

ACCURATE AND EASY EVALUATION OF AEROBIC MICROBIAL-DEGRADABILITY OF BIODEGRADABLE PLASTICS UNDER CONTROLLED SOIL

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Introduction

Environmental disruption brought about by industrial waste is now of a global concern. That is, pollution of soil and pollution of ocean to be caused by dumping industrial waste into the soil or at the sea can be causative of disruption of ecosystem including the human society, giving raise to a serious social problem. Under such circumstances, degradation of organic waste to be achieved by microorganisms is the cleanest purification system that occurs in the realm of nature. Accordingly, to substrate those substances which are now chemically synthesized with those having microbial-degradability can be a useful measure for solving or improving the problem of environmental disruption induced by industrial waste as described above.

As an example of such substance having microbial degradability, biodegradable plastics that are decomposed by soil or water microorganisms are under development and are sharing some part of organic industrial waste. The biodegradable plastics are expected: (1) to be stable during the period of use, and (2) to be decomposed soon in the soil by microorganisms after disposal. Although it is possible to measure the period during which biodegradable plastics can be used stably, there is established no standardized familiar method of measuring the rate of degradation in biodegradable plastics to be caused by microorganisms, so that evaluation results of biodegradable plastics are of inconsistency, which is a main cause of hindering development of high-quality biodegradable plastics. There exists so far no familiar evaluation apparatus relating to total degradation of organic matters in the soil and in compost into CO₂. As carbon dioxide analytical methods, following methods are generally known: (1) quantitative determination of CO₂ captured in a strong alkaline solution by means of neutralization titration; and (2) quantitative determination of CO₂ captured in a gas reservoir by use of gas analyzer. However, since the volume or weight of the organic matter to be tested ranges widely from 1 cc to several hundreds of ton, and besides organic matters are of different compositions, there is reported no standardized test method. Accordingly, the test method (1) or (2) is selected depending on the respectively subjects to be tested, with no established standardized means for evaluating biodegradability. Authentic method is stipulated under ASTM (American Society for Testing and Materials D5209-92, D5338-92)¹, in which the biodegradable plastics are allowed to be decomposed by activated sewage sludge microorganisms to measure the amount of CO₂ formed by decomposition by means of titration. Especially, JIS K6953 based on ISO 14855^{2,3} under controlled composting conditions was established in May 2000. It is an excellent test-method with automatically calibrating the evolved carbon dioxide by the gas chromatography, but presents some difficulties. First, the part of reactor is on a large scale and the apparatus is expensive. Second, it costs a great deal to examine the specimens for developing biodegradable polymers, compounds and manufactured goods. The present study is to carrying out an accurate and easy evaluation of microbial-degradability of organic matters with a simplified apparatus in a laboratory scale.

Experimental

Materials. The compost manufactured by Yahata Bussan Co., Ltd. (Shizuoka, Japan) was used as inoculum and was subjected to

selection using a 6.5-mesh official sieve. The resulting compost under 6-mesh was used as such. The aged farmyard compost was made from wasted mushroom bed and poultry-manure. The chemical properties were total dry solid (42%), volatile solid (70%), pH (7.0), total organic carbon (12%), total nitrogen (1.7%), and C/N ratio (6.8), respectively. The biodegradable polymer (250 μ m-pass) was prepared by mixing the granular polymer and dry ice pieces by a blender. As the cellulose, a filter paper powder (40 mesh) manufactured by K.K. Tokyo seisakusho was used. As the sea sand (grain size: 20 to 35 mesh), 5 Kg of Wako's reagent grade sand was rinsed three times with distilled water, and the resulting sand which was dried at 100°C was used. The role of sea sand is involved for the advantages of the water-holding and soil texture between the inoculum and test material. All the components were mixed with gently and then put into a reactor.

Apparatus. An apparatus (MODA: Microbial Oxidative Degradation Analyzer⁴) is designed for evaluating microbial-degradability of an organic matter, the apparatus comprising: a reaction column maintained constantly at a fixed temperature and containing an organic matter as an evaluation subject or a cellulose as an evaluation reference together with a prescribed microbial source; means for forming saturated water vapor, connected to one end of the reaction column, the means being provided with first CO₂ adsorption means and feeding CO₂-free saturated water vapor into the reaction column; and an adsorption column, connected to the other end of the reaction column, the adsorption column containing second CO₂ adsorption means as shown in Figure 1. The amount of CO₂ can be determined by measuring the weight gain in the adsorption means having adsorbed CO₂. As described above, it is extremely easy to determine the weight if CO₂ by measuring the weight gain in the adsorption means caused by adsorption of CO₂, and even inexperienced operators can carry out easy measurement.

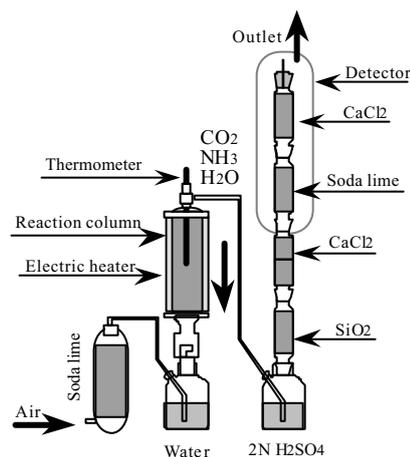


Figure 1. Scheme of apparatus MODA

Cellulose biodegradation test Rinsed sea sand, compost, cellulose and water according to the formulation shown in Table 1 were taken to a 5-liter plastic vessel. Then the resulting mixture was started well, and consequently introduced to the reaction column in the apparatus shown in Figure 1 to measure the amount of CO₂ formed under the following conditions: quantity of air flow: 40ml/min; reaction temperature: 58°C. Measurement of CO₂ carried out for 46 days, and the sample in the reaction cylinder was subjected to turning after 7, 14, 21, 28, 35 days of the reaction. The turning was carried out by transferring the sample in the reaction column into a 5-liter plastic vessel and stirring it well therein.

At the same time, the water content of the sample was adjusted at 35~40%. After completion of turning, the sample was returned into the reaction column.

Biodegradation test of biodegradable polymers. Difference in the biodegradability depending on the structure of biodegradable polymer was examined. Sea sand rinsed with distilled water, compost and a polymer were taken into a 5-liter plastic vessel according to the formulation shown in Table 2, and after the resulting mixture was stirred

well, it was introduced to the reaction column to measure the amount of CO₂ formed under the same condition of the cellulose biodegradation test. Each polymer was rinsed three times with distilled water, and the thus treated polymer from which the excess of water was decanted off was used. The water content in a reactor was adjusted at 40%.

Table 1. Cellulose biodegradation test

Test No.	Ratio of components(g)			
	cellulose	compost (dry weight)	sea sand	water
1, 2	0	60	320	195
3, 4	5	60	320	195
5, 6	10	60	320	195
7, 8	15	60	320	195

Table 2. Biodegradation test of polymer

No	Polymer derivative	Mixing ratio		
		polymer (g)	Compost (cc)	Sea sand (cc)
1	3-Hydroxybutyrate	10	250	250
2	Cellulose	10	250	250
3	Polybutylene succinate adipate	10	250	250
4	Starch derivative	10	250	250
5	Control	0	250	250

Results and Discussion

Cellulose biodegradation test. The results of measurement of amount of evolved CO₂ and the estimated biodegradability³ are shown in Figure 2 and 3, respectively. The measurements were carried out at the end of the experiment within the prescribed time intervals during 46 days. The total amount of CO₂ evolved from the test material for 46 days was increased with the amount of cellulose. The relation was shown as the linear equation $y=1.230x+9.559$ with the correlation coefficient 0.994. The percentage of biodegradation was over the 70 % biodegradability of cellulose as a reference material established by ISO 14855. It was considered that the biodegradability over 70% was based on the advantages of the water-holding and soil texture between the inoculum and test material by the addition of sea sand. The high linearity showed the stable and constant activity of microorganisms in reaction column to be maintained during the experimental period.

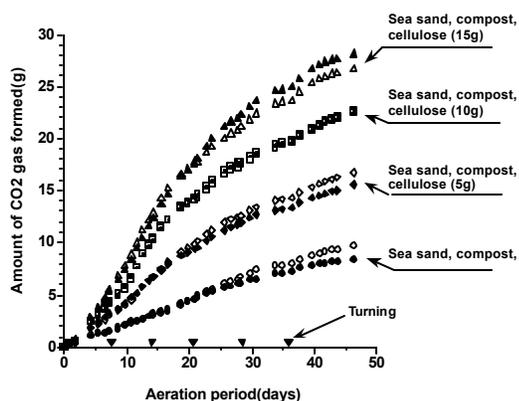


Figure 2. Biodegradation of cellulose in controlled soil at 58°C

Biodegradation test of biodegradable polymers. Figure 4 shows the biodegradability of biodegradable plastics in controlled soil at 35°C. Difference in the biodegradability depending on the structure of biodegradable polymer was distinctly observed. Poly 3-hydroxybutyrate

showed the high biodegradability than cellulose. As the starch derivative polymer mixed with PVA, the biodegradation proceeded slowly in this experiment. Polybutylene succinate adipate (#3030) showed the same biodegradability as cellulose at the end of the experiment during 38 days, but the time lag with biodegradation was observed.

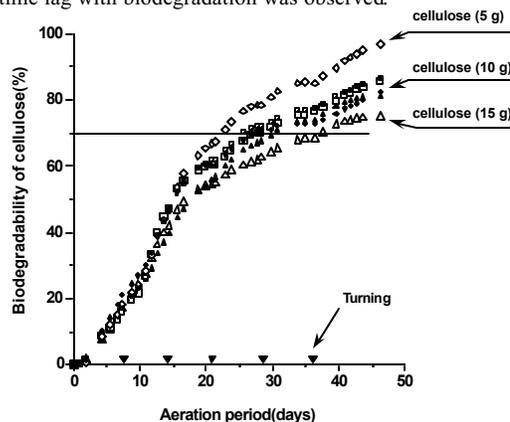


Figure 3. Biodegradability of cellulose in controlled soil at 58°C.

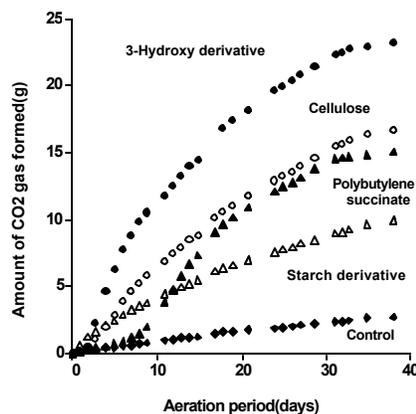


Figure 4. Biodegradability of biodegradable plastics in controlled soil at 35°C.

Conclusions

Cellulose biodegradation test showed the amount of cellulose in reaction column to be determined in an error with few percent. It was obvious from the biodegradability over 70% that the advantages of the water-holding and soil texture between the inoculum and test material was gained by the addition of sea sand to compost. The results of this method by using MODA-apparatus were approximately coincided with JIS K6953 (ISO14855). In conclusion, under the controlled composting condition during the experiment, this method demonstrates an effective screening test method for the compostability of plastic materials in a laboratory scale.

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