

FTIR Spectroscopic Characterization of Low-Density Polyethylene (LDPE) Using the KBr (Potassium Bromide) Pellet Method

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Received 29 January 2026 | Revised 23 February 2026 | Accepted 22 March 2026 | Available Online 18 April 2026

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Citation: Adedayo O. Tella, Favour O. Olanrewaju, and Tomere Daubotei (2026). FTIR Spectroscopic Characterization of Low-Density Polyethylene (LDPE) Using the KBr (Potassium Bromide) Pellet Method. *Life Science Review*.

DOI: <https://doi.org/10.51470/LSR.2026.10.01.118>

Abstract

Background: Low-Density Polyethylene (LDPE) is a ubiquitous thermoplastic which are usually derived from ethylene monomers, and occupies a prominent position among plastic polymers, and it presents a persistent ecological challenge due to improper disposal and environmental accumulation. Despite its recalcitrance, certain microbial populations have evolved or emerge with the metabolic capacity to utilize LDPE as a primary carbon and energy source, thus offering a potential biotechnological pathway for waste remediation. Despite its enormous utility, the very properties that make LDPE commercially desirable, chemical stability and resistance to biological and environmental degradation render it a persistent and accumulating pollutant in terrestrial and aquatic ecosystems. The continuous and indiscriminate disposal of LDPE waste has resulted in its progressive accumulation in the environment, including in soils, freshwater systems, and marine environments, where it poses a severe ecological threat to biota, biodiversity, and ecosystem function (16). The recalcitrant nature of LDPE to conventional degradation pathways is attributed its high molecular weight, hydrophobic character, and the absence of functional groups that would render it susceptible to enzymatic hydrolysis under ambient environmental conditions (17, 6). Consequently, LDPE and related polyolefin may persist in the environment for centuries, fragmenting progressively into micro plastics and Nano plastics that infiltrate food chains and accumulate in living organisms (2).

Methodology: This research investigated the biodegradation of LDPE by isolating and characterizing specialized microorganisms from plastic-polluted dumpsite soils. Using enrichment techniques, the isolates were cultivated in both axenic (single) and consortium (mixed) cultures containing pre-treated LDPE strips in aseptically method. The degradation process was conducted at room temperature under a constant agitation of (120 rpm). The extent to which the polymer degraded was quantified through microbial load monitoring and gravimetric weight loss analysis, while the structural modifications were determined and identified via Fourier Transform Infrared (FTIR) spectroscopy.

Results: Thirteen distinct isolates were identified, belonging to the genera *Bacillus* (5), *Aspergillus* (5), *Penicillium* (2), and *Fusarium* (1). Among these isolates identified, *Pseudomonas aeruginosa*, *Penicillium chrysogenum*, *Aspergillus niger*, and *Fusarium oxysporum* demonstrated superior survival traits during the preliminary biodegradative screening. *P. chrysogenum* exhibited the highest individual microbial load of (1.9084 to 2.1762 Log CFU/mL), whereas the *Aspergillus niger* and *Pseudomonas aeruginosa* consortium showed the highest overall density of (2.7958 to 2.6883 Log CFU/mL). The highest gravimetric weight loss (5%) was recorded for both *A. niger* in isolation and the *A. niger/P. aeruginosa* consortium. FTIR spectra established and strengthened these findings, with *A. niger* producing the most significant peak reduction at 3801.82 cm⁻¹, which is followed closely by *P. aeruginosa* at 3840.28 cm⁻¹.

Conclusion: The synergistic action of the *Aspergillus niger* and *Pseudomonas aeruginosa* mixed culture proved highly effective in altering the polymer's structural integrity. These findings suggest that these specific microbial strains hold significant promise for mitigating the environmental impact of polyethylene waste accumulation.

Keywords: LDPE, Biodegradation analysis, FTIR spectroscopy, *Aspergillus niger*, *Pseudomonas sp.*

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1.0 Introduction to FTIR Characterization of LDPE

The identification and structural analysis of synthetic polymers are foundational to materials science, particularly for ubiquitous plastics like Low-Density Polyethylene (LDPE). Among the various analytical suites available, Fourier Transform Infrared (FTIR) spectroscopy stands as a primary diagnostic tool due to its high sensitivity to molecular vibrations and functional group arrangements. By measuring how a polymer sample absorbs infrared radiation, researchers can create a molecular fingerprint that reveals chemical composition, crystallinity, and the presence of oxidative degradation (31).

1.1 Fourier Transform Infrared (FTIR) Spectroscopy as an Analytical Tool for Polymer Characterization

Fourier Transform Infrared (FTIR) spectroscopy is among the most widely employed analytical techniques in polymer science, offering rapid, non-destructive, and highly informative characterization of molecular structure and functional group composition (40, 21). The fundamental principle of FTIR spectroscopy lies in the selective absorption of infrared radiation by molecular bonds undergoing vibrational transitions; each functional group absorbs IR radiation at characteristic frequencies that correspond to its bond energy and molecular environment, producing a unique spectral "fingerprint" of the material under analysis (12).

In the context of polymer characterization, FTIR spectroscopy affords critical insights into the nature and relative abundance of functional groups, the degree of oxidative degradation, structural alterations induced by thermal or biological processing, and the discrimination of polymers belonging to the same chemical family (13, 24). The technique has been applied across a broad range of polymeric systems for quality control, degradation studies, material identification, and the monitoring of chemical modifications induced by processing or environmental exposure (2). For polyethylene specifically, FTIR has been extensively validated as a tool for distinguishing LDPE from HDPE and linear low-density polyethylene (LLDPE) on the basis of characteristic band intensities and positions (9, 22).

A comprehensive FTIR dataset published in 2024, encompassing 3,000 spectra of the six most globally prevalent industrial plastics including PET, HDPE, PVC, LDPE, PP, and PS underscores the analytical centrality of FTIR spectroscopy in the discrimination and identification of synthetic polymer types across scientific and industrial applications (22). The excitation of vibrational energy by infrared radiation provides detailed molecular structural information pertaining to functional groups, interaction modes, and inter-chain relationships within polymer samples, all of which are encoded in the characteristic absorption bands resolved in an FTIR spectrum (22).

1.2 The Chemical Profile of LDPE

LDPE is a thermoplastic characterized by a high degree of short and long-chain branching.

This branching prevents and enables the polymer chains from packing tightly into a rigid crystalline structure, thus resulting in a material that is not only flexible, translucent, but possesses a lower density compared to its linear counterpart, HDPE. During an FTIR spectrum procedure, LDPE is identified by specific hydrocarbon signatures such as: C-H Stretching: Here, the prominent peaks typically appear between 2850 and 2950 cm^{-1} .

C-H Bending (Scissoring): The sharp peaks appear near 1460–1470 cm^{-1} .

C-H Rocking: Specific vibrations appear around 720 cm^{-1} which often provide clues regarding the crystallinity nature of the sample (19).

1.3 Research Justification and Objectives

Given the escalating threat posed by LDPE waste accumulation in the Nigerian environment and the global need for bioremediation strategies, the present study aims to characterize the structural properties of LDPE samples before and after microbial treatment using FTIR spectroscopy with the KBr pellet preparation method. The study employs enrichment culture techniques to isolate LDPE-degrading microorganisms from dumpsite soils, screens and identifies the most effective isolates including species of *Bacillus*, *Aspergillus*, *Penicillium*, *Fusarium*, and *Pseudomonas* and utilises FTIR spectroscopic analysis to confirm and elucidate the structural changes undergone by LDPE films following microbial degradation in both single and mixed culture systems. This work contributes to the expanding knowledge base on the biodegradation of synthetic polymers and offers insights into the potential application of microbial consortia, particularly *Aspergillus niger* and *Pseudomonas aeruginosa*, in the bioremediation of polyethylene waste in contaminated environments.

1.4 The KBr Pellet Method in FTIR Analysis

The preparation of solid samples for FTIR transmission spectroscopy has historically relied upon the potassium bromide (KBr) pellet technique, a methodology that remains a gold standard for obtaining high-quality, well-resolved infrared spectra of solid materials (30). The KBr pellet method involves the intimate grinding of a finely powdered sample with spectroscopically pure KBr powder, typically at a sample-to-KBr mass ratio of 1:100 to 1:200 followed by compression under high hydraulic pressure (8–10 tonnes) to form a transparent disc of approximately 13 mm diameter and 1–2 mm thickness (30).

The rationale for employing KBr as the matrix material lies in its unique optical properties: KBr is an ionic salt whose crystal lattice vibrations occur at frequencies far below the mid-infrared range (400–4000 cm^{-1}) used for functional group analysis, rendering it entirely transparent to IR radiation within the analytically relevant spectral window (5, 27).

The Pelletization process addresses the fundamental challenge of analyzing solid polymer samples, which, owing to their opacity, cannot be analyzed directly in transmission mode without appropriate sample preparation. By dispersing the analytes homogeneously in a transparent KBr matrix, the method ensures that infrared radiation passes through the sample unimpeded by particle scattering, thereby yielding spectra of high signal-to-noise ratio with sharp, well-resolved absorption peaks and a flat baseline (20, 39).

Notable advantages of the KBr pellet method include its suitability for a wide range of solid sample types, its capacity to produce spectra of exceptional resolution appropriate for spectral library matching and structural elucidation, and its compatibility with quantitative analysis based on Beer–Lambert relationships when uniform pellet geometry is maintained (30, 20). The primary limitation of the technique is the hygroscopic nature of KBr, which readily absorbs atmospheric moisture, potentially introducing broad water absorption bands near 3400 cm^{-1} and 1600 cm^{-1} that may obscure spectral features of analytical significance (30, 5). To mitigate this effect, KBr powder must be thoroughly dried prior to use, and sample preparation should be conducted expeditiously to minimize atmospheric exposure (28).

While the Attenuated Total Reflectance (ATR) accessory has gained considerable popularity in recent decades for the analysis of solid polymers owing to its simplicity and minimal sample preparation requirements, the KBr pellet method retains significant advantages for detailed structural analysis, spectral library comparisons, and studies where the highest achievable spectral resolution and sensitivity are required (39, 20). In the context of LDPE characterization, the KBr pellet method permits thorough spectral interrogation of the polymer matrix, facilitating the identification of both major structural features and subtle functional group changes associated with oxidative or biological degradation. While modern techniques like Attenuated Total Reflectance (ATR) are common, the Potassium Bromide (KBr) pellet method remains a classic transmission-based technique valued for its ability to produce high-resolution spectra with minimal background noise.

1.5 Significance of Characterization

Analyzing LDPE via the KBr method was not merely an exercise in identification; but it is essential for quality control and environmental monitoring in LDPE degradation. For instance, the appearance of a carbonyl peak ($\text{C}=\text{O}$) near 1715 cm^{-1} often indicates photo-oxidative degradation, which forms a critical factor when assessing the age or environmental impact of plastic waste (1). By utilizing FTIR, scientists can ensure that the LDPE meets specific industrial standards or track how its molecular integrity changes over time under various stressors.

2.0 Materials and Methods

2.1 Sample Preparation and Instrumentation

The structural analysis of the Low-Density Polyethylene (LDPE) samples was performed using a Buck Scientific M530 FTIR spectrometer. To ensure high-quality spectral resolution, the KBr (Potassium Bromide) pellet technique was employed using:

1. Preparation: Since KBr is IR-transparent (which makes it not to absorb radiation in the mid-IR region), it thus serves as an ideal matrix. The LDPE sample is typically reduced to a fine powder often via cryogenic grinding and mixed with spectroscopic grade KBr.

2. Compression: The mixture was subjected to high pressure using a hydraulic press to form a thin, translucent disk.

3. Analysis: The infrared beam was passed directly through the pellet. This transmission approach often yields more distinct peaks for quantitative analysis compared to surface-level techniques, as it accounts for the bulk properties of the polymer (11).

Initially, 0.5 g of the LDPE sample was combined with an equal mass (0.5 g) of spectroscopic-grade KBr. This mixture was thoroughly homogenized using a manual mortar and pestle until a consistent, fine particulate was achieved. The resulting powder was then subjected to high pressure in a mechanical pelletizing press to produce a thin, translucent disk suitable for transmission analysis (42).

2.2 Analytical Protocol

Before analyzing the LDPE samples, a background scan of a pure KBr pellet was conducted. This step was critical in order to subtract the spectral signature of the KBr matrix and atmospheric interference (such as CO_2 and water vapor) from the final results. After it was calibrated, the sample pellets were placed in the instrument's inner compartment. The absorption spectra were recorded across a range of 4000 to 650 cm^{-1} , with results monitored in real-time and processed for functional group identification.

3.0 Results and Spectral Interpretation

The FTIR analysis in this research provided a molecular capture of the chemical changes induced by microbial action over time. The Control sample represented the pure polymer, while subsequent tests tracked and monitored the impact of various fungal and bacterial isolates during the research.

3.1 Characteristic Absorption Bands (Baseline)

The control spectrum (Fig. 5) established the fundamental backbone of the LDPE, characterized by: C-H Methylene Stretching: 2899.95 cm^{-1} , C=O/C=C Stretching: 1627.48 cm^{-1} , C-Cl and C-O-C/C-O Fingerprints: Found at 1083.24 cm^{-1} and 1192.87 cm^{-1} respectively.

3.2 Evidence of Biodegradation

The introduction of microbial agents (*Penicillium chrysogenum*, *Aspergillus niger*, *Fusarium oxysporum*, and *Pseudomonas aeruginosa*) resulted in significant spectral shifts as shown in the Figures and tables below; at Four weeks (Figure 1-5), at the Termination stage (Figure 6-9), and for Consortium (Figure 10-12) and with the control showing the backbone and the significant shift made in the obtained spectra as observed in: Bond Disappearance: Several samples showed the loss of traditional hydrocarbon bonds, particularly in the 1100–1600 cm^{-1} range, suggesting enzymatic cleavage of the polymer chain. New Functional Groups: The appearance of peaks near 3800–3900 cm^{-1} (O-H stretching) and 1790 cm^{-1} (Carbonyl groups) indicates the formation of alcohols, ketones, or carboxylic acids, a common byproducts of oxidative biodegradation. Consortium Effect: The combination of fungal and bacterial agents (e.g *A. niger*, *P. aeruginosa*) showed the most aggressive reduction in peak intensities, signaling a synergistic effect in breaking down the LDPE matrix (23,41).

AT FOUR WEEKS

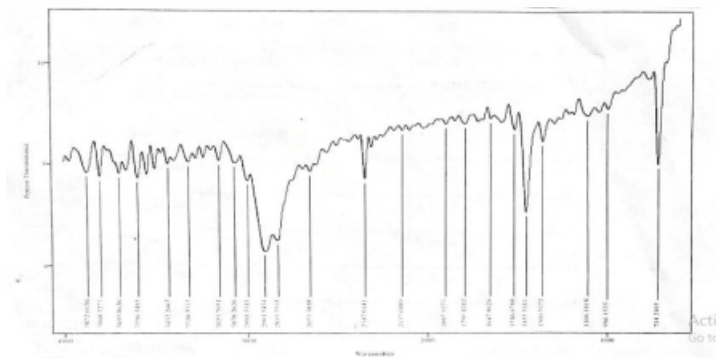


Fig 1: Spectroscopy result of biodegraded LPDE (*Penicillium chrysogenum*)

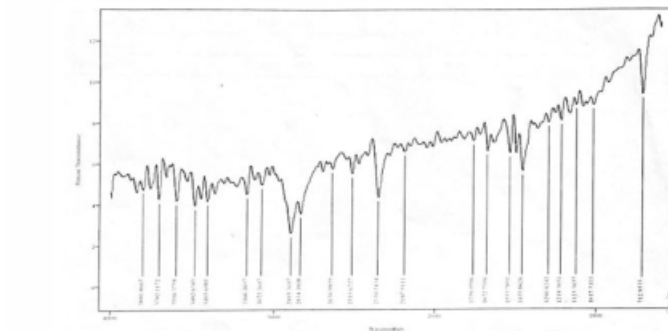


Fig 2: Spectroscopy result of biodegraded (*Aspergillus niger*)

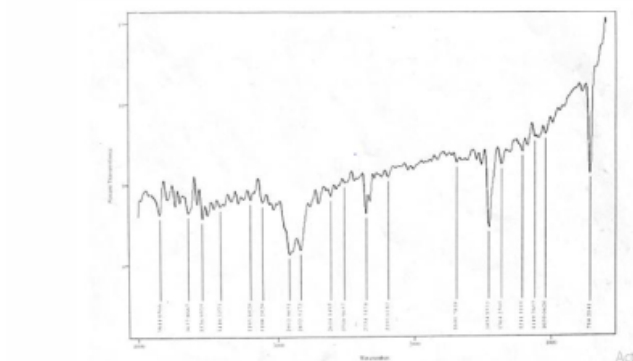


Fig 3: Spectroscopy result of biodegraded LPDE (*Fusarium oxysporum*)

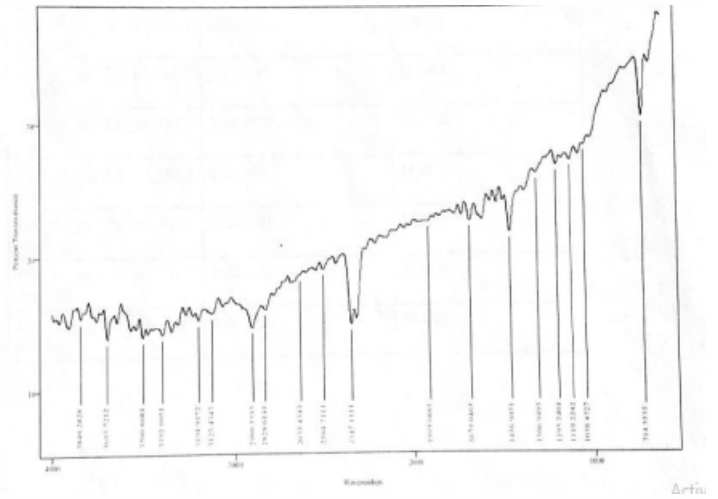


Fig 4: Spectroscopy result of biodegraded LPDE (*Pseudomonas aeruginosa*)

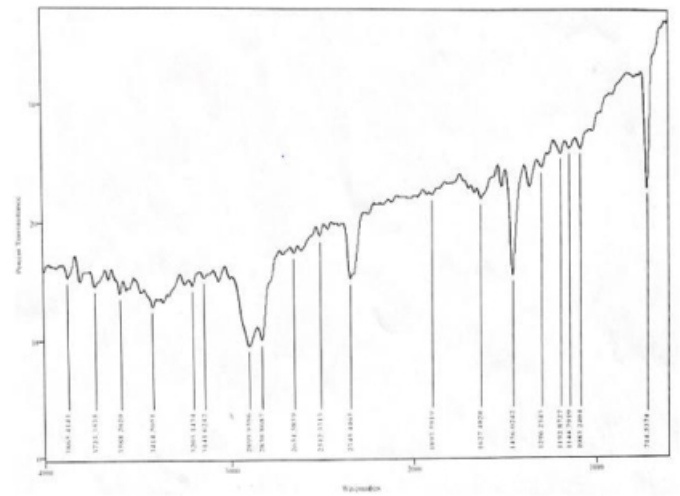


Fig 5: Spectroscopy result of Control

AT TERMINATION

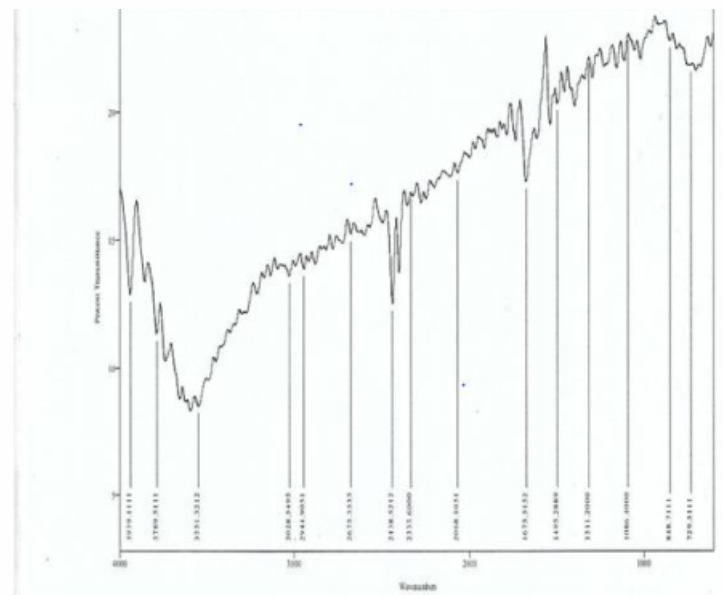


Fig 6: Spectroscopy result of biodegraded LPDE (*Penicillium chrysogenum*)

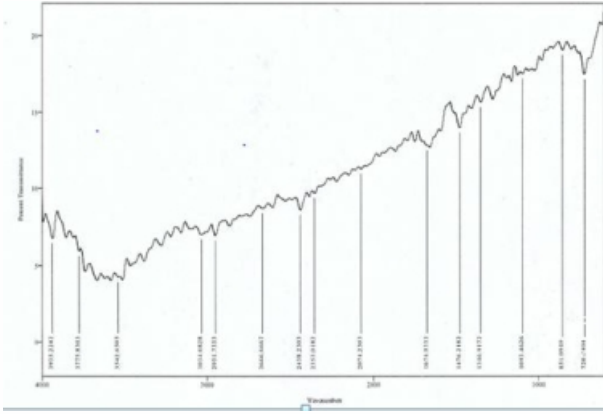


Table 4: Spectroscopy result of biodegraded LPDE (*Pseudomonas aeruginosa*)

Peak (Cm ⁻¹)	Possible Bond Type	Functional Group
1679.04	C=O stretch	Aldehyde, ketone, carboxylic acid
1456.90	C=O, C=C stretch	Carboxylates, amides, Alkenes, aromatic Compound
1306.94	C-N stretch	Aromatic (primary & secondary) amine
1195.2	C-O-C, C-O	Polysaccharides
714.55	C-Cl	alkyl, halides
2900.53	C-H	Methylene
3840.28	O-H	Bonded and non-bonded hydroxyl water group

Table 5: Spectroscopy result of LPDE (Control)

Peak (Cm ⁻¹)	Possible Bond Type	Functional Group
1627.48	C=O, C=C	Carboxylates, amides, Alkenes, Aromatic Compounds
1296.23	C-N stretch	Aromatic (primary & secondary)
1192.87	C-O-C, C-O	Polysaccharides
1083.24	C-Cl	Alkyl, halides
714.53	C-Cl	Alkyl, halides
2899.95	C-H	Methylene
3865.41	O-H	Bonded and non-bonded hydroxyl Group and water

AT TERMINATION

Table 6: Spectroscopy result of biodegraded LPDE (*Penicillium chrysogenum*)

Peak (Cm ⁻¹)	Possible Bond Type	Functional Group
1495.28	C=C, C=O	Carboxylates, amides, Alkenes, aromaticCompounds
1311.20	C-N	Aromatic (primary & secondary)
1086.40	C-O-C, C-H	polysaccharides & hemicelluloses
848.71	C-Cl	alkyl, halides
1675.5	C=O	Aldehyde, ketone, Carboxylic acid
2675.35	C-H	Methylene
3939.11	O-H	Bonded and non-bonded hydroxyl Group and water

Table 7: Spectroscopy result of biodegraded LPDE *Aspergillus niger*

Peak (Cm ⁻¹)	Possible Bond Type	Functional Group
1476.21	C=C, C=O	Carboxylates, amides, Alkenes, Aromatic compounds
1346.91	N-O	Nitrate
1093.46	C-O-C, C-O	Polysaccharides
851.09	C-Cl	Alkyl, halides
1674.93	C=O	Aldehyde, ketone, Carboxylic acid
2666.66	C-H	Methylene
3955.22	O-H	Bonded and non-bonded hydroxyl group and water

Table 8: Spectroscopy result of biodegraded LPDE *Fusarium oxysporium*

Peak (Cm ⁻¹)	Possible Bond Type	Functional Group
1484.25	C=C, C=O	Carboxylates, amides, Alkenes, Aromatic compounds
1311.89	C-N stretch	Aromatic (primary & secondary)
1085.81	C-O-C, C-H stretch	polysaccharides & hemi cellulose
854.14	C-Cl stretch	Alkyl, halides
1684.00	C=O	Aldehyde, ketone, carboxylic acid
2671.71	C-H stretch	Methylene
3939.97	O-H	Bonded and non-bonded hydroxyl group & water

Table 9: Spectroscopy result of biodegraded LPDE *Pseudomonas aeruginosa*

Peak (Cm ⁻¹)	Possible Bond Type	Functional Group
1484.92	C=O, C-H	Carboxylates, amides, Alkenes, Aromatic compounds
1086.21	C-O, C-O-C	Polysaccharides
852.05	C-Cl stretch	Alkyl, halides
1670.24	C=O	Aldehyde, ketone, Carboxylic acid
2667.20	C-H	Methylene
3929.11	O-H	Bonded and non-bonded hydroxyl group and water

FOR CONSORTIUM

Table 10: Spectroscopy result of biodegraded LPDE *Penicillium chrysogenum* and *Pseudomonas aeruginosa*

Peak (Cm ⁻¹)	Possible Bond Type	Functional Group
1485.59	C=O, C=C	Carboxylates, amides, Alkenes, aromatic compounds
1271.01	C-O-C, C-O	Polysaccharides
849.64	C-Cl	Alkyl, halides
1682.98	C=O	Aldehyde, ketone, carboxylic acid
2673.93	C-h	Methylene
3943.2	O-H	Bonded and non-bonded hydroxyl group and water

Table 11: Spectroscopy result of biodegraded LPDE *Aspergillus niger* and *Pseudomonas aeruginosa*

Peak (Cm ⁻¹)	Possible Bond Type	Functional Group
1480.69	C=O, C=C	Carboxylates, amides Alkenes, Aromatics compounds
1132.05	C-O-C, C-O	Polysaccharides
722.90	C-Cl stretch	Alkyl, halides
1796.20	C=O stretch	Aldehyde, ketone, carboxylic acid
1695.35	C=O, C-C	Carboxylates, amide, alkenes, aromatic compounds
2824.48	C-H	Methylene
3942.24	O-H	Bonded and non-bonded hydroxyl group and water

Table 12: Spectroscopy result of biodegraded LPDE *Fusarium oxysporium* and *Pseudomonas aeruginosa*

Peak (Cm ⁻¹)	Possible Bond Type	Functional Group
1484.61	C=O	Aldehyde, ketone, Carboxylic acid
1324.96	N-O	Nitrate
1050.98	C-O-C, C-H	Polysaccharides
850.90	C-Cl	Alky, hylides
1679.89	C=O, C-C	Carboxylates, amides, Alkenes, aromatic compound
2665.67	C-H	Methylene
3947.11	O-H	Bonded and non-bonded hydroxyl group and water

4.0 Discussion

4.1 Microbial Degradation Mechanisms

Polyethylene remains one of the most challenging synthetic polymers to remediate due to its high molecular weight and hydrophobic (water-repellent) nature (7). However, our studies demonstrate and reveal that specific soil-borne microorganisms can utilize LDPE as a primary carbon and essential energy source.

Microbes, particularly fungi such as *Aspergillus* and *Penicillium*, secrete extracellular enzymes known as (depolymerases). These enzymes are critical in process such as biofilm degradation, and the environmental breakdown of synthetic plastics. Tools like the DePolymerase Predictor (DePP) are now being used by researchers to identify these enzymes within bacterial genomes (10). Functionally, these enzymes cleave long-chain carbon bonds into smaller monomers that can be assimilated into the microbial cell for energy (34). Our observations align with previous findings by (29), where fungi exhibited superior degradation efficiency compared to bacterial isolates, likely due to their hyphal growth which facilitates for better physical penetration of the polymer matrix.

The degradation of LDPE is significantly enhanced by the filamentous nature of fungi such as *Aspergillus* and *Penicillium*. Unlike many bacterial isolates, these fungi utilize hyphal extension to penetrate the polymer matrix, increasing the surface area available for enzymatic contact.

To facilitate this, fungi secrete hydrophobins that mediate the interface between the hydrophobic LDPE surface, and the aqueous extracellular environment (38, 14).

This attachment allows for localized oxidative stress, where the fungi promote the formation of oxygenated functional groups. The presence of carbonyl (1679.89 cm^{-1}) and hydroxyl (3947.11 cm^{-1}) peaks in our FTIR spectra confirms this oxidative transition. By introducing these polar groups, the fungi effectively lower the polymer's hydrophobicity, rendering the long-chain hydrocarbons susceptible to further cleavage by secreted depolymerases for subsequent cellular assimilation (35, 37).

4.2 The Role of Microbial Consortia

A key finding in this research was that the microbial consortium outperformed individual strains. The metabolic diversity of a multi-species community allows for a more "complete" breakdown, as the byproducts produced by one species may be more easily consumed by another. This is evidenced by the disappearance of primary bonds and the emergence of various oxygenated intermediates in the FTIR spectra of mixed cultures (26, 3).

5.0 Conclusion and Recommendations

5.1 Conclusion

This study confirms that *Aspergillus niger*, *Penicillium chrysogenum*, *Fusarium oxysporum*, and *Pseudomonas sp.* possess the enzymatic machinery required to degrade LDPE. FTIR analysis provided clear evidence of structural alteration, specifically through the reduction of methylene peaks and the emergence of hydroxyl and carbonyl signatures. Fungal isolates generally demonstrated a higher efficacy than bacteria, though the combined consortium yielded the most significant weight loss and chemical modification over the 60-day period.

5.2 Recommendations

To transition these findings from the laboratory to commercial application, further research is required to:

1. Optimize Environmental Conditions: Determine the ideal pH, temperature, and nutrient levels to maximize microbial activity.
2. Pre-treatment Protocols: Explore eco-friendly pre-treatments (like UV exposure) to further weaken the polymer chains before microbial inoculation.
3. Scalability: Investigate the performance of these consortia in large-scale landfill or composting environments.

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