

## Study on Properties of Polymer Blends from Polypropylene with Polycaprolactone and Their Biodegradability

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**ABSTRACT:** The main problem in preparation of polymer blends is compatibility between polymers mixtures with different properties. This paper describes properties of polymer blends prepared from modified polypropylene (PP) and poly- $\epsilon$ -caprolactone (PCL). The preparation of these polymer blends involves modification of PP by oxidation with hydrogen peroxide. A part of the polymer blends consisting oxidized PP and PCL was compatible polymer blends, and the highest compatible polymer blends was obtained in the PP/PCL ratio of 70/30 (% w/w) with low crystallinity. The mechanical properties of polymer blends decreased with increasing of PCL proportion in the polymer blends. Enzymatic degradability of PP-PCL polymer blends were lower than that of PCL, and increased slowly with increasing of PCL content in the polymer blends. The use of oxidized PP in preparation of polymers blends with PCL effectively improves the degradability of the polymer blends due to the increase of compatibility of each component in polymer blends. Furthermore, biodegradation process might occur through hydrolysis of PCL and compatible parts of polymer blends to result in hydroxyl and carboxylate functional groups in the end of polymer chain.

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**KEY WORDS** Polypropylene / Poly- $\epsilon$ -caprolactone / Polymer Blends / Compatibility / Biodegradation /

Increasing volumes of synthetic polymers are manufactured for various applications, mostly for packaging materials. Disposal of the used polymers has been accumulated under the land surface. Unlike natural polymers, most synthetic polymers can not be decomposed by microorganisms, hence the landfill approach becomes inefficient, and other plastics waste management should be found. A study of the biodegradation properties of synthetic polymers has become very important. New techniques have been developed to obtain environmentally friendly polymers. A modification of non-biodegradable synthetic polymers with a biodegradable polymer producing environmentally friendly polymers is an established technique to solve this problem. The main problem in modification of non-biodegradable synthetic polymers with biodegradable polymers is compatibility between polymers mixtures with different properties.

Polypropylene (PP) is a thermoplastic polymers produced commercially as plastic materials and difficult to be decomposed by microorganisms in environment (1). Despite its many commercial application, isotactic polypropylene suffers a major deficiency that is poor interaction with other materials. Furthermore, attempts to blend polypropylene with other polymers have been unsuccessful for much the same reasons, *i.e.*, incompatibility of the two polymers. It has been demonstrated that the addition of polar groups to a

polyolefin can improve adhesion of polyolefin to many materials (2). PP forms very poor blends with other polymers because it has no chemical functionalities. Without any specific interactions, PP is unlikely to form miscible blends or even to adhere to other polymers (3). There are several examples of PP blended with polyesters (4,5). Typically when PP is blended with the liquid crystalline polyester (LCP), the mechanical strength is improved due to fiberlike LCP phases in the flow direction. By addition of some maleic anhydride modified PP in the melt blending of PP and LCP, impact and tensile strength of blends significantly improved (4). Blends of PP and poly(ethylene terephthalate) (PET) have likewise been improved by the addition of acrylic acid modified PP (5). Therefore, the presence of chemical functionalities such as carbonyl and hydroxyl groups would be useful to increase interaction such components in a polyblend.

The biodegradable synthetic polymers being developed so far are aliphatic polyesters, such as poly- $\epsilon$ -caprolactone (PCL) (6,7). PCL has a low melting point (60 °C), high crystallinity and brittleness, which limit its practical application (8). However, PCL is very attractive due to a valuable set of properties, such as a high permeability, the lack of toxicity, biodegradability, and a capacity to be blended with various commercial polymers over a wide composition

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range (9,10). PCL can be easily prepared by ring-opening polymerization of  $\epsilon$ -caprolactone monomer in the presence of distannoxane derivatives catalyst (7,11,12).

The aim of the experiments is designed to improve the properties of PP as well as its biodegradability by blending with polyesters. In the previous study have been studied structure and properties of polymer blends between PP and synthetic poly-(R,S)- $\beta$ -hydroxybutyrate (PHB) (13). The main problem in preparation of these polymer blends is compatibility between polymers mixtures with different properties. In this paper is presented new results on thermal and mechanical properties, morphology, and biodegradation behavior of polymer blends of oxidized PP with PCL. The original PP-PCL blend is also fabricated for purposes of comparison. The structure-property relationship of the blends films were studied by Fourier transmission infrared (FT/IR), differential scanning calorimeter (DSC), X-ray diffraction (XRD), tensile testing, and biodegradation test.

## EXPERIMENTAL

### Materials

$\epsilon$ -Caprolactone monomer ( $\epsilon$ -CL) was obtained commercially from Aldrich Chemical. Co., dried over  $\text{CaH}_2$  and distilled under reduced pressure. Dibutyltin oxide ( $\text{C}_4\text{H}_9$ )<sub>2</sub>SnO and dibutyltin dichloride ( $\text{C}_4\text{H}_9$ )<sub>2</sub>SnCl<sub>2</sub> as catalyst precursors were also obtained from Aldrich Chemical Co., whereas polypropylene is a commercial product of the Polyolefin Company (PT. Trypolyta, Indonesia) with a melt flow index of 1.5 g/10 min. An enrichment of culture was established with activated sludge collected from a chemical sewage treatment plant at Biotechnology Research Center, Institut Teknologi Bandung, Indonesia. A sub-enrichment culture inoculated with an aliquot of this culture was made in liquid mineral salts medium containing standard reagent. The culture was grown with shaking at 30 °C, and then the degradation ability of the culture to degrade standard reagent was determined by bacterial growth through monitoring the optical density at 600 nm. An agar medium prepared from the mineral salts by addition of 1.5% (w/vol) bacteriological agar. Mineral salts contained (per liter): 1 g of  $\text{KH}_2\text{PO}_4$ , 1 g of  $\text{K}_2\text{HPO}_4$ , 0.04 g of  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ , 0.004 g of  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ , and 1 g of  $(\text{NH}_4)_2\text{SO}_4$  in deionized water (14). The pH of the medium was adjusted to 7.4 with sodium hydroxide. The agar medium was transferred to test tubes (10 mL/tube) and sterilized at 121 °C for 20 min.

**Catalyst Preparation.** A mixture of dibutyltin oxide (60.0 mmol), dibutyltin dichloride (20.0 mmol), and 95% ethanol was refluxed. After 6 h, the resulting

transparent solution was concentrated to give a white powder, which was pulverized and then exposed to ambient atmosphere overnight to convert the partially formed 1-ethoxy-3-chlorotetrabutyl-distannoxane into the corresponding 1-hydroxy-3-chlorotetrabutyl-distannoxane. Recrystallization of the crude product in hexane at 0 °C produced crystals within the melting point range of 110–120 °C.

**Poly- $\epsilon$ -caprolactone Preparation.** Preparation of PCL was done in the same condition with preparation of synthetic poly-(R,S)- $\beta$ -hydroxybutyrate (PHB) (7,13). 1-Hydroxy-3-chlorotetrabutyl-distannoxane ( $1.2 \times 10^{-4}$  mol) in hexane was first added to the reactor and then hexane was evaporated by heating in silicone oil bath at 80 °C under vacuum for 6 h.  $\epsilon$ -CL monomer (0.06 mol) was added to the reactor at room temperature. The mixture was degassed during three freeze-thaw cycles and finally sealed under vacuum. Polymerization reactions were carried out at 100 °C for 4 h. The resulting mixture was dissolved in chloroform (100 mL g<sup>-1</sup> of initial monomer), and polymer solution was stirred for 12 h at room temperature and refluxed for half an hour. The clear solution was then concentrated (10 mL g<sup>-1</sup> of initial monomer), followed by precipitation in diethyl ether to give a white solid of PCL (6.50 g) in 95.0% yield with molecular weights  $M_n = 25,900$  and  $M_w = 81,700$ .

**Modification of Polypropylene.** Modification of polypropylene was carried out by oxidation in the presence of hydrogen peroxide. Polypropylene (2.5 g) was mixed with  $\text{H}_2\text{O}_2$  (30%) and stirred for 4 h at various temperatures. The polymer was separated from  $\text{H}_2\text{O}_2$  with a filter paper. The solid polymer was washed with  $\text{Na}_2\text{CO}_3$  (0.001 mol L<sup>-1</sup>) and water repeatedly, and then the polymer was dried in a vacuum oven for 24 h.

**Polymer Blends Preparation.** Polymer blends were prepared by casting of polymer solution in various compositions. The oxidized PP and PCL powders were blended in xylene at 110 °C. The mixture was stirred for 2 h until it become homogenous, and then xylene was evaporated and dried under vacuum at room temperature. The polyblend was compression molded with a hot press at 140 °C into films and plates with thicknesses of 0.2 mm and 3.2 mm, respectively.

### Polymers Characterization

FT/IR (Shimadzu 8501) was used to characterize the chemical structure of the modified PP and its polymer blends. X-Ray diffraction (Diano type 2100E) was used to determine the degree of crystallinity of the polymers formed. A Perkin Elmer differential scanning calorimeter (DSC, Model DSC-7) was used to determine their thermal behavior. All the scans were carried out from -50 °C to 200 °C at a heating rate

of  $20^{\circ}\text{C min}^{-1}$ . The tensile specimens were prepared from compression-molded films. The tensile properties of the specimens were determined according to ASTM standard using a tensile tester machine/autograph (Shimadzu AGS 500D) at room temperature with single tension and a rate of  $5\text{ mm min}^{-1}$ .

In the study of their biodegradation behavior, film of polymer sample ( $2.5\text{ cm} \times 2.5\text{ cm}$  with a thickness of  $0.2\text{ mm}$ ) and solid mineral salts medium described above were sterilized separately at  $1.5\text{ atm}$  of steam, and then placed in a Petri dish under a sterilized condition and allowed to stand for 24 h. The appropriate dilutions of the culture suspension of activated sludge were spread onto solid mineral salts medium containing polymer sample, and then incubated at  $37^{\circ}\text{C}$  in various times. The initial weight of the polymer sample was measured prior to the degradation tests. The film specimens were periodically removed, washed with distilled water and ethanol (95%), and dried under vacuum for 4 h. Their weights were measured after the film specimens were dried to constant weight.

## RESULTS AND DISCUSSION

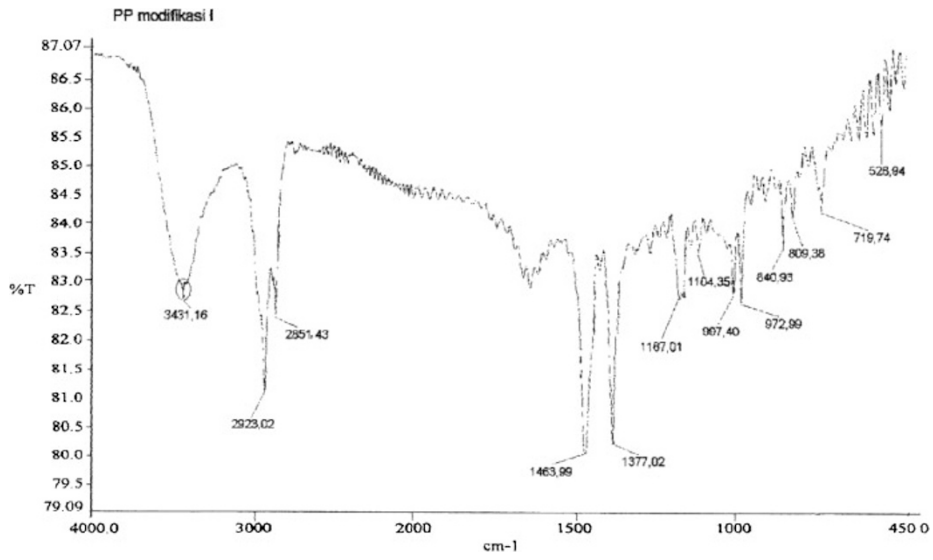
Polymer blends of modified PP with PCL were prepared by casting polymer solutions, followed by compression molding into thin films. The modified PP was obtained by oxidation with hydrogen peroxide resulting carbonyl and hydroxyl groups on the polymer chain (13). The FT/IR spectra of pure oxidized PP and PCL film specimens are showed in Figure 1a and 1b, respectively. The oxidized PP specimen shows absorption peaks at wave number of  $3431\text{ cm}^{-1}$  and  $1724\text{ cm}^{-1}$ , which are associated with the stretching vibration of hydroxyl and carbonyl groups. Oxidation of PP with hydrogen peroxide effectively improves polar functional groups of PP. The FT/IR spectra of oxidized PP and PCL polymer blends shows the presence of superposition and displacement of characteristic peaks of polymer blend components (Figure 1c). The peak intensity of hydroxyl group for polymer blends at wave number of  $3431\text{ cm}^{-1}$  decreases compared to that of hydroxyl group for pure oxidized PP (Figure 1a). The decrease in peak intensity of hydroxyl group of oxidized PP might be associated to the reaction between oxidized PP with PCL through van der Waals force or hydrogen bonding between carbonyl and hydroxyl groups of the components in the polymer blends.

The typical DSC curves of oxidized PP, PCL, and oxidized PP-PCL polymer blends in various compositions can be seen in Figure 2. The melting temperatures of oxidized PP and PCL were determined to be  $175.5^{\circ}\text{C}$  and  $74.7^{\circ}\text{C}$ , respectively. However, the

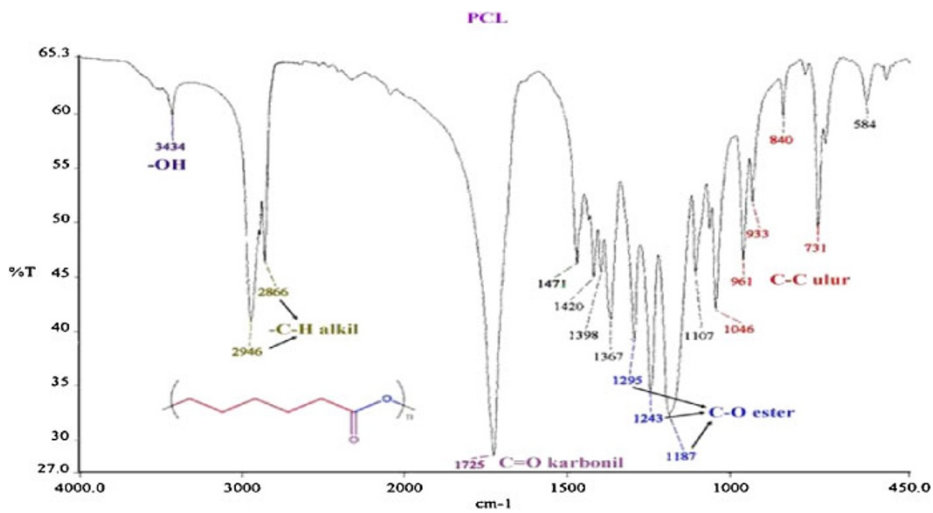
DSC curves of polymer blends show two peaks associated with the melting temperatures of their component polymers. This indicates that the oxidized PP and PCL are immiscible polymer blends. Similarly, the original PP also forms immiscible blends with PCL. The relevant DSC results of the polymer blends of oxidized PP-PCL in various compositions are summarized in Table I. Furthermore, the fusion enthalpy of oxidized PP phase decreases with increasing of PCL content in the polymer blends, whereas the fusion enthalpy of PCL phase increases. These results are associated with the composition of their component in polymer blends. We also observed that in polymer blend with PP/PCL composition of 70/30 (% w/w), the fusion enthalpies of the oxidized PP and PCL phases are smaller than those of the original PP and PCL phases. It is known that a reduction in the fusion enthalpy generally results in a decrease in the crystallinity of the polymer blends. Hence the use of oxidized PP in preparation of PP-PCL blends can lead to a reduction in crystalline phase of polymers forming a small amount of compatible polymer blend.

In addition, the observed fusion enthalpy of oxidized PP and PCL phases in various compositions are lower than the calculated fusion enthalpy. Those results might be caused by the presence of a small amount of oxidized PP and PCL to form compatible polymer blends. Based on the difference between calculated and observed fusion enthalpies, the percentage of compatible polymer blends obtained from modified PP and PCL can be predicted in which the highest percentage of the compatible polymer blends is obtained in PP/PCL ratio of 70/30 (% w/w) (Table I).

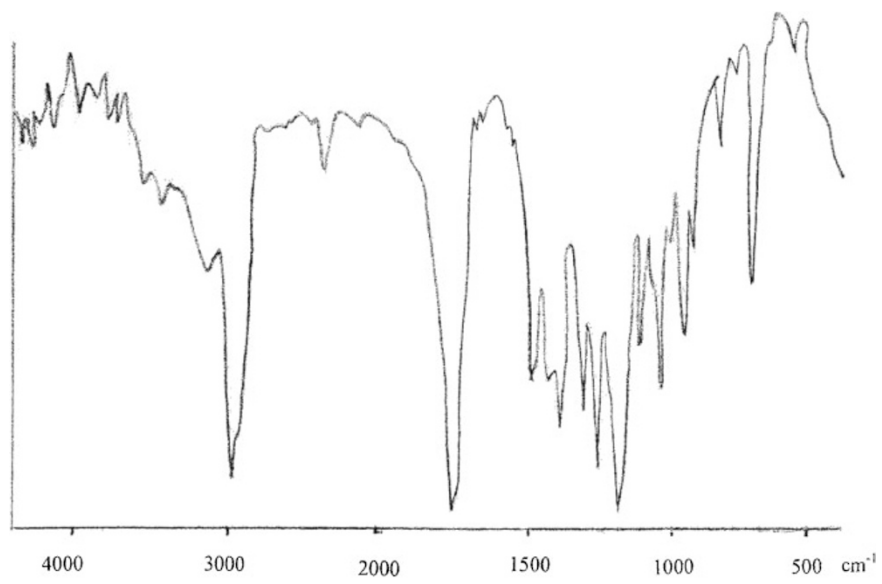
The analysis of crystallinity indicates that the addition of a small amount of PCL until 30% (w/w) in polymer blends result in a decrease in the degree of crystallinity (Table II). The decrease of crystallinity might be associated to the interaction between modified PP and PCL forming compatible polymer blends. Moreover, the crystallinity of the original PP-PCL polymer blend in PP/PCL ratio of 70/30 (% w/w) is higher than that of the oxidized PP-PCL polymer blend. This means that the interaction between the PP and PCL can be improved considerably by using oxidized PP in preparation of PP-PCL polymer blends. The highest compatible polymer blend of modified PP-PHB was found in a PP/PHB ratio of 90/10 (% w/w) (13). This was caused by the presence of a small amount of PHB in the amorphous parts of PP, so that the structure of the PP component becomes more regular and denser indicated by the highest crystallinity and mechanical properties of the PP-PHB polymer blend. Furthermore, the crystallinity of oxidized PP-PCL polymer blends then increases with in-



(a)

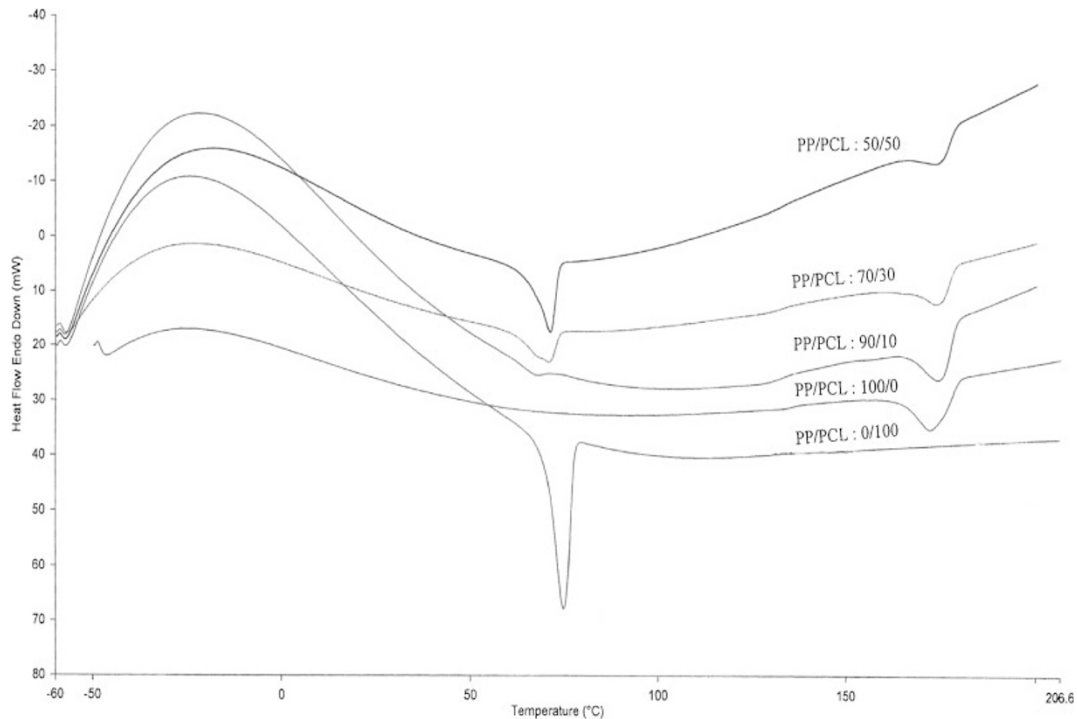


(b)



(c)

**Figure 1.** (a) FT/IR spectra of PP after oxidation with hydrogen peroxide. (b) FT/IR spectra of PCL. (c) FT/IR spectra of polymer blend with PP/PCL ratio of 50/50 (% w/w).



**Figure 2.** DSC thermogram of PP-PCL polymer blends in various compositions.

**Table I.** Thermal properties of oxidized PP-PCL polymer blends with various compositions determined by differential scanning calorimeter (DSC)

| Sample<br>(PP/PCL)<br>(w/w) | Observed <sup>a)</sup> |                     |                        |                     | Calculated <sup>b)</sup> |                     | Compatible <sup>c)</sup><br>Component |      |
|-----------------------------|------------------------|---------------------|------------------------|---------------------|--------------------------|---------------------|---------------------------------------|------|
|                             | PP                     |                     | PCL                    |                     | PP                       | PCL                 | $\Delta H$ (J/g)                      | (%)  |
|                             | T <sub>m</sub><br>(°C) | $\Delta H$<br>(J/g) | T <sub>m</sub><br>(°C) | $\Delta H$<br>(J/g) | $\Delta H$<br>(J/g)      | $\Delta H$<br>(J/g) |                                       |      |
| 100/0                       | 175.5                  | 54.1                | —                      | —                   | 54.1                     | —                   | —                                     | —    |
| 90/10                       | 174.6                  | 44.8                | 67.2                   | 8.6                 | 48.7                     | 10.9                | 6.2                                   | 10.4 |
| 70/30                       | 174.1                  | 27.3                | 70.8                   | 23.9                | 37.9                     | 32.8                | 19.5                                  | 27.6 |
| 70/30 <sup>d)</sup>         | 175.2                  | 38.5                | 72.9                   | 30.1                | 41.7                     | 32.8                | 5.9                                   | 7.9  |
| 50/50                       | 174.2                  | 26.0                | 71.1                   | 42.7                | 27.1                     | 54.7                | 13.1                                  | 16.0 |
| 0/100                       | —                      | —                   | 74.7                   | 109.4               | —                        | 109.4               | —                                     | —    |

<sup>a)</sup>Determined by observation of DSC curves, <sup>b)</sup>Determined by calculation, <sup>c)</sup>Determined by the difference between observed and calculated fusion enthalpies, <sup>d)</sup>Polymer blend prepared between original PP and PCL ( $\Delta H$  of original PP = 59.5 J/g).

**Table II.** Crystallinity and mechanical properties for PP-PCL polymer blends with various compositions

| Sample<br>(PP/PCL) (w/w) | Crystallinity<br>(%) <sup>a)</sup> | Mechanical properties <sup>b)</sup> |                |         |
|--------------------------|------------------------------------|-------------------------------------|----------------|---------|
|                          |                                    | $\sigma$ (MPa)                      | $\epsilon$ (%) | E (MPa) |
| 100/0                    | 43.6                               | 23.2                                | 32.8           | 70.5    |
| 90/10                    | 38.3                               | 13.2                                | 26.8           | 49.4    |
| 70/30                    | 39.9                               | 5.8                                 | 25.4           | 23.0    |
| 70/30 <sup>d)</sup>      | 46.0                               | 4.9                                 | 22.5           | 21.8    |
| 50/50                    | 51.9                               | 2.6                                 | 21.1           | 12.2    |
| 0/100                    | 69.0                               | —                                   | —              | —       |

<sup>a)</sup>Determined by XRD, <sup>b)</sup>Determined by tensile testing, and <sup>d)</sup>Polymer blend prepared between original PP and PCL.

creasing PCL content more than 30% in the polymer blends. It is known that PCL has high crystallinity, hence the increase of PCL content leads to an increase in the crystallinity of PCL part region, and also a reduction in interaction or miscibility between modified PP and PCL.

Mechanical properties of the oxidized PP-PCL polymer blends films are showed in Table II. The Young's modulus (E) and tensile strength ( $\sigma$ ) at room temperature of oxidized PP films were 70.5 MPa and 23.2 MPa, respectively. When oxidized PP was blended with PCL, which has high crystallinity, both mechanical values of polymer blends film decreased with increasing the fraction of PCL component. In addi-

**Table III.** Polymer weight loss after biodegradation with active sludge for various incubation times

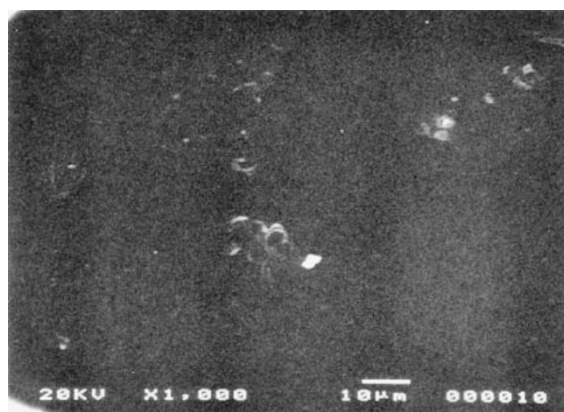
| Samples<br>PP/PCL (w/w) | Polymer weight loss after biodegradation (%) |      |      |      |      |      |
|-------------------------|--|------|------|------|------|------|
|                         | 5 d  | 10 d | 20 d | 30 d | 40 d | 60 d |
| 100/0                   | 0.0  | 0.7  | 1.5  | 1.7  | 1.7  | 2.0  |
| 90/10                   | 0.0  | 0.8  | 2.2  | 3.5  | 3.5  | 5.6  |
| 70/30                   | 0.7  | 2.2  | 3.3  | 3.8  | 8.4  | 15.3 |
| 70/30 <sup>d)</sup>     | 0.2  | 1.2  | 2.1  | 3.0  | 3.4  | —    |
| 50/50                   | 1.4  | 3.7  | 5.2  | 8.7  | 10.7 | 16.6 |
| 0/100                   | 2.0  | 10.5 | 22.3 | 38.3 | 45.5 | 50.0 |

<sup>d)</sup>Polymer blend prepared between PCL and original PP.

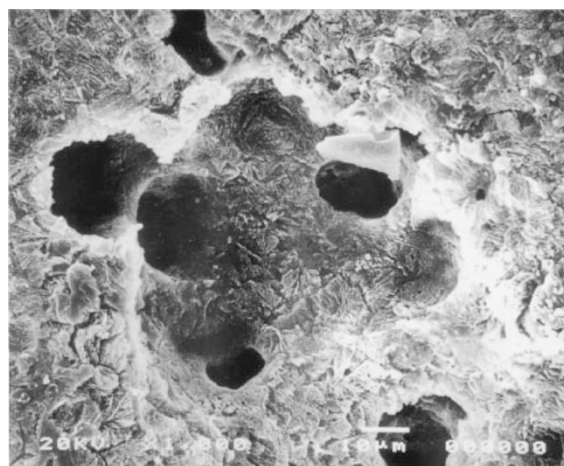
tion, the elongation required for breaking ( $\varepsilon$ ) slightly decreased from 32.8% to 21.1% as the fraction of PCL component increased from 0 to 50% (w/w). Thus, the oxidized PP-PCL polymer blends films become less flexible and tougher as the fraction of PCL component increased. This mechanical property of polymer blends might be attributed to the high crystallinity and brittleness of PCL, and this property was approved by the difficulty of PCL to form polymer films. In contrast, the Young's modulus and tensile strength of polymer blend film prepared from original PP-PCL in ratio of 70/30 (% w/w) were 21.8 MPa and 4.9 MPa, respectively. These values were lower than those of oxidized PP-PCL polymer blend film in the same ratio. This observation could be attributed to the lower compatibility and weaker adhesion of original PP and PCL in polymer blend.

The results of the enzymatic degradation of oxidized PP-PCL polymer blends by the culture of activated sludge in agar medium are shown in Table III. The rate of degradation is generally affected by crystallinity, blend phase morphology, structure, and chemical composition (6,15). Polymers with a lower degree of crystallinity would be decomposed faster by enzymatically hydrolysis. In this work, the degree of degradation is determined from weight loss, express in percentage (% weight loss).

It is apparent that the oxidized PP and PCL polymer blend in PP/PCL ratio of 70/30 (% w/w) is hydrolyzed with higher weight loss than polymer blend prepared from the original PP-PCL with the same ratio. PCL in compatible part of polymer blends should have lower crystallinity than PCL part, so PCL in compatible part of polymer blends should be hydrolyzed easier than PCL part. Thus, the higher weight loss of oxidized PP-PCL polymer blend is related to the compatibility of each component in polymer blends. The enzymatic degradability of the oxidized PP-PCL polymer blends is lower than that of pure PCL and bacterial PHB (as a standard) (13), and it increases slowly with increasing PCL content in the polymer blends (Table III). PCL can be easily decom-



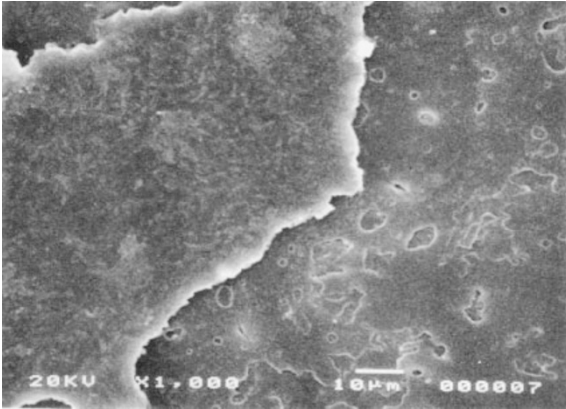
**Figure 3.** Micrograph of the film surface of PCL (enlargement 1000  $\times$ ) before biodegradation.



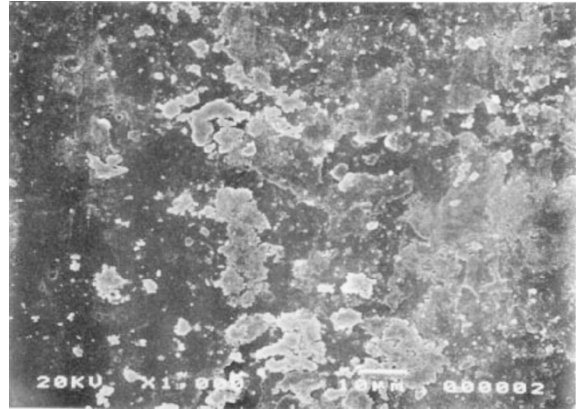
**Figure 4.** Micrograph of the film surface of PCL (enlargement 1000  $\times$ ) after biodegradation for 30 d.

posed by microorganism because the backbone chain of PCL can be hydrolyzed. This result was approved by analysis of scanning electron microscopy (SEM) (Figures 3 and 4).

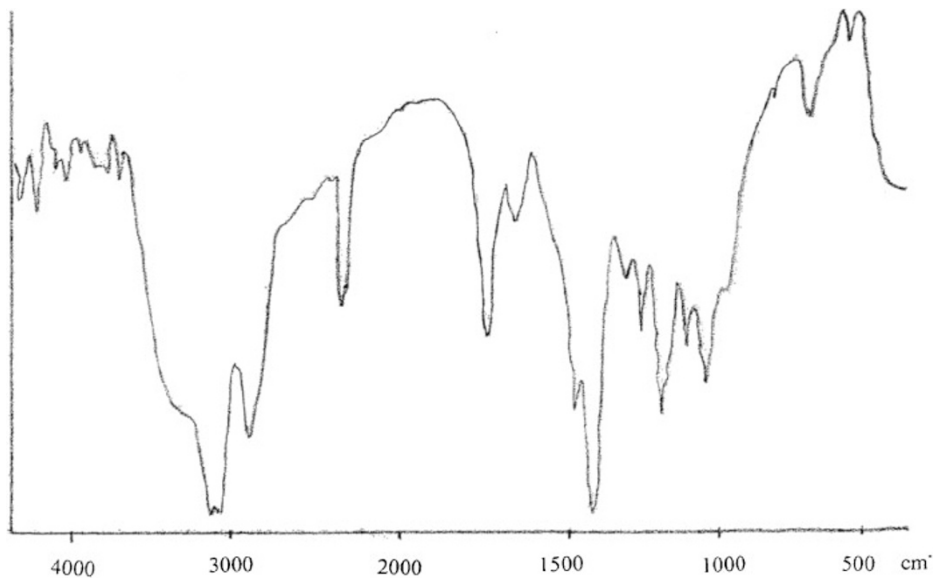
The pure oxidized PP was practically not decomposed by microorganisms for incubation time of 60 days due to the hydrophobic property of PP, which is resistant to enzymatic attack. The modification of



**Figure 5.** Micrograph of the film surface of polymer blend with PP/PCL ratio of 50/50 (% w/w) (enlargement 1000 ×) before biodegradation.



**Figure 6.** Micrograph of the film surface of polymer blend with PP/PCL ratio of 50/50 (% w/w) (enlargement 1000 ×) after biodegradation for 30 d.



**Figure 7.** FT/IR spectra of polymer blend with PP/PCL ratio of 50/50 (% w/w) after biodegradation for 30 d.

PP by oxidation with hydrogen peroxide is not sufficient to produce hydrophilic groups such as carbonyl and hydroxyl groups that would normally facilitate interaction between polymers and enzymes. However, in the polymer blend of oxidized PP-PCL, the oxidized PP might be decomposed slowly by microorganisms. It is clearly observed that the weight loss of the oxidized PP-PCL polymer blend in PP/PCL ratio of 70/30 (% w/w) is about 5.0% higher than that of the original PP-PCL polymer blend in the same ratio after incubation time of 40 d. Thus, the blending PCL with oxidized PP leads to more weight loss by incubation time. The use of oxidized PP in preparation of polymers blends with PCL effectively improves the degradability of the polymer blends due to the increase compatibility of each component in polymer blends. In the polymer blends, the presence of phase

separation between PCL and oxidized PP was observed by SEM analysis (Figure 5). Due to the presence of phase separation in polymer blends, PCL and compatible parts of polymer blends might be decomposed by microbial enzyme. It is apparent that microorganisms is difficult to attack PP in the polymer blends, but SEM analysis showed that the film surface of polymer blends has been destroyed by microorganisms (Figure 6).

In addition, the FT/IR spectra of oxidized PP-PCL polymer blend with a PP/PCL ratio of 50/50 (% w/w) after incubation time of 30 d showed the significantly decrease of absorption peak intensity of carbonyl group observed at wave number of  $1725\text{ cm}^{-1}$  (Figure 7). This indicates that the amount of carbonyl group in the chain of polymer blends decreases as a result of biodegradation process by microorganism.



It is also noted that the wide and broad absorption peaks appeared at wave number of 2800–3600 cm<sup>-1</sup> after degradation biologically. The existence of this absorption peaks is associated to hydroxyl and carboxylate functional groups in the end of polymer chain due to the hydrolysis of PCL and compatible parts of polymer blends.

### CONCLUSION

A part of the polymer blends obtained between oxidized PP and PCL were compatible polymer blends, and the highest compatible polymer blends was obtained in PP/PCL ratio of 70/30 (% w/w) with low crystallinity. The mechanical properties of polymer blends decreased with increasing PCL proportion in the polymer blends due to the high crystallinity and brittleness of PCL. Enzymatic degradability of oxidized PP-PCL polymer blends were lower than that of PCL, and increased slowly with increasing PCL content in the polymer blends. The use of oxidized PP in preparation of polymers blends with PCL effectively improves the degradability of the polymer blends due to the increase of compatibility of each component in polymer blends.

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