ENHANCED KNOWLEDGE IN SCIENCES AND TECHNOLOGY
e-ISSN: 2773-6385

EKST

Vol. 6 No. 1 (2026) 30-38
https://publisher.uthm.edu.my/periodicals/index.php/ekst

Comparative Study on the Surface Morphology and Structural Degradation of Bio-based Polylactic Acid and Conventional High-Density Polyethylene

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DOI: https://doi.org/10.30880/ekst.2026.06.01.005

Article Info

Received: 27 February 2026
Accepted: 7 May 2026
Available online: 9 July 2026

Keywords

HDPE, PLA, FTIR, Digital Microscope, Hydrolytic and Photo-Oxidative Degradation

Abstract

This work studied the surface morphology and molecular structure of bio-based and conventional plastics, specifically polylactic acid (PLA) and commercial-grade high-density polyethylene (HDPE). The degradation process was conducted over a period of 45 days under controlled hydrolytic conditions and natural sunlight exposure. The FTIR result revealed the formation of a new hydroxyl (-OH) peak at 3396 cm^{-1} in PLA after degradation, indicating ester bond cleavage and the formation of degradation products. HDPE showed a very minimal spectral change, revealing its resistance to both hydrolytic and photo-oxidative degradation remained stable. The surface morphology analysis proved that HDPE had a comparatively constant and intact structure, while PLA showed increased roughness and surface defects. Overall, the results show that PLA is more particularly susceptible to environmental degradation compared to HDPE, which provides insights on how conventional and bio-based polymers degrade differently.

1. Introduction

Plastic production began commercially in the 1930s, and its utilization has never stopped growing since resulting in significant environmental issues as they are not biodegradable. Conventional plastics are manufactured using fossil fuels and are not biodegradable in nature. Polypropylene (PP) and polyethylene (PE) products that are commonly used in our daily lives contribute the greatest to most of the plastic waste in the environment. Among them, high-density polyethylene (HDPE) extensively utilized due to its low-cost manufacturing, strong mechanical properties and resistance to chemicals. However, its linear polymer chains pack tightly, resulting in a high degree of crystallinity that attributed to its long-term environmental persistence by making it resistant to natural degradation [1]

As the petroleum-derived lack the ability to biodegradable, they possess a higher carbon footprint and the accumulation of plastics in the ecosystem which results in significant environmental challenges. Therefore, it is the only right thing to change to biodegradable polymers instead of conventional plastics, Consequently, it is extremely necessary to switch to biodegradable polymers instead of conventional plastics. Polylactic acid (PLA), an aliphatic polyester, is a biobased plastic and is commonly used on medical, packaging, and agricultural applications [2]. PLA is more susceptible to degradation under environmental conditions compared to conventional plastics as it contains hydrolytically unstable ester bonds within its polymer backbone [3].

Polymer degradation has a significant impact on its molecular structure and chemical composition. HDPE shows minimal degradation as it lacks the chromophoric groups, which are light-absorbing functional groups

capable of initiating photochemical reactions under UV or visible radiation, whereas PLA primarily undergoes hydrolytic degradation due to its ester bond cleavage. As a result, HDPE degradation typically limited to surface oxidation or additive-related reactions.

Therefore, the aim of this study is to compare the molecular structure and surface morphology of bio-based and non-biobased thermoplastic polymers, specifically commercial-grade high-density polyethylene (HDPE) and polylactic acid (PLA). Fourier Transform Infrared Spectroscopy (FTIR) and optical surface imaging were used to investigate changes in functional groups and surface properties before and after hydrolytic and photo-oxidative degradation under identical experimental conditions.

2. Materials and Methods

The instruments used in the analyses were Fourier Transform Infrared (FTIR) Spectroscopy (Cary 630, Agilent Technologies Inc., USA), Forced Convection Oven (DK-800, Taiwan), Opto-Digital Microscope (DSX100, Olympus Corporation, Japan)

2.1 Sample Preparation

Plastic films of commercial-grade HDPE and PLA were cut and standardized to a thickness of 0.25 mm and was dried in an oven at 50°C for 1 hour. It was then stored in a desiccator prior to analysis.

2.2 Fourier Transform Infrared Spectroscopy Analysis

A Fourier Transform Infrared (FTIR) analysis was done to confirm the chemical composition and to identify the characteristics functional groups present in each of the polymer's samples before and after degradation analysis. Fourier Transform Infrared (FTIR) Spectroscopy was used as it is found in the Chemistry Shared Laboratory 1, Universiti Tun Hussein Onn Malaysia (UTHM) Pagoh. The settings are 4000 to 650 cm^{-1} with a resolution of 4 cm^{-1} and 32 scans per sample. A background scan with an empty cell was performed before each sample measured.

2.3 Surface Morphology Analysis

Surface morphology analysis was conducted by using the Opto-Digital Microscope to evaluate the roughness of the surface, homogeneity, the presence of surface defects such as cracks or voids and any visible degradation or erosion before and after the degradation exposure. The sample commercial-grade HDPE and PLA films was cut 2 cm × 2 cm pieces. Then it was oven dried at 50°C for around 1 hour to remove moisture. The surfaces of HDPE and PLA samples were examined at optical magnifications of 6×,10×, 20×, and 30× to assess the changes.

2.4 Degradation Analysis

The biodegradation test was conducted to assess the effect of photo-degradation and hydrolytic degradation on both samples. The sample was submerged in 250 mL beaker filled with distilled water at room temperature for around 45 days. Another separate set of samples was exposed to natural sunlight in an indoor environment at Universiti Tun Hussein Onn Malaysia (UTHM), Malaysia. The samples were placed next to a window, allowing continuous exposure to sunlight over a period of 45 days, including day and night cycles. The exposure was conducted in an air-conditioned environment with uncontrolled light intensity, humidity, and ambient temperature. After exposure, all samples were dried at 50°C for around 1 hour and it was reanalysed by using the Fourier Transform Infrared (FTIR) Spectroscopy and Opto-Digital Microscope.

3. Results and Discussion

3.1 Fourier Transform Infrared Spectroscopy Analysis

The primary biodegradation mechanism for PLA is hydrolytic degradation, which occurs when moisture breaks the ester bonds in PLA chains. PLA absorbs water, which initiates hydrolysis, despite its great resistance to water due to its steric shielding effect and hydrophobic nature of its methyl side group. Additionally, as a heterochain polymer, the backbone of PLA is known to have hydrolytically degradable ester bonds, which makes it more susceptible to hydrolysis [7]. When PLA is exposed to water conditions, its hydrolysis process causes the ester bonds in the polymer chain to randomly reacts and leading to a breakage of the chain into a shorter fragment that terminate with a carboxyl group on one side and a hydroxyl group on the other [8]. Random chain scission and chain-end hydrolysis are two possible reaction processes that could take place during the hydrolysis process [11]. The terminal ester bonds break down more quickly than those in the backbone due to the different in reactivities of the backbone and terminal ester groups [10] . The chain length affects the reaction's overall kinetic constant.

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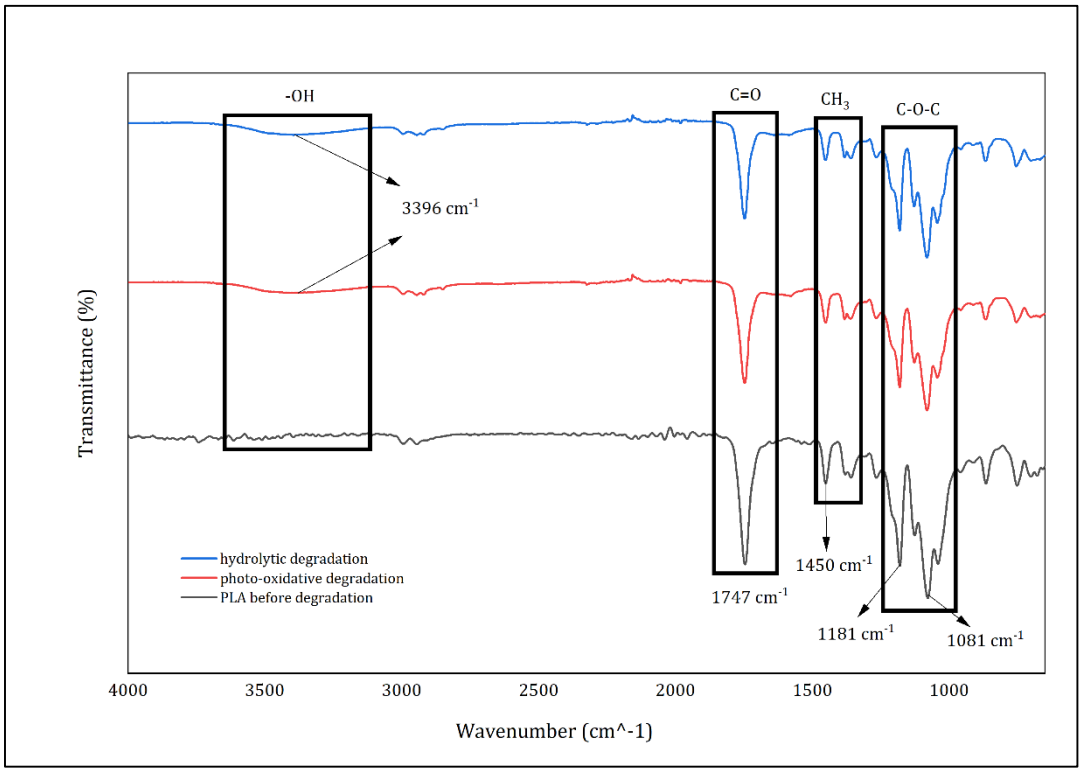

Fig. 1 FTIR spectra of PLA before and after hydrolytic and photo-oxidative degradation

The FTIR spectra of PLA before degradation and after hydrolytic and photo-oxidative degradation are presented in Fig. 1. To qualify changes, the band at 1747 cm^{-1}, 1450 cm^{-1}, 1181 cm^{-1} and 1081 cm^{-1} was normalized as it was the characteristics of PLA [14]. The characteristic bands of PLA before degradation present in the spectra are summarized in Table 1. After undergoing hydrolytic and photo-oxidative degradation, a slight broadening and increased intensity appears at 3396 cm^{-1} which corresponds to the hydroxyl group (-OH) at the molecular end groups [12]. The changes are due to occurrence of ester bond hydrolysis where the water penetrates the polymer and initiates internal chain scission that leads to the formation of hydroxyl and carboxyl end groups [13].

Table 1 Wavenumber and functional groups present in PLA before degradation [17]

Wavenumber (cm^{-1})	Functional Group
1747	(C=O) stretching
1450	(C-H) stretching
1181	C-C(O)-C stretching
1081	(C-O) stretching

It was observed that after the degradation, there was a very weak peak in the region of 1579 cm^{-1} which indicates the appearance of a complicated band. These changes are typically described as the formation of new carbonyl moieties due to the cleavage of ester bond in the PLA back bone [17]. Despite its incredibly low intensity, this band's appearance only after degradation indicating the beginning of small structure structural changes linked to early-stage degradation. Furthermore, a decrease in intensity was noted in the carbonyl band, which is centered at 1747 cm^{-1}, as well as in the band at 1181 cm^{-1} (C-O-C), which is associated with the linkage of repeating units of PLA. When taken together, these results suggest an increase in the concentration of -COOH and -OH end groups due to hydrolysis and the occurrence of degradation process.

In contrast, commercial-grade HDPE did not show a pronounced spectral change after degradation. This is due to the fact that HDPE was a long linear polymer with limited branching and a high degree of crystallinity [21]. Fig. 2 showed the FTIR spectra of commercial-grade HDPE before degradation and after hydrolytic and photo-oxidative degradation. The standard bands of commercial-grade HDPE before degradation that appear in the spectra are summarized in [18]. The band at 2913 cm^{-1}, 2846 cm^{-1}, 1738 cm^{-1}, 1463 cm^{-1}, 1239 cm^{-1} and 721 cm^{-1} were normalized to compare the spectral changes. The asymmetric and symmetric stretching vibrations of methylene group (CH2), correspondingly attributed to the strong intensity peak at 2913 cm^{-1} and 2846 cm^{-1}

[20]. Its crystalline structure is indicated by the band observed at 721 cm^{-1} and 1463 cm^{-1} [19]. These peaks confirm the polyethylene backbone structure.

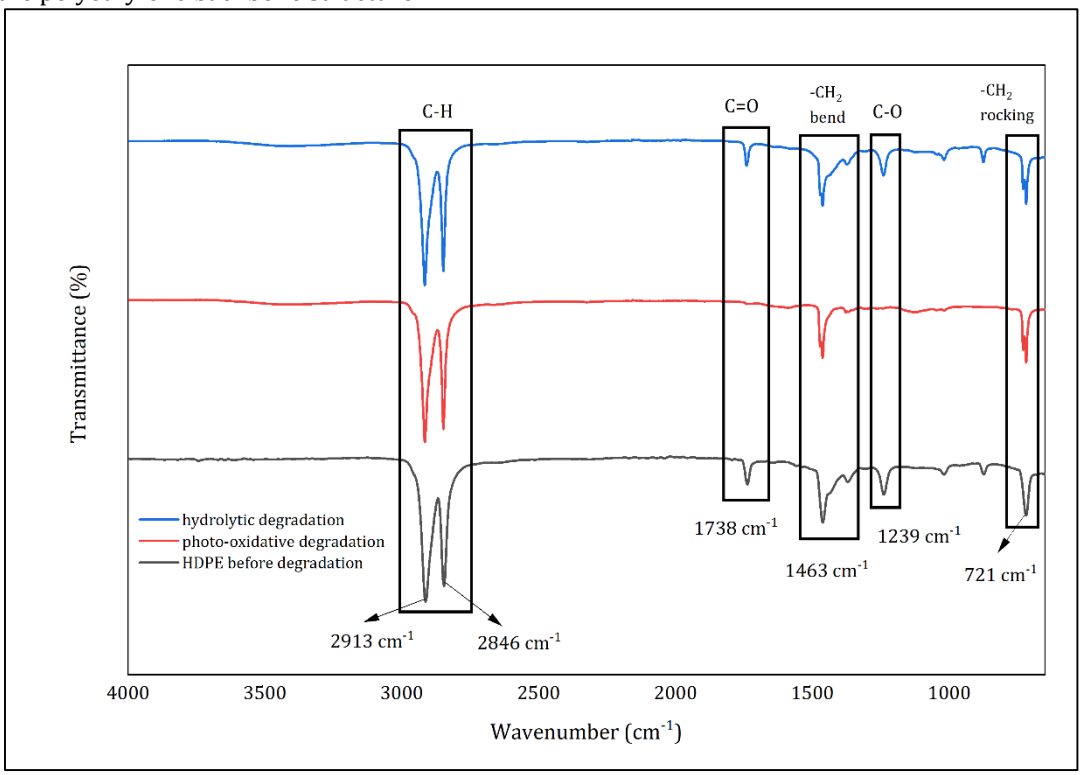

Fig. 2 FTIR spectra of commercial-grade HDPE before and after hydrolytic and photo-oxidative degradation

Weak intensity of bands that are not characteristics of pure HDPE were observed at 1738 cm^{-1} and 1239 cm^{-1}. These bands are probably associated with trace carbonyl and C-O functional groups that come from processing additives or minor pre-existing oxidation which are commonly detected in commercial-grade polyethylene. The intensities of both bands are low which indicates that it has a low concentration of additives.

The distinctive HDPE peaks were not significantly changed during the hydrolytic degradation indicating HDPE's resistance to hydrolytic degradation. The persistence of the distinctive CH2 absorption bands, however, shows that photo-oxidative degradation didn't significantly change the HDPE backbone chemically. The weak non-characteristics bands at 1738 cm^{-1} and 1239 cm^{-1} suggest the depletion or degradation of additive-related functional groups.

Table 2 Wavenumber and functional groups present in commercial-grade HDPE before degradation [18]

Wavenumber (cm^{-1})	Functional Group
2913	(=CH2) asymmetric stretching
2846	(=CH2) symmetric stretching
1738	(C=O) stretching
1463	=CH2 bending
1239	C-O stretching
721	-CH2 rocking

Several studies state that HDPE is a non-absorbing polymer as it lacks chromophoric species that can absorb in the range of the solar light. Although this fundamental principle of photochemistry is occasionally overlooked, light absorption is required to initiate photochemical reactions [22]. Nevertheless, photochemical reactions that leads to limited radical formation may occur at the defect sites or in additive-related chromophoric species. These radicals may react with oxygen, extract hydrogen from the macromolecular chain, or add unsaturated groups that leading to crosslinking [24], however, in this study, as there are not significant changes in the characteristics of HDPE FTIR bands, which conclude that the said processes appeared to be limited.

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3.2 Surface Morphology Analysis

The morphology of the PLA samples before and after hydrolytic and photo-oxidative degradation with a different magnification are presented in Fig. 3. The PLA sample before degradation shows a smooth and uniform structure with no significant defects, indicating a homogenous and intact polymer structure prior to degradation with a higher magnification to analyse and determine structural changes in the sample surface (Fig. 3a and Fig. 3b). Subsequent to the exposure of the degrading treatment for 45 days, Fig. 3c to Fig. 3f shows a significant change from one sample to another as uneven texture, increased in roughness and the formation of localized defects such as small pits, wrinkled area and dark spots were spotted. These morphological features suggest that the occurrence of degradation at the polymer surface, which can be attributed to photo-oxidative reactions and hydrolytic cleavage of ester bonds.

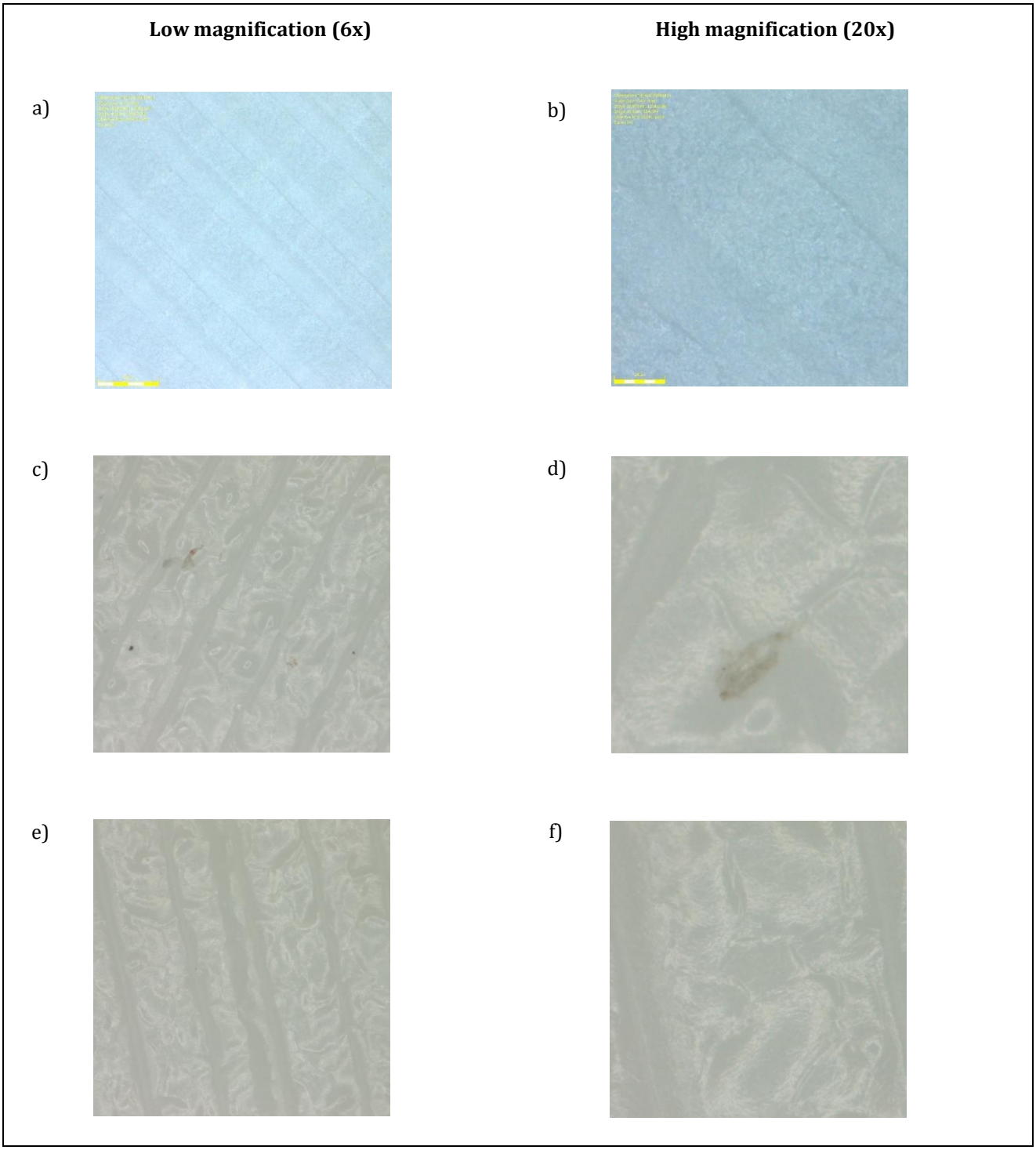


Fig. 3 Opto-digital microscope images of PLA at 6x and 20x magnifications showing (a, b) before degradation, (c, d) after photo-oxidative degradation, and (e, f) after hydrolytic degradation

In general, the degraded PLA samples appeared brittle even though there was no evidence of spontaneous cracking. As mentioned in the previous studies, water penetrates through gaps and cracks, leading to material's expanding and initiating its hydrolytic degradation [25]. As shown in Fig. 3c and Fig. 3e, the surface of PLA films was getting rougher that may be attributed to degradation-induced chain scission and the possibility for secondary crystallization. The FTIR finding support this statement as they showed changes in characteristic functional groups of PLA, indicating ester bond cleavage and chemical modification of polymer chain. The increasing of chain mobility that was attributed to the formation of shorter polymer chains contributing to the surface rearrangement and the observed morphological changes [26].

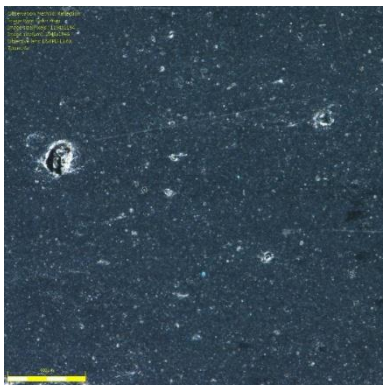
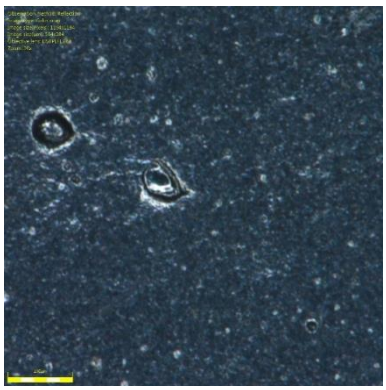
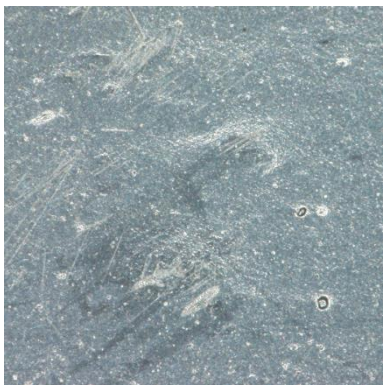
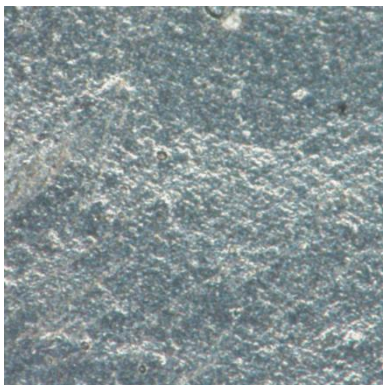
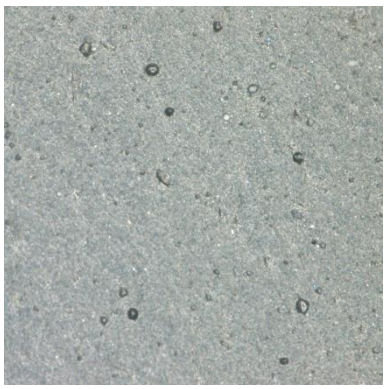
Low magnification (6x)	**High magnification (20x)**
a) 	b 
c) 	d) 
e) 	f) 

Fig. 4 Opto-digital microscope images of HDPE at 6x and 20x magnifications showing (a, b) before degradation, (c, d) after photo-oxidative degradation, and (e, f) after hydrolytic degradation

In contrast, there is no significant changes in the surface morphology of HDPE samples were observed before and after hydrolytic and photo-oxidative degradation as illustrated in Fig. 4. With a slight surface imperfection, the degraded and undegraded HDPE sample showed comparatively smooth surface. No severe cracking, pitting or fragmentation was detected in, Fig. 4c to Fig. 4f, even after 45 days of degradation treatment. This suggests that, under these particular conditions, the degradation process has little to no impact on HDPE's surface structure.

The strong resistance of HDPE to degradation can be attributed to its high strength properties as it has a long linear polymer chain with a high degree of crystallinity and low branching, which limit the access of degrading agents such as water and oxygen and prevent chain mobility [21]. Several studies also state that HDPE are impermeable as the dense packing of macromolecules crystalline phase restricts chain mobility and consequences reduces the free-volume pathways for water and oxygen to penetrate the material [27]. These findings are in line with the FTIR results, which revealed no significant changes in bands intensities or formation of new bands after the degradation, indicating that the polymer backbone went through a minimal chemical modification. Although a small band attributed to additives was seen, it does not result in any changes in the surface morphology of HDPE. Overall, the surface morphology and FTIR analyses reveal that HDPE is not significantly impacted by hydrolytic and photo-oxidative degradation within the studied duration.

4. Conclusion

This study investigated the degradation behaviour of polylactic acid (PLA) and high-density polyethylene (HDPE) by using ATR-FTIR and surface morphology analysis. Following hydrolytic and photo-oxidative degradation, PLA exhibited noticeable changes, as evidenced by the production of hydroxyl and carbonyl-related groups as well as increased surface roughness and defects. HDPE, on the other hand, showed strong resistance to degradation with very minimal changes in both surface morphology and chemical structure due to its structural properties. These findings confirm that PLA is more susceptible to environmental degradation than HDPE, demonstrating how conventional and bio-based polymers degrade differently under the same circumstances. Although the results are favourable, conclusions are stated with appropriate restraint. Key limitations include short-term degradation period, which does not fully represent environmental variability, as well as the lack of advanced characterization techniques such as Scanning Electron Microscopy (SEM) and detailed thermal analysis to further investigate decomposition behaviour. Further work should focus on extending the degradation duration under more realistic environmental conditions and incorporating interval-based analyses to capture the progression of degradation, as this study primarily reflects the initial stage. Moreover, systematic variation of environmental parameters, including pH, temperature, and UV intensity should be considered to provide a deeper insight of the degradation mechanisms and long-term environmental stability of PLA and HDPE.

Acknowledgement

This research was made possible by the Faculty of Applied Sciences and Technology, Universiti Tun Hussein Onn Malaysia. I would like to thank all the laboratory and assistant engineers for making this learning experience smooth and fulfilling.

Conflict of Interest

Authors declare that there is no conflict of interest regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** *Nur Insyirah Rosli, Saliza Asman;* ***data collection:*** *Nur Insyirah Rosli;* ***analysis and interpretation of results:*** *Nur Insyirah Rosli, Saliza Asman;* ***draft manuscript preparation:*** *Nur Insyirah Rosli, Saliza Asman. All authors reviewed the results and approved the final version of the manuscript.*

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