Evaluation of Mechanical Properties of Polylactic Acid (PLA) Films Over One and Ten Months of Aging

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Abstract

Polylactic acid (PLA) is a thermoplastic, biodegradable, and bioactive polymer obtained from renewable resources such as beets and potatoes. PLA is regarded as a polymer that is nearly brittle, which can restrict its applications in the packaging industry. The mechanical properties of this polymer can be improved by adding nanoparticles and plasticizers. In this research, zinc oxide nanoparticles (1 wt% of PLA), Polyethylene glycol 400 (20 wt% of PLA), and Polysorbate 80 (0.25 wt% of the solution) were used to improve the mechanical properties of PLA films. The effects of these materials on the films were measured at two time points: the first month and the tenth month, with the aim of investigating physical aging, a precursor to polymer degradation. Statistical analysis was performed on the mechanical properties measured during these periods to identify significant differences between the produced films. Results showed that the highest tensile strength (82.99± 1.90 MPa, neat PLA), elongation at break (76.82± 27.22 %, PLA/PEG/ZnO), toughness (20.13± 7.89 J cm^{-3}, PLA/PEG/ZnO), and Young's modulus (2.74± 0.10 GPa, neat PLA) were observed in the first month. Analysis of variance results regarding the effect of time on each film revealed that in most cases, the mechanical properties did not change significantly after ten months. Based on the stress-strain curves, it was found that the neat PLA film is among the resistant materials. The PLA/Polysorbate/ZnO film exhibited brittle behavior in the tenth month. The remaining samples exhibited characteristics that fell between resistant and ductile materials in both the first and tenth months.

Keywords: Mechanical properties, Physical aging, PLA, ZnO nanoparticles

Introduction

Polylactide is a relatively inexpensive polymer obtained from sources such as corn, sugarcane, and beets. The usage of polylactic acid has grown significantly in the plastic industry due to its superior mechanical properties (Ahmed, Hiremath, & Jacob, 2016; Heydari-Majd *et al.*, 2019). Mechanical properties are important parameters in food packaging. The ideal film should be both resistant and tough, which, of course, is not possible in reality; therefore, deformation is preferred over fracture (Heydarian, Ahmadi, Dashti, & Normohammadi, 2022; Leon *et al.*, 2017; Odian, 2004). Packages with high tensile strength, Young's modulus, and elongation at break have a longer shelf life and are less likely to be damaged during transportation (Aljilji *et al.*, 2020; Sangroniz *et al.*, 2019). Despite all the advantages of PLA, some problems, such as brittleness and a weak barrier to water vapor, gas, and light, need to be improved (Yu *et al.*, 2021). Zinc oxide nanoparticles, when used as a filler, can improve the mechanical properties, water vapor barrier, UV protection, and antibacterial properties of PLA (Guz *et al.*, 2017; Yu *et al.*, 2021). Zinc oxide is a compound that, when used in plastic materials in contact with food, does not pose a risk to human health at concentrations of up to 2 wt% (Guz *et al.*, 2017). Plasticizers can be used to reduce the brittleness of polylactic acid films. Polyethylene glycol and polyoxyethylene sorbitan (Polysorbate 80) are non-toxic plasticizers that are suitable for use in the food industry (Khairuddin *et al.*, 2016; Parreidt *et al.*, 2018).

Physical aging is essentially the alteration in polymer behavior with passing time, such as

mechanical property modifications, under conditions where no external factors like change in temperature and stress exist to influence the film's characteristics (Cangialosi, 2024). Physical aging does not necessarily lead to degradation. Sometimes aging can mean the formation and stabilization of new materials (Izdebska, 2016). Physical aging in biodegradable films is a progressive deterioration process where polymeric materials, especially polysaccharide-based films, systematically lose their functional properties during usage. This intrinsic aging mechanism compromises mechanical integrity and performance characteristics, rendering films increasingly unsuitable for industrial applications (Havstad *et al.*, 2023; Janik *et al.*, 2023). Investigating the various properties of biodegradable films over time, particularly mechanical characteristics crucial to film integrity, is of paramount importance in understanding their practical applicability and functional sustainability.

Due to its importance, mechanical properties have been investigated in many articles. For example, in one research study, the mechanical properties of polylactic acid/polyethylene glycol (6000, 10000, and 20000) films were measured. Researchers found that as the molecular weight of polyethylene glycol increased, the elongation at break decreased while the tensile strength and Young's modulus increased (F.-J. Li *et al.*, 2015). In a study, the mechanical properties of PLA-based composites were investigated to quantitatively model their mechanical behavior (Mirkhalaf & Fagerstrom, 2021). The effect of polyethylene glycol as a plasticizer on the mechanical and thermal properties of polylactic acid/polybutylene succinate films was investigated. The results of the study showed that polyethylene glycol is a suitable plasticizer for PLA, as it decreases the glass transition temperature and increases elongation at break (Pivsa-Art *et al.*, 2016). The effect of 1%, 3%, and 5% ZnO nanoparticles on the morphological and mechanical properties of PLA films was investigated by researchers. According to their report, Young's modulus increased with the increasing percentage of nanoparticles (Shafiee-Nasab, Tabari, & Azizi, 2018). In a study, the physical and mechanical properties of PLA/ZnO nanoparticle films with acetyl tributyl citrate plasticizer were investigated. It was found that with the addition of 3 wt% of nanoparticles, the elongation at break decreased (Tang *et al.*, 2020). The mechanical and thermal properties, as well as the decomposition of polylactic acid composites, were investigated over a period of 180 days. By adding iron powder to the composites, the tensile strength increased, while the addition of magnesium powder resulted in increased elongation at break (Oksiuta *et al.*, 2020). Various research studies have been conducted regarding the investigation of physical aging, with several key studies being highlighted. A research investigation explored the impact of nine-month storage on starch film mechanical properties. The study revealed significant increases in tensile strength and Young's modulus during the extended storage period (Guaras, Alvarez, & Luduena, 2019). Effect of physical aging on mechanical properties of 3D printed elastomeric Polyurethane was investigated. The results showed that physical aging could not affect strength in about 8 months (Schwarz *et al.*, 2022). In a comprehensive study, researchers investigated the mechanical behavior of poly lactic acid/poly hydroxybutyrate films over a 30-day duration. The tensile strength and elongation at break of the films were above 20 MPa and 270%, respectively, with no significant changes observed during the 30-day period (Briassoulis, Athanasoulia, & Tserotas, 2022). The primary objective of this research is to investigate the mechanical properties of poly lactic acid films over a ten-month period, focusing on the impact of physical aging on mechanical characteristics. The data for the first month are similar to those from our previous study, and we aim to compare them with the data from the tenth month (Tajari, Sadrnia, & Hosseini, 2023). Stress-strain curves were meticulously generated to analyze film behavior during tensile testing. The

comprehensive experimental approach provides robust insights into the mechanical durability of PLA films under prolonged storage conditions.

Material and Methods

Materials

The PLA granules (197,000 g mol^{-1}) was provided by FKuR Kunststoff GmbH, Germany. Zinc oxide nanoparticles, with an average diameter of 35 to 45 nanometers, were purchased from the Iranian Nanomaterials Pioneers Company (Iran). Polyethylene glycol 400 (PEG 400) and polysorbate 80 were obtained from Merck (Germany). Dichloromethane (DCM) was supplied by Dr. Mojallali Company (Iran).

Preparation of films

Experiments were conducted in the post-harvest laboratory of the Biosystems Engineering department at Ferdowsi University of Mashhad, Iran. PLA granules were placed in an oven at a temperature of 60℃ for 24 hours to completely lose their moisture. To make the film with a 15 cm diameter, 0.625 g of PLA and about 31 ml of dichloromethane were used. The amount of polyethylene glycol was set at 20 wt% of PLA, the amount of polysorbate 80 was set at 0.25 wt% of the solution, and ZnO was set at 1 wt% of PLA, according to the articles of other researchers (Falqi *et al.*, 2018; Heydari-Majd *et al.*, 2020; Jantrawut *et al.*, 2017; Luangtana-Anan, Nunthanid, & Limmatvapirat, 2010; Shankar, Wang, & Rhim, 2018). The solutions were stirred for 12 hours with a stirrer (H-M101, Sabalan Azmai Tehran, Iran) at room temperature. For the films with nanoparticles, the ZnO nanoparticles and dichloromethane were first sonicated with an ultrasonic probe (400W/20kHz, Topsonics, Iran) for 10 minutes and then added to the solution. The actual power used in the experiment was 200 watts. The final solution was poured into a glass plate and dried at room temperature for 24 hours to facilitate easy film removal from the plate. The compositions of the produced films are shown in Table 1.

Table 1- Compositions of produced films and their abbreviation

Abbreviation	Composition
PEG/PS/ZnO	PLA+ Polyethylene glycol 400+ Polysorbate 80+ ZnO NPs
PS/ZnO	PLA+ Polysorbate 80+ ZnO NPs
PEG/ZnO	PLA+ Polyethylene glycol 400+ ZnO NPs
Neat PLA	PLA

Determination of film properties and characterization

Mechanical properties

The mechanical properties of the films were measured according to ASTM D882-02, both in the first month and after ten months (ASTM, 2012). The samples were in the form of rectangles (2 × 10 cm^2) and were conditioned for moisture. Tests were conducted using a texture analyzer (H5 KS, Manchester, U.K.), with an initial grip distance of 50 mm and a strain rate of 50 mm min^{-1}. Tensile strength (TS), elongation at break (EB), toughness (T), and Young's modulus (YM) were calculated using the following equations:

$$TS = \frac{F_{max}}{A} \quad (1)$$

where F_{max} is the maximum load and A is the cross-sectional area of the sample.

$$EB = \frac{L_f - L_0}{L_0} \times 100 \quad (2)$$

here, L_0 is the initial length and L_f is the final length of the sample.

$$T = \frac{\text{Work}}{\text{Volume}} \quad (3)$$

work is the area under the force-displacement curve and volume refers to the volume of the sample.

$$YM = \frac{L0(\Delta F)}{A0(\Delta L)} \quad (4)$$

where, L_0 is the initial length (50 mm), A_0 is the original cross-sectional area, and ΔF and

ΔL are, respectively, the amounts of force and length changes in the linear part of the force versus length changes graph (Abubakar *et al.*, 2022; Ching, Keesan, & Muhamad, 2022; He, Duan, & Wang, 2020; Jantrawut *et al.*, 2017; Lin *et al.*, 2020).

Statistical analysis and interpretation of data

The results were analyzed using Minitab software version 18 (Minitab Inc, USA). The data were represented as a mean value ± standard deviation of a triplicate of each measurement. A significant difference between means was evaluated using one-way ANOVA, and Tukey and Fisher LSD tests were used for comparing mean values. Tukey's test was employed to assess significant differences between various films, while Fisher's test was used to examine the effect of time on each film. Curves were drawn in Excel 2013 software.

Results and Discussion

Mechanical properties

The results of investigating the mechanical properties of films in the first and tenth month, and the significant differences between films, are observable in Figures 1-4.

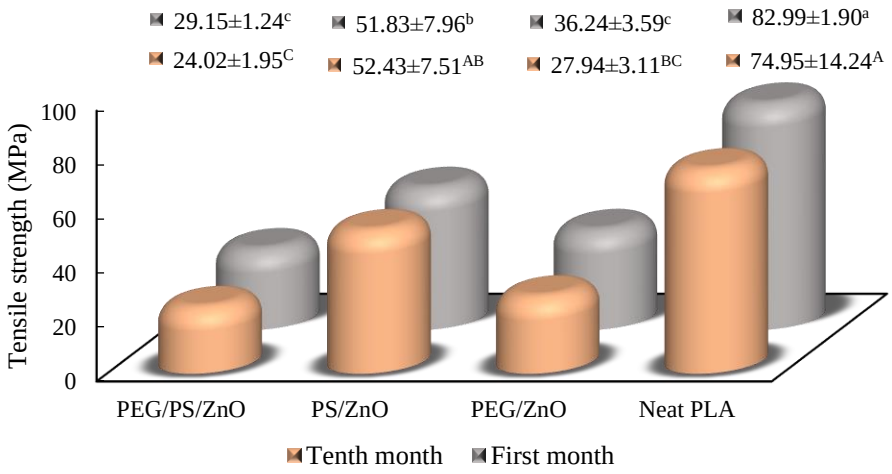

Fig. 1. Tensile Strength of films produced in the first and tenth months
Values are reported as mean± standard deviation. Lowercase letters show significant differences between films in the first month, and capital letters show significant differences between films in the tenth month ($p< 0.05$)

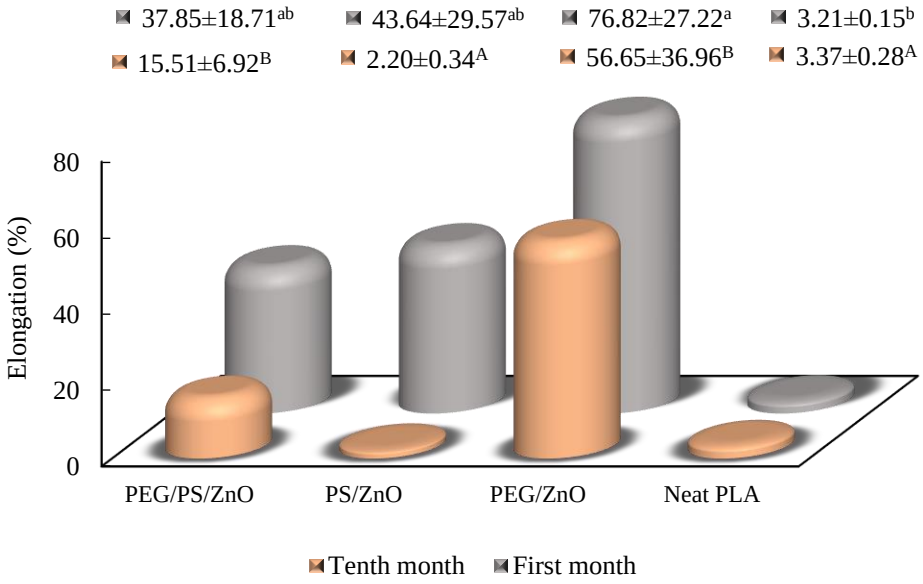

Fig. 2. Elongation at break of films produced in the first and tenth months
Values are reported as mean± standard deviation. Lowercase letters show significant differences between films in the first month, and capital letters show significant differences between films in the tenth month ($p< 0.05$)

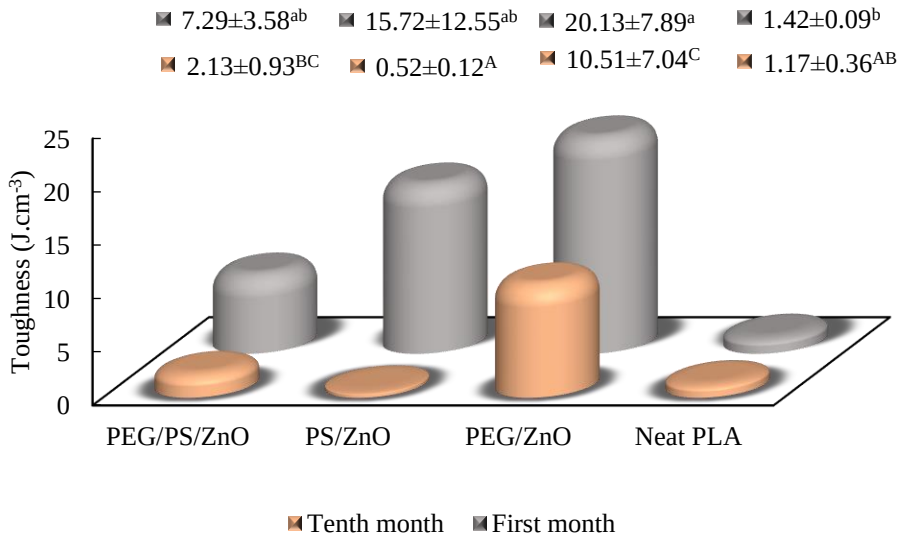

Fig. 3. Toughness of films produced in the first and tenth months
Values are reported as mean± standard deviation. Lowercase letters show significant differences between films in the first month, and capital letters show significant differences between films in the tenth month ($p< 0.05$)

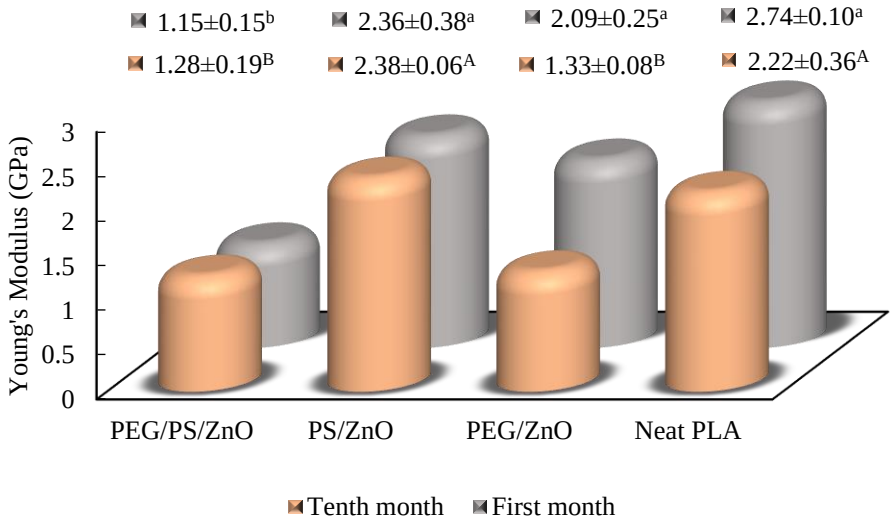

Fig. 4. Young's modulus of films produced in the first and tenth months.
Values are reported as mean ± standard deviation. Lowercase letters show significant differences between films in the first month, and capital letters show significant differences between films in the tenth month ($p < 0.05$).

For the first month, there is no significant difference between PEG/PS/ZnO and PEG/ZnO in terms of tensile strength, while the rest have a significant difference. Plasticizer reduces the tensile strength of films by reducing hydrogen bonds in intermolecular bonds (Maulana, Mubarak, & Pujiastuti, 2021). The highest elongation at break was related to PEG/ZnO (76.82 ± 27.22%), which was not significantly different from PEG/PS/ZnO and PS/ZnO. The toughness value for the neat PLA film is 1.42± 0.09 J cm^{-3}, which has a significant difference only with PEG/ZnO. The Young's modulus in PEG/PS/ZnO is significantly smaller than in the other samples because it contains polyethylene glycol 400 and Polysorbate 80. Adding more plasticizers makes the films more flexible or reduces the Young's modulus (Jantrawut *et al.*, 2017). ZnO nanoparticles can reduce the interaction between PLA chains and increase elasticity (W. Li *et al.*, 2017). The values of tensile strength, Young's modulus, and elongation at break for PLA/1wt% ZnO nanoparticles were reported as 44± 5 MPa, 2.90± 0.2 GPa, and 3.3± 0.7%, respectively (Pantani *et al.*, 2013). In a study, the toughness value for neat PLA was reported as 2.19± 0.36 J cm^{-3} (He *et al.*, 2020).

There was no significant difference between the tensile strength of the neat PLA and PS/ZnO in the tenth month. The lowest tensile strength value was for PEG/PS/ZnO (24.02± 1.95 MPa), which was not significantly different from PEG/ZnO. Regarding the elongation at break, the samples containing polyethylene glycol 400 had a significant difference from the other two types. PS/ZnO has the lowest toughness value (0.52± 0.12 J cm^{-3}), which is not significantly different from neat PLA. The young's modulus of PS/ZnO and neat PLA did not have a significant difference, but they had a significant difference with the films containing Polyethylene glycol 400. In a study, it was reported that the plasticizing effect of glycerol on increasing elongation at break and decreasing Young's modulus in chitosan films is greater than that of Tween 80 (Ziani *et al.*, 2008).

Examination of time's effect on mechanical properties, as presented in Tables 2 and 3, reveals minimal significant variations.

Table 2- Tensile strength and elongation at break of produced films

Films	Tensile strength (MPa)		Elongation at break (%)	
	First month	**Tenth month**	**First month**	**Tenth month**
PEG/PS/ZnO	29.15± 1.24[a]	24.02± 1.95[b]	37.85± 18.71[a]	15.51± 6.92[a]
PS/ZnO	51.83± 7.96[a]	52.43± 7.51[a]	43.64± 29.57[a]	2.20± 0.34[b]
PEG/ZnO	36.24± 3.59[a]	27.94± 3.11[a]	76.82± 27.22[a]	56.65± 36.96[a]
Neat PLA	82.99± 1.90[a]	74.95± 14.24[a]	3.21± 0.15[a]	3.37± 0.28[a]

Letters show significant differences caused by the effect of time in each film ($p < 0.05$)

Table 3- Toughness and Young's modulus of produced films

Films	Toughness (J cm^{-3})		Young's modulus (GPa)	
	First month	**Tenth month**	**First month**	**Tenth month**
PEG/PS/ZnO	7.29± 3.58[a]	2.13± 0.93[a]	1.15± 0.15[a]	1.28± 0.19[a]
PS/ZnO	15.72± 12.55[a]	0.52± 0.12[b]	2.36± 0.38[a]	2.38± 0.06[a]
PEG/ZnO	20.13± 7.89[a]	10.51± 7.04[a]	2.09± 0.25[a]	1.33± 0.08[b]
Neat PLA	1.42± 0.09[a]	1.17± 0.36[a]	2.74± 0.10[a]	2.22± 0.36[a]

Letters show significant differences caused by the effect of time in each film ($p < 0.05$)

Significant differences are observed in tensile strength in PEG/PS/ZnO, elongation at break and toughness in PS/ZnO, and ultimately Young's modulus in PEG/ZnO. Given that no changes have occurred in the mechanical properties of neat PLA film over time, it can be concluded that the observed variations are attributable to the presence of plasticizers and nanoparticles. Based on the results, the effect of plasticizers was more pronounced compared to nanoparticles. Film containing polysorbate 80 as a plasticizer exhibits the most significant time-dependent changes in elongation at break and toughness compared to other film types. Unexpected complications including molecular segregation, substance migration, and surface evaporation represent critical challenges inherently associated with plasticizer incorporation. These intricate phenomena progressively transform diverse film properties throughout temporal progression (Eslami *et al.*, 2023). Over a 90-day research period, researchers studied the mechanical properties of chitosan films. The findings revealed that tensile strength generally increased across all film samples as time progressed, though the increments were not statistically significant in some instances. They consider moisture loss as the primary reason for changes in strength values. The lubricating effect of water on films with polar groups occurs through hydrogen bonding, which leads to structural softening and increased molecular mobility. With water loss, the film transitions to a more brittle state (Leceta *et al.*, 2015). The results of the 14-month study revealed that the Young's modulus of polylactic acid-based films remained unaffected by storage time (Tajari, Sadrnia, & Hosseini, 2024).

Resistant materials like PLA are sufficiently strong but exhibit little elongation at break. The suitable polymer film for packaging should be flexible plastic. Flexible plastic is characterized by elastic behavior initially, followed by plastic behavior when more force is applied. As shown in Fig. 5, materials that exhibit plastic behavior do not demonstrate elastic behavior, resulting in permanent deformations. For ductile materials, both elastic and then plastic behaviors can be observed (Fig. 5). The area under the graph for brittle materials is small, indicating the low energy required for failure or low toughness.

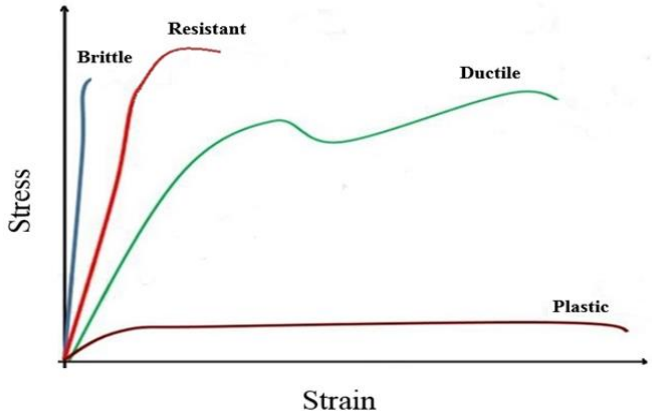

Fig. 5. Stress-strain curve showing different mechanical behavior in polymer films
Curve reconstructed according to(Shahid & Gukhool, 2020)

Figures 6-13 show the stress-strain curves related to the tensile test for the samples in the first and tenth months. PEG/PS/ZnO (Figs. 6 and 7) and PEG/ZnO (Figs. 10 and 11) exhibited behavior between that of resistant and ductile materials in both the first and tenth months. PS/ZnO behaved similarly to the others in the first month, as shown in Fig. 8, while it displayed behavior between brittle and resistant materials in the tenth month (Fig. 9). Neat PLA (Figs. 12 and 13), which has no plasticizer, remained consistently resistant. Plasticizers can increase the amorphous areas and reduce the brittleness of films by increasing the free volume (Liu & Zhang, 2011). According to the results, it can be concluded that the effect of Polysorbate 80 as a plasticizer is not very stable.

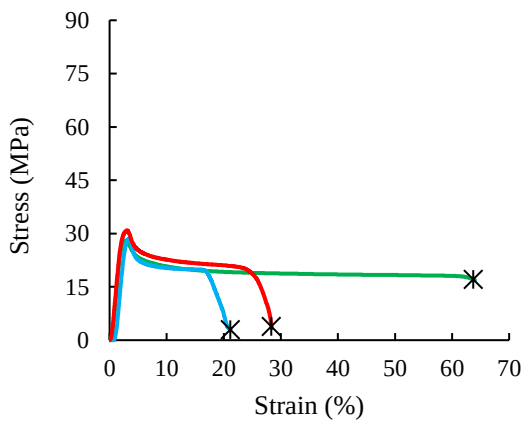

Fig. 6. The stress-strain curve related to the tensile test of PEG/PS/ZnO in the first month; different colors represent different repetitions

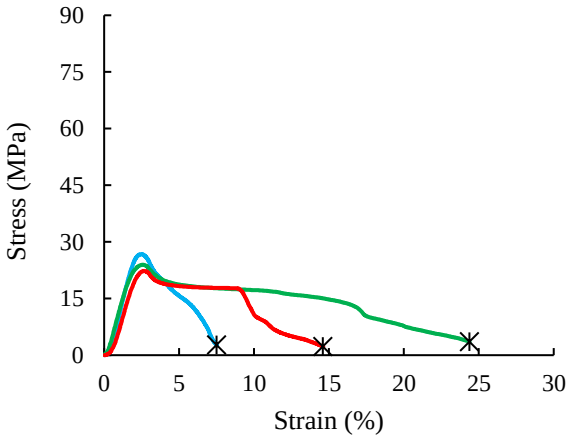

Fig. 7. The stress-strain curve related to the tensile test of PEG/PS/ZnO in the tenth month; different colors represent different repetitions

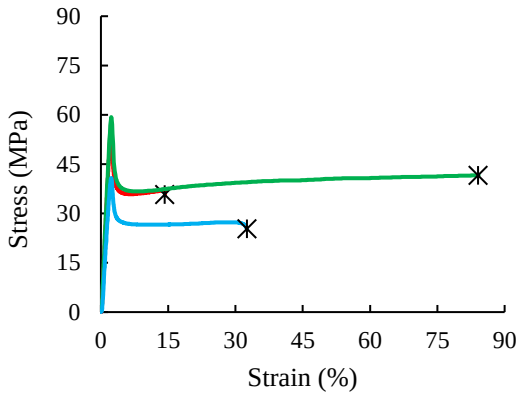

Fig. 8. The stress-strain curve related to the tensile test of PS/ZnO in the first month; different colors represent different repetitions

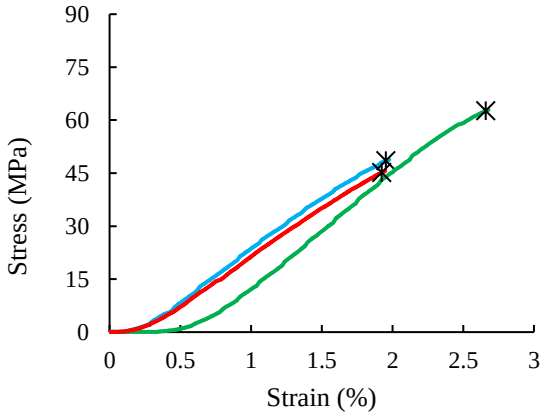

Fig. 9. The stress-strain curve related to the tensile test of PS/ZnO in the tenth month; different colors represent different repetitions

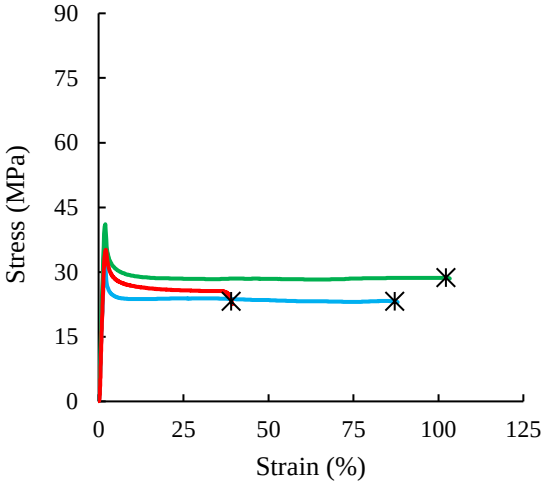

Fig. 10. The stress-strain curve related to the tensile test of PEG/ZnO in the first month; different colors represent different repetitions

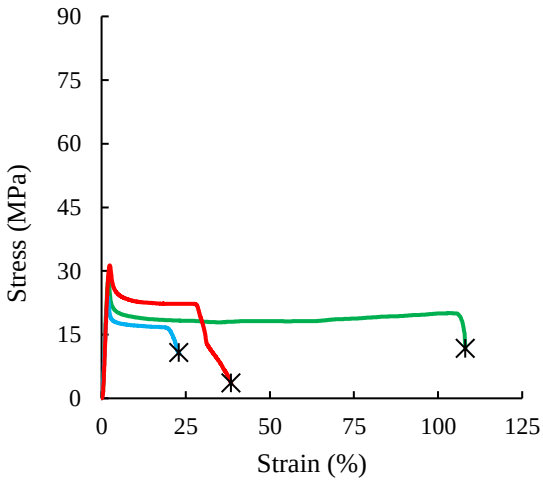

Fig. 11. The force-displacement curve related to the tensile test of PEG/ZnO in the tenth month; different colors represent different repetitions

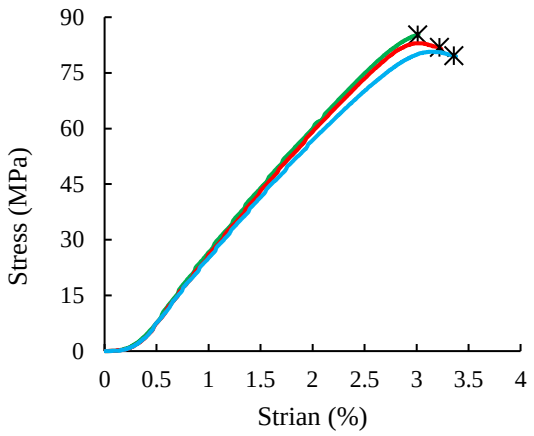

Fig. 12. The stress-strain curve related to the tensile test of neat PLA in the first month; different colors represent different repetitions

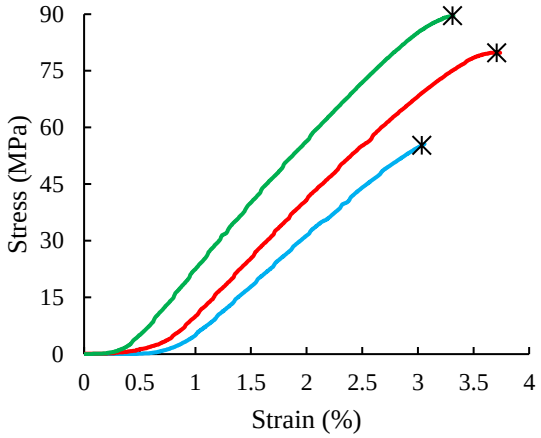

Fig. 13. The stress-strain curve related to the tensile test of neat PLA in the tenth month; different colors represent different repetitions

A significant decrease in a specific part of the cross-sectional area of the sample, often associated with yielding or some form of plastic deformation, is called necking. An increase in the force applied to the sample leads to a decrease in the cross-sectional area, and as the cross-sectional area decreases, the stress increases. Necking occurs when the stress increases beyond the material's capacity to bear the load due to work hardening. After the yield point, necking begins and continues until the point of fracture. Brittle specimens break at the yield point, whereas with increasing film ductility, the fracture point occurs at higher strain (Meyers & Chawla, 2008; Song, 2022). In PEG/PS/ZnO and PS/ZnO (first month) and PEG/ZnO films, necking occurs; however, in PS/ZnO (tenth month) and neat PLA, which are almost brittle, necking does not occur.

Fracture points are marked in Figs. 6-13. At the fracture point, materials physically separate. The maximum strain occurs at this point, but the stress may be less than the ultimate strength. Brittle materials break linearly at a critical load, while other materials resist crack propagation and thus break softly (Maeda *et al.*, 2021).

Conclusion

The aim of this research was to investigate the mechanical properties of polylactic acid films over a ten-month period to examine their physical aging. The investigation of mechanical properties in the first and tenth months showed that nanoparticles and plasticizers could affect mechanical behavior. In the first month, the highest elongation at break value was related to the film containing nanoparticles and polyethylene glycol, which was equal to 76.82%, while the lowest elongation at break, at 3.21%, was associated with the neat polylactic acid film. The stress-strain curves indicated that the presence of a plasticizer could reduce the brittleness of the films; however, this effect disappeared over time for the film that contained only Polysorbate 80 as a plasticizer. Analysis of variance results revealed that the effect of time on most mechanical properties was not statistically significant, with the most pronounced time-related changes observed in the film containing Polysorbate 80 plasticizer. Based on the results obtained over ten months, it can be seen that the tensile strength and Young's modulus of the produced films have acceptable values for use in the packaging industry, but the issue of brittleness in the films still remains.

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Authors Contribution

N. Tajari: Conceptualization, Methodology, Data acquisition, Data pre and post processing, Statistical analysis, Software services, Validation, Visualization, Review and editing services

H. Sadrnia: Supervision, Validation

F. Hosseini: Validation, Review and editing services

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ارزیابی خواص مکانیکی فیلم‌های پلی‌لاکتیک اسیدی، اندازه‌گیری‌شده در ماه اول و دهم

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چکیده

پلی‌لاکتیک اسید یک پلیمر گرمانرم، زیست‌تخریب‌پذیر و زیست‌فعال است که از منابع تجدیدپذیر مانند چغندر و سیب‌زمینی به‌دست می‌آید. پلی‌لاکتیک اسید پلیمری با ماهیت نسبتاً شکننده است که این ویژگی می‌تواند کاربردهای آن را در صنعت بسته‌بندی محدود سازد. خواص مکانیکی این پلیمر می‌تواند با افزودن نانوذرات و نرم‌کننده‌ها بهبود یابد. در این تحقیق، نانوذرات اکسید روی به میزان یک درصد وزنی پلیمر، پلی‌اتیلن گلیکول ۴۰۰ بیست درصد وزنی پلیمر و پلی‌سوربات ۸۰، ۰/۲۵ درصد وزنی محلول برای بهبود خواص مکانیکی فیلم‌های پلی‌لاکتیک اسید استفاده شدند. اثر این مواد بر فیلم‌ها در دو بازه زمانی مختلف، ماه اول و ماه دهم، با هدف بررسی پیری فیزیکی به‌عنوان مقدمه برای تخریب پلیمر، مورد ارزیابی قرار گرفت. تحلیل‌های آماری بر روی خواص مکانیکی اندازه‌گیری‌شده در این دوره‌ها انجام شد تا تفاوت‌های معنادار میان فیلم‌های تولیدشده شناسایی گردد. نتایج نشان داد که بالاترین استحکام کششی (۸۲/۹۹±۱/۹۰ مگاپاسکال، فیلم پلی‌لاکتیک اسید خالص)، ازدیاد طول در نقطه شکست (۷۶/۸۲±۲۷/۲۲ درصد، فیلم پلی‌لاکتیک اسید/پلی‌اتیلن گلیکول/نانوذرات اکسید روی)، چقرمگی (۲۰/۱۳±۷/۸۹ ژول بر سانتی‌مترمکعب، فیلم پلی‌لاکتیک اسید/پلی‌اتیلن گلیکول/نانوذرات اکسید روی)، مدول یانگ (۲/۷۴±۰/۱۰ گیگاپاسکال، فیلم پلی‌لاکتیک اسید خالص) در ماه اول مشاهده شد. نتایج تحلیل واریانس در مورد تأثیر زمان بر هر فیلم نشان داد که در اکثر موارد، خواص مکانیکی پس از ده ماه تغییر معناداری نداشتند. منحنی‌های تنش–کرنش، نشان داد که فیلم پلی‌لاکتیک اسید خالص یک ماده مقاوم است. فیلم دارای نانوذرات و پلی‌سوربات در ماه دهم رفتار شکننده‌ای از خود نشان داد. سایر نمونه‌ها در هر دو ماه اول و دهم رفتاری بین مواد مقاوم و انعطاف‌پذیر نشان دادند.

واژه‌های کلیدی: پلی‌لاکتیک اسید، پیری فیزیکی، خواص مکانیکی، نانوذرات اکسید روی

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