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Rating on Biodegradability of Microwave-Induced Low-Density Polyethylene-Totally Degradable Plastic Additives (LDPE–TDPA) in Petri Dish Screen Test by ISO 846

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Abstract

The environmental concern on accumulation of throwaway plastic materials imposes the synthesis of oxo-biodegradable plastic such as Low-Density Polyethylene–Totally Degradable Plastic Additives (LDPE–TDPA) plastic polymer as a waste management option for polymer in the environment in recent years.

In order to enhance the biodegradability of LDPE–TDPA, initial oxidation through microwave induction was conducted for its disintegration by using a domestic microwave oven of 950 W at the frequency of 2.45 GHz and indicated 17.02% decreased value of its tensile strength. The resulted microwave induced plastic was then exposed to the activated sludge microorganisms withdrawn from wastewater treatment plant under anaerobic condition in solid environment in petri dishes.

The assessing the bacteria and fungal resistance was conducted by the rating scheme based on visual assessment used by ISO 846. Weight loss of 28-day duration in petri dish test recorded that microwave induced film reached 3.92% while untreated film occurred 1.82%. Surface morphological changes and weight losses of biodegraded plastic films showed the assimilation of activated sludge microorganisms based on SEM analysis and spectral imaging analysis. The rating level of microbial resistance to both plastic films was identified as 3 due to more than 25% cover of microorganisms on the surface.

Key words: *LDPE–TDPA,* microwave *. induction, activated sludge microorganisms, rating by ISO 846.*

Introduction

Due to public awareness to growing plastic wastes as litter an interest in degradable plastics turns to solve this concern. Two types of degradable plastics are currently being introduced as biodegradable plastic: (a) biodegradable plastics, including starch-based polymers and polyesters, that

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are designed to break down through the actions of microorganisms; and, (b) oxo-biodegradable plastics with prodegradant additives that are designed to break down under the influence of thermal and ultraviolet light, before final biodegradation takes place through the activities of microorganisms. Plant based cellulosic plastic, bacterial based plastic such as polyhydroxy β alkanoate (PHA), and starch based plastic undergo into the first category of biodegradable plastics and totally degradable prooxidant containing plastic like Low density polyethylene–totally degradable plastic additives (LDPE–TDPA) consists of the second category of oxo-biodegradable plastics.

The direct incorporation of oxidative additives (prodegradants) such as transition metals and ketone containing materials into the backbone of Polyethylene (PE) polymer facilitates the disintegration and subsequent microbial colonization. Initial thermal or photodegradation enhances the eventual biodegradation of LDPE containing prodegradants by molecular fragmentation into low molecular weight compounds such as ketones. aldehydes and carboxylic acids. The resulting low molecular products are accessible to microorganisms (Dupret, David, and Daro, 2000; Chiellini, Corti, and Swift, 2003).

The present study evaluated the biodegradability of microwave induced-LDPE–TDPA by exposing activated sludge from wastewater treatment plant through petri dish screen test in mineral salt agar media under anaerobic condition following rating scheme based on visual assessment used by ISO 846

Materials and Methods

Low-Density Polyethylene-Totally Degradable Plastic Additive (LDPE–TDPA) plastic samples were obtained from Planet Friendly Plastics Incorporation (PFPI), Philippines.

Activated sludge inoculum was withdrawn from the station 1 wastewater treatment plant of Yuchengco Building of De La Salle University (DLSU), Manila.

Microwave Induction

Plastic films were cut into strips (3 x 15 cm^2) and placed into a teflon vessel, measuring 8 × 19 cm^2, and then put onto the turning disc (rotating reflector) inside the microwave cavity and irradiated under atmospheric pressure for four hours. After microwave induction, the films were recut into smaller (3 x 3 cm^2) pieces.

Biological Treatment

Preparation of Inoculum

The freshly withdrawn activated sludge was filtered and decanted. The inoculum solution was prepared to 2:8 (v/v) with distilled water, aerated, and then used within six hours of sampling.

Petri Dish Screen Test in Solid Anaerobic Condition

The Petri Dish Screen Test was performed based on ISO 846 (International Organization for Standardization). The test involved placing the plastic material on the surface of a mineral salts agar in a petri dish containing no additional carbon source (Seal, 1995). Mineral salt agar media contains in g/L as follows: potassium dihydrogenphosphate, 0.7; dipotassium hydrogenphosphate, 0.7; magnesium sulphate heptahydrate, 0.7; ammonium nitrate, 1; sodium chloride, 0.005; ferrous sulphate, 0.002; zinc sulphate, 0.002; manganese sulphate, 0.001; and agar, 15 and adjusted to pH 7.

Petri dishes were sterilized in the autoclave at 120°C for 30 min. Agar nutrient was poured into the sterilized petri dishes up to 6 mm deep. The preweighed test polymers with a size 3×3 cm^2 were then placed on the solidified agar surface and 1 ml of the inoculum solution was sprayed both on the agar surface and on the test polymer using a sterilized atomizer. The petri dishes were sealed and incubated at a constant temperature range of 28–30°C for 28 days. The growth was monitored and recorded for each week. This test was done in a triplicate procedure for each sample.

Characterization of Microwave Induced- and Biodegraded Microwave Induced- LDPE–TDPA

Surface Morphological Changes by SEM Analysis and Surface Analysis by Spectral Imaging System

The surface morphology before and after being subjected to microwave induction and biological degradation was observed using JEOL JSM-5310 Scanning Electron Microscope (SEM) and the new Spectral Imaging-System, SD-300 with Dual Mode Optical Head.

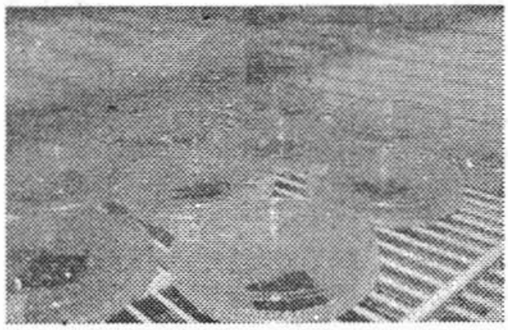
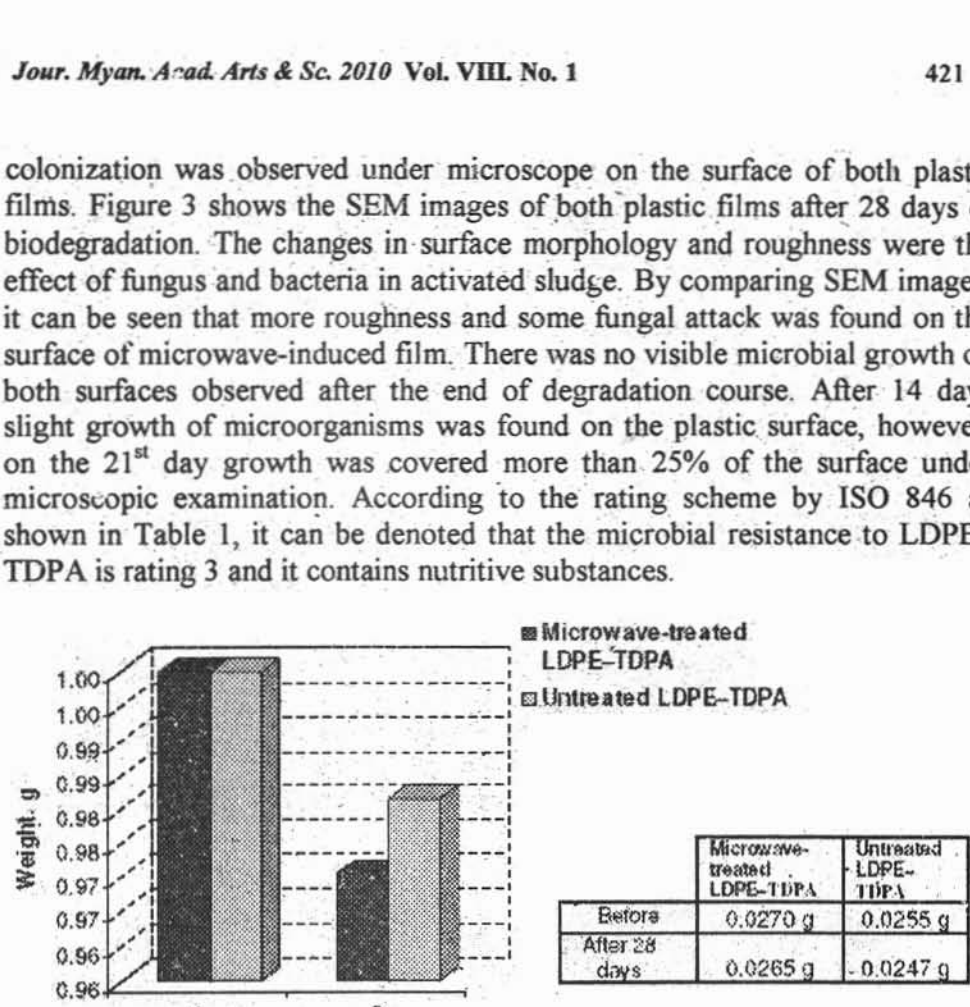


Figure 1 Petri Dish Screen Test

Results and Discussion

A maximum four hours of microwave induction showed 17.02% decrease in tensile strength from 14.1Mpa and no change in elongation at 25mm. Untreated and microwave-induced LDPE–TDPA plastic samples were then exposed to laboratory controlled simulated petri dish screen test under anaerobic condition using fresh activated sludge as inoculum from station 1 wastewater treatment plant. Activated sludge is a mixture of microorganisms involving bacteria, protozoa, and higher microorganisms such as rotifers. The pH of activated sludge could affect the enzymes that generate the biochemical reaction in the bacteria in activated sludge. The pH range in present sludge was at an optimum level of 6.84-7.2 that is proper for the microorganisms in activated sludge and the BOD was below 10 mg/L.

Biodegradability was evaluated by performing petri dish screen test for its ready biodegradability at 28–30°C based on ISO 846 procedure. To establish the resistance to microbial attack on the plastic materials plastic films a 3×3 cm^2 were placed on the surface of agar in petri dishes containing no additional carbon source, sprayed with activated sludge inoculum and incubated at 28–30°C for 28 days. Weight loss and surface morphology changes were assessed weekly on the 7^{th}, 14^{th}, 21^{st}, and 28^{th} day. Weight loss of LDPE–TDPA first occurred during the sampling on the 7^{th} day, whereas no apparent weight loss was observed in microwave induced- LDPE–TDPA, however, after 28 days, weight losses were found apparently in both plastic films as shown in Figure 2. The weight losses were observed 3.92% or 0.02475 g of the original 0.0255 g for microwave induced film and 1.85% or 0.0265 g of the original 0.027 g for untreated film. For the first week, little colonization was observed under microscope on the surface of both plastic films. Figure 3 shows the SEM images of both plastic films after 28 days of biodegradation. The changes in surface morphology and roughness were the effect of fungus and bacteria in activated sludge. By comparing SEM images, it can be seen that more roughness and some fungal attack was found on the surface of microwave-induced film. There was no visible microbial growth on both surfaces observed after the end of degradation course. After 14 days slight growth of microorganisms was found on the plastic surface, however, on the 21st day growth was covered more than 25% of the surface under microscopic examination. According to the rating scheme by ISO 846 as shown in Table 1, it can be denoted that the microbial resistance to LDPE–TDPA is rating 3 and it contains nutritive substances.



	Microwave-treated LDPE-TDPA	Untreated LDPE-TDPA
Before	0.0270 g	0.0255 g
After 28 days	0.0265 g	0.0247 g

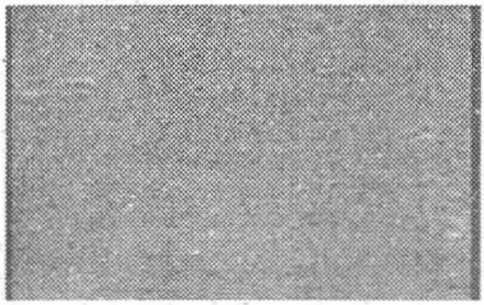
Figure 2 Weight Loss Profile of Petri Dish Test 1. Before and 2. After 28 days

Table 1 Rate Scheme for Assessing Microbial Resistance by ISO 846

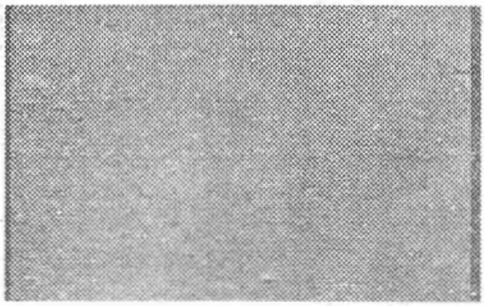
Visual Assessment	Rating	Evaluation
No growth apparent even under a microscope	0	Not a nutritive medium for microorganisms
Growth invisible or hardly visible to the naked eye, but clearly visible under the microscope	1	Contains nutritive substances

Visual Assessment	Rating	Evaluation
Slight growth covering less than 25% of the specimen surface	2	Not resistant to fungal attacks and contains nutritive substances
Growth covering more than 25% of the specimen surface	3	Not resistant to fungal attacks and contains nutritive substances

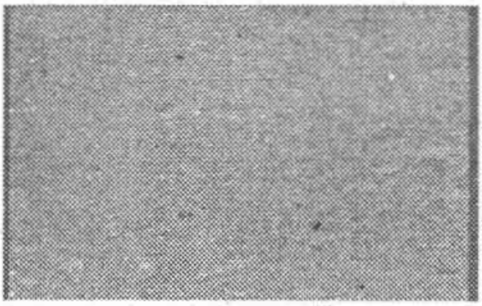
Source: ISO 846



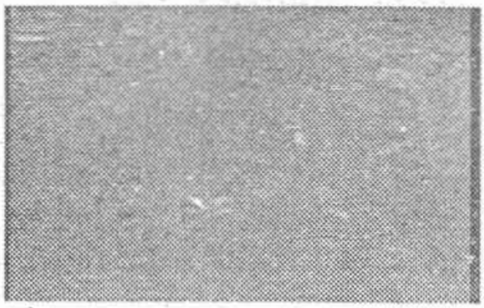
Untreated Film at Day 0



Untreated Film at Day 28



Microwave Induced Film at Day 0



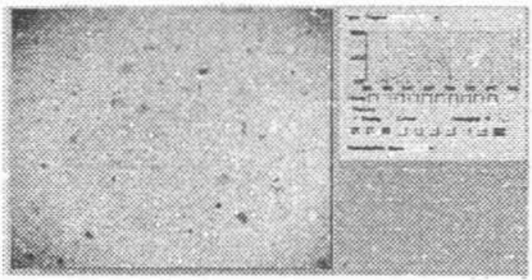
Microwave Induced Film at Day 28

Figure 3 SEM at 3500x of Petri Dish Test Samples for Untreated LDPE–TDPA and Microwave-Induced LDPE–TDPA Before and After 28 Days of Biodegradation

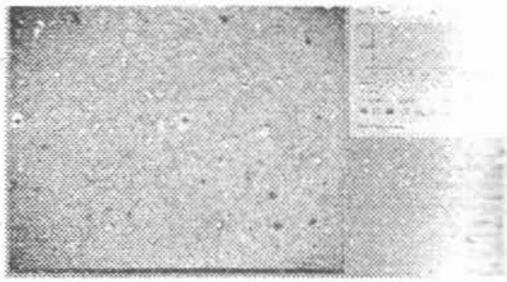
Aside from SEM analysis, surface analysis was also conducted using spectral imaging system, SD-300 with Dual Mode Optical Head. Spectral images present the color intensity versus wavelength of the spectrum. One part of test specimens was taken under spectral microscope and the results were interpreted as three spectral units indicated by green color for white matters of the plastic surface; blue color for black matters; and red color for the rest part of the surface as shown in Figure 4. The white matters of the plastic films may represent the attack of microorganisms and their assimilation on the plastic films whereas black matters may represent the disintegrated fragments resulted from microwave induction. For the rest of plastic surface it may represent a clear zone that showed no evidence of microwave induced effect and no bioassimilation by the microorganisms. Therefore, the rest of the surface resembled the untreated film, no bioassimilation and no stress by microwave heating of the plastic film surface would occur so that white and black matters would be lesser. For the microwave-induced LDPE–TDPA film, there would have lesser white matter and greater black matters because of the stress on the surface resulted by microwave induction. On the other hand, biodegraded microwave-induced film would show higher white matters and lesser black matters. Moreover, the biodegraded untreated LDPE–TDPA would have lesser white and black matters on the surface. A summary of the results of spectral images is shown in Table 2. According to the above mentioned assumption, the results in Figure 4 proved that the biodegraded microwave-induced LDPE–TDPA had a higher white matters and a lesser black matters, indicating higher bioassimilation and biodegradation had occurred on its surface. For biodegraded untreated film a higher white matters occurred, however, the black matters were observed to increase so that biodegraded untreated film did not follow the assumption given for a lesser black matter. These observations could be due to some other components present in LDPE–TDPA such as stabilizers and other prodegradants. But the biodegraded microwave-induced LDPE–TDPA showed a lesser black matters and a higher white matters in petri dish screen test.

Table 2 Summary of the Results of Spectral Images

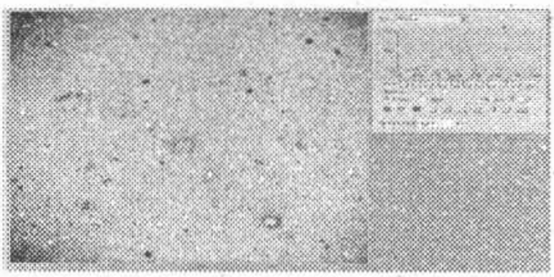
Plastic Film	Description	
	Intensity of white matter	**Intensity of black matter**
Untreated LDPE–TDPA	7713	4454
Untreated film in Petri Dish Screen Test	9026	5212
Microwave-Induced LDPE–TDPA	6614	3827
Microwave-Induced film in Petri Dish Screen Test	8232	2402



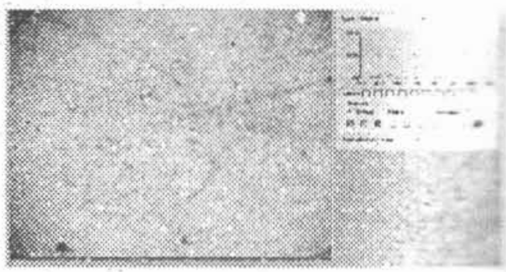
Untreated Film at Day 0



Untreated Film at Day 28



Microwave Induced Film at Day 0



Microwave Induced Film at Day 28

Figure 4 Spectral Images: Untreated Film and Microwave-Induced Film in Petri Dish Test

Conclusions

After microwave induction it was observed that changes in mechanical properties, such as tensile strength, indicate that LDPE–TDPA can be oxidized by heat generated by microwave. Petri dish screen test under anaerobic degradation of microwave-induced film showed two times weight loss of untreated film. Both the untreated and microwave-induced LDPE–TDPA plastic film reached the rating level of 3 for ready biodegradability test defined by ISO 846.

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