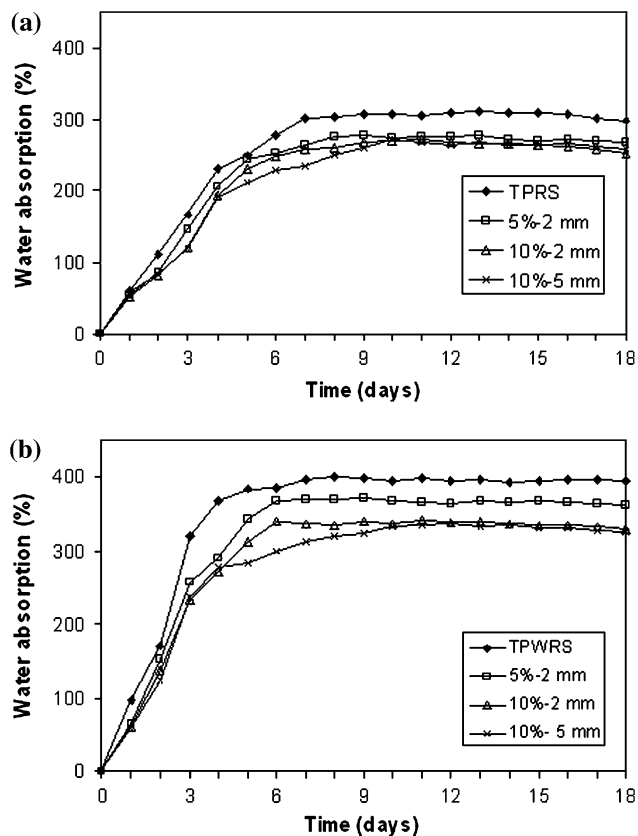
Water sensitivity is an important criterion for many applications of TPS materials. The results of water absorption experiments, performed at the temperature of 30 °C and 50% relative humidity, are shown in Fig. 6. It can be seen that the TPRS and TPWRS samples without cotton fiber reinforcement showed the increase of water absorption at the first stage, but the more rapid water absorption was found in the TPWRS sample because of the greater content of the more branched structure of amylopectin molecule. After that, the percentage water absorption tended to increase slowly and reached its maximum within 7 days for the TPRS sample and 4 days for the TPWRS sample.

The addition of cotton fiber into the TPRS and TPWRS matrices clearly caused the decrease of water absorption, as



**Fig. 5** Mechanical properties of the TPRS and TPWRS composites reinforced by different contents and lengths of cotton fibers **a** stress at maximum load, **b** Young's modulus, and **c** strain at maximum load

presented in Fig. 6. The result is caused by different absorption characteristics between the TPS and 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 the starch [18]. 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 TPRS and TPWRS matrix as shown by IR spectra in Fig. 1. It should be mentioned that water absorption of the TPRS and TPWRS composites was relatively high, compared with the water absorption of the native starch and cotton fiber. This could be because starch



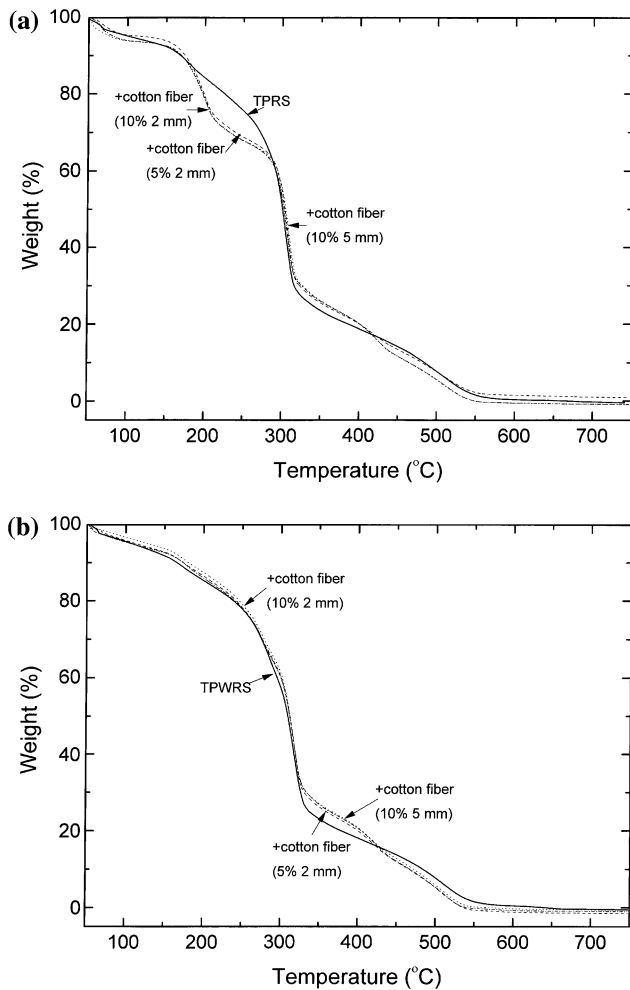
**Fig. 6** Water absorption of **a** TPRS and **b** TPWRS composites determined at 30 °C and 50% RH

granules have been gelatinized and retrograded, as shown by XRD patterns in Figs. 2, 3. The lowest water absorption was obtained from the TPRS matrix with the reinforcement of 10%wt cotton fiber. However, cotton fiber length did not show the significant effect on the water absorption of the TPRS and TPWRS composites.

#### Thermal Properties

Thermogravimetric analysis of the composite materials was carried out to assess their thermal stability and degradation temperatures. The two weight losses, at around 100 °C and 160 °C were related to the volatilization of water and glycerol, respectively (Fig. 7). The maximum degradation step at 290–310 °C is assigned to the degradation of starch [5]. Cotton fiber also showed the last degradation step at the temperature of approximately 400 °C.

It can be seen in Table 1 that the TPRS and TPWRS composites reinforced by cotton fiber promoted an increase in the onset decomposition temperature of starch. This could be described as the TPRS and TPWRS is phase compatible with cotton fiber by the increase of hydrogen bond linkages as shown in FTIR spectra in Fig. 1. The

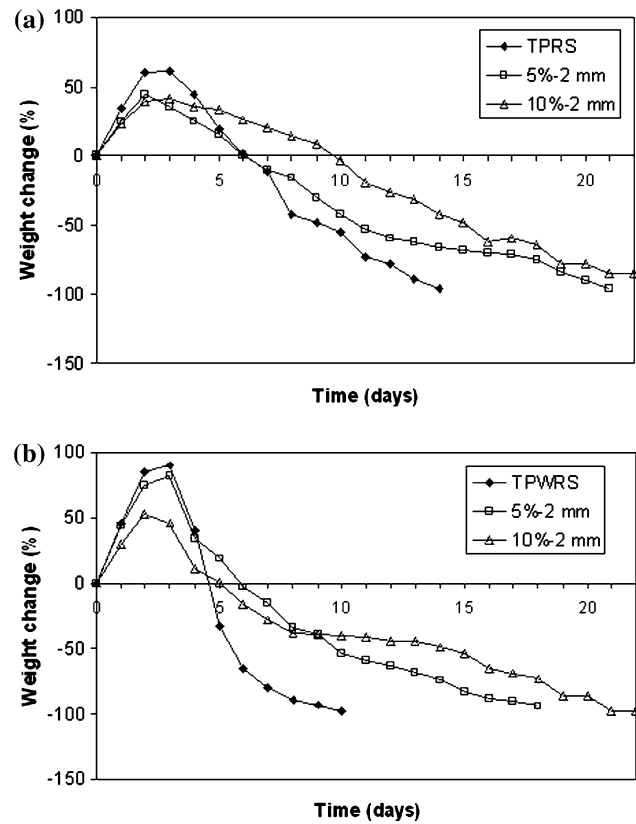


**Fig. 7** TGA thermograms of **a** TPRS and **b** TPWRS composites

addition of low amounts of cotton fibers to the TPRS and TPWRS matrix also resulted in a significant increase in the thermal stability of the composites as indicated by the decrease of percentage weight loss (Table 1), regardless of the cotton fiber contents and lengths. This can be explained by the higher stability of the cotton fibers and particularly by the good compatibility between the two carbohydrate components of the composites [4, 5]. It should be noted

**Table 1** Onset degradation temperatures and percentage weight losses of different TPRS and TPWRS composites

Samples	Onset degradation temperature ( °C)			Weight loss (%)
	Step 1 (glycerol)	Step 2 (starch)	Step 3 (fiber)	
TPRS	165.2	294.3	–	59.8
+5%-2 mm	173.5	296.3	400.5	44.9
+10%-2 mm	174.9	297.9	400.7	44.3
+10%-5 mm	170.4	301.0	403.4	48.7
TPWRS	145.5	292.5	–	65.8
+5%-2 mm	150.4	294.9	398.0	63.0
+10%-2 mm	158.7	295.0	407.3	61.7
+10%-5 mm	159.8	295.6	400.2	60.3



**Fig. 8** Weight changes of **a** TPRS and **b** TPWRS composites under soil burial test

that a strong shoulder in the thermogram existed at the temperature range of 150–200 °C was observed only in the TPRS composites. The observed strong shoulder in the thermograms could be probably because of different amounts of non-absorbed glycerol by cotton fiber and starch. This observation was also reported in the TPS/Eucalyptus composite prepared from corn starch [5].

### Biodegradation

Biodegradable properties of different TPRS and TPWRS composites can be determined by soil burial test. Figure 8



showed the percentage weight change of different TPRS and TPWRS composites. All of the composites, showed the increment in weight after a few days of soil immersion due to water absorption from the soil. The result was related to the water uptake as shown in water absorption property in Fig. 6. After that, the sample weight tended to decrease and then the starch sample started to degrade by micro-organisms existed in soil. Especially, the TPWRS sample without cotton fiber reinforcement degraded within 10 days, but the TPRS sample without cotton fiber reinforcement degraded within 14 days. Regardless of starch type, the percentage weight change in the TPRS and TPWRS composites reinforced by cotton fiber was lower than that of the TPRS and TPWRS samples without cotton fiber reinforcement and the TPRS and TPWRS composites degraded within approximately 20 days. This is due to the phase compatibility of the TPRS and TPWRS matrix and cotton fiber reinforcement.

## Conclusions

Thermoplastic starch could be prepared from rice and waxy rice starches. It was found that, without cotton fiber reinforcement, the TPRS sample (higher content of amylose) showed higher stress at maximum load, Young's modulus, lower water absorption, higher thermal stability and slower biodegradation in soil than the TPWRS sample (lower content of amylose). For both TPRS and TPWRS composites, mechanical properties increased; however, water absorption decreased by the cotton fiber reinforcement. Thermal degradation temperature and thermal stability of the TPRS and TPWRS composites were also improved by reinforcing with cotton fibers. For biodegradable property, cotton fibers could extend the decay of the TPRS and TPWRS matrices. Cotton fiber contents and lengths greatly affected only the mechanical properties of both TPRS and TPWRS composites. The best optimum properties were

obtained from the TPRS with 10% and 2 mm cotton fiber reinforcement.

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