Assessment and Applications of Multi-Degradable Polyethylene Films as Packaging Materials

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Abstract Degradation performance of environmentally friendly plastics that can be disintegrated by combination of sunlight, microbes in soil, and heat produced in landfills was evaluated for use in industries. Two multi-degradable master batches (MCC-101 and MCC-102 were manufactured, separately mixed with polyethylene using film molding machine to produce 0.025 mm thick films, and exposed to sunlight, microbes, and heat. Low- and high-density polyethylene (LDPE and HDPE) films containing MCC-101 and MCC-102 became unfunctional by increasing severe cleavage at the surface and showed high reduction in elongation after 40 days of exposure to ultraviolet light. LDPE and HDPE films showed significant physical degradation after 100 and 120 days, respectively, of incubation at 68±2°C. SEM images of films cultured in mixed mold spore suspension at 30°C and 85% humidity for 30 days revealed accelerated biodegradation on film surfaces by the action of microbes. LDPE films containing MCC-101 showed absorption of carbonyls, photo-sensitive sites, at 1710 cm⁻¹ when exposed to light for 40 days, whereas those not exposed to ultraviolet light showed no absorption at the same frequency. MCC-101-based LDPE films showed much lower M_w distribution after exposure to UV than its counterpart, due to agents accelerating photo-degradation contained in MCC-101.

Keywords: multi-degradable master batch (M/B), low density polyethylene (LDPE), high density polyethylene (HDPE), food packaging film, photolysis, biodegradability, thermal degradability, mulching test

Introduction

The materials commonly called plastics are synthetic polymers, which are chemically formed and relatively new compared to natural materials such as tree, paper, stone, metal, and glass. Given a wide range of properties, including processing capabilities and duration, plastics have rapidly become ubiquitous in our everyday life and are used in all industries, contributing to the economic development and modernization of the human life. Although plastic-based flexible packaging materials and containers are crucial in the food industry, their use in food packaging also sparks concerns for environmental pollution. Because most plastics used for food packaging are semipermanent and non-degradable, releasing deadly poisonous dioxins when incinerated and land-filled as well as emitting air pollutants when not completely combusted (1-3). Thus, massive waste plastic materials, including vinyl bags, Styrofoam, and plastic containers, are cited as the culprit of environmental pollution (1-3). Therefore, development of environmentally friendly plastics is a key to overcoming these harmful effects of plastics. By making plastics environmentally degradable by sunlight, soil microorganisms or heat of landfill gas, the use of plastics would be safer and more convenient.

The use of degradable plastic materials for shopping bags and plastic containers is mandatory in developed countries, including the US, Japan, Germany, and Italy (4, 5), as a part of the effort to develop a new degradable polymer and commercialize degradable plastic products. Degradable plastics are divided into three typesbiodegradable, bio-disintegrated, and photodegradablebased on raw materials and reaction mechanism in chemical composition (6-9). Biodegradable plastics are manufactured from biopolymer materials such as polyhydroxyl butyric acid (PHB), pullulan, and hyaluronic acid (HA), or naturally derived substances such as alginate, cellulose, and chitin. These compounds of biological origins are combined with other polymer materials in the manufacturing process. Biodegradable plastics do not need to be incinerated and buried in a landfill, because they decompose when in contact with water, carbon dioxide, and methane gas through the actions of microorganisms. the decomposition process taking from several months up to 2 years. However, biodegradable plastics are not seen as the replacement for plastics used in food packaging, because they are weak in terms of tensile strength, water resistance, and processing efficiency, as well as being costly (6). Bio-disintegrated plastics are made by integrating naturally degradable compounds such as cornstarch with synthetic polymers such as polyethylene (PE), polypropylene (PP), and polystyrene (PS), and various degradation accelerators are added to increase their degradability (7). Although bio-disintegrated plastics are not costly, they cannot be used for food packages due to lower durability and strength, limiting their use to shopping and garbage bags. The amount of degradation accelerators is so limited that they not only degrade slowly but also are not completely degradable, compared with biodegradable plastics. Photodegradable plastics disintegrate into small

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pieces when exposed to sunlight. In other words, the ultraviolet (UV) radiation disintegrates the polymer structure and eventually lowers the physical properties of resin and polymer molecules, leading to further degradation. Adding transition metal catalysts, oxidation accelerators, and photosensitive materials to the main fraction of polymeric species for accelerating photodegradation makes plastics photodegradable; by inducing carbonyl radicals working as photo-sensitive sites, they break down when exposed to UV light (290-350 nm). Depending on their chemical composition, photodegradable plastics absorb different wavelength of light and degrade at different speed. The 290 nm wavelength region is considered adequate to break the covalent bond between carbon and hydrogen (8, 9). Although production of photodegradable plastic products is cost effective, contemporary technology allows the production of films only. The problems related to photodegradable plastics are that they remain nondegradable in landfills due to the lack of sunlight and that they are inextricably dependent upon the environment to degrade. Moreover, the use of photosensitive additives raises the concern for heavy metal toxicity (9).

A new type of degradable plastics, called "multi-degradable plastics" with combined properties and capabilities of the three plastics mentioned above, have been developed and commercialized in developed countries, including Canada, the US, and Japan. However, these plastic materials have not been widely used in commercial applications due to high price (10). Following three steps can explain the degradation mechanism of multi-degradable polymers. Firstly, the structure of multi-degradable plastics becomes porous owing to the decomposition of biodegradable materials contained in the plastics, causing decreased strength, elongation percentage, and expanding effect of a surface area. Secondly, auto-oxidation reaction is triggered by oxidation accelerators, eventually decreasing molecular weight, and plastic

structure disintegrates into small pieces. Lastly, the biodegradation takes place by the actions of microorganisms or enzymes. However, degradation of multidegradable plastics do not necessarily follow the three phases in order under natural conditions. All degradation processes can occur simultaneously or codependently.

The purpose of this study was to assess environmental degradability, such as biodegradability, photodegradability, and thermal degradability, and safety of three kinds of multi-degradable plastics, high- and low-density polyethylene (HDPE & LDPE), which can be used for garbage and shopping bags, agricultural mulching film and various packages of household products.

Materials and Methods

Master batch (M/B) manufacturing All materials used for the production of functional master batch multidegradable through a combination of biodegradation, photo-degradation, and thermal degradation are presented in Table 1. LDPE (grade 530G manufactured by Samsung Total Petrochemicals, Seoul, Korea) was mixed with cornstarch, poly-caprolactone (PCL), photo-degradation additives, and benzophenone in a kneader. For the first reaction, two rotors were kept at 20 and 25 rpm for 30 min. After adding various additives such as chemical degradation accelerator, radical initiator, and auto-oxidizer to the master mix, the second reaction was performed for 20 min. The mixture was then passed into a single-screw extruder (Brabender Stand-Alone/E19-25D, Duisburg, Germany) at 170°C. Master batch pellets with diameters ranging from 2 to 3 mm (MCC-101) were produced. The abovementioned manufacturing method of M/B yielded many polymer radicals caused by the high shear stress produced in the kneader following polymer chain scission. These polymer radicals then reacted with oxygen, aiding in the formation of photo-sensitive agents able to absorb

Table 1. Compositions of master batch

Functions	Materials	Compositions (%)	
Biodegradable polymer	Polycaprolactone (PCL)	10.0	
Biodegradable polymer	Corn starch	20.0	
Biodegradable polymer	Tapioca starch	10.0	
Inorganic filler	CaCO ₃	30.0	
Stabilizer (antioxidant)	Benzene propanoic acid	0.6	
Degradation accelerator	1st Transition metals	0.2	
Degradation accelerator	2nd Transition metals	0.2	
Oxidation accelerator	Aromatic ketone group (benzophenone, etc.)	0.3	
Oxidation accelerator	Organic acids (citric acid, etc.)	0.4	
Oxidation accelerator	Sugars (glucose, etc.)	0.3	
Oxidizable component	Unsaturated fatty acids (oleic acid, etc.)	2.0	
Lubricant	Waxes	3.0	
Adhesive agent	Low density polyethylene (LDPE) for MCC-101 High density polyethylene (HDPE) for MCC-102	23.0	
Total		100.0	

sunlight (8, 11). The production of master batch based on HDPE (grade F120A, Samsung Total Petrochemicals, Seoul, Korea) followed the same procedures mentioned above. The mixture was then passed into a single-screw extruder, and turned into master batch pellets (MCC-102) through a batch hot cut process conducted at 200 to 220°C.

Film manufacturing The master batch pellet (MCC-101) was mixed with LDPE in the kneader at different proportions, constituting 26% (plus additional 4% transition metal), 28% (plus additional 2% transition metal), and 30% total volume using a blender (1400-OOBBIN, Yoojin Engineering, Seoul, Korea). The mixture was then moved to the hopper, and the processing temperature was maintained at a range of 160 to 170°C to produce films with average thickness of 0.025 mm using a T-Die film molding machine (Cast Film & Sheet Chill Roll Machine, LABTECH Engineering, Bangkok, Thailand). In addition, 30% of the MCC-102 master batch was blended in HDPE, and the mixture was then moved to the hopper. The 0.025-mm thick films were produced at 190 to 220°C.

Safety assessment as packaging materials The safety of test films as packaging materials were evaluated in compliance with the guidelines set by Korea Health Industry Development Institute (KHIDI). The heavy metal content (mg/L), potassium permanganate consumption (mg/L), lead concentration (mg/kg), dry residues (mg/L), and cadmium concentration (mg/kg) of the three master batches were measured in accordance with the standard test methods for plastics implemented by the KHIDI (12).

Assessment of photo-degradation The photo-degradability of resin was measured based on American Society for Testing and Materials (ASTM D) 3826-98 method (13). A 20-W mercury lamp (F40T10BLB, Sankyo Denki, Tokyo, Japan) with strong emission in wavelength region of 352 was installed in a box (160×50×50 cm), which was made in-house. Film samples cut to the size of 13×80 mm were then fixed at 10 cm below the lamp and exposed to air. The samples were collected at 7-day intervals, and changes in elongation were measured six times each using the Instron Universal Testing Machine (Instron 5565, Canton, MA, USA) by pulling films to the point of film rupture at 100 mm/min and comparing the stretched length with the original length. Changes in elongation were averaged.

Assessment of thermal degradation To measure thermal

degradation, film samples were cut to the size of 13×80 mm and put into an incubator ($68\pm2^{\circ}C$ and 85% humidity). Samples were collected at 7-day intervals, and changes in elongation were measured six times each and averaged.

Assessment of mold degradation The degrading activities of five mold strains (Korea Research Institute of Bioscience and Biotechnology) were assessed using ASTM G21-96 method (14). All strains were sub-cultured twice on each medium listed in Table 2. Newly formed spores were picked with a platinum inoculating wire loop and shaken carefully to spread them for filtering. After filtering the liquid out, mold strains were partitioned and placed into sterile flasks, to which the same amount of each mold was added and diluted into spore suspension containing $1,000,000 \pm 200,000$ spores/mL liquid. The number of spores was measured by picking a small portion of the mixed spore suspension with a yellow tip and using Hemacytometer (Surerier, Lauda-Koenigshofen, Germany). The counting plate consisted of nine 1 mm² wells. The average number of spores was calculated by summing up figures shown at four corners of the wells. The computation was repeated four times to ensure uniform number of spores in 1 mL liquid. Film samples (13×80 mm) were placed on top of the minimum nutrientsalt medium, and the mixed spore suspension was thoroughly sprayed over the film samples and the medium. After arranging the weight deviation of each treatment at ± 0.001 g, four replicates of each treatment were cultured at 28 to 30°C and humidity of 85% for 60 days. During this period, changes in weight of film samples were tracked every 20 days to calculate the average weight. To measure the weight loss of film samples, the samples were taken from the medium and washed in distilled water, and sterilized by soaking in 1% HgCl₂ liquid for 2 min. The samples were washed in 85°C distilled water and showered with 75% alcohol to remove the molds from the surfaces of films. After sterilizing, samples were dried in the oven at 105°C for 3 to 4 hr until no changes in the weight of samples were found. Concomitantly, growth of microorganisms was evaluated by examining the surface morphology of samples with a scanning electronic microscope (Phillips XL30 ESEM-FEG, Nottingham, Holland).

Mulching test To investigate degradability of films in natural environments, LDPE and HDPE films manufactured with a 30% MCC-101 and 30% MCC-102,

Table 2. Medium for the culture of fungi

Fungi	ATCC No. ¹⁾	Medium
Aspergillius niger	9642	Potato Dextrose Agar
Penicillium pinophilum	11797	Harrold's M40Y
Trichoderma virens	9645	Potato Dextrose Yeast Agar
Aureobasidium pullulans var. pullulans	15233	Sabourauds Agar
Penicillium funiculosum	11797	Potato Dextrose Agar

¹⁾Available from Korean Collection for Type Cultures.

respectively, were cut to the size of 60×40 cm, and the plastic mulching was tested. A 10-cm high nursery bed was made in the garden after eliminating weeds and was then mulched with films. Degradability of films was tracked for 4 months using a digital camera.

Interpretation of infrared absorption spectrum (FTIR) To investigate the effect of the functional master batch for LDPE (MCC-101) on chemical changes associated with UV exposure and changes in chemical structure during photo-degradation, IR spectra of plastic films before and after UV exposure were observed as follows using an FTIR (Perkin-Elmer 1760X, Wellesley, MA, USA). After impurities were removed, film samples were washed in sterile water for 2 hr and dried in a vacuum dry oven for 24 hr. The dried film samples were treated with p-xylene solvent, and extracted for 2 hr using a Soxhlet extractor. After removing the extraction residue, the extracted liquid was put into the vacuum dry oven to evaporate all solvents. The remaining material was turned into a 0.15mm thick film in a hot press at 170°C, and the IR spectrum was observed using the FTIR.

Changes in molecule weight A decrease in molecular weight of MCC-101 based LDPE samples before and after UV exposure was measured using a gel permeation chromatography (GPC, Waters 150C, Milford, MA, USA) and a refractive index (RI) as a detector. A calibration curve was completed using THF as a carrier solvent and a standard LDPE sample.

Results and Discussion

Safety as food packaging materials The safety of LDPE resin (MCC-101) and HDPE resin (MCC-102) for use as additives of food packages was evaluated through the test conducted in accordance with procedures specified by the government-run health industry control unit. The test results showed that both master batches were qualified for use in food packaging (Table 3).

Assessment of photo-degradation Generally, tensile strength and elongation are seen as important barometers in estimating photo-degradability, and the ultimate tensile strength and elongation values at the break point were statistically compared to determine photo-degradability. Elongation is more closely related to degradability (15, 16). Changes in average elongation of each test sample toward cross direction (CD) and machine direction (MD)

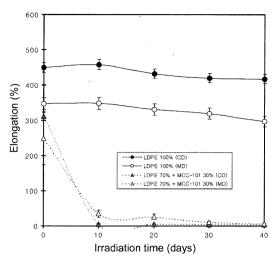


Fig. 1. Changes in elongation of LDPE films during UV irradiation.

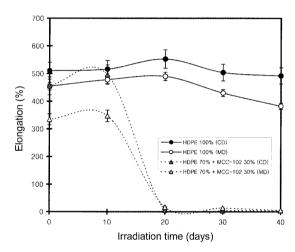


Fig. 2. Changes in elongation of HDPE films during UV irradiation.

during the periods of UV irradiation are shown in Fig. 1 and 2. LDPE film, which was made with 30% MCC-101, sustained only 0.5% of the original elongation for both directions after 40 days of UV exposure. Although the MCC-102-based HDPE film showed a higher original elongation in MD compared with CD, only 0.5% of original elongation remained for both directions after 40 days of UV exposure. In addition, a slight increase in elongation was observed for the MCC-102-based HDPE

Table 3. Results for safety tests for MCC-101 and MCC-102 as a material of food packaging

Items	Contents			
	MCC-101	MCC-102	Regulation	
Amount of heavy metal	Trace	Trace	below 1.0 mg/L as Pb	
Amount of lead (Pb)	0.9 mg/L	0.6 mg/L	below 100 mg/kg	
Consumption of KMnO ₄	Trace	Trace	below 10 mg/L	
Evaporating residue	3.0 mg/L	4.0 mg/L	below 30 mg/L	
Amount of cadmium (Cd)	Trace	Trace	below 100 mg/L	

film after 10 days of exposure, due to the cross-linking structure formed at the early stage of photo-oxidation reaction, as suggested by previous studies (17, 18).

Considering ASTM D3826 standard, which defines that degradation process is complete when the elongation reaches below 5%, MCC-101-based LDPE and MCC-102-based HDPE demonstrated that almost complete photo-degradation occurred in 20 days. In the case of LLDPE, almost 10% of original elongations was maintained after 1 year of UV exposure (17). Thus, degradable master batches contained in films played a key role in accelerating photo-degradation by enabling photosensitive materials such as transition metals, benzophenone, and other oxidation accelerators to undergo photo-degradation in the presence of UV light (Table 1). These results suggest the possibility that multi-degradable films can be broken down and disintegrated into small fragments with time by the action of natural factors, including wind, rain, animals, and plants.

Assessment of thermal degradation The thermal degradation of test films demonstrated results similar to those presented in the previous section, although films showed slower changes in elongation. Changes in average elongation, which was computed after measuring elongation six times for each film, were presented according to various time exposure lengths (Fig. 3 and 4). MCC-101-based LDPE-1 lost almost all of its original elongation in 100 days. MCC-102-based HDPE film took 120 days until the original elongation completely disappeared. These results showed that the extent of the damage of thermoplastics (LDPE and HDPE) increased with increasing temperature, which was triggered by the addition of degradable master batches. Generally, promoting various chemical reactions, the heat produced under most predictable conditions breaks thermoplastic resin. The most significant degradation sequence starts from the formation of free radicals such as -OH caused by small amounts of iron ions contained in the plastics. Free radicals react with polymers, leading to the

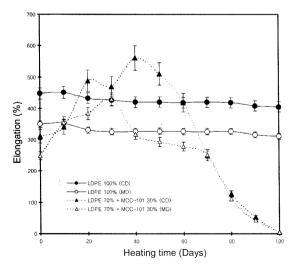


Fig. 3. Changes in elongation of LDPE films during heat treatment ($68 \pm 2^{\circ}$ C).

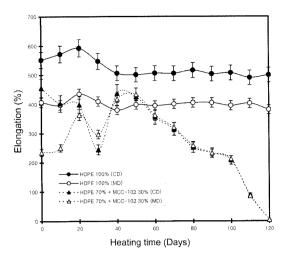


Fig. 4. Changes in elongation of HDPE films during heat treatment ($68 \pm 2^{\circ}$ C).

production of new free radicals, which then react with the C-C bonds, accelerating the disintegration of polymers into monomers such as alcohol and ketone (19, 20). Transition metals, copper and vanadium, which are contained in degradable master batches, are known as catalysts in the promotion of chemical reactions involving free radicals. It is also assumed that heat-induced degradation of films was accelerated by the catalytic role of copper and vanadium contained in MCC-101 and MCC-102 master batches.

Assessment of biodegradation Due to the shorter time requirement and consistent control of environmental conditions, the assessment of degradation utilizing direct injection of cultured molds is considered superior to the traditional degradability evaluating method through soil reclamation. Therefore, the accelerated method is widely used as a pre-step to the assessment of biodegradation in the real world for the development of degradable plastics (21, 24). Our study aimed at determining the effects of five different types of mold, which are abundant in the soil, according to assessment methods for plastic resins set by ASTM. Images from scanning electron microscopy (SEM) showed more active mold growth on the surface of LDPE and HDPE films, including function mater batches, after the injection of cultured fungi (Fig. 5b and 6b), compared to the pre-injection films (Fig. 5a and 6a). As claimed by Cole (25), microbes appear to be capable of penetrating into micropores, such as the surface of plastic films, when they exist under appropriate conditions. Examination of the test revealed that microbes first disintegrate compounds such as cornstarch, PCL, and carbohydrate. As a result, cleavages increased at the surface and in the structure of the films, and molecule mass of the polymer decreased, allowing a more complete degradation.

Interpretation of mulching test After mulching nursery beds with LDPE and HDPE films, each containing 30% functional mater batches, periodical variations were observed (Fig. 7 and 8). The test films began showing a physical degradation in part of the film, and the breakdown increased as time passed (Fig. 7b and 8b).

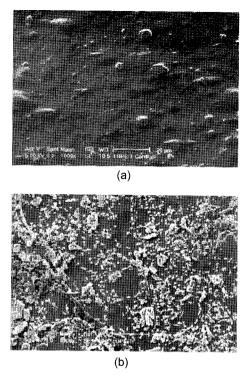
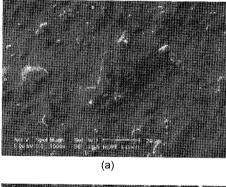


Fig. 5. Scanning electron micrographs of the surface of LDPE 70% + MCC-101 30% (a) before and (b) after 30 days of microbial attack.



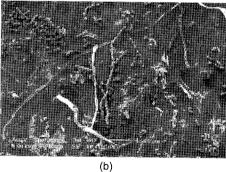
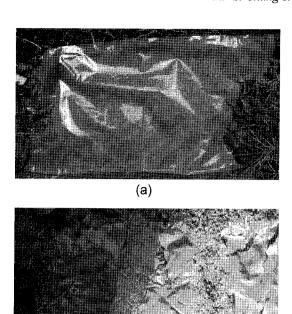


Fig. 6. Scanning electron micrographs of the surface of HDPE 70% + MCC-102 30% (a) before and (b) after 30 days of microbial attack.

After 4 months of mulching, the films were mostly depleted (Fig. 7c and 8c). These results showed that the degradation of test films occurred via multiple processes in natural environment without undercutting the growth of



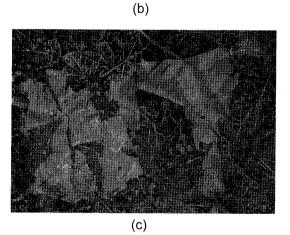
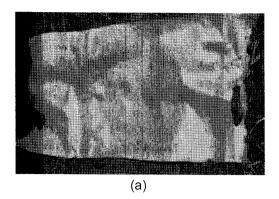


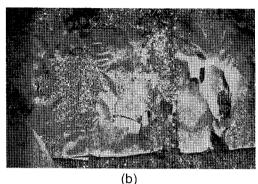
Fig. 7. Visual changes of LDPE 70% + MCC 30% film during mulching test. (a) initial (b) after 2 months and (c) after 4 months mulching in the yard.

plants.

Interpretation of infrared absorption spectrum (FTIR)

As shown by the changes in mechanical properties, photooxidation occurred in test films that underwent photodegradation. To confirm this finding, chemical changes associated with UV radiation were investigated by comparing UV-exposed and -unexposed test films. After 40 days of exposure to UV light, the IR absorption spectrum representing carbonyl group at 1710 cm⁻¹ was observed in the film Fig. 9b, whereas the spectrum representing carbonyl group was not found in the film not exposed to UV Fig. 9a. These results confirmed that the LDPE films containing functional master batches and additional transition metal used in the study successfully induced photo-oxidation, proving their photo-degradability. The results also support the findings of our previous studies (26, 27) that claimed that the manufacturing





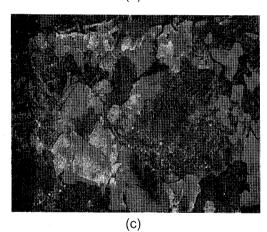
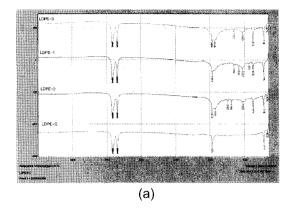


Fig. 8. Visual changes of HDPE 70% + MCC 30% film during mulching test. (a) initial (b) after 2 months and (c) after 4 months mulching in the yard.

process of master batches used in this study is capable of inducing photo-sensitive sites through shearing stress mechanism. Consequently, such a formation of carbonyl absorption layer is deemed as a functional group that is able to induce photo-oxidation by absorbing UV light at the region of long wavelength greater than 300 nm (17), and the increment of carbonyl group implies that Norrish Type II reaction is underway (28).

Changes in molecule weight Forty days following exposure to UV radiation, changes in molecule weight of the test films were investigated (Table 4). The average molecular weights (M_w) of LDPE-C (LDPE 100%), LDPE-2 (LDPE 70% + MCC-101 26% + additional transition metal 4%), and LDPE-3 (LDPE 70% + MCC-101 28% + additional transition metal 2%) were 187,000.



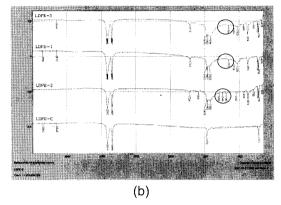


Fig 9. IR spectra of LDPE films exposed to UV irradiation for (a) 0 days and (b) 40 days (LDPE-C : LDPE 100%, LDPE-1 : LDPE 70% + MCC-1 30%, LDPE-2 : LDPE 70% + MCC-101 26 % + additional transition metal 4%, LDPE-3: LDPE 70% + MCC-101 28% + additional transition metal 2%). Peaks in the circle represent carbonyl group.

Table 4. Changes in molecular weight of low density polyethylene (LDPE) films before and after 40 days UV exposure

-		Mn ⁴⁾	Mw ⁵⁾	Mz ⁶⁾	MWD ⁷⁾
LDPE-C ¹⁾	before UV exposure	39,000	187,000	576,000	4.81
	before UV exposure after UV exposure	38,000	184,000	581,000	4.64
LDPE-2 ²⁾ a	before UV exposure	48,000	189,000	584,000	3.94
	after UV exposure	7,500	22,010	52,000	2.91
	before UV exposure after UV exposure	2,700	13,000	32,000	4.71

¹⁾LDPE 100%.

189,000, and 190,000 before exposure to UV light, whereas those exposed to UV light for 40 days showed 184,000, 22,000, and 13,000, respectively, showing almost no changes in Mw for the 100% LDPE film and a significant reduction in Mw for the LDPE films containing the functional materials. These results explain that longchain polymers with relatively high molecular weight

²ILDPE 100/0. ²ILDPE 70% + MCC-101 26% + additional transition metal 4%. ³ILDPE 70% + MCC-101 28% + additional transition metal 2%.

⁴⁾Number average molecular weight.

⁵⁾ Average molecular weight. -average molecular weight.

⁷⁾Molecular weight distribution.

transformed into short-chain molecules through chain scission caused by the auto-oxidation in the presence of UV, due to the functionality of photo-sensitive materials in the films. The short-chain molecules can be further degraded by the action of microbes, resulting in biodegradation.

The present study assessed photo-degradability, thermal degradation, and biodegradability of multi-degradable plastics that can be used for food packages such as singleuse food containers, trays, cups, plates, and pouches. The mulching capability of these plastics, considered the most essential part of soil testing, was also determined. The degradation process of waste plastics, however, occurs under the combined influences of various factors in real world. For instance, landfilling test is one of the primary methods for estimating the degradability of plastic wastes. Therefore, further studies are necessary to estimate the degradability of plastics under the conditions that reflect a real waste management environment by simultaneously exploring UV-based technique, heat treatment, and mold culture. In addition, because this study used mold strains only for the biodegradability assessment, the effects of anaerobic and non-anaerobic bacteria and birds on degradability of plastics should be further investigated.

Acknowledgments

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