

## Effects of Temperature and Compost Conditions on the Biodegradation of Degradable Polymers

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**Abstract** The effectiveness of current biodegradation test methods for degradable polymers under controlled composting conditions was studied in regards to the test temperature and compost condition. When biodegradability tests for the natural (starch, cellulose, PHB/HV) and synthetic (PCL, SG, PLA) polymers were conducted at temperature levels of 35 and 55°C with compost cured at ambient temperature, the degradations of cellulose and starch were higher at 35°C because of the priming effect. On the other hand, degradations of other polymers were higher at 55°C. In the biodegradation test at 55°C, compost harvested right after the thermophilic degradation stage showed higher biodegradation activities than the cured compost for both the synthetic aliphatic polyester (SG) and a natural polymer, cellulose. These results suggest that the biodegradation test conducted at 55°C with the compost, harvested right after the thermophilic degradation stage during composting, showed the highest biodegradation activity under controlled composting conditions.

**Key words:** Compost, biodegradation of polymer, biodegradation test

The recycling of organic wastes through composting has received much attention to effectively solve problems dealing with solid waste, both in Korea and European Union [6]. Composting is a biological process in which thermophilic and mesophilic microorganisms degrade organic materials and produce compost that is beneficial to soil [7]. Generally, composting processes go through a fast decomposing thermophilic stage followed by a slow degrading curing stage for a fully matured compost [8]. Easily degradable materials such as sugar and lipids are initially degraded at the thermophilic stage, while hardly-degradable materials remain until the curing stage that is maintained at the mesophilic temperature [10].

Biodegradable polymers have been regarded as a good alternative in solving the solid waste problem caused by non-degradable synthetic polymers such as polyethylene and polystyrene. Because of their biodegradability, they can be easily treated in solid waste treatment systems such as composting or anaerobic digestion [4]. Since treatment of solid waste through composting is increasingly popular, development of biodegradable polymers which can be treated in a composting process has become an attractive subject in the biodegradable polymer society.

To promote the usage of biodegradable polymers, the development of incorporating proper evaluation methods is critical. Biodegradability tests of degradable plastics under a specific waste treatment condition indicate whether these plastic materials can be treated under the given conditions. Many standard test methods have been issued for biodegradable polymers, including the method to measure the biodegradability under controlled composting conditions [1-3, 5]. In this test, biodegradability is measured at thermophilic temperature (58°C) using a compost cured at ambient temperature. Since the compost is produced through both thermophilic and mesophilic stages, there exist both mesophilic and thermophilic microorganisms in the compost. Therefore, it is not clear whether the test condition reflects the characteristics of the microorganisms in the compost or not.

In this study, we compared the biodegradation of polymers at two different culture temperatures of 35 and 55°C, using composts prepared in various conditions, to find the proper test condition for biodegradability. As test polymers, both synthetic and natural polymers were used.

### MATERIALS AND METHODS

#### Substrate for Compost

Major raw material for composting was food wastes generated from the cafeteria in our Institute, The Korea Institute of Science and Technology. Food wastes collected

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for two to three days were mixed with the bulking agent (sawdust) and seed (compost produced in our lab.). The substrate mixture for composting consisted of 50% food waste, 20% bulking agent, and 30% seed.

### Composting Reactors

An acrylic column of 29-cm diameter by 50.7-cm length equipped with an air distribution system at the bottom was used as a composting reactor. The reactor was insulated with polyethylene foam in order to reduce the loss of heat generated through biological decomposition of organic matters. One temperature sensor was placed in the center of the compost to measure the temperature during the process of composting. Wet air was supplied from the bottom through an air distribution system with variable flow rates (0.5 to 4.3 l/min) to control the reactor temperature below 70°C. For the curing reaction, acrylic reactors with a volume of 2 l were used.

### Composting Conditions

Twenty kilograms of the substrate mixture was placed into the composting reactor and aeration started. To ensure an aerobic condition, the oxygen concentration in the exhaust gas was closely monitored and the aeration rate was adjusted to maintain the O<sub>2</sub> concentration to be at least 1%. The compost was completely mixed when the compost temperature dropped during the thermophilic degradation stage. After the thermophilic degradation stage, the compost was cured at an ambient temperature of 15°C.

### Polymer Samples

Both a synthetic aliphatic polyester, poly-(butylene succinate-co-adipate) (SG, grade 2109, SKI Corp, Korea) and a natural polymer, cellulose, were used as major plastic samples. The polymer samples were in the form of a film of 50–100 µm thickness or in powder. Cellophane film was used for the cellulose experiment. For the test temperature experiment, polycaprolactone (PCL, Tone 787, Union Carbide, U.S.A.), poly-lactic acid (KIST, MW 49,000), poly-hydroxybutyrate-co-hydroxyvalerate (PHB/HV, Aldrich, U.S.A.), and starch were also used.

### Biodegradability Test

The test method was based on the American Standard for Testing and Materials (ASTM) D5338-92 [3] with a minor modification. An acrylic column of 10.5-cm diameter by 25-cm length which was equipped with an air distribution system at the bottom was incorporated as a test reactor. In the reactor, 5% (w/w) of the polymer sample was mixed with a compost. The test reactors were placed in a water bath controlled at either 35 or 55°C. Air was supplied to the reactors at a rate of 50–100 ml/min after carbon dioxide was removed by an alkaline solution. Carbon dioxide generated by biodegradation of both compost and polymer

samples was collected by 0.4 N KOH solution and measured by titration with an HCl solution [12].

### Analysis

During the composting, substrate samples were collected after complete mixing and analyzed for volatile solid (VS) content, C/N ratio, pH, and microbial population of the compost. VS content was determined by measuring the change of the weight after 24 h incubation at 550°C in an oven. Carbon and nitrogen contents were determined by an elemental analysis. To measure the microbial numbers for both mesophiles and thermophiles in the compost, compost samples were taken from various locations after mixing the compost. Then, each compost sample was mixed completely and suspended in a saline solution, and then spread onto the Plate-Count Agar (Difco, U.S.A.) plates. Plates for one compost sample were cultivated for 3 to 5 days at two different temperatures of 30°C and 55°C for mesophiles and thermophiles, respectively. Temperature and gas composition (O<sub>2</sub>, CO<sub>2</sub>, NH<sub>3</sub>) in the exhaust gas during composting were closely monitored daily. Gas concentration was measured using the Gastec detector column (Gastec, Japan).

Biodegradation of polymers was defined as the percentage of the cumulative CO<sub>2</sub> production to the total CO<sub>2</sub> evolution (theoretical quantity), when all the carbon in the test material was converted to CO<sub>2</sub> [3]. CO<sub>2</sub> produced during the biodegradation test was absorbed in 0.4 N KOH solution and then precipitated with BaCl<sub>2</sub>. The alkaline solution was titrated with 0.05 N HCl to determine the absorbed CO<sub>2</sub>.

## RESULTS AND DISCUSSION

### Composting of Food Waste

The characteristics of food waste, bulking agent (sawdust), and seed compost are shown in Table 1. Since the carbon to nitrogen (C/N) ratio of food waste was only 8.1, sawdust was mixed at a final composition of 20% (wet weight) to improve both C/N ratio and substrate structure. The mature compost, which was produced previously in our laboratory with the same substrate mixture and method as in this study, was also mixed by 30% for proper initiation of composting. By the addition of sawdust and mature

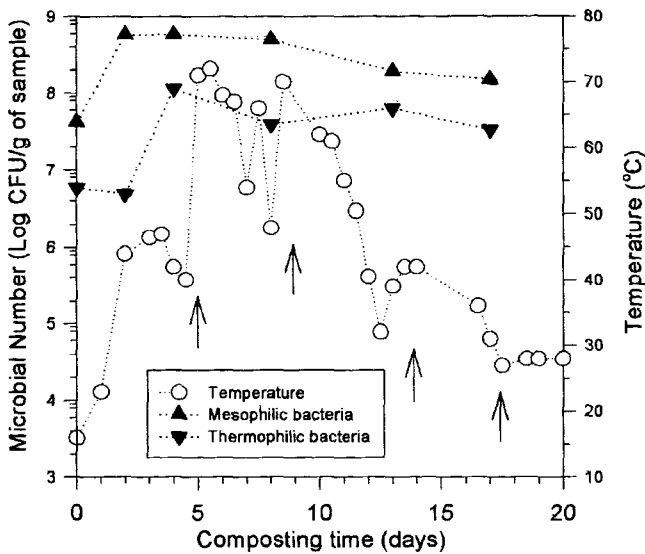
**Table 1.** Characteristics of substrate mixture for the composting experiment.

Material	Total solid (%)	Volatile solid (%)	C/N	Composition in mixture (%)
Food waste	21.5	81.2	8.1	50
Sawdust	90.5	99.5	44.2	20
Seed compost	31.7	81.3	16.8	30
Mixture	32.5	89.5	15.9	100

compost, the C/N ratio of the substrate mixture was improved as much as 15.9.

The changes of temperature and number of microbes are shown in Fig. 1. Immediately after composting started, the temperature of the reactor rose to 45°C in two days and dropped slightly after 4 days. After mixing the substrate, the temperature increased again and reached up to 70°C in a day and was maintained over 50°C for 6 days. Although the temperature dropped again below 50°C on the 8th day, the temperature of 70°C was recovered by yet another process of mixing. On the 12th day, the temperature dropped to the mesophilic range and did not rise again to the thermophilic range even after mixing. When the temperature dropped below 30°C after a slight increase above 40°C by mixing on the 12th day, the substrate mixture was harvested at day 17 and curing was started at the ambient temperature of 15°C.

The initial numbers of both mesophilic and thermophilic bacteria were  $6 \times 10^6$  and  $4 \times 10^5$  colony forming units (CFU)/gram of sample, respectively. The number of mesophilic bacteria increased as composting began and reached to  $6 \times 10^7$  CFU/g in two days and decreased slightly thereafter, while the thermophilic bacteria showed a two-day lag phase and increased to  $8 \times 10^6$  CFU/g. Mesophilic bacteria did not decrease significantly in the thermophilic stage, which suggests that the temperature inside the composting reactor might vary significantly depending upon the position and, as a result, mesophiles could survive in some position where the temperature was not as high as shown in the temperature measurement. According to Nakasaki *et al.* [11], the number of mesophilic bacteria decreased by one order in the thermophilic stage when they conducted

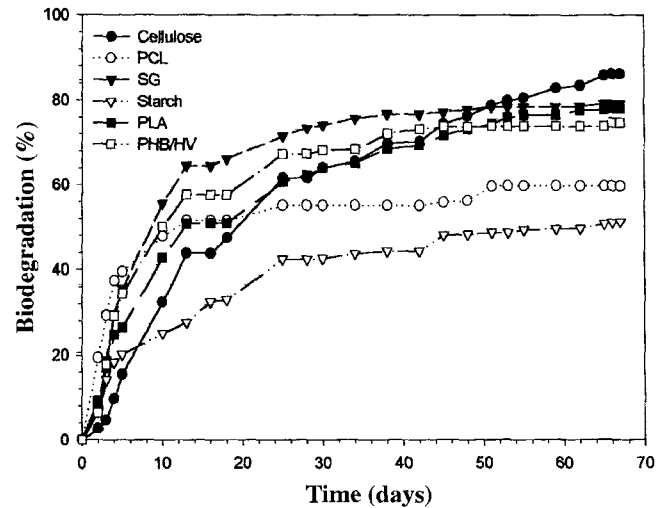


**Fig. 1.** Changes of temperature and microbial population during composting of food waste. Solid arrows indicate the turning time of composting materials while the dotted arrow indicates the start of the curing stage.

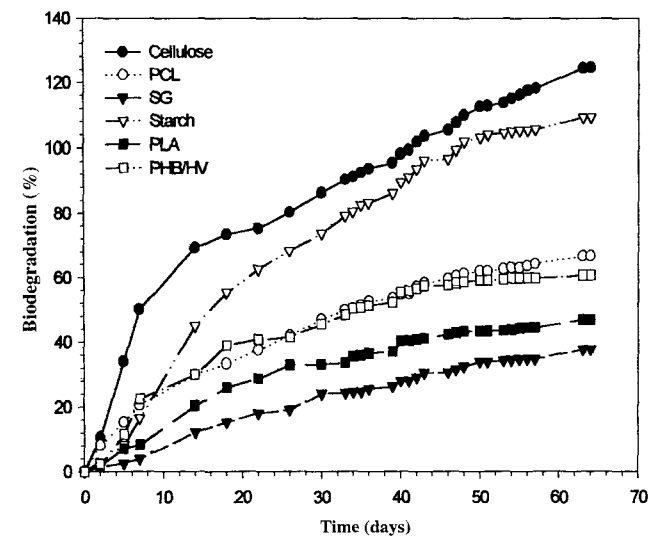
composting with sewage sludge. During the curing stage, the number of bacteria for both mesophile and thermophile decreased. It is important to note that thermophiles decreased more than mesophiles, but the level of decrease was not significant.

**Effect of Test Temperature on Biodegradation**

Since it was suggested in the International Standard Organization (ISO) draft that the biodegradation test be performed at the thermophilic temperature of 58°C with compost cured at an ambient temperature, the study was conducted to reveal whether or not the thermophilic test temperature was better than the mesophilic temperature [4]. For this study, the biodegradation test was performed



**Fig. 2.** Biodegradation of various polymers at 55°C with cured compost.



**Fig. 3.** Biodegradation of various polymers at 35°C with cured compost.

at two different temperatures of 35 and 55°C with the cured compost (Figs. 2 and 3). In this test, various natural (starch, cellulose, PHB/HV) or synthetic (PCL, SG, PLA) polymers were used. During the 66 days of the degradation test, all the polymers, with an exception of cellulose and starch, showed a higher biodegradability at 55°C than at 35°C, while biodegradations for both cellulose and starch were higher at 35°C than at 55°C. In another test, the degradation of cellulose at both temperatures was similar but those of the synthetic aliphatic polyesters (SG, PCL) were consistently shown to be higher at 55°C than at 35°C (data not shown). Since the compost cured at an ambient temperature of 15°C was used and the biodegradation test was performed at 55°C, these results may suggest that thermophilic microorganisms utilizing natural polymers lost some of their activities during the curing stage. On the contrary, it seems that different microorganisms were involved in the degradation of the synthetic polymers and these thermophilic bacteria did not lose their activities during the curing stage. Studies on the characteristics of the thermophilic microorganisms which degrade either the natural or the synthetic polymer will be required to verify our explanation.

It is interesting to note that the final degree of biodegradation for both cellulose and starch exceeded 100% at 35°C. In the case of the cellulose, it seems that biodegradation reached the plateau phase in 24 days of cultivation with a biodegradation degree of about 80%. After 24 days, carbon dioxide was produced with a constant rate of a continual flow, and, as a result, the biodegradation degree reached over 120% after 66 days of cultivation. When the volatile solid (VS) content of the remaining compost was measured after the test, the VS varied from 63.4% of the total solid to 72.6% (Table 2). The VS of the compost was the lowest with cellulose and increased in the order of starch, blank, and SG. Since the final degree of biodegradation of SG was below 40%, the remaining SG seemed to have contributed to the higher VS value compared to the blank. This result confirmed the fact that cellulose and starch caused more degradation of the compost. According to Jyunkai *et al.* [9], the addition of glucose to the soil stimulated the degradation of soil organics as well as glucose, and, as a result, CO<sub>2</sub> production exceeded the amount expected by only the degradation of glucose. They called this phenomenon the "priming effect"

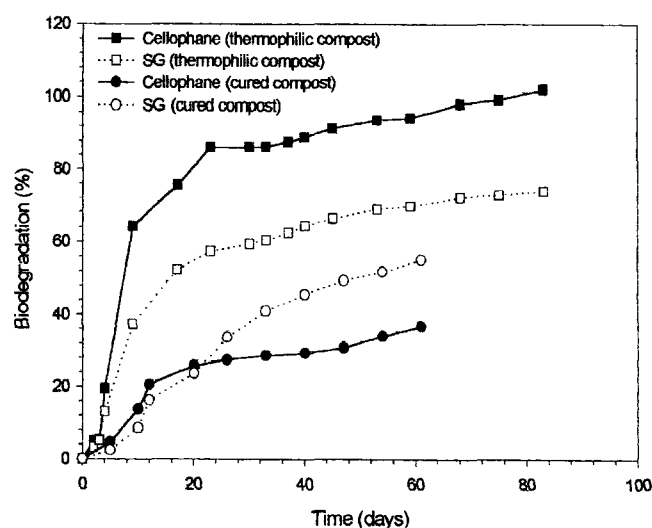
**Table 2.** Final volatile solid (VS) value for composts with various polymers after a biodegradation test at 35°C.

Polymer sample	Final VS (% of total solid)
Starch	66.2
Cellulose	63.4
SG	72.6
Blank	69.6

and also demonstrated that other compounds such as formate, benzoate, n-hexadecane, and bis (2-ethylhexyl) phthalate did not cause any priming effect. Since glucose is a monomer for both starch and cellulose and was generated during degradation, our observation may have been due to the priming effect. Our results also reveal that this priming effect was not observed at the thermophilic temperature (55°C) with the same compost and polymer samples. Except for the priming effect, the test temperature of 55°C represents a higher biodegradability than 35°C for the composting test with a cured compost.

### Effect of Compost Characters on Biodegradation

In the previous section, it was shown that microbial populations changed during the composting process. This observation suggests that the microbial activity for the biodegradation of polymers may vary depending upon the composting stage. To compare the microbial degradation activities at different composting stages, compost samples harvested right after the thermophilic degradation stage or cured for one month at 15°C were collected as inocula. In this test, cellulose and SG were used as representatives of natural and synthetic polymers, respectively. Biodegradation test was conducted at 55°C. As shown in Fig. 4, the compost harvested right after the thermophilic degradation stage revealed higher degradation activities for both polymers, compared to the cured compost. These results are not surprising since the biodegradation activity of the thermophilic microorganisms was higher at the thermophilic degradation stage during composting. The compost harvested after the thermophilic stage produced about 130 mg of carbon dioxide per gram of volatile solids for the first 10



**Fig. 4.** Comparison of biodegradabilities of cellulose and SG with compost harvested either after the thermophilic stage (thermophilic compost) or after curing for one month at ambient temperature (cured compost). Temperature for the biodegradation test was 55°C.

days, which was in the range of 50 to 150 mg per gram suggested by the ISO [4]. This result suggested that compost taken right after the thermophilic degradation stage could be a better inoculum for the biodegradation test than the compost cured at an ambient temperature.

The effect of compost condition on the biodegradation was higher for cellulose than that of the SG, a synthetic polymer. Cellophane biodegradation varied significantly depending upon the compost. On the contrary, biodegradation for the synthetic polymer was similar in both composts. That is, cellulose-degrading microorganisms seem to be more sensitive to the temperature change than the synthetic polymer degrading microorganisms.

## CONCLUSION

The effects of test temperature and compost condition on the biodegradability of degradable polymers in the composting test were studied. When biodegradability tests for the natural (starch, cellulose, PHB/HV) and synthetic (PCL, SG, PLA) polymers were performed at the temperatures of 35 and 55°C, along with the compost made from food waste and cured at an ambient temperature, degradations of cellulose and starch were higher at 35°C than at 55°C due to the priming effect, while the degradations of other polymers were higher at 55°C. In the biodegradation test at 55°C, the compost harvested immediately after the thermophilic degradation stage showed higher biodegradation activities compared to the cured compost for both the synthetic aliphatic polyester (SG) and a natural polymer, cellulose. These results reveal that the biodegradation test at 55°C with compost harvested right after the thermophilic degradation stage represent the highest biodegradation activity under the controlled composting condition. Based on these results, we suggest that the current biodegradability test of polymers with cured compost under the controlled composting condition should be re-evaluated in view of compost activity.

## Acknowledgments

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