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RESEARCH ARTICLE

The effect of soil burial degradation on the mechanical and thermal properties of poly (lactic acid)/graphene nanoplatelets nanocomposites

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Abstract - In this research, the effect of soil burial degradation of poly (lactic) acid (PLA)/graphene nanoplatelets (GNP) nanocomposites on its mechanical and thermal properties was reported. PLA is a polyester made with two conceivable monomers, or building blocks: lactic acid and lactide. Much research has been done about PLA that has been tested and mixed with other materials, such as graphene and other nanofillers. For this research, graphene nanoplatelets are blended into PLA using the melt-blending method to enhance PLA properties. The effect of degradation on the mechanical and thermal properties of PLA/GNP is reported. The mechanical properties observed after the degradation process indicate that the tensile strength decreased by 80.29% for PLA and by 70.90% for PLA/GNP. At the same time, thermogravimetric analysis shows that the degradation process reduced the thermal stability of both neat PLA and PLA/GNP nanocomposites. X-ray diffraction analysis revealed no new peak formation of PLA/GNP before and after degradation. However, the XRD peak intensity of the degraded PLA/GNP was higher, indicating a shift in crystalline arrangement.

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1. Introduction

A typical occurrence, polymer degradation is a substantial alteration in a polymer's chemical and physical characteristics brought about by various environmental stresses. Heat, radiation, chemicals, mechanical stress, biological degradation, hydrolysis, and oxidation are some of these variables. Once initiated, these hydrolytic or oxidative pathways often proceed autocatalytically at the molecular level, leading to irreversible, progressive deterioration of the molecular chain structure and loss of structural integrity. These physical and chemical alterations are usually undesirable in most industrial applications, leading to premature mechanical failure and chemical disintegration [1]. Polylactic acid (PLA) is a biodegradable polyester derived primarily from agricultural feedstocks such as corn. PLA has been developed for use in textiles, food packaging, and other engineering applications [2]. The use of carbon-based nanofillers has emerged as a promising strategy to reduce the PLA polymer matrix's intrinsic brittleness and poor thermal stability, enabling broader applications. Graphene is a free-standing two-dimensional structure made up of hexagonally organised sp2-hybridised carbon atoms in a honeycombed network lattice. Small stacks of graphene layers form graphene nanoplatelets (GNP), a flexible and affordable reinforcement for polymers that often outperform alternative fillers such as carbon fibre, carbon nanotubes, and nanoclays [3]. Because it is more economically viable for mass manufacturing than more costly carbon nanotubes or fullerenes, graphene is considered one of the most promising carbon-based nanomaterials for industrial applications. Compared with other carbon-based nanomaterials (such as carbon nanotubes, fullerenes, and carbon nanofibers), graphene's mass-production capacity offers great potential for commercial applications [4]-[5]. The literature has long documented graphene-based polymer nanocomposites with conventional petroleum polymers such as polypropylene, polyamide, and polyethene [6]-[7]. Most research has demonstrated that GNPs can effectively strengthen PLA mechanically at loadings below 1 weight per cent [8]-[10]. Researchers are increasingly interested in developing PLA/graphene nanocomposites due to the global trend toward sustainable materials. The use of GNPSs was claimed to act as a nucleating agent, effectively accelerating crystallisation kinetics and improving the thermomechanical profile of the matrix, despite PLA's reputation for sluggish crystallisation and a low heat distortion temperature. Furthermore, graphene's large aspect ratio and superior intrinsic conductivity offer multifunctional advantages that are rarely achievable with traditional fillers, such as enhanced thermal performance and electromagnetic shielding.

Notwithstanding the physical and chemical benefits conferred by GNP incorporation, the long-term durability of PLA/GNP nanocomposites remains a crucial area of research [11]-[12]. The interaction between the hydrophobic surface of graphene and the relatively polar PLA matrix can markedly affect moisture transport rates, ultimately leading to hydrolytic degradation of the PLA molecular chain. Although GNP can serve as a deterrent to the permeation of water molecules and oxygen, the existence of geometric edges may induce localised stress that promotes micro-cracking under sustained mechanical loading. Therefore, understanding the synergistic relationship between the reinforcement advantages and the degrading effects is crucial for predicting the service life of PLA/GNP nanocomposites, especially in outdoor environments. Moreover, biodegradation through soil burial requires special attention due to diverse soil conditions and variable environmental factors that substantially affect material stability. Therefore, the present study will investigate the effect of soil burial degradation on the mechanical and thermal properties of PLA/GNP nanocomposites

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through systematic soil burial analysis utilising natural soil from UMPSA Gambang. Understanding the synergistic relationship between the reinforcement benefits of GNPs and the resulting degradation is essential for predicting the service life of PLA/GNP nanocomposites in real-world environments. This research is expected to reveal the underlying mechanisms of ageing and biodegradation in PLA/GNP nanocomposites, paving the way for managing PLA nanocomposite waste as multifunctional materials for various commercial and high-tech applications.

2. Materials and Methods

2.1 Materials

Commercial-grade PLA (grades 3052D & 7001D) and GNP were purchased from Sigma-Aldrich, USA. The surface area of GNP was 750 m^2/g.

2.2 Preparation of PLA/GNP Nanocomposites

The PLA/GNPs were pre-mixed in sealed containers and manually agitated before drying. PLA/GNPs were dried in an oven at 80 °C overnight to eliminate moisture and prevent hydrolytic deterioration. Our prior research demonstrated that GNP at loadings below 1 wt.% provides the most effective mechanical reinforcement [10]. Consequently, for the initial comparative analysis, PLA/GNP containing 1.0 wt.% GNP was chosen as the formulation to be evaluated against the pure PLA sample. PLA and 1.0 wt.% GNP were combined using the melt blending process in a Haake twin-screw extruder. The temperature in the heating zone during the compounding of all blends was set to 180, 190, 190, 200, 200, and 190 °C, with screw rotation fixed at 50 rpm and a mixing duration of 20 min. After mixing, a hot-and-cold moulding press was used to fabricate test samples that met the specified criteria. The hot moulding press was set to 180 °C for 20 min at 150 kg/cm^2.

2.3 Soil Burial Degradation Test

Soil burial was used to assess the biodegradability of nanocomposites, as soil microorganisms can enzymatically degrade starch and cellulose particles under appropriate conditions. The specimens were buried in composted soil in a gardening polybag outdoors. They were situated outdoors adjacent to the Chemical Engineering Laboratory of Universiti Malaysia Pahang, Al-Sultan Abdullah, Kuantan, Malaysia. The experiment was conducted from November to December, during the East Coast Peninsular Malaysia monsoon season, characterised by an average temperature of 24-28 °C and a relative humidity of 87% [13]. This research assessed the degradation of pure PLA and PLA/GNP nanocomposites over 14 and 28 days, respectively.

2.4 Analysis

X-ray diffraction (XRD) analysis was performed using an X-ray diffractometer (Rigaku, Tokyo, Japan; model D-MAX 25600 HK). The 2θ range of 10-50 was analysed on a specimen sample at a scan speed of 4°/min, using Cu K radiation ($\lambda = 1.54$ Å). The tensile measurements were conducted in accordance with ASTM D638 using a Shimadzu AG-1 Series universal testing machine equipped with a 5 kN load cell. The PLA/GNP nanocomposite specimens were tested at a crosshead speed of 10 mm/min under ambient conditions. A grip separation of 115 mm and an extensometer gauge length of 50 mm were utilised. Tensile strength and elongation at break were reported as the average values from five specimens per formulation. The thermogravimetric analysis (TGA) was conducted to examine the thermal stability of the graphene nanoplatelet material and the proportion of volatile components by monitoring the weight change of a sample heated at a constant rate. The analysis was performed using a TGA Q500 instrument from TA Instruments under a nitrogen atmosphere. The temperature increment range for analysis was set from 25 °C to 600 °C at a rate of 10 °C/min. The mass loss of specimens was documented as a function of temperature. The loss of mass resulted from chemical reactions or changes in physical properties during heating, including PLA degradation and the emission of volatile compounds as the temperature rose [8,10].

3. Results and Discussion

3.1 Soil Burial Degradation Test

Polymer biodegradability is a critical characteristic of their commercial applications. This study evaluated the degradation of pure PLA and PLA/GNP nanocomposites in an outdoor soil burial experiment over 28 days. From the weight-loss data shown in the graphs in Figure 1, the weight loss after degradation of the pure PLA sample was higher than that of the PLA/GNP nanocomposite sample. The results also indicate that the weight loss of all samples increased with prolonged burial time. Although both pure PLA and PLA/GNP samples exhibit degraded behaviour, the results further indicate that the presence of graphene reduces weight loss compared with pure PLA. Higher weight loss for pure PLA is expected due to its homogeneous nature, which facilitates microbial access to the polymer chain. In contrast, the incorporation of GNP can disrupt the homogeneity of the PLA matrix, hence creating barriers that hinder microbial penetration towards the polymer chain. This phenomenon does not increase the polymer’s degradation rate. Pinto reported a comparable observation in their investigation regarding the impact of GNP on the permeability of the polymer matrix [9].

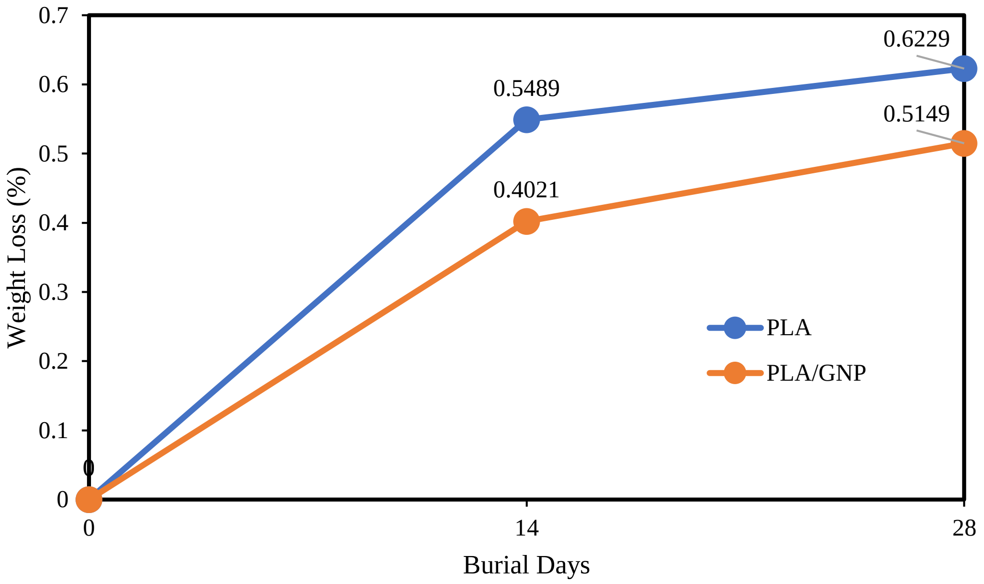


Figure 1. Weight loss of PLA and PLA/GNP for the soil burial experiment method for 28 days

3.2 Mechanical Testing

Tensile tests were performed to determine the tensile strength and elongation at break of each formulation, as shown in Figures 2 and 3, respectively. This test was conducted on samples before degradation and after 14 and 28 days of degradation processes. Figure 2 illustrates that pure PLA exhibits a tensile strength of 17.2943 MPa before the degradation process, 7.5931 MPa following 14 days of degradation, and 5.0335 MPa after 28 days of degradation. The incorporation of 1 wt.% GNP enhanced the tensile strength to 26.4181 MPa relative to neat PLA before degradation, representing a rise of over 50% in magnitude. Compared with neat PLA, higher tensile strength was observed after 14 and 28 days of deterioration. The increase in tensile strength is primarily due to effective stress transfer from GNPs within the PLA matrix, as observed in various studies [14]-[15]. On the other hand, Figure 3 illustrates a notable reduction in the elongation at break of PLA-reinforced GNP, primarily attributable to the rigidity of the GNP nanofiller. The incorporation of rigid elements, such as GNP, enhances the stiffness of nanocomposites. This rigidity limits the capacity of polymer nanocomposites to undergo plastic deformation, thereby reducing elongation at break.

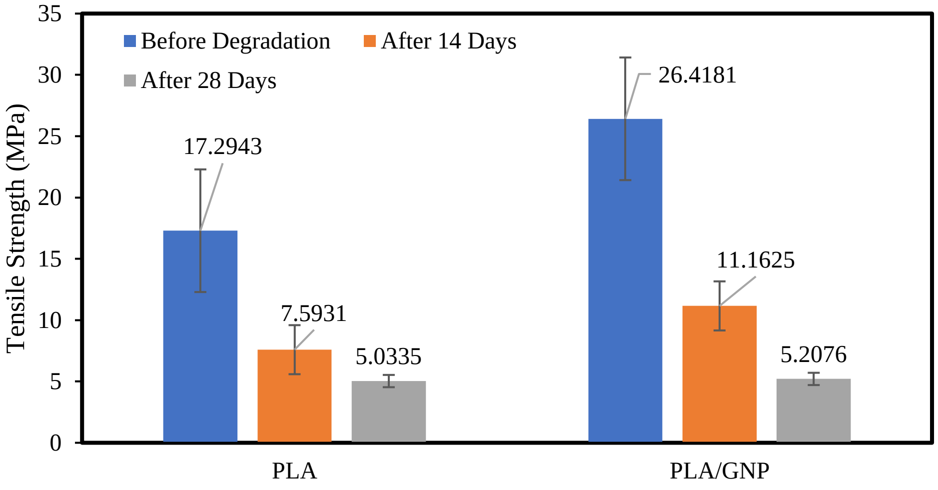


Figure 2. Tensile strength before degradation and after 14 and 28 days of degradation tests

3.3 Thermogravimetric Analysis

Figure 4 illustrates the Thermogravimetric curve, while Table 1 summarises the T5, T10, and T50 temperatures. The TGA curves in Figure 4 indicate that the breakdown temperature of the nanocomposites begins at approximately 330 °C and rises rapidly to 380 °C before degradation begins. The results indicated a decrease in temperature for samples following 14 days of deterioration, showing that soil burial degradation affects the thermal stability of the polymer sample. The decomposition temperature of the nanocomposites, after 24 days of deterioration, initiates at approximately 350 °C and rises rapidly to 400 °C. The TGA curves in Figure 4 unambiguously indicate that the PLA/GNP exhibits superior thermal stability compared to pure PLA. As has been widely observed in other research, graphene acts as an effective thermal barrier. However, as the polymer degrades, the relative concentration of graphene might increase, and it tends to agglomerate, disrupting the thermal barrier capacity. Degradation also converts long polymer chains into shorter ones, leaving an exposed microcrack [16]. The presence of microcracks facilitates the penetration of oxygen into the matrix, thereby lowering thermal stability. These complex phenomena contributed to a decrease in the thermal stability of 28-day-degraded PLA/GNP.

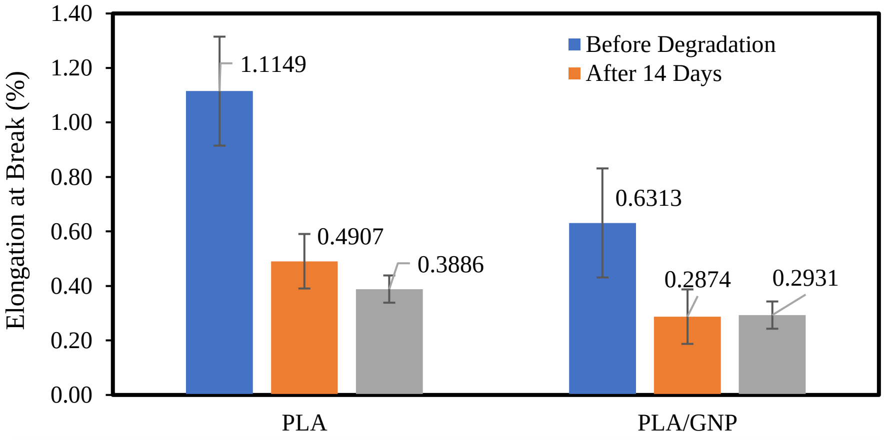


Figure 3. Elongation at break before degradation and after 14 and 28 days of degradation test

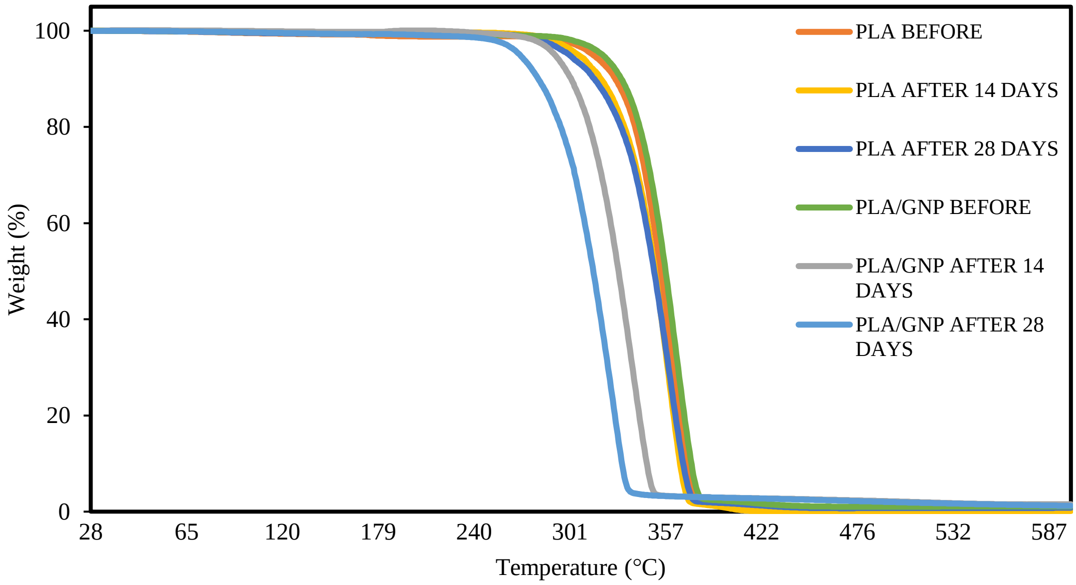


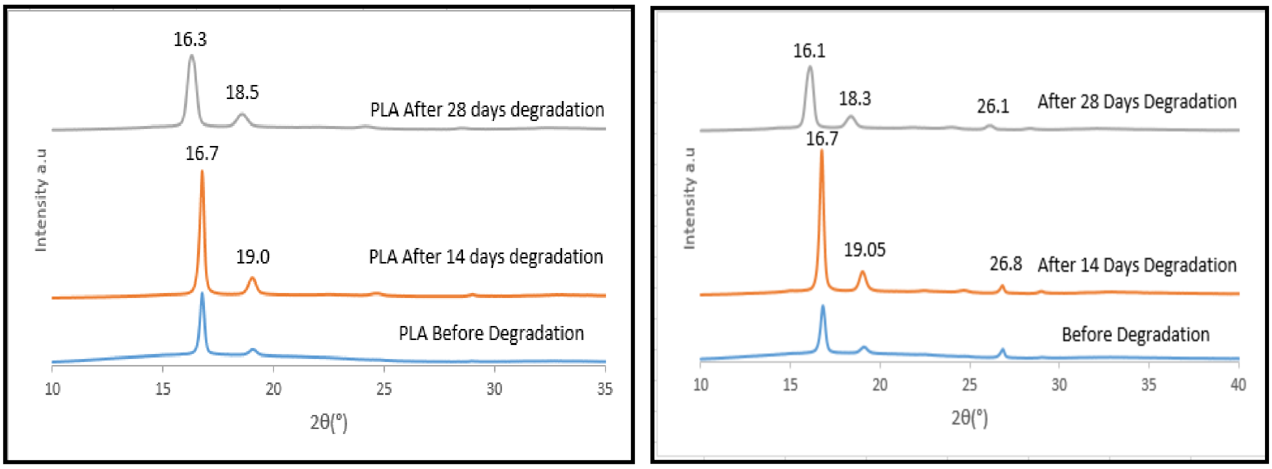
Figure 4. Comparison of TGA curves for PLA and PLA/GNP before and after 14 and 28 days of degradation processes

Table 1. T5, T10 and T50 temperatures of pure PLA and PLA/GNP samples before and after 14 and 28 days of degradation processes

Sample		T_5 (°C)	T_{10} (°C)	T_{50} (°C)	Char Residue
Before Degradation	PLA	315.20	328.86	356.12	0.9160
	PLA/GNP	320.80	3332.43	356.87	1.0968
After 14 Days	PLA	304.97	319.04	353.62	0.1533
	PLA/GNP	290.65	300.45	328.91	1.5317
After 28 Days	PLA	300.65	316.67	353.62	0.7790
	PLA/GNP	269.51	280.11	314.05	1.1930

3.4 X-Ray Diffraction Analysis

The crystalline (α form) characteristic peaks at $2\theta = 16.76$ ° and $2\theta = 19.07$ ° were visible in the neat PLA in Figure 5(a). For PLA, no new peak emerged to distinguish the results before and after deterioration. Nevertheless, an extra peak that can be associated with stacked graphene layers emerged at $2\theta=26.8°$ in the PLA-GNP nanocomposite (Figure 5(b)) [16]. Interestingly, the PLA/GNP sample shows a pattern that is nearly identical to that of clean PLA. The XRD result after 14 days of degradation shows increased diffraction intensity compared to the pre-degradation result. This is likely due to faster degradation of PLA's amorphous regions, leading to a relatively higher crystalline content in the matrix. The result after 28 days of degradation shows that the characteristic peaks of neat PLA have slightly shifted to 2θ = 16.3 ° and 2θ = 18.5 °. A shift to a lower angle indicates an increase in the d-spacing (d) between polymer chains. This observation also suggests that as the PLA matrix degrades, the lattice arrangement changes, thereby disrupting the tight packing of the crystalline structure [17].



(a) (b)

Figure 5. X-Ray Diffraction for (a) PLA before and after the degradation process, (b) PLA/GNP before and after the degradation process

4. Conclusions

Based on the study, incorporating Graphene Nanoplatelets into the PLA matrix significantly enhances the material's structural and thermal integrity. However, as biodegradation proceeds, ductility and thermal stability decrease. While soil burial tests confirmed that all samples degraded over 28 days, the GNPs acted as a physical barrier, hindering microbial penetration and resulting in lower weight loss than the more homogeneous neat PLA. As for the mechanical properties analysis, the presence of 1 wt.% GNP reinforcement provided a substantial 50% increase in tensile strength through efficient stress transfer. However, the presence of GNP has also increased the stiffness and rigidity of PLA nanocomposites, leading to a significant drop in elongation at break. From TGA analysis, the PLA/GNP nanocomposites exhibited superior thermal stability compared to neat PLA under all conditions, providing evidence that GNPs serve as an effective thermal barrier. However, the degradation of the PLA matrix significantly reduces the thermal stability of all PLA/GNP nanocomposite samples, primarily due to molecular-level changes in the polymer chain. These molecular changes were supported by XRD analysis, which revealed that although degradation primarily attacked amorphous regions, it eventually led to lattice distortion and a change in crystalline structure.

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Declaration of Competing Interest

The author declares no conflicts of interest.

CRediT Authorship Contribution Statement

M.S.Z. Mat Desa: Conceptualisation, Methodology, Validity, Formal analysis, Investigation, Resources, Data curation, Writing – review and editing, Supervision, Project administration, Funding acquisition

S.M.A.D. Syed Mohd Azmir: Methodology, Formal analysis, Investigation, Resources, Data curation, Writing – original draft, Visualisation

M. Bijarimi: Conceptualisation, Methodology, Validity, Formal analysis, Investigation, Resources, Data curation, Supervision

M. Yusop: Resources, Data curation, Writing – review and editing, Visualisation, Supervision, Project administration

S.H. Kamarudin: Resources, Data curation, Writing – review and editing

Availability of Data and Materials

The datasets generated and/or analysed during the current study are available from the corresponding author on reasonable request.

Ethics Statement

This study did not involve human participants or animal subjects. Ethical approval was therefore not required for this research.

Generative Artificial Intelligence Declarations

The authors claim that artificially intelligent-assisted technologies, such as generative AI, were not used to generate content, ideas, or theories. We have just utilised AI to enhance readability and refine the language. This was used with extreme human control and oversight. The authors take full responsibility for reviewing and approving the content.

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