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Photo-Oxidative Degradation and Hydrolytic Degradation of Micro-Graphite Filled Poly(lactic acid) Composites

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Abstract: In a specific range of electrical conductivity, poly(lactic acid)/PLA has the potential to be developed into environmentally friendly antistatic packaging after a modification process. PLA was blended in a mini single screw extruder at 180°C with different compositions of micro-graphite (0, 0.5, 1, and 1.5 %wt.). This report discusses the degradability of PLA composite, i.e., photo-oxidative degradation and hydrolytic degradation. The weight loss, thermal properties, and cross-section morphology of the tested specimens were monitored periodically. During the degradation test, micro-graphite could be released from the composite, leaving a rough surface and reducing the weight of the composite. Differential scanning calorimetry (DSC) test exhibited that the presence of micro-graphite did not influence the melting temperature of the composition studied. However, the onset temperature of the melting point showed a slight shift of about 2-4°C. Bulk crystallinity demonstrated a considerable dependence on the micro-graphite loading (0-1.5%wt). However, there were two contradictory phenomena after both degradation tests. UV exposure could stimulate the fragmentation of PLA chains, break the crystal structure and increase the embrittlement. Thus, crystallinity tended to decrease during photo-oxidative degradation. In hydrolytic degradation, degradation firstly occurred in the amorphous regions and was ongoing within the studied range of time (0-20 weeks). Thus, the bulk crystallinity of composite tended to increase

Keywords: Photo-oxidative Degradation, Hydrolytic Degradation, Poly(Lactic Acid), Micro-Graphite, Composite

INTRODUCTION

The biodegradable polymers used in the world have grown to 60 percent or more than 1.2 million tons that are dominated by poly(lactic acid) (PLA) and poly(hydroxy alkanoates) (PHAs) (Balla et al. 2021). This growth provides an alternative solution to environmental problems due to the massive use of conventional petroleum-based polymers. Researchers from academia and industry are also playing a role in the growth of biodegradable polymers by adapting their properties (Elsawy et al. 2017).

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PLA is an aliphatic thermoplastic polyester obtained by polymerization of lactic acid (2-hydroxy propionic acid). As a monomer, lactic acid is mainly derived from renewable resources such as starch, corn, cellulose, etc. (Casalini et al. 2019; Jin et al. 2019). PLA has good mechanical properties, biocompatibility, composability, and biodegradability (Naser et al. 2021). Its mechanical properties can be compared with polypropylene (PP), poly(ethylene terephthalate) (PET), and polystyrene (PS). Thus, PLA has become the most promising polymer applied in various applications, such as packaging, bottles, containers, automotive, etc. (Reichert et al. 2020).

Nowadays, the use of polymers is dominated as packaging about 42% of plastic production (Geyer et al. 2017). Antistatic packaging is a unique packaging that protects electronic components or devices from electrostatic discharge (ESD). The ESD can cause damage to electronic components in the form of total failure or decreased performance (Cho et al. 2018).

ESD occurs when there is a transfer of electric charge between objects with different electrostatic potentials. Antistatic packaging plays a role in preventing the occurrence of ESD by inhibiting the accumulation of electric charge and dissipating static electricity slowly (de Souza Vieira et al. 2021).

With its superior properties, PLA allows being developed as an environmentally friendly antistatic packaging after modifying its electrical conductivity properties. Braga et al. (2021) explained that the main requirement of antistatic packaging is its electrical conductivity in the range of 10^{-11}-10^{-6} S/cm (Braga et al. 2021).

Our previous research concluded that PLA could be increased its electrical conductivity by adding carbon-based fillers (Esa Nur Shohih et al. 2020). Several researchers also reported similar results when adding fillers to PLA, such as multi-walled carbon nanotubes (MWCNT) (Lin et al. 2013; Mai et al. 2013), carbon black (CB) (Silva et al. 2019), and graphite (Sullivan et al. 2014).

PLA modification may affect biodegradability, so further research about biodegradability is needed. Generally, the percentage of biodegradation depends mainly on the nature of the medium and not on the preparation procedure (Copinet et al. 2003). Besides that, it depends on temperature, pH, molecular weight, dimensions of the sample, morphology, presence of humidity, crystallinity, and the filler or reinforcement particles (Codari et al. 2012; González-López et al. 2020; Gorrasi et al. 2018; Li et al. 2016).

The first step of PLA degradation is related to hydrolysis due to the presence of ester groups on the PLA backbone, which facilitates it. However, the degradation rate of PLA will be increased by placing the polymer under a composting medium rather than in soil or seawater (Rosli et al. 2021). Furthermore, the most critical cause of the natural degradation of polymer is solar radiation which can break polymer bonds (Litauszki et al. 2019). Mucha et al. (2014) reported structural, molecular order, and glass transition changes in neat PLA and its composites with UV exposure. Intrinsic viscosity and crystallinity can also be affected by photodegradation. Biodegradability also can be observed through its electrical characteristics (Mucha et al. 2014). Mai et al. (2013) studied the hydrolytic degradation behavior of PLA/carbon nanotubes (CNT) composites using electrical resistivity as a sensor. The result showed a relationship between electrical resistivity and degradation levels of the polymer (Mai et al. 2013).

This article focuses on a discussion about the degradability of PLA/micro-graphite composites, which is part of our research on the development of conductive polymer composite (CPC) in various applications such as semiconductor materials, antistatic packaging, etc. (Kaavessina et al. 2020; Esa Nur Shohih et al. 2020; Esa N. Shohih et al. 2020). Two kinds of degradation were observed and compared: photo-oxidative degradation and hydrolytic degradation. The samples were placed in two different conditions: exposed in the open air and buried in the soil under atmospheric conditions (Vohlídal 2021). Several analyzes were carried out to evaluate sample changes over a specific time.

EXPERIMENTAL PART

Materials

Poly(lactic acid) as a base of biocomposites was obtained from Shenzhen Cadit Plastic Material Co., Ltd., with a commercial named CADIT KD-195. This granular polymer has a glass transition temperature (Tg) of 60-62 °C. Micro-graphite, as a filler, is the product of Sigma Aldrich, with maximum particle size is about 37 µm. All materials in this study were used directly without any pre-treatment.

Table 1. Nomenclature of PLA Composite

Sample	PLA (wt%)	Micro-graphite (wt%)
PLA*	100	0
PLAG05	99.5	0.5
PLAG10	99	1
PLAG15	98.5	1.5

*Pure PLA after fed in a mini extruder

Samples Preparation

Composites were prepared via melt blending in a mini single screw extruder to form a noodle-like cylinder. The method and conditions of the sample preparation were as in our previous article (Kaavessina et al. 2020). Table 1 tabulates the composition and the nomenclatures of the PLA/micro-graphite composites.

Degradation tests

The photo-oxidative degradation test was done by placing samples in such a way in the open air that allows for direct contact with day and night weather changes. A hydrolytic degradation test was done by burying samples in the soil (pH 6.5) with a depth of approximately 5 cm and watering about 10 ml of water every morning. The length of samples was prepared at about 2 cm (Φ = 3 mm).

Both degradation tests were carried out simultaneously in the same period between March 1 - July 19, 2021. The temperature and humidity of the environment were not controlled. Extreme weather occurred during this transitional period from rainy to dry season. The average temperature and relative humidity (RH) were 31 °C and 74%. At each degradation time, samples were taken and washed intensively with distilled water at room temperature before being dried. The following equation measured the percentage of residual weight:

$$\text{Residual Weight (\%)} = \left[1 - \left(\frac{W_{initial} - W_{final}}{W_{initial}}\right)\right] \times 100\% \qquad (1)$$

where, $W_{initial}$ and W_{final} are the weight of the samples before and after the degradation test, respectively.

Characterizations

Bulk crystallinity was analyzed using

Differential Scanning Calorimetry (DSC), Shimadzu DSC60 (Japan). The condition of thermal analysis was operated under a nitrogen atmosphere at a heating rate of 10 °C /min with a temperature range of 30-300 °C. Morphology of samples was observed under scanning electron microscopy (SEM), JEOL JCM-7000 Japan, at 10 kV.

RESULTS AND DISCUSSION

As shown in Figure 1, the residual weight shows a decreasing trend in each sample as the degradation time. At a glance, the presence of micro-graphite affects the photo-oxidative degradation of samples. As seen in week 20 (Figure 1), pure PLA still showed a residual weight of 97.01%. Compared with the PLAG05, PLAG10, and PLAG15, the residual weight decreased to 96.68%, 96.53%, and 95.88%, respectively. It could be explained that the micro-graphite was released from the matrix and reduced the residual weight of the sample directly during the degradation test. The release of micro-graphite induces a sharp reduction in the residual weight of samples. It indicates that the wettability of the PLA matrix to the micro-graphite decreased because PLA has started to degrade (Koffi et al. 2021). Further explanations will be visually analyzed using SEM images.

The same phenomenon can be seen in Figure 2 when the degradation test was carried out by burying the sample in the soil as deep as 5 cm. Residual weight was seen to decrease during the degradation test and the presence of micro-graphite loading.

In comparison between Figures 1 and 2, the residual weight showed a significant difference in the same degradation period. Photo-oxidative degradation shows that the residual weight of the sample is lower than the hydrolytic degradation. This phenomenon indicates that photo-oxidative degradation has more effect in degrading poly(lactic acid). This tentative statement will be further analyzed by looking at the thermal and morphological properties of the PLA composite before and after degradation.

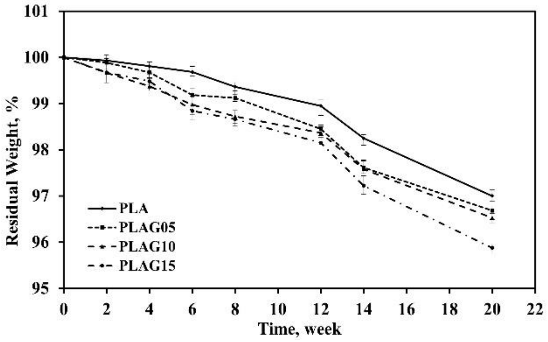

Fig. 1: Photo-Oxidative Degradation of PLA Composites

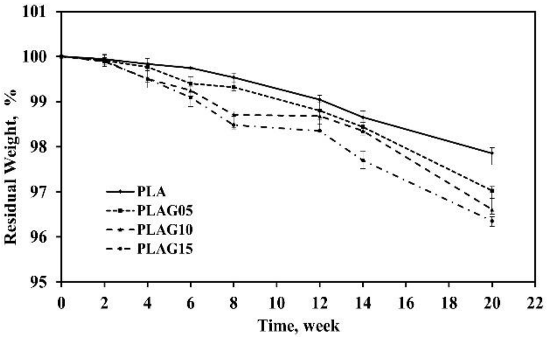

Fig. 2: Hydrolytic Degradation of PLA Composites

As known, thermal properties of polymers depict other properties of thermoplastic polymers such as molecular weight, crystallinity, intermolecular bonding, cross-linking, etc. (Balani et al. 2014). This thermal test was used to detect poly(lactic acid) thermal properties changes, which indicates a degradation process. Tables 2 and 3 show the thermal analysis results before and after the degradation test. The temperature glass transition (Tg) of PLA is relatively constant in the presence of fillers (micro-graphite). Both photo-oxidative degradation and hydrolytic degradation showed a similar trend. It is possible because only a small amount of micro-graphite was added to the PLA matrix. This phenomenon was also found when PLA was added by hydroxyapatite with more quantity (0-20% wt), as reported in our previous work (Kaavessina et al. 2015). Tg can be concluded in constant value due to only shifting about 1-2 °C.

The melting temperature read is the meeting point between the historical effect of heating and the presence of filler (Zare et al. 2019). PLA was melted during the sample preparation and added micro-graphite simultaneously in the single screw extruder.

In general, processing thermoplastic polymers at a melting state will lower the polymer's melting point due to the possibility of cutting polymer molecular chains. However, the presence of filler will act as a nucleating agent, which tends to increase crystallinity or increase the melting temperature (Sullivan et al. 2014). Tables 2 and 3 show a slightly decreasing trend in melting temperature. It means that the presence of micro-graphite is not significant to the melting temperature. A small amount of micro-graphite may cause this phenomenon.

Onset melting temperature (Tm_{onset}) is monitored to lower temperatures when the micro-graphite was blended in poly(lactic acid) and after both degradation tests. For example, the Tm_{onset} of pure PLA is changing from 165.3 °C to 164.3 °C (PLAG05), 163.5 °C (PLAG10), and 161.9 °C (PLAG15). Indirectly, it indicates a change in the molecular weight of PLA that may be caused by the chain scission of PLA, as mentioned above.

Comparing Tables 2 and 3 shows that the photo-oxidative degradation is a more significant shift in melting temperature and onset melting temperature. It may be explained that during photo-oxidative degradation, samples were getting the combined action of UV light and oxygen/water in a humid environment. While in the hydrolytic degradation, samples were only getting hydrolysis reaction (Araújo et al. 2013; Copinet et al. 2003; Elsawy et al. 2017; Zaaba et al. 2020). This result is in accordance with the Feldman explanation (Feldman 2002).

Further, Feldman (2002) explained that impurities in the polymer might act as photo-initiators to generate complex chain reactions. These impurities can be in the form of filler added or carbonyl groups from thermal processing (Feldman 2002). As seen in Tables 2 and 3, the presence of micro-graphite also affects shifting temperature. A small filler ratio (0-1,5%wt) gives a temperature shift of about 1-3 °C.

As shown in Tables 2 and 3, the crystallinity (Xc) of PLA tends to increase in the presence of micro-graphite. Again, this increase in crystallinity might be due to filler acting as a nucleating agent. A similar result was reported in our previous work on PLA/hydroxyapatite composite (Kaavessina et al. 2015).

There was a different tendency of crystallinity after both degradations. Photo-oxidative degradation causes decreasing bulk crystallinity. Contrary, the bulk crystallinity tends to increase with degradation time during hydrolytic degradation. For example, the crystallinity of PLAG05 before degradation is about 22.92%. Respectively, it became 21.42% and 23.55% after photo-oxidative degradation and hydrolytic degradation for 20 weeks.

Table 2. Thermal Properties of the Composites Before and After Photo-Oxidative Degradation Test

Sample	Time (Week)	T_g (°C)	$T_{m\ onset}$ (°C)	T_m (°C)	ΔH_m (J/g)	X_c (%)
PLA	0	61.2	165.3	179.8	-19.27	20.57
	6	60.9	163.3	178.7	-20.21	21.57
	12	61.1	162.4	176.8	-19.12	20.41
	20	61.2	160.1	177.3	-19.01	20.29
PLAG05	0	60.8	164.3	179.3	-21.37	22.92
	6	61.1	161.1	178.1	-20.67	22.06
	12	60.9	160.7	177.9	-19.93	21.27
	20	60.9	158.8	176.2	-20.07	21.42
PLAG10	0	61.3	163.5	179.0	-21.69	23.38
	6	60.9	159.5	178.8	-21.42	22.86
	12	61.1	160.1	176.4	-20.89	22.29
	20	61.3	166.7	175.2	-20.29	21.65
PLAG15	0	60.9	161.9	179.1	-22.29	24.15
	6	61.1	161.2	177.2	-21.79	23.26
	12	61.2	156.8	177.6	-21.89	23.36
	20	61.1	155.7	175.4	-20.79	22.19

Table 3. Thermal Properties of the Composites Before and After Hydrolytic Degradation Test

Sample	Time (Week)	T_g (°C)	$T_{m\ onset}$ (°C)	T_m (°C)	ΔH_m (J/g)	X_c (%)
PLA	0	61.2	165.3	179.8	-19.27	20.5
	6	61.3	163.8	179.6	-21.30	22.73
	12	61.2	162.6	179.2	-22.07	23.55
	20	61.2	162.6	179.1	-21.97	23.45
PLAG05	0	60.8	164.3	179.3	-21.37	22.92
	6	61.1	163.6	19.0	-21.78	23.36
	12	61.3	162.3	179.6	-21.56	23.12
	20	61.2	162.1	179.5	-22.89	23.55
PLAG10	0	61.3	163.5	179.0	-21.69	23.38
	6	61.2	161.9	179.1	-21.98	23.69
	12	61.2	161.5	179.2	-22.36	24.10
	20	61.1	161.1	179.8	-22.21	23.94
PLAG15	0	60.9	161.9	179.1	-22.29	24.15
	6	60.9	161.2	180.0	-22.48	24.36
	12	61.0	161.0	179.2	-22.84	24.75
	20	61.2	160.5	179.5	-23.51	25.47

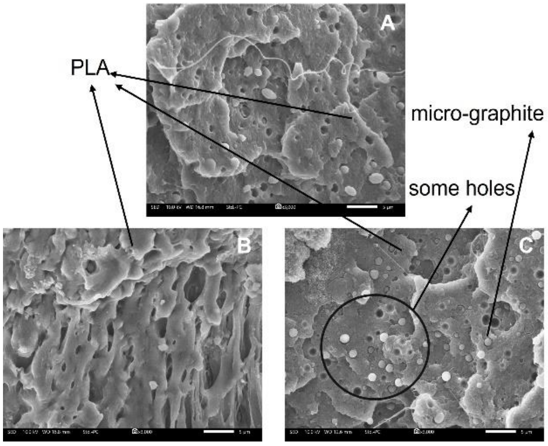

Fig. 3: Cross-section Morphology of PLAG15 Before (A) and After Hydrolytic Degradation (B) and Photo-oxidative Degradation (C) Tests for 20 Weeks.

In hydrolytic degradation, degradation first occurs in the amorphous regions, as explained by others (Kaavessina et al. 2015; Tsuji et al. 2012). It released the amount of amorphous phase within the studied time range (0-20 weeks). Thus, the bulk crystallinity of composite tended to increase, as seen in Table 3. However, the combination of UV-light, oxygen/water, and impurities allows the polymer to weather when it is done in photo-oxidative degradation (Vohlídal 2021). Thus, crystallinity tends to decrease, as shown in Table 2.

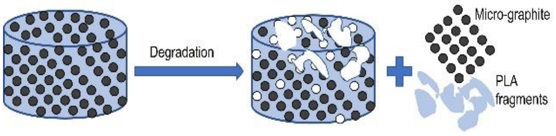

Fig. 4: Schematic of PLA/micro-graphite fragmentation

Figure 3 shows the cross-section surface of the PLAG15 sample before and after degradation for 20 weeks. The comparison in Figure 3 shows that Figure 3(C) looks more rigid than Figure 3(B). Weathering caused chain-breaking/scission resulting in embrittlement and a rougher surface of PLA, as seen in Figure 3(C). UV exposure stimulates the fragmentation of PLA chains and decreases the wettability of the PLA matrix to the micro-graphite.

Both fragmentation and decreased wettability facilitate the release of micro-graphite during surface peeling, as depicted (see Figure 4). Thus, correlated with the residual weight, photo-oxidative degradation delivered a lower residual weight than that of hydrolytic degradation, as discussed above.

CONCLUSIONS

Poly(lactic acid)/micro-graphite were prepared via melt blending in a mini single screw extruder. The presence of micro-graphite may act as a nucleation agent of poly(lactic acid) matrix, which is monitored from a degree of crystallinity. Photo-oxidative degradation of PLA/micro-graphite composite gives the combined action of UV light and oxygen/water in a humid environment. Thus, it allows the polymer weathering, which causes chain-breaking/scission resulting in embrittlement and a rougher surface of PLA. In comparison, hydrolytic degradation only gives a single water action via hydrolysis. In general, the results of this study are part of showing that PLA can be applied as antistatic packaging with the advantage of being more environmentally friendly.

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