

# Tensile Strength Characteristics of Biodegradable Plastics Made from Sago Starch-Extraction Residue

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**Abstract** Biodegradable plastics were applied for the effective use of sago starch-extraction residue, which has so far been treated as waste. Sago starch-extraction residue and palm oil were reacted for esterification in the presence of an acid catalyst at 160°C and 2 hours. Unreacted oil was removed to make the ester by mixing ethanol and hexane (9:1), and esterified plastics were thus obtained. Thermoplasticity of plastics, which was characterized as plastics, was added to the original (untreated) sago starch-extraction residue by esterification. Biodegradable plastics made from sago starch-extraction residue and palm oil (P-SP) were evaluated on the basis of their esterification degree according to their saponification, thermal softening point, and tensile strength. When the acid catalyst concentration increased from 0 to 1 mol l<sup>-1</sup> HCl, the esterification degree of P-SP tended to increase gradually. The esterification degree of P-SP, which was producing by using 1 mol l<sup>-1</sup> acid catalyst concentration, was 3.23 mmol g<sup>-1</sup>. The thermal softening points of P-SP (1 mol l<sup>-1</sup> HCl) and the untreated sago starch-extraction residue were almost 220 and 260°C, respectively. In particular, the thermal softening point decreased down to 40°C by esterification in this study. The tensile strength value of P-SP, which ranged from 2.59 to 5.86 MPa, showed a lower value than those of commercial biodegradable plastics. It is concluded that the tensile strength of P-SP must be necessary to improve before P-SP can be used to make plant seedling pots. P-SP will be a useful biodegradable plastic for use in the manufacture of plant seedling pots in tropical areas, where sago palm and oil palm grow well.

**Key words:** esterification, sago residue, thermoplasticity, tensile strength

## サゴヤシ澱粉抽出残渣から製造した生分解性プラスチックの引張強度特性

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**要約** 廃棄物として扱われているサゴヤシ澱粉抽出残渣を有効利用するために生分解性プラスチックを製造した。サゴヤシ澱粉抽出残渣とパーム油を酸触媒存在下で160°C、2時間反応させ、エステル化を行った。作製したエステル化物からエタノールとヘキサンの混合液 (9 : 1) を用いて未反応油を除去し、エステル化プラスチックを得た。エステル化によりプラスチックの特性である熱可塑性が付加され、原材料 (未処理) のサゴヤシ澱粉抽出残渣と比較して熱可塑性は増加した。サゴヤシ澱粉抽出

残渣とパーム油から製造した生分解性プラスチック (P-SP) をケン化によるエステル化度、熱軟化点そして引張強度に基づいて評価した。酸触媒 (塩酸) 濃度が0から1 mol l<sup>-1</sup>に上昇するにつれてP-SPのエステル化度は徐々に上昇する傾向を示した。1mol l<sup>-1</sup> 塩酸を触媒として用いて製造したP-SPのエステル化度は3.23 mmol g<sup>-1</sup>であった。P-SP (1 mol l<sup>-1</sup> 塩酸) と未処理サゴヤシ澱粉抽出残渣の熱軟化点は、それぞれ約220、260℃であり、本研究ではエステル化反応により、特に熱軟化点が40℃低下した。P-SPの引張強度は2.59から5.86 MPaであり、市販の生分解性プラスチックより低い値を示した。P-SPの育苗ポットへの適応には引張強度の改善が必要と考えられたが、サゴヤシと油ヤシが良好に生育可能な熱帯地域において、P-SPは育苗ポットに適した有用な生分解性プラスチックの一つとなるであろう。

キーワード エステル化、サゴヤシ澱粉抽出残渣、熱可塑性、引張強度

## Introduction

Plastics derived from petroleum are used for many purposes. The annual worldwide production of plastic in 2004 was 224 million tons. Plastics production in Japan in 2004 was 14.08 million tons (Japan Plastics 2006). The source of most plastics is petroleum. These plastics have caused environmental pollution. For example, burning plastic produces air pollution, which has a negative effect on human health. They are usually hard to degrade or don't degrade in natural conditions. For this reason, the manufacture and use of biodegradable plastics are important.

Sago palm (*Metroxylon sagu*) grows in the lowland of tropical areas of Southeast Asia with 10 degrees north and south of the equator. It accumulates more than 200 kg starch per palm in its stem, and the starch is used as a food staple. Its biomass, including bark, leaves, and pith, is also useful. It is expected that sago is one of the useful plants which has potential for solving food crises and for contributing to economic development in tropical wetland countries. When sago starch is processed, large quantities of agricultural waste accumulate, and effective means of disposal have not been established.

Many researchers, including Funakoshi et al. (1979), Kiguchi (1990), Mohammad et al. (1999), and Shiraishi (1983), have reported on the production of biodegradable plastics from plant residue. These plastics were estimated by the characteristics such as mechanical and thermal properties. These plastics

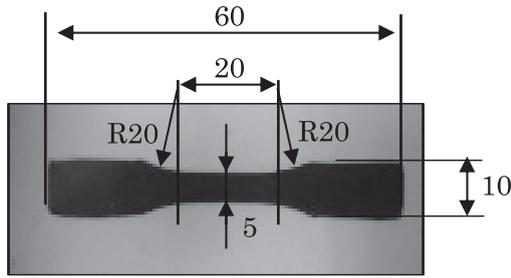
were evaluated by their mechanical and thermal properties (Japanese Standards Associations 2003).

The objectives of this research are to produce biodegradable plastics made from sago starch-extraction residue and to investigate the physical properties of biodegradable plastics, including the esterification degree, thermal softening point and tensile strength for use of these plastics in the manufacture of fast-biodegradable seedling pots.

## Materials and Methods

### *Production of biodegradable plastics made from sago starch-extraction residue*

The sago starch-extraction residue was cut into small fragments by a grinder (Kiya Seisakusho, Ltd.). To remove the lipid from the residue, a mixture of ethanol and benzene (1 : 2) was refluxed using a Soxhlet's extractor for 24 hours. Ten g of residue was esterified with 120 g of palm oil with 7.5 ml of 0 to 1 mol l<sup>-1</sup> HCl using an oil bath at 160°C for 2 hours, and the unreacted oil was removed using a mixture of ethanol and hexane (9 : 1) (Sasaki, 1999). The sago biodegradable plastics made from palm oil were called P-SP. P-SP was molded into a dumbbell shape with a hot press under 215 kPa at 140 for 1 hour with 1, 2, 3-propanetriol triacetate (triacetin) (50 % ( g triacetin / g P-SP × 100)). Produced P-SP powder was used to measure the esterification degree, thermal degradation point and thermal softening point. On the other hand, a dumbbell shape of P-SP was used for the tensile strength test (Fig. 1).



**Fig. 1.** Dumbbell-shaped specimen of P-SP (1 mol l<sup>-1</sup> HCl)  
Unit:mm; Thickness: 1mm; R: radius

### Measurement of esterification degree (*Ed*)

A mixture of 0.3 g of P-SP and 25 ml of 0.5 mol l<sup>-1</sup> KOH (ethanol solution) was put into a flask attached to a reflux condenser and heated at 60°C in a water bath for 1 hour. After cooling, 25 ml of 0.5 mol l<sup>-1</sup> HCl and phenolphthalein were added in the flask. The amounts of fatty acids that were generated by saponification were determined by neutralization titration with 0.5 mol l<sup>-1</sup> NaOH (Sasaki 2003). The esterification degree was calculated as follows:

$$Ed = \frac{0.5 \times f \times (N_1 - N_0)}{W},$$

where *Ed* is the esterification degree (mol g<sup>-1</sup>); *N*<sup>0</sup>, the amount of 0.5 N NaOH for titration for blank (ml); *N*<sup>1</sup>, the amount of 0.5 N NaOH for titration (ml), and *W*, the mass of the sample (g).

### Thermal characteristics of P-SP

#### 1) Thermogravimetry (TG)

The thermal gravimetric behavior, including the thermal degradation, of P-SP (used with 1 mol l<sup>-1</sup> HCl as the acid catalyst), P-SP (0.5 mol l<sup>-1</sup> HCl), and the untreated (original) sago starch-extraction residue was evaluated from room temperature to 600°C using a thermal gravimetric analyzer (SHIMADZU TG-20) (Watanabe et al. 1997). The P-SP and untreated sago starch-extraction residue samples were dried in a vacuum drying oven at 60°C for 24 hours. After sieving using a 60-mesh sieve, about 10 mg of the dried P-SP and sago starch-extraction residue samples were used for the measurement of the thermogravimetry. The measurement was performed

under a helium atmosphere (50 ml min<sup>-1</sup>) at a temperature increase rate of 10°C min<sup>-1</sup>, and the TG curve of each sample was obtained. The thermal degradation point was obtained from the intersection of the baseline and tangent for the TG curve.

#### 2) Thermal softening point

The thermal softening points (°C) of P-SP (1 mol l<sup>-1</sup> HCl), P-SP (0.5 mol l<sup>-1</sup> HCl), and the sago starch-extraction residue were investigated by means of a penetration test using a thermal mechanical analyzer (Sasaki 2003). The P-SP and untreated sago starch-extraction residue samples were dried in a vacuum drying oven at 60°C for 24 hours. After sieving through a 60-mesh sieve, the sample was filled into a glass tube (ϕ 7mm) and installed in the mantle heater. A glass pin (ϕ 4mm) was installed vertically on the sample, and penetration of the pin due to a rise in the temperature was measured using a dial gauge. The measurement was performed under the air at a temperature increase rate of 3°C min<sup>-1</sup>, and the thermal softening curves of each sample were obtained. The thermal softening point was calculated from the intersection of the baseline and tangent for the thermal softening curve.

The penetration (%) was calculated as follows:

$$Penetration (\%) = \frac{P}{h} \times 100,$$

where *P* is the distance of penetration of the pin into the sample (cm) and *h* is the height of the sample which was filled in a glass tube (cm).

#### Differential scanning calorimetry (DSC)

The differential scanning calorimetry (DSC) was performed using a differential scanning calorimeter (SHIMADZU DSC-50). Three to four mg each of the dried P-SP (1 mol l<sup>-1</sup> HCl), P-SP (0.5 mol l<sup>-1</sup> HCl), and sago starch-extraction residue were added into an aluminum DSC pan, and a standard reference pan (not containing a sample) was also prepared. These pans were heated from room temperature to 600°C. The measurement was performed in a helium atmosphere with 20 to 30 ml min<sup>-1</sup> at a temperature increase rate

of 10 °C min<sup>-1</sup>, and a DSC curve of each sample was obtained.

### Tensile strength of P-SP

A tensile testing apparatus, as shown in Figure 2, was used to determine the tensile strength of P-SP and commercial biodegradable plastics at room temperature. Three kinds of P-SP (P-SP (1 mol l<sup>-1</sup> HCl), P-SP (0.75 mol l<sup>-1</sup> HCl), and P-SP (0.5 mol l<sup>-1</sup> HCl)) and three kinds of commercial biodegradable plastics, Polycaprolactone + Polybutylene succinate adipate (PCP) (Tokai Kasei Corporation, Japan), Starch (ST) (Chubu Nozai Co., Ltd., Japan), and Starch + Polycaprolactone (SPC) (Tokai Kasei Corporation, Japan), were used for the tensile strength test. Five replications for each sample of the tensile strength value were performed. Corn was the source of the starch used for commercial biodegradable plastics (ST). The tensile strength (MPa) was calculated as follows (JIS K 7176 1994):

$$\sigma = \frac{F}{A},$$

where  $\sigma$  is tensile strength (MPa), F, the load (N), and A, the initial cross section of the specimen (mm<sup>2</sup>).

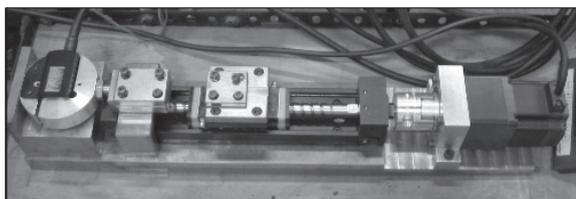


Fig. 2. Tensile testing apparatus

## Results

### Appearance of P-SP

The appearance of biodegradable plastics is important for commercial use. Table 1 shows the color of the sago starch-extraction residue and biodegradable plastics (P-SP) resulting from the use of different acid catalyst concentrations. The color of the

original sago starch-extraction residue was light yellow-orange (7.5YR 8/3) (Oyama and Takehara 2001) to dull orange (7.5YR 7/4). On the other hand, the color of P-SP changed from dull orange (P-SP (non catalyst)) to black (P-SP (1 mol l<sup>-1</sup> HCl)) when the acid catalyst concentration increased. P-SP was bulkier than the unreacted sago starch-extraction residue. Dumbbell-molded P-SP, which was made without HCl (P-SP (non catalyst)), collapsed easily, and the surface was rough. However, the surface of P-SP (1 mol l<sup>-1</sup> HCl) was smoother.

Table 1. Color of untreated sago starch-extraction residue and sago biodegradable plastics made from palm oil (P-SP) with 0 to 1 mol l<sup>-1</sup> HCl by esterification

Sample	Color
Untreated*	dull orange (7.5 YR 7/4)
P-SP (non catalyst)	dull orange (7.5 YR 6/4)
P-SP (0.25 mol l <sup>-1</sup> HCl)	dull orange (7.5 YR 5/4)
P-SP (0.5 mol l <sup>-1</sup> HCl)	brown (7.5 YR 4/4)
P-SP (0.75 mol l <sup>-1</sup> HCl)	dark brown (7.5 YR 3/4)
P-SP (1 mol l <sup>-1</sup> HCl)	black (7.5 YR 1.7/1)

\* Untreated: untreated sago starch-extraction residue.

### Esterification degree (Ed) of P-SP

Table 2 shows the amounts of 0.5 mol l<sup>-1</sup> NaOH to neutralize the fatty acids generated by saponification. The esterification degree was higher in all reaction products (P-SP) than in the untreated sago starch-extraction residue. When the acid catalyst concentration increased, the esterification degree of P-SP tended to increase gradually. However, the esterification degree of P-SP (1 mol l<sup>-1</sup> HCl) was less than that of P-SP (0.75 mol l<sup>-1</sup> HCl).

Table 2. Changes in the esterification degree of P-SP when using different acid catalyst concentrations

Sample name	Acid catalyst (HCl) concentration mol l <sup>-1</sup>	Esterification degree (Ed) mmol g <sup>-1</sup>
P-SP (non catalyst)	0.00	1.82
P-SP (0.25 mol l <sup>-1</sup> HCl)	0.25	2.53
P-SP (0.5 mol l <sup>-1</sup> HCl)	0.50	2.60
P-SP (0.75 mol l <sup>-1</sup> HCl)	0.75	3.67
P-SP (1 mol l <sup>-1</sup> HCl)	1.00	3.23
Untreated*	—	1.84

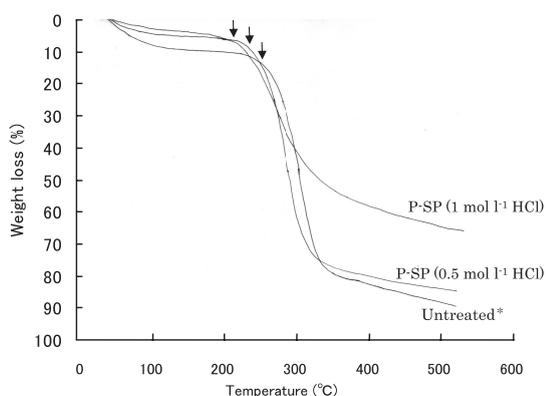
\* Untreated: untreated sago starch-extraction residue.

The esterification degree was calculated by the amount of 0.5 mol l<sup>-1</sup> NaOH used for the titration divided by the sample weight.

## Thermal characteristics of P-SP

### 1) Thermogravimetry (TG)

Figure 3 shows the thermogravimetric curves for PSP (1 mol l<sup>-1</sup> HCl), P-SP (0.5 mol l<sup>-1</sup> HCl), and the sago starch-extraction residue, and Table 3 shows the thermal degradation point of these samples. The thermal degradation points of P-SP (1 mol l<sup>-1</sup> HCl), PSP (0.5 mol l<sup>-1</sup> HCl), and the untreated sago starch-extraction residue were almost 228, 248, and 260°C, respectively (arrow in Fig. 3). When the esterification degree in Table 1 increased, the thermal degradation points of the samples tended to decrease gradually.



**Fig. 3.** Thermogravimetric curves of P-SP (1 mol l<sup>-1</sup> HCl), P-SP (0.5 mol l<sup>-1</sup> HCl), and the sago starch-extraction residue.  
\*Untreated: untreated sago starch-extraction residue.  
Arrow: thermal degradation point.

**Table 3.** Thermal degradation point of P-SP

Sample	Thermal degradation point (°C)	SD**
Untreated*	259.6	0.57
P-SP (0.5 mol l <sup>-1</sup> HCl)	248.3	0.35
P-SP (1 mol l <sup>-1</sup> HCl)	228.3	1.06

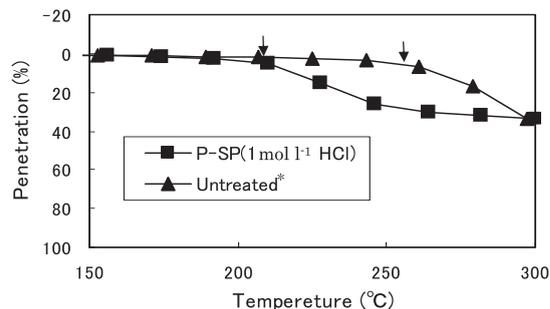
\* Untreated: untreated sago starch-extraction residue.

\*\* SD: standard deviation.

The thermal degradation point was obtained from the intersection of the baseline and tangent for the thermogravimetric curve.

### 2) Thermal softening point

Figure 4 shows the thermal softening curve for P-SP (1 mol l<sup>-1</sup> HCl) and the untreated sago starch-extraction residue, and Table 4 shows the thermal softening points of P-SP (1 mol l<sup>-1</sup> HCl), P-SP (0.5 mol l<sup>-1</sup> HCl), and the untreated sago starch-extraction residue. The thermal



**Fig. 4.** Thermal softening curves of P-SP (1 mol l<sup>-1</sup> HCl) and the untreated sago starch-extraction residue.  
\*Untreated: untreated sago starch-extraction residue.  
Arrow: thermal softening point.

softening points for P-SP (1 mol l<sup>-1</sup> HCl), P-SP (0.5 mol l<sup>-1</sup> HCl), and the sago starch-extraction residue were around 220, 240, and 260°C, respectively (arrow in Fig. 4). The thermal softening points for P-SP (1 mol l<sup>-1</sup> HCl) and P-SP (0.5 mol l<sup>-1</sup> HCl) were lower than that for the untreated sago starch-extraction residue. Consequently, the thermal softening point decreased as a result of the esterification, and, when the esterification degree increased, the thermal softening point of the sample tended to decrease.

Furthermore, the thermal softening points of P-SP (1 mol l<sup>-1</sup> HCl) and P-SP (0.5 mol l<sup>-1</sup> HCl) occurred at lower temperature than thermal degradation point. This result revealed that thermal softening behavior of P-SP occurred before P-SP was carbonized by heating.

Therefore, both P-SP (1 mol l<sup>-1</sup> HCl) and PSP (0.5 mol l<sup>-1</sup> HCl) showed slight thermal softening behavior, which is common in plastics. On the other hand, the thermal softening point and thermal degradation point of the untreated sago starch-extraction residue occurred at similar temperatures. Therefore, the sago starch-extraction residue did not show any of the characteristics of plastics.

**Table 4.** Thermal softening point of P-SP

Sample	Thermal softening point (°C)	SD**
Untreated*	259.3	1.91
P-SP (0.5 mol l <sup>-1</sup> HCl)	238.5	14.2
P-SP (1 mol l <sup>-1</sup> HCl)	220.1	8.11

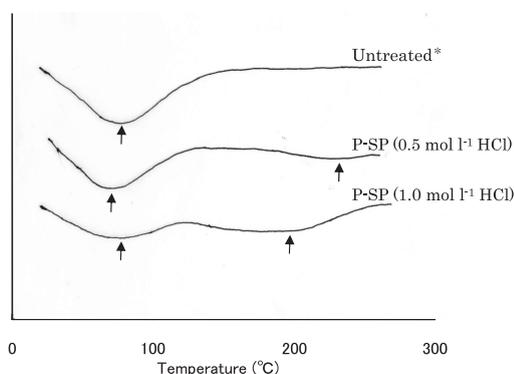
\* Untreated: untreated sago starch-extraction residue.

\*\* SD: standard deviation.

The thermal softening point was obtained from the intersection of the baseline and tangent for the thermal softening curve.

### Differential scanning calorimetry (DSC)

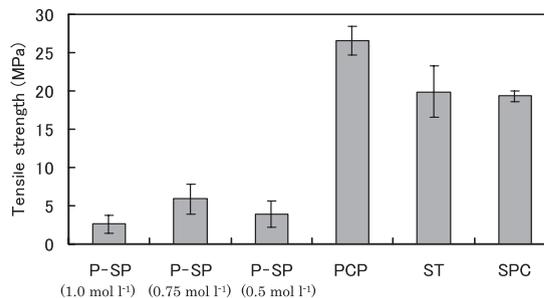
Figure 5 shows the DSC curves of P-SP (1 mol l<sup>-1</sup> HCl), P-SP (0.5 mol l<sup>-1</sup> HCl), and the untreated sago starch-extraction residue. All samples showed an endothermic peak at about 90°C. This peak was derived from the evaporation of water. Furthermore, P-SP (1 mol l<sup>-1</sup> HCl) showed an endothermic peak at about 130 to 270°C, and P-SP (0.5 mol l<sup>-1</sup> HCl) showed it at about 200 to 270°C. It is suspected that these peaks are derived from the thermal molecule motion of the P-SP, which is affected by the esterification degree. However, the endothermic peak area of P-SP (1 mol l<sup>-1</sup> HCl) and P-SP (0.5 mol l<sup>-1</sup> HCl) was different.



**Fig. 5** DSC curves of P-SP (1 mol l<sup>-1</sup> HCl), P-SP (0.5 mol l<sup>-1</sup> HCl), and the unreacted sago starch-extraction residue. \*Untreated: untreated sago starch-extraction residue. Arrow: endothermic peak.

### Tensile strength of P-SP

Figure 6 shows the tensile strength values of three kinds of P-SP samples, P-SP (1 mol l<sup>-1</sup> HCl), P-SP (0.75 mol l<sup>-1</sup> HCl), and P-SP (0.5 mol l<sup>-1</sup> HCl), and three kinds of commercial biodegradable plastics. The tensile strength values of P-SP (0.5 mol l<sup>-1</sup> HCl), P-SP (0.75 mol l<sup>-1</sup> HCl), and P-SP (1 mol l<sup>-1</sup> HCl) were 3.86, 5.86, and 2.59 MPa, respectively. Each P-SP sample had an almost identical tensile strength value. However, the tensile strength values of commercial biodegradable plastics (PCP, ST, and SPC) were 26.6, 19.9, and 19.3 MPa, respectively. Consequently, the tensile strength value of P-SP was lower than those of commercial biodegradable plastics. The P-SP samples used in this study did not have large differences in tensile strength as a result of esterification.



**Fig. 6** Tensile strength value of P-SP samples obtained using different acid catalyst concentrations (0.5, 0.75, and 1 mol l<sup>-1</sup> HCl) and commercial biodegradable plastics, Polycaprolactone + Polybutylenesuccinate adipate (PCP), Starch (ST), and Starch + Polycaprolactone (SPC). Bar: standard deviation (n = 5).

### Discussion

#### Plasticization of sago starch-extraction residue by esterification

For esterification reaction, the acid catalysts stimulated not only the esterification reaction but also the hydrolysis of ester. Therefore, a slight decrease in the esterification degree of P-SP (1 mol l<sup>-1</sup> HCl) compared with P-SP (0.75 mol l<sup>-1</sup> HCl) seemed to be derived from the hydrolysis of ester by increasing the concentration of the acid catalyst. The thermal softening point of P-SP showed a tendency to decrease with the esterification degree. Funakoshi et al. (1979) reported the thermal melting point of esterified cellulose (lauroyl cellulose), and the thermal softening point of lignin decreased with increasing the ester content. The thermal softening points of P-SP (1 mol l<sup>-1</sup> HCl) and P-SP (0.5 mol l<sup>-1</sup> HCl) took place at slightly lower temperatures than those of the thermal degradation point and the thermal softening point of the original sago starch-extraction residue; thus, both P-SP (1 mol l<sup>-1</sup> HCl) and P-SP (0.5 mol l<sup>-1</sup> HCl) showed thermal softening behavior as plastics.

For DSC analysis, P-SP (1 mol l<sup>-1</sup> HCl) and P-SP (0.5 mol l<sup>-1</sup> HCl) showed an endothermic reaction from about 130 to 270°C and about 200 to 270°C, respectively. These endothermic reactions were derived from the thermal softening behavior and the temperature range of these endothermic reactions seemed to be affected by esterification degree.

Endothermic reactions, such as melting and thermal degradation, occur as a result of the heating process (Saito 1990). Wunderlich (1964) reported that the endothermic reaction is thought to involve a librational motion. Therefore, the endothermic peaks observed in this research were derived from the thermal molecular motion of P-SP.

### *Tensile strength of P-SP*

The tensile strength of P-SP was about 2.5 to 6 MPa, which was lower than that of commercial biodegradable plastics (about 19 to 27 MPa). Kani et al. (1998) reported that the tensile strength of commercial biodegradable plastics used in their research was about 20 to 70 MPa. The improvement of the tensile strength should be necessary to produce plant seedling pots from P-SP.

The density of commercial biodegradable plastics (PCP, ST, and SPC) calculated from the surface area, thickness, and weight of each specimen was 1.10, 1.02, and 0.93 g cm<sup>-3</sup>, respectively, and the tensile strength of commercial biodegradable plastics showed a tendency to decrease with decreasing the density of the specimen. The density of P-SP (1 mol l<sup>-1</sup> HCl), PSP (0.75 mol l<sup>-1</sup> HCl), and P-SP (0.5 mol l<sup>-1</sup> HCl) was 0.81, 0.82, and 0.86 g cm<sup>-3</sup>, respectively. Common commercial plastics derived from petroleum, such as polystyrene and polypropylene, have the density of 1.04 to 1.05 and 0.9 to 0.91 g cm<sup>-3</sup>, respectively (Plastics Processing Databook, 1988). When compared with the density of common commercial plastics, the P-SP produced in this study showed low density. Sasaki (2003) reported that the main component of P-SP was cellulose. Cellulose has been used as a filler or reinforcement in plastics (JIS K 6899-2). Consequently, improvement of the tensile strength of P-SP can be expected by increasing the amount of the filling of P-SP and thus the density of P-SP.

An objective of this research was to produce biodegradable plastics made from sago starch-extraction residue for use in the manufacture of fast-biodegradable plant seedling pots. Therefore, it is

necessary to improve the tensile strength of P-SP to about 20 MPa, which is the lowest value of tensile strength of commercial biodegradable plastics, before P-SP can be used in the manufacture of plant seedling pots.

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