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The study of degradation and mechanical properties of poly(lactic) acid (PLA) based 3D printed filament

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Abstract

Additive manufacturing, commonly known as 3D printing technology, has become one of the mainstream processes in the manufacturing industry due to its advantages over conventional manufacturing, which have piqued the public's interest. This study aims to focus on the influence of thermal conditions on crystallization towards mechanical properties of 3D printed poly(lactic) acid (PLA) degradation samples with 100% infill. As for the degradation profile, the highest weight loss recorded by the samples was 0.7%, observed in samples buried in soil with an abiotic medium for one month. The exposure of degraded samples to high temperature during drying affected their crystallinity, resulting in significant changes in strains, particularly between week 1 and week 2, where strains dropped significantly from 7.33% to 4.28%, respectively. In conclusion, it has been demonstrated that degradation for PLA material still can occur in an abiotic medium, albeit at a slower rate compared to a biotic medium due to the presence of additional microorganisms and bacteria. Besides, the post-heat treatment process on PLA degradation samples affects their crystalline structure, resulting in significant changes in mechanical properties, particularly especially strains. Therefore, it can be concluded that different materials exhibit distinct mechanical properties.

1. Introduction

Since its inception in 2011, when the concepts of the Fourth Industrial Revolution 4.0 were first unveiled to the public, this industrial revolution has evolved from theoretical notions to real-world applications. The Fourth Industrial Revolution, commonly known as IR 4.0, comprises nine pillars: robotics, simulation, cloud computing, system integration, Internet of Things (IoT), cyber-security, augmented reality, big data and additive manufacturing [1]. Among these, additive manufacturing stands out as a new approach offering numerous advantages over conventional manufacturing. These include shorten development period, ultra-customization, increased flexibility in production environment, faster decision-making through decentralization, and most importantly, promotion of sustainability and resource efficiency in ecological contexts [2, 3].

Additive manufacturing, also known as 3D printing, is the process of making objects using a computer-controlled device based on a desired computer-aided design (CAD) file [4]. There are various classifications of

additive manufacturing, including vat-polymerization, sheet lamination, directed energy deposition, binder jetting, material jetting, powder bed fusion, material extrusion [5]. 3D printing helps reduce production costs and waste of raw materials, promotes energy-friendly usage and shortens fabrication time for specific applications [6]. Its high customizability to construct intricate and complex design is one of the most prominent features of this technology, making it particularly attractive to manufacturers, especially in the research and development. However, it is important to note that different 3D printing techniques in yields different results.

Fused Deposition Modelling (FDM), a sub-category of material extrusion, stands out among various 3D printing techniques for its widespread use in producing functional prototypes using various thermoplastics [7]. Its ability to generate complicated geometrical parts in a clean and safe manner, suitable even for office environments, makes it highly favoured [8]. Additionally, FDM offers several advantages including affordability, cost-effectiveness, on-demand manufacturing, a wide variety of materials and the elimination of post-processing requirements compared to other techniques [9]. The FDM process begins by melting thermoplastic material or filament into a liquid–solid state, which is then selectively deposited through a nozzle following the cross-sectional geometry of the parts [10]. This layer-by-layer approach enables the direct production of 3D parts from a CAD model. Currently, 3D printing technology, particularly FDM, is extensively used for mass customization and fabrication of open-source design across numerous applications, including agriculture, healthcare, automotive, locomotive, and aviation [11–14]. Moreover, amidst the Coronavirus disease 2019 (COVID-19) pandemic, 3D printing has emerged as a critical tool in enhancing healthcare and our overall response to the crisis. Medical devices such as personal protection equipment (face shields and respirators), testing devices (nasopharyngeal swabs), and medical devices (ventilator parts including valve and components) are all being produced using 3D printing technology [15]. The crisis has demonstrated the potential of 3D printing to contribute to a more sustainable and environmentally friendly future.

To ensure consistent production of high-quality products, 3D printing relies on the use of high-quality of materials, akin to other manufacturing process. This technology can fabricate fully functioning parts from a diverse range of materials including ceramics, metals, polymers, composites and functionally graded materials [16]. Among these materials, poly (lactic) acid (PLA) stands out as a promising option, often derived from renewable resources such as corn starch and sugar cane [17]. PLA has gained prominence due to its favourable properties, including ease of fabrication, not-toxicity, biocompatibility and good in mechanical strength [18]. Importantly, PLA is degradable and recyclable, contributing to sustainability goals [19]. As 3D printing technology advances, the sustainability element becomes significantly important for maintaining environmentally friendly practices while preserving performance standards tailored to specific applications.

The use of biopolymer as primary materials in manufacturing process is increasingly recognized as a key strategy for promoting environmental sustainability. The development of biopolymer materials has garnered significant interest among researchers, driven by growing concerns over environmental issues associated with conventional polymers waste products. In Malaysia, heightened awareness of waste management has been portrayed by initiatives of Ministry of Energy, Science, Technology, Environment, and Climate Change (MESTECC) roadmap's towards zero single-use plastics campaign [20]. Aligned with the United Nations Sustainable Development Goals (SDGs), this campaign aims to promote sustainable development by fostering the economic growth while preserving ecosystems [21]. In the broader context, environmental degradation processes are influenced by two correlated conditions: biotic and abiotic environment. Biotic degradation involves the breakdown of the polymer molecules aided by microorganism and bacteria, while abiotic degradation occurs through chemical processes in the absence of living organisms, influenced by factors such as humidity, temperature, pH value and ultraviolet (UV) light [22]. Additionally, molecular weight, glass transition temperature (T_g) and crystallinity of the biopolymer also play significant roles in the degradation profile.

In this work, the research focuses on the biodegradability of 3D printed samples buried under a soil and left exposed to natural weather for 31 days. The assessment will evaluate the degradation rate of 3D printed samples and examine the tensile properties of these samples after exposure to specific thermal temperatures. This analysis is crucial important for understanding the impact of high temperature on degraded polymers and their tensile properties during the recycling process, aiming to achieve environmental sustainability while maintaining performance standards required for specific applications.

2. Materials and methods

This research investigated the impact of soil-based environments on the biodegradability of 3D printed PLA samples. Additionally, the influence of thermal conditions on these samples was examined by assessing their tensile properties using a universal tensile machine (UTM). To ensure data reliability, triplicate sets of 3D printed PLA samples were produced for each week of the experiment, spanning from first week until forth week.



Figure 1. The samples were buried in the transparent box and exposed under natural environment.

Table 1. Ender 3 Pro set-up parameters.

Parameters	Descriptions
Nozzle temperature	200 °C
Bed temperature	50 °C
Infill percentage	100%
Infill pattern	Lines

2.1. Preparation of samples

A commercialized white PLA filament (Flashforge, Zhejiang, China) was used as a material feedstock in this research. The samples were designed as rectangular and dumb-bell shape in an open 3D software (Blender, Amsterdam, Netherlands) for degradation and tensile measurement respectively. The tensile design concordance to the ASTM D-638 (Type-I) with a dimension of 165 mm × 13 mm × 3.2 mm. Then, a slicer software (Cura, Utrecht, Netherlands) was used to convert the Standard Tessellation Language (.stl) into a readable format (.gcode) for a 3D printer (Ender 3 Pro, Creality, Shenzhen, China). The 3D printer was used to print the samples according to certain parameters. The parameters were set as followed in the table 1.

2.2. Degradation and tensile

For the degradation test, triplicate of 3D printed samples were printed for four weeks. The samples were weighed using analytical balance and recorded. Then, they were buried in a transparent box fill with garden soil that was sieved through a 2 mm mesh filter for unwanted particles at a depth of 10 cm. The transparent box was let to expose under natural environment as shown in figure 1.

The triplicate samples were extracted from the soil each week to measure the weight loss as an indication of the degradation process. Upon extracting all the 3D printed PLA samples, the samples were washed with distilled water to remove dirt from soil residue. Then, samples were exposed to 80 °C for 24 h in the oven before being weighted again. It was to understand the correlation between degradation and thermal treatment on crystallization of the degraded samples. The percentage of weight loss were calculated by using equation (1) whereas the W (%) is the percentage of weight loss, W_i is the initial weight and W_f is the final weight.

$$W(\%) = \frac{W_i - W_f}{W_i} \times 100\% \quad (1)$$

The final weight after being dried were recorded before doing tensile measurement. The samples were let to cool down at room temperature. The stress–strain measurement was conducted using UTM (Instron 5566, Massachusetts, United States) equipped with a 30 kN load and at a speed of 50 mm min^{−1} as shown in figure 2. The load direction relative to the layer direction in printing the samples was 45°.

3. Results and discussion

3.1. Influence of abiotic mechanism on weight loss of 3D printed PLA with 100% infill

Polymers have various ways for biodegradation depends on their types. The carbon chain polymers undergo peroxidation process first before proceed to oxo-biodegradation of low molar mass compound within the



Figure 2. Tensile measurement using Instron 5566.

Table 2. The percentage of weight reduction loss throughout a month.

Week	Initial weight (g)	Final weight (g)	Percentage of reduction (%)
1	9.2185	9.2005	0.20
2	9.2076	9.1761	0.37
3	9.2897	9.2420	0.51
4	9.3071	9.2410	0.71

polymer structures [23]. However, some polymers experience biotic or abiotic process before going to hydrodegradation process as their main degradation mechanism especially for hetero-chain polymers such as poly(lactic) acid (PLA) and polyhydroxyalkanoates (PHA) [24]. The environmental degradation of PLA is affected by its material properties such as the molecular weight, crystallinity, glass transition, and by environmental factors such as humidity, temperature, pH value, and the presence of enzymes or microorganisms during degradation [25]. In order to simulate the real compost environment, the degradation experiment was set up by using common soil from the garden as the abiotic medium without any additional bacteria or microorganisms until the end of the experiment.

Table 2 below shows the results of weight loss of samples that had been buried for a month. The results show that no significant weight loss was observed between samples from week 1 until week 4 but still show the sign of degradation with an increase of the percentage of weight reduction from samples in the first week until the last week although the rate for the samples to degrade was very slowed as shown in figure 3. This was due to low moisture content in the soil which one of the main factors that encouraged hydrolysis and assimilation of PLA [26]. Another study found that some bleached patches appeared on the surface of PLA samples only after 60 days of testing, with a modest increase in weight loss [27]. According to the same work, the small changes in weight reduction and physical appearance presented a slow rate of PLA degradation in the common soil. The sluggish rate of hydrolysis at low temperatures, as well as the random distribution of PLA-degrading microbes in the soil

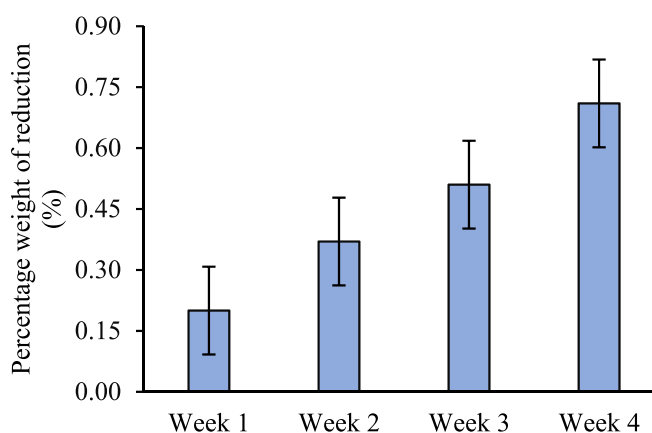


Figure 3. Percentage of weight reduction (%) over a month.

environment were a few factors that contributed to this situation. Based on the results, it can be seen that the highest weight loss was recorded during the fourth week, which was left buried for a month in the soil with 0.71% percentage of weight reduction, while other samples such as P1 recorded the lowest weight loss with only 0.20%. P2 and P3 samples each recorded 0.37% and 0.51% respectively.

Past studies reported that medium and mechanisms that were used for identifying the degradation rate of biodegradable polymer had resulted in a significant difference between them especially in terms of the weight loss, tensile stress and strain of the polymer. Other researchers had reported that additional bacterial or microorganisms into the soil for degradation of polymer gave a significant change to the physical appearance of the samples plates. It was found that the clear PLA plates used in the experiment reacted well with the biotic medium as the samples' corner of the plates got corroded as reaction with the mixture of land soil and bacterial. This was later finally justified by the 6% percentage of weight reduction that was recorded from initial weight within a month [28]. Besides, another previous study also identified that degradation of PLA in medium with enrichment of microorganisms was found to be faster than abiotic medium at elevated temperature of 45 °C and 50 °C implying that PLA degradation was accelerated by the presence of bacteria in the environment. This was verified by the rapid reduction of samples' tensile stress in mixture of soil and microbes compared to abiotic medium as the tensile stress was absent after at 50 °C only after 36 days of testing.

Although in a previous study, there was no justification for the acceleration of degradation rate in the presence of microorganism for high molecular weight PLA as no major difference was discovered between biotic and abiotic mechanisms when PLA was incubated at various temperatures [29]. However, based on the comparison with the results obtained above, it was justified that the using of abiotic medium without the presence of any microorganisms or bacteria contributed to the slow rate of degradation which later would affect the percentage of weight reduction compared to the biotic medium that enriched with microorganisms [22]. Therefore, it is generally approved that degradation of PLA involved two distinct way of mechanisms with chemical hydrolysis in the presence of water (abiotic) and the presence of microorganisms (biotic) as the primary pathway for degradation of heterochain polymer [26].

3.2. Influence of thermal on crystallization towards mechanical properties of 3D printed PLA degradation samples with 100% infill

The experiment was designed to study the influence of thermal treatment on crystallization towards mechanical properties of 3D printed PLA degraded samples from week 1 until week 4. Figure 4 below shows the results of tensile stress–strain curve of each samples after been exposed at 80 °C. Based on the results it can be observed that there was only a less changes in tensile strength especially between the samples in week 1 and week 2. The degraded samples in week 1 recorded 45.89 MPa while the samples in week 2 recorded a slight difference at a value of 46.98 MPa. The trend continued in week 3 with 48.91 MPa was recorded but slightly decreased in week 4 with 47.94 MPa. As the tensile stress for each samples increased, it indicated that the temperature played a big role on the mechanical properties for each of the degraded samples.

Besides, the figure 5 below shows the tensile strains between samples from week 1 until week 4 which were consistently decreased. From the results, it a significant decreased of samples' strains were observed especially between the samples in week 1 and week 2. Week 1 samples recorded the highest strains with a 7.33% while for the samples in week 2, the result was only 4.28% which presented a great loss of strains in the first two weeks of the experiment. The results keep decreased for the remaining samples for week 3 and week 4 with 3.93% and

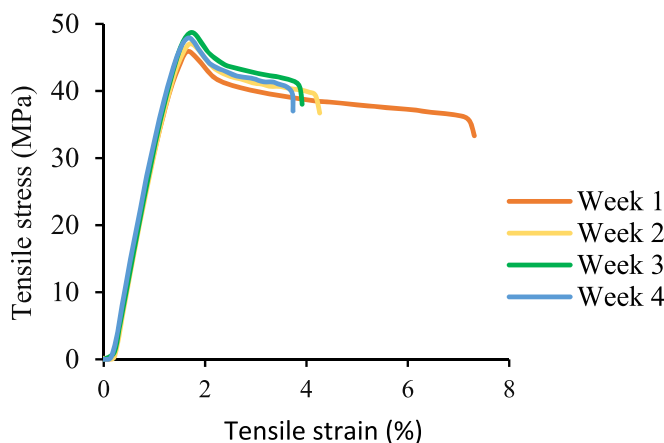


Figure 4. The tensile stress–strain curve for the degradation samples.

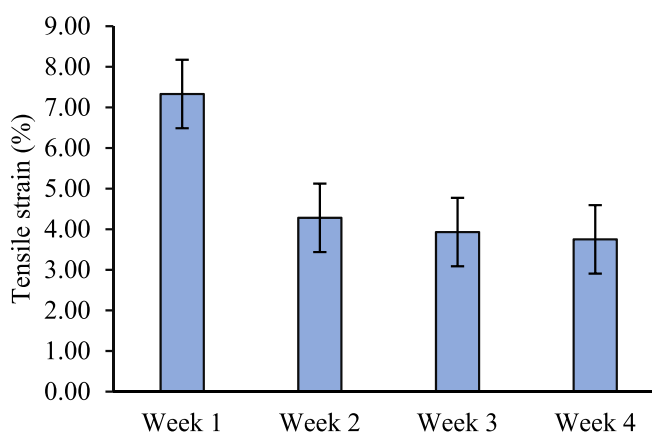


Figure 5. The tensile strain over the weeks.

3.75% were recorded respectively. This indicated that the brittleness in each samples was increased due to a significant decrease of their strains which related to the crystallinity structure of the polymer.

The crystallinity level is one of the main features that indicates the amount of crystalline area with respect to amorphous region in the polymer structure. There are many polymer's mechanical characteristics that affected by degree of crystallinity such as hardness, young modulus, tensile stress, stiffness, cease and melting points [30]. As we know, PLA was a semi-crystalline polymer that contained many amorphous regions in its structure. However, according to previous study, the structure will change whenever high temperature was implemented to the structure of the polymer. When the implemented temperature was higher than the glass transition temperature of the polymer, the specific heat of PLA falls as the crystallinity increases and because of that the specific heat capacity of the amorphous phase is greater than the crystal phase [31].

PLA samples initially structured with amorphous regions due to low degree of crystallinity as portrayed by the previous study. However, after undergo degradation process, the amorphous regions were directly experienced chain scissions and relaxation in the structure with increased of six-fold chains rapidly to form crystalline region at 65 °C. This led to an increase of the tensile strength in the early phase of degradation that also can be seen in the early stage of this current study [32]. However, greater degree of crystallinity in polymer structure directly decreased the strains value exponentially as crystalline is more than amorphous regions, resulted to stiff polymer composition that reduced its capability to elongate. This could imply that during the early stages of degradation occurred, the changes in degree of crystallinity had affected more on the mechanical features of polymer than lost in its molecular weight, which would be consistent with polymer-chemistry studies for conventional manufactured samples [33].

4. Conclusions

Additive manufacturing has become one of the main component pillars in The Fourth Industrial Revolution (IR 4.0). Its main advantage is the ability to freely construct and personalize the thing with the help of 3D modelling software, making it a viable alternative to subtractive manufacturing. However, since this kind of manufacturing is still considered new, the filament or resin used in it requires a lot of study to compete with products from traditional production. From the result, it can be concluded that degradation still occur in abiotic medium but at a slow rate. In future work, the study of combination between biotic and abiotic medium is very important in order to identify the effect of microorganism towards the rate of degradation. Last but not least, the current study also established that the post-heat treatment process on PLA degrades samples affecting its crystallinity structure while resulting in significant changes in mechanical properties especially the strains. Therefore, it can be simplified that different materials have their own mechanical properties.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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