

The Disintegration Rate of Traditional and Chemically Modified Plastic Films in Simulated Fresh- and Sea-Water Environments

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SYNOPSIS

Six plastic films with varying chemical compositions in three different accelerated weathering environments were evaluated for disintegration. The films included two traditional films (LDPE and polystyrene), three with enhanced photodegradability (2% ECO and 10% ECO and LDPE with a vinyl ketone graft), and one with enhanced biodegradability (LDPE with 6% corn starch). The films were exposed to UVA-340 bulbs to simulate sunlight while in fresh water, salt water, or no water conditions. Disintegration of the films was determined by monitoring the change in physical properties of tensile strength at break, elongation at break, and toughness at break at selected intervals throughout the exposure period. Both the chemical composition of the film and the exposure environment produced significant differences in disintegration rates. Two of the films with enhanced photodegradability (2% and 10% ECO copolymers) disintegrated more rapidly than did the other films in this study. The environment where no water was present resulted in the fastest disintegration rate for the films when compared with the fresh-water environment. However, two films, LDPE and LDPE with 6% corn starch, degraded more rapidly in the salt-water environment than in the other two environments due to the presence of oxidation catalysts in the water system. © 1993 John Wiley & Sons, Inc.

INTRODUCTION

There is growing concern over the contamination of our environment. Recent investigations have shown plastics to be a major contributor to the debris found on land and in water systems.¹ The advantages of plastics over traditional natural products such as fibers and metals have resulted in their obtaining an increasing share of the market.² Traditional plastics have been engineered to be inert to the environment and therefore may take centuries to degrade. There is mounting evidence that plastic debris poses a hazard to wild resources in fresh water and at sea.³⁻⁶ The contamination of plastics that are inherently inert to the environment is a threat to the ecosystems of these environments. Increased concern over the fate of plastics has renewed interest in the development and evaluation of plastics that are susceptible to disintegration by weathering.

There are no specific standards for weathering, but it is rather a complex system and can include one or any combination of actions on the substance by living organisms: light, heat, oxygen, water, ionizing radiation, or chemical reagents.

Disintegration of polyethylene films in outdoor exposure is primarily a result of the light-initiated thermooxidative disintegration of the polymer. The autooxidative pathway for polyethylene has been discussed extensively.⁷ Oxidation is initiated by the generation of free radicals of functional groups, additives, or impurities present in the polymer. Hydroperoxides are also thought to play a role in the initiation and the propagation of autooxidation, and other initiation mechanisms have been proposed. Oxygen reacts with the radical to form a hydroperoxy radical that transforms into a polymeric hydroperoxide group by hydrogen abstraction from the polymer. Each propagation cycle creates hydroperoxides that can photolytically or thermally dissociate to provide further initiator species. Each initiation leads to multiple propagation cycles until dispro-

portionation or inactivation due biradical combination terminates the radicals. The propagation step in this sequence of reactions is associated with main-chain scission event. Simultaneous cross-linking by radical-radical interaction may also occur.

There are several methods of modifying polyethylene to enhance disintegration. To enhance photoinduced disintegration, carbonyl groups are incorporated into the polymer chain either by copolymerization of carbon monoxide with ethylene to produce a copolymer (ECO) or vinyl ketone molecules are introduced as a graft to the polymer chain. Another common method of enhancing the disintegration rate is by using metal compound additives to enhance the autooxidation process.⁸⁻¹⁰

To enhance the biodegradability of a plastic, a biodegradable additive can be incorporated into the polymer structure prior to extrusion. The most common biodegradable additive is corn starch. Studies by Griffin have shown this to be an effective method in enhancing the disintegration rate of polyethylene.¹¹

There is controversy in the discussion of this area of research (degradable plastics) due to the inconsistent use of terms by researchers and the general media. For this paper, terms will be used as defined here. These are consistent with definitions currently under review by ASTM. Disintegration is the physical breakdown of the plastic and can be monitored by measuring tensile properties, tear strength, elongation, energy at break, and other suitable measurements. Disintegration can be a result of changes in the chemical structure or because of physical forces. The term degradation reflects the chemical changes and can be determined by measuring average molecular weight, functional groups, and other chemical characteristics. Disintegration as a result of physical forces (i.e., wind, animals) is deterioration and does not greatly affect the chemical properties previously described. Disintegration may be a result of one or a combination of these processes. Frequently, it is possible to identify the initial primary mechanism of disintegration.

In this study, films of traditional and chemically modified polymeric films were placed in simulated fresh- and salt-water environments and exposed to accelerated sunlight. Disintegration was monitored by measuring selected physical properties at predetermined intervals throughout the exposure period. The relationship between the chemical composition of the film and the exposure environment on film disintegration was examined. The disintegration rates of plastics exposed to simulated sunlight in aquatic environments are expected to be dif-

ferent from the rates of those exposed only to the light source. Pegram and Andradý evaluated the differences between marine and land environments and reported that polymeric materials degraded more slowly in the sea than on land.¹² One difference in the disintegration rates of plastic on land and at sea is attributed to the fact that plastics exposed to sunlight build up heat and become significantly warmer than the surrounding air temperature, therefore enhancing thermooxidative reactions.^{12,13} Other factors also identified as influencing degradation rates of polymeric materials on land and at sea were the plasticizing action of small quantities of sorbed water and fouling of the plastics. A thorough discussion of this topic was given in the previous study.¹² There are no reported studies comparing the disintegration rates between plastics in aquatic and marine environments.

MATERIALS AND METHODS

This study examined the disintegration rates of six different plastic films during exposure to accelerated weathering using UV fluorescent tubes (UVA-340) in simulated fresh- and salt-water environments. The films represented both traditional plastics and those that had been chemically altered to enhance degradability. Three replications, each containing three samples, were completed in this study.

Plastics

Plastic films of six different chemical compositions were evaluated in this study and are listed in Table I. Two of the films were traditional plastics commonly used in packaging (A and E), three were modified to have enhanced photodegradability (B, C, and D), and one was modified to have enhanced biodegradability (F). The first five plastics (A-E) were supplied by Dow Chemical Co. (Freeport, TX), and the sixth plastic (F) was a U-Bag[®] manufactured and marketed by Archer Daniel Midland (Decatur, IL). These bags with enhanced degradability were produced by blending silted intact starch grains and other additives with low-density polyethylene and were marketed as U-Bags. The plastics were cut into sheets of 20.3 × 27.9 cm, and they were of similar thicknesses (Table I).

Environments

Eighteen 30-gallon glass aquaria (30.5 × 91.5 × 30.5 cm) were placed on benches in a temperature-con-

Table I Plastic Films: Description and Characteristics

Plastic	Thickness (m × 10 ⁻⁵)	
	Mean	SD
(A) Low-density polyethylene (LDPE)	5.83	0.15
(B) 2% ethylene carbon monoxide copolymer (2% ECO)	6.54	0.16
(C) 10% ethylene carbon monoxide copolymer (10% ECO)	6.97	0.12
(D) Polyethylene with vinyl ketone graft (PE + graft)	4.58	0.14
(E) Polystyrene (PS)	4.55	0.23
(F) Polyethylene with 6% corn starch (PE + CS)	4.28	0.09

trolled greenhouse ($24 \pm 4^\circ\text{C}$). The aquaria were set up in four groups of four aquaria and one group of two. Each group was placed under a bank of eight UVA-340 bulbs from Q-Panel Co. (Cleveland, OH). The UVA-340 lamp emits radiation beginning at approximately 295 nm and peaking at approximately 350 nm. This is considered the best artificial solar source to simulate light in the lower wavelengths. The bulbs were mounted in shoplight-type fixtures. The fixtures were mounted in wooden frames and suspended so the lamps were 5.1 cm above the aquarium (7.6 ± 0.5 cm above the water surface). Acceptable water level tolerances (7.6 ± 0.5 cm from lamps) were labeled on each aquarium, and the water level was monitored throughout the exposure period. Some water loss occurred due to evaporation and when the samples were removed for testing. Water was added as necessary to maintain the acceptable level in each aquarium. Black shade cloths completely covered the lights and aquaria systems to prevent exposure to any outside light during the exposure period.

UV/Fresh Water

All aquaria were filled with tap water, and the chlorine was allowed to dissipate. The aquaria were then inoculated with bacteria collected from 20 temporary and permanent water sources throughout downstate Illinois to introduce microorganisms typically found in fresh-water systems. Aquaria were cross-inoculated by transferring approximately 1 L aliquots of water to each aquarium to maintain an aquatic environment equal in bacteria, algae, and aquatic invertebrates. Cross-inoculation was necessary to establish and maintain these populations in all tanks. As is true with any natural fresh-water environment (i.e., pond, lake, river), the composition varies not only from source to source, but also from location

to location in the same source. In this study, the goal was to introduce microorganisms typically found in fresh-water systems into our laboratory systems. However, it was virtually impossible to assure that microbial populations in all tanks were identical. The aquaria were allowed to condition for several days. Plate counts were used to ensure that similar bacteria, algae, and aquatic invertebrates were present in each aquarium throughout the study.

UV/Salt Water

The salt-water system was simulated by filling clean aquaria with tap water, and the chlorine was allowed to dissipate. The salinity was increased to 1.021 ± 0.002 using Instant Ocean®, a commercially available product. Instant Ocean contains sodium chloride, calcium chloride, magnesium chloride, sodium carbonate, potassium chloride, sodium sulfate, and trace metals including iron and manganese. Bacteria commonly found in marine environments were added to each aquarium and the cross-inoculation of the water from the aquaria was completed as described for the fresh-water system. In both water environments, the plastic films were randomly floated on the surface of the water, 7.6 ± 0.5 cm from the lamps, and no overlapping of the films occurred.

UV/No Water

In addition to the fresh- and seawater environments, one environment with exposure to the accelerated simulated light and no water was established. In this environment, the films were placed on white foam core board 7.6 cm from the bulbs. The film samples were randomly placed with respect to aquaria group and location to offset any inherent biases in the experimental design.

Exposure Intervals

Preliminary studies were conducted to establish exposure intervals. In an uninterrupted exposure period (115 h in the environment expected to be the harshest, UV/no water), only the 10% ECO copolymer (B) reached the brittle point, at 11.5 h. Based on these data, plastic B was exposed for a maximum of 16 h with sampling intervals at 0, 4, 8, and 16 h. The remaining five plastic films were each exposed for 240 h with sampling intervals of 0, 60, 120, 180, and 240 h. Physical properties of the plastics were measured at each testing interval.

Analysis

At each testing interval, three samples of each plastic were removed from the appropriate aquaria, and three 5 × 15.2 cm specimens in the machine and

transverse directions were cut from each sample using a twin-blade cutter. The samples were tested for tensile strength, elongation, and energy at break (TEB) according to modified ASTM Test Method D 882: Test Method for Thin Plastic Films. An Instron tensile tester, table model, equipped with a data analysis package from Hyperplot, was used to calculate the TEB. The modifications included changing the grip distance to 5.1 cm and the pull speed to 12.7 cm/min. This change in testing procedure was made after the first replication of the UV/no water environment was completed. Therefore, this environment contains only two replications rather than three. The change was made to reflect a proposed recommendation by the ASTM Committee on Degradable Plastics.¹⁴ Elongation is considered by some to be a more appropriate parameter than is tensile strength for measuring physical degradation because it reflects the brittleness and con-

Table II Mean Tensile Strength^a for All Plastic Films in All Environments at Each Testing Interval

Environment	Hours of Exposure	Tensile Strength (MPa) (SD)				
		Films				
		LDPE	2% ECO	PE-graft	PS	PE + CS
I ^{b,c}	0	> 200	> 200	> 200	28 (5)	> 200
	60	*	11 (6)	> 200	36 (6)	> 200
	120	> 200	11 (3)	> 200	38 (9)	> 200
	180	> 200	10 (9)	> 200	36 (9)	> 200
	240	> 200	10 (3)	> 200	34 (7)	> 200
II ^b	0	> 200	> 200	> 200	28 (5)	> 200
	60	105 (50)	9 (3)	> 200	50 (27)	95 (32)
	120	87 (32)	9 (4)	> 200	58 (32)	95 (41)
	180	116 (62)	10 (7)	> 200	59 (28)	101 (42)
	240	102 (28)	9 (3)	> 200	59 (25)	77 (28)
III ^b	0	> 200	> 200	> 200	28 (5)	> 200
	60	> 200	15 (14)	> 200	70 (13)	> 200
	120	> 200	8 (7)	> 200	68 (15)	> 200
	180	158 (70)	0	> 200	65 (15)	> 200
	240	137 (84)	0	> 200	62 (16)	> 200
10% ECO	Hours of Exposure	Environment				
		I	II	III		
		0	> 200	> 200	> 200	
		4	10 (1)	12 (5)	12 (2)	
		8	5 (0)	0	0	
		12	0	0	0	
16	0	0	0			

^a Values based on maximum load for plastics LDPE, 10% ECO, 2% ECO, PE-graft, and PE + CS.

^b I = UV/freshwater; II = UV/saltwater; III = UV/no water.

^c *n* = 18 due to changes in testing procedures.

* Not possible to calculate mean.

sequent tendency of the plastic to fragment.¹⁵ The plastic is considered embrittled when the average elongation at break is less than 5%. Because of the limited maximum extension length of the equipment, the maximum elongation, tensile strength, and TEB were not obtainable for five of the plastic films (all except polystyrene [PS]); therefore, the results reported for these samples are the maximum obtainable values, and the calculations are based on this value. This limitation may have prevented detecting the initial change of properties but did not influence the determination of the brittle point, as defined by ASTM.

RESULTS AND DISCUSSION

The tensile strength at break, elongation at break, and TEB are reported for all films in Tables II, III,

and IV, respectively. Statistical analysis showed significant differences between disintegration rates of the various films and by environment.

Environments and Disintegration

Films exposed to the UV/fresh-water system showed the least change in the physical properties during exposure. This supports the proposed theory that the presence of water reduced heat buildup. The lower temperature reduced the potential for thermooxidative reactions and polymer chain scission. There were no indications that the bacteria present in this water system were using the film as a food source or contributing significantly to the disintegration process. If bacteria were using the film for food, they would have attached themselves to the surface of the films as has been reported in previous

Table III Mean Percent Elongation^a for All Plastic Films in All Environments at Each Testing Interval

Environment	Hours of Exposure	Elongation (%) (SD)				
		Films				
		LDPE	2% ECO	PE-graft	PS	PE + CS
I ^{b,c}	0	> 1200	> 1200	> 1200	13 (2)	> 1200
	60	> 1200	134 (16)	> 1200	21 (15)	> 1200
	120	> 1200	48 (34)	> 1200	12 (8)	> 1200
	180	> 1200	18 (12)	> 1200	17 (2)	> 1200
	240	> 1200	1 (0)	> 1200	29 (17)	> 1200
II ^b	0	> 1200	> 1200	> 1200	13 (2)	> 1200
	60	> 1200	59 (29)	1125 (428)	43	678 (300)
	120	> 1200	46 (32)	1112 (462)	38	772 (362)
	180	> 1200	38 (19)	1168 (385)	43	800 (451)
	240	> 1200	40 (2)	1178 (247)	43	762 (216)
III ^b	0	> 1200	> 1200	> 1200	13 (2)	> 1200
	60	> 1200	32 (30)	> 1200	15 (12)	> 1200
	120	> 1200	15 (13)	> 1200	20 (22)	> 1200
	180	966 (307)	0	> 1200	23 (18)	> 1200
	240	833 (437)	0	> 1200	16 (12)	> 1200

10% ECO	Hours of Exposure	Environment		
		I	II	III
	0	> 1200	> 1200	> 1200
	4	47 (27)	96 (47)	9 (8)
	8	7 (3)	44 (4)	0
	12	0	38 (2)	0
	16	0	28 (2)	0

^a See footnote a to Table II.

^b See footnote b to Table II.

^c See footnote c to Table II.

Table IV Energy at Break (TEB)^a for All Plastic Films in All Environments at Each Testing Interval

Environment	Hours of Exposure	TEB (mJ/m ³) (SD)				
		Films				
		LDPE	2% ECO	PE-graft	PS	PE + CS
I ^{b,c}	0	> 700	> 700	> 700	7 (3)	> 700
	60	> 700	33 (15)	> 700	111 (38)	> 700
	120	> 700	19 (15)	> 700	120 (42)	> 700
	180	> 700	8 (12)	> 700	86 (43)	> 700
	240	> 700	9 (3)	> 700	64 (24)	> 700
II ^b	0	> 700	> 700	> 700	7 (3)	> 700
	60	330 (198)	4 (2)	635 (158)	4 (2)	239 (125)
	120	255 (200)	3 (3)	598 (238)	4 (3)	292 (138)
	180	369 (153)	3 (1)	661 (202)	4 (3)	304 (206)
	240	318 (112)	3 (0)	674 (215)	4 (2)	228 (172)
III ^b	0	> 700	> 700	> 700	7 (3)	> 700
	60	> 700	23 (6)	> 700	7 (4)	> 700
	120	> 700	11 (9)	> 700	7 (3)	> 700
	180	580 (255)	0	> 700	95 (26)	> 700
	240	503 (306)	0	> 700	111 (32)	> 700

10% ECO	Hours of Exposure	Environment		
		I	II	III
	0	> 700	> 700	> 700
	4	37 (24)	4 (2)	25 (7)
	8	4 (8)	3 (0)	0
	12	0	3 (0)	0
	16	0	3 (0)	0

^a See footnote a to Table II.^b See footnote b to Table II.^c See footnote c to Table II.

research on biodegradable plastics.¹⁶ There was zero-to-little fouling of the samples in this study.

In the UV/salt-water environment, there was a more rapid loss of properties for two of the films, LDPE and PE + CS, than in the other two environments in this study. This was unexpected as it was reasonable to expect that any heat buildup that would accelerate disintegration would be dissipated as was demonstrated in the UV/fresh-water environment. In evaluating the chemical composition of the Instant Ocean used to prepare the simulated salt-water environment, there were small quantities of metals present. If the plastics initially sorbed small quantities of water-containing metals, the oxidative reaction would have been initiated, resulting in change in the physical properties of the film. Although it was not possible to obtain the exact composition of the trace metals, both iron and man-

ganese were included. Previous research has shown that these metals can enhance degradation of the polymer.¹⁷ The UV energy is absorbed by the metal complexes, thus releasing the metal ions, which act as catalysts to break the polymer chains. The decrease in elongation was noted at the first testing interval (60 h) in the UV/salt-water environment, and no significant additional disintegration was observed. This type of initial dramatic loss, followed by little or no loss, is consistent with previous research. It is hypothesized that the metal components react with the fatty-acid-like material of the polymer, forming peroxides or free radicals and causing scission of the polymer chain. Over time, these may be leached into the surrounding environment, and the disintegration process is compromised.

The PE-graft film also exhibited a different disintegration pattern in the UV/salt-water environ-

ment than in the other two environments. There was a slight reduction in elongation during the exposure period in the UV/salt-water environment, but there was no significant loss.

The 2% and 10% ECO copolymers (B and C) showed no significant differences between the environments. However, the films in the UV/no water environment were embrittled more quickly than in the other two environments. This was expected as water was not present and heat buildup occurred, enhancing the thermooxidative reactions and accelerating the disintegration process. Had testing intervals been closer together for both of the ECO copolymers, significant differences due to environment may have been detected.

Chemical Composition and Disintegration

The two ECO films (B and C) had significantly faster rates of disintegration than did the other films evaluated in this study. These were the only films to become embrittled during exposure. The rapid disintegration rate was expected as these films contained carbonyl groups sensitive to rupture by UV light. Carbonyl groups absorb UV energy in the range of 270–360, thus raising the electrons to higher orbitals and energy levels, reducing the stability. Most photodegradation of polyethylene follows Norrish-type reactions, resulting in the rapid reduction of molecular weight and an accompanying loss in mechanical and physical properties.¹⁷ As the percentage of carbonyl groups increases, the disintegration rate increases, as there is the potential of the UV energy to break the polymer chain at each carbonyl site, making the plastic brittle. In this study, the 10% ECO copolymer lost physical properties significantly faster than did the 2% ECO copolymer due to the increase in the number of carbonyl functional groups. The other film with enhanced photodegradability, PE-graft (D), was expected to degrade at a rapid rate as well, but no significant decrease in the physical properties was seen in the physical properties of this film. In the salt-water environment, there was a change in the elongation as the metal salts attacked the polyethylene backbone of the polymer chain, resulting in chain scission. Information on the percent carbonyl groups incorporated into this polymer was not available.

The PE + CS film showed no loss of elongation in two of the three environments: UV/fresh water and UV/no water. However, there was a significant loss in elongation and the other measured physical properties in the UV/salt-water environment as

previously discussed. The PE + CS was developed to be susceptible to attack by living organisms, as research has shown that hydrolytic degradation takes place when polymers containing hydrozable groups (such as starch) are exposed to moisture.¹⁸ If hydrolysis is achieved enzymatically, then the process is usually considered to be biodegradable. Guillet¹⁹ confirmed that polymer fragments are indeed metabolized by soil microorganisms; however, in this study, there was no indication that the starch component was contributing to the disintegration process. Scanning electron microscopy revealed on the film's surface no detectable pitting, which has been present in other research evaluating the disintegration of starch-containing polyethylene.¹⁶ A series of investigations on biodegradation of polyethylene have been previously completed, and results have shown that the disintegration of polyethylene is affected by preliminary irradiation for a UV source and the presence of photodegradable additives.²⁰ As the molecular chains are broken, the molecular weight is reduced, and the number of chain ends increase and the plastic becomes more susceptible to biological degradation by microorganisms. In this study, the lack of fungal growth on the film indicated lack of attack by microorganisms. The degradation in a UV/salt-water environment can be attributed to the presence of oxidizing metals as previously discussed.

The LDPE film (A) was not modified to have enhanced degradation and showed no loss of properties in the UV/fresh-water environment. There was some disintegration at the 180 h testing interval of the UV/no water environment, as heat buildup contributed to the thermooxidative reaction. Also, as previously discussed, the presence of metals in the UV/salt-water environment enhanced the disintegration of the film.

The PS film (E) had a lower initial elongation than did the other films in this study. In each environment, the properties of the film initially increased. This can be attributed to the formation of free radicals, resulting in cross-linking as described in the introduction of this paper.

CONCLUSIONS

The chemical composition of the film and the environment had significant impacts on the physical properties of the polymeric materials evaluated in this study. The two ECO copolymer films disintegrated more rapidly than did the other films evaluated here due to their sensitivity to UV light. The

polystyrene film exhibited an increase in properties due to the cross-linking of free radicals that were photoinduced. The polyethylene with a vinyl ketone graft showed no significant change in properties under any conditions in this study.

In the UV/salt-water environment, the disintegration rate increased significantly for two of the films, PE + CS and LDPE, due to the presence of autooxidizing metals. The PE-graft also showed a significant change in properties in this environment. Because the rapid disintegration rate of the two ECO copolymer films, it was not possible to determine if the salt-water environment significantly impacted these films.

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