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Development and Characterızatıon of Polylactıc Acıd (Pla)

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Abstract

Today, the usage areas of polymer materials are quite wide. It is not possible to recycle petroleum-based polymers. Synthetic polymers have wide usage areas; This makes synthetic polymers the most important cause of environmental pollution. In particular, disposable products are produced from synthetic polymer materials and then recycled in nature for many years; It has pushed us to seek a solution to this problem. At the same time, decreasing oil resources pose a raw material threat for the production of oil-based products. Therefore, it is aimed to minimize the use of synthetic polymers used in many industries such as automotive, household appliances, food and pharmaceutical packaging, and to contribute to the increase of biopolymer studies. In this study, the production and mechanical characterization of Polylactic Acid, a natural polymer, was performed. PLA, which is a biodegradable polymer as well as a natural polymer, is thought to be an alternative to synthetic polymers in many sectors. Pure natural PLA samples were molded with an injection device and mechanical characterization was performed. The results of tensile and 3-point bending tests were interpreted. As a result of the mechanical tests of PLA, a biocomposite was produced using chestnut shells to improve the mechanical properties and the mechanical properties of this biocomposite were examined. It was concluded that 25% chestnut shell reinforcement improved the mechanical properties of PLA.

Key Words: Synthetic Polymer, Natural Polymer, PLA, Biodegradable, Biocomposite

1. Introduction

Most of the commonly used plastics such synthetic polymers are manufactured from petroleum and its allied components. Global environmental concerns and regulations have triggered researchers to develop biodegradable and biocompatible materials which could replace conventional petroleum-based products [1]. Bio composites are considered the best alternative materials to address these issues and ensure an eco-friendly and sustainable environment [2]. The first partially biodegradable composites were prepared in the 1890s using thermoplastic resin and cellulose fibers [3]. The advantageous characteristics of biocomposites (i.e., biodegradability, no toxicity, renewability, low density, good mechanical properties, economical, etc.) have drawn the attention of researchers and scientists as potential alternatives for petroleum-based products [4].

There are numerous natural and synthetic biodegradable polymers available such as poly-lactic acid (PLA), poly-lactic-co-glycolic acid (PLGA), polyethylene glycol (PEG), poly-hydroxy butyrate (PHB), polycaprolactone (PCL), etc. That can be used as matrix materials for the development of bio composites, among these, PLA is the most suitable and promising candidate to replace petroleum-based plastics, because it is renewable. It should not be forgotten that polymers such as PET and PVC, which are widely used and frequently encountered in many sectors today, are petroleum-based and it is known that it is not possible to recycle these synthetic polymers. Some of the synthetic polymers are burned in order to prevent environmental pollution, and they cause global warming due to the CO2 gas released as a result of burning. Environmental pollution, climate change and decreasing oil resources are the main reasons that lead to the search for alternatives to synthetic polymers. The most used and promising biopolymer in biopolymer studies, which have increased in the last 20 years, is PLA. The most frequently used biopolymer in academic studies is PLA. PLA studies are increasing day by day because it is easy to transport, easy to shape, economical and as a result of the studies carried out so far, it is thought to be a rival to synthetic polymers in many sectors.

In a study, biodegradable PLA, different forms of PLA, PGA, PCL and PHB were used, their mechanical properties were examined and the ideal biodegradable polymer matrix for a flax fiber reinforced composite was investigated. The structural formulas of the 4 basic biopolymers are as follows. PGA is difficult to use as a matrix because its high melting point and density restrict production conditions. The reason why PCL is not preferred is that its melting temperature is not suitable for environments where it may be exposed to high temperatures [5]. A. Rubio-López et al. In his study on fully biodegradable PLA and linen production, impact strength and residue testing, the thickness of PLA/linen laminates was determined as a parameter and their mechanical properties were investigated. Depending on the laminate thickness, post-impact change has been observed, ranging from barely visible damage to hole formation. It was found that the compressive strength of the sample with a diameter of 12.7 mm was higher compared to the sample with a diameter of 20 mm. By increasing the diameter of the PLA/linen sample, the area to be damaged and the energy to be absorbed increased, decreasing the compressive strength. The mechanical properties of the fully degradable PLA/Flax composite were compared with conventional composites; Some properties of PLA/Flax composite have been found to be more advantageous. Considering the impact strength results, it was concluded that PLA/Flax biodegradable composites can be used for industrial applications, but more studies are needed [6].

The research focused on strengthening of PLA bio composites with chestnut nutshell. By means of this study; it better understood that PLA and its bio composites in t

2. Material and Method

Filameon brand PLA, produced from corn starch, was used in the study. PLA in granule form Riiz Makine Arge Proje Dan. And Cons. Singing. And Trade. Ltd. Ltd. It was supplied from the company named (Fig. 1). The preferred PLA was chosen to be opaque in color and suitable for production by injection. Chestnuts grown in Bartın province, dried in September 2022 and kept in the freezer were used for the chestnut shell. Images of the products used are given below.

Figure 1: Filameon brand PLA in granule form

The study was carried out in 4 steps in total. Injection molding of pure PLA, mechanical testing, production of PLA + chestnut shell biocomposite, mechanical testing of the biocomposite. The table summarizing the stages of the study is given in Table 1.

Table 1 Main stages of the study

1. STAGE	Molding of Pure PLA with Injection Molding Device Tensile and 3 Point Bending Tests of the Produced Samples
2. STAGE	Grinding PLA + Chestnut NutShells Dehumidifying Products in the Oven Preparation of the Mixture by Weighing on a Precision Scale Profile Production with Etruder Injection Molding of Samples Tensile and 3 Point Bending Tests of Biocomposite Samples

Chestnut shell reinforced PLA biocomposite samples were produced to improve the mechanical values of pure PLA samples. Granulated PLA and chestnut shells were ground in a mechanical mixer and brought to the same dimensions. The mixture was prepared as 75% PLA and 25% chestnut shell, and the mixture was weighed on a DENSI brand precision scale.

2.1. Production of Samples

Firstly, granular PLA produced from corn starch with a melting temperature of 175 oC was used in the study.

2.1.1. Injection Device

It is a device in which granular polymer materials are melted and molded with pressure and temperature. Granules are added from the feeding zone and become molten. The polymer material in molten liquid form is transferred to the mold part and shaped. The shape to be given can be varied by changing the mold. The most commonly used plastic processing method is the injection molding method. In our study, granular PLA and chestnut shell reinforced PLA composite were molded with different injection devices. BOY brand 22A model laboratory type injection device was used for molding pure PLA (Figure 2). Molding of chestnut shell reinforced PLA composites was done with TORUN brand injection device (Figure 3).

Figure 2: BOY Brand injection device

Figure 3: Injection machine

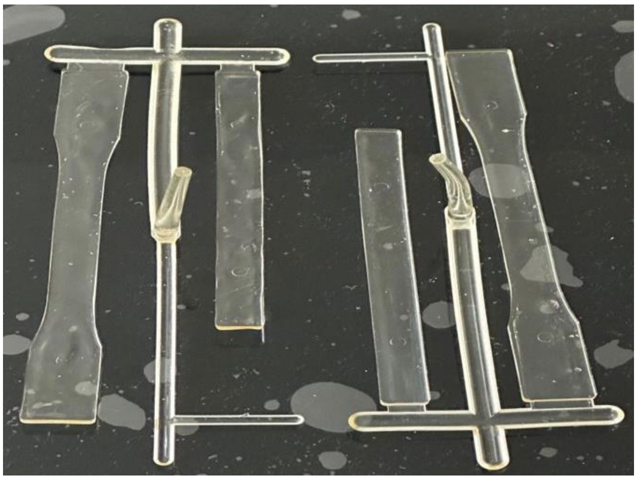

Figure 4: Injection molded test samples

2.1.2. Oven

Oven is a laboratory oven used to dehumidify polymer materials before production. Ovens can be set at different temperatures according to the material to be dehumidified. It is used for pre-production preparation processes such as dehumidifying, heating and cooking the material. Since the chestnut shells were dried without being placed in the freezer in September 2022, the shells were not dried again before the grinder. Before extrusion, the mixture was kept in an INSTRON brand oven at 105oC for 24 hours and its moisture was removed.

2.1.3. Extrusion

Extrusion is a plastic shaping method in which rod, pipe, strip and profile structures are produced. Polymer materials are melted with pressure and temperature and advanced to the shaping section to produce profile shaped materials. It is the most frequently used production method, especially for pipe production. The variety of products produced by the extrusion method, including complex products, is quite large. In our study, the PLA + chestnut hell mixture, which was kept in an oven and dehumidified, was shaped as a profile by extrusion and cooled by immersion in water. The produced profiles were cut and turned into granules

2.2. Mechanical Tests

Tensile and 3-point bending tests were performed on the samples molded with an injection device. As a result of the tests, bending strength (MPa), bending modulus (MPa), tensile strength (MPa) and tensile modulus (MPa) information were obtained.

2.2.1. Tensile Device

Tensile testing is applied to measure the behavior and strength of many products such as metal, polymer, rubber, fabric and ceramics under the influence of tensile force. By applying force to the sample in the axial direction, the amount of elongation and the force applied at the breaking point are taken into account. Thus, the maximum force that the material can withstand is reached (Material Bilimi.Net, 2023). There are standards set for different materials to be tensile tested. ASTM D3039, ASTM D638, ISO 527, ASTM D412, ASTM C297, ASTM E8 are some of these standards (İnnoma, 2023). In our study, the samples were prepared and tested according to the ISO 527 standard (Fig. 5). SHIMADZU brand tensile device was used for molded pure PLA samples, and ZWICK brand tensile device was used for chestnut shell reinforced PLA biocomposite samples.

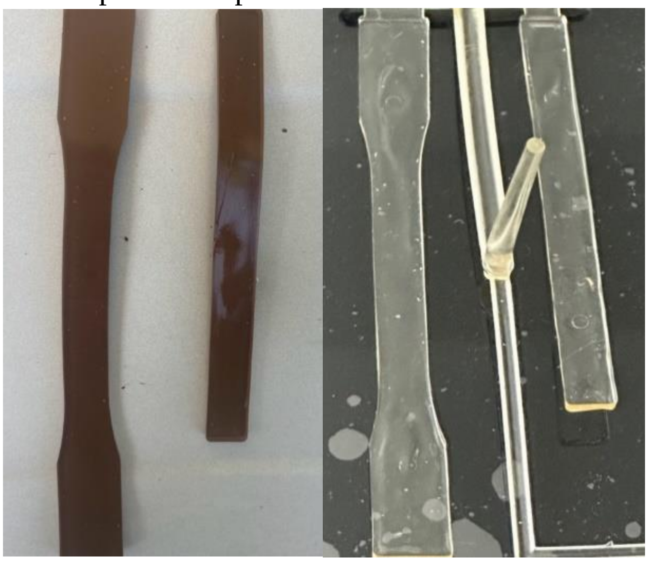

Figure 5: Test samples

2.3. Three Point Bending Test

3-point bending test is one of the tests performed to examine the mechanical values of the material. With the 3-point bending test, the bending strength and bending modulus of the material are reached [7]. There are ASTM D790, ASTM D6272, ASTM D7264, ISO 178, ISO 14125, ASTM C1161, ASTM C393 standards for 3-point bending test. In our study, the samples were produced and tested according to ISO 178 standard. The same devices used for the tensile test were used for the 3-point bending test. SHIMADZU brand tensile device was used for pure PLA, and ZWICK brand tensile device was used for chestnut shell reinforced PLA biocomposite samples. The sample to be tested is placed on the table and force is applied from its midpoint. Bending strength is calculated by taking into account the shape changes as a result of the applied force. Since force is applied to the sample from different directions during the test, the sample is exposed to both compressive and tensile stress. The schematic representation of the 3-point bending test is as follows (Fig. 6).

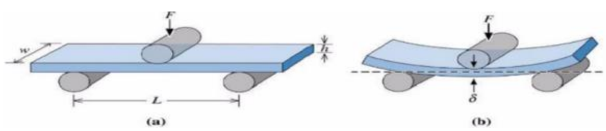

Figure 6: Schematic Representation of 3 Point Bending Test [7]

The samples are seen exposed to the 3-point Bending Tests in Fig.7.

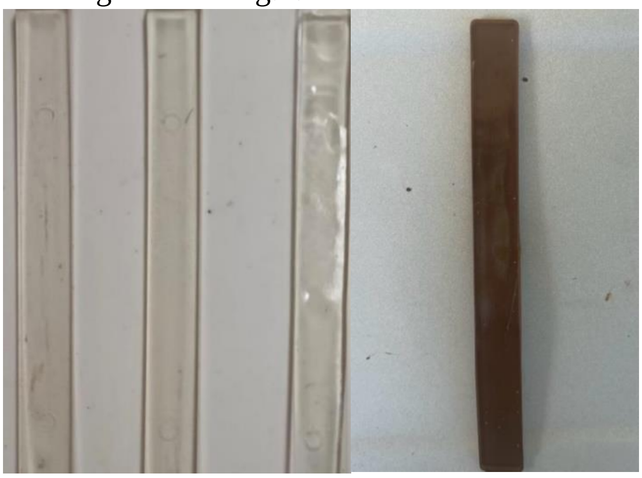

Figure 7: Three Point Bending Test Specimens

3. Discussion

3.1. Tensile Test Results

The samples are seen exposed to thr tensile tests in Fig. 8. Tensile test values of pure PLA and chestnut shell reinforced PLA biocomposite are given in the table below (Table 2 and Table 3). The six samples were produced for each group and the result was accepted as the average value. As a result of the tensile test, tensile strength and tensile modulus/elasticity modulus were reached.

Table 2 Tensile Test Results of Pure PLA Samples

Sample name	Sample thickness (mm)	Samples Width (mm)	Stress Strength (MPa)	Strain (mm)	Elasticity Modül (GPa)
PLA1	3.27	12.69	57.50	15.54	3.70
PLA2	3.2	12.67	57.40	25.98	2.21
PLA3	3.21	12.59	61.00	15.32	3.98
PLA4	3.22	12.64	59.90	24.95	2.40
PLA5	3.2	12.59	54.80	15.65	3.50
PLA6	3.22	12.64	60.60	18.36	3.30
Average Value			58.53	19.3	3.18

Figure 8: Images of Biocomposite Samples Before and After Tensile Test

Table 3. Tensile test results of biocomposite samples

Sample	Sample thickness (mm)	Samples Width (mm)	Maximum Force (Mpa)	Strain (mm)	Elasticity Modül (Gpa)
Pure PLA 1	3.2	12.55	186.42	14.86	12.54
Pure PLA 2	3.3	12.62	192.5	15.26	12.61
Pure PLA 3	3.0	12.59	182.2	15.20	11.98
Pure PLA 4	3.18	12.69	198.70	15.68	12.67
Pure PLA 5	3.21	12.65	179.5	14.94	12.01
Pure PLA 6	3.23	12.67	187.93	14.7	12.78
Average Value			187.87	15.1	12.43

4.2. 3. Point Bending Test Findings

Table 4.3 and Table 4.4 give the 3-point bending test results of pure PLA and biocomposite samples. 6 samples were molded for each group and the result was accepted as the average value. The maximum force applied to the sample, bending strength and bending modulus values are given in the tables. The force applied before the sample changes shape is called the maximum force and can also be called a measure of the toughness of the material. The amount of resistance of the sample to force before breaking also gives information about its toughness and brittleness. If the material's resistance to the applied force before rupture is high, we can say that the material is ductile; if it is low, we can say that the material is brittle.

Table 4. Three-point bending test results of pure PLA samples

Sample	Sample thickness (mm)	Samples Width (mm)	Maximum Force (Mpa)	Flexural Strength (Mpa)	Bending Modul (Mpa)
Pure PLA 1	3.19	12.33	163.50	246.28	462.03
Pure PLA 2	3.20	12.36	163.53	244.20	514.77
Pure PLA 3	3.17	12.23	152.19	234.05	454.84
Pure PLA 4	3.21	12.39	157.44	233.08	499.85
Pure PLA 5	3.17	12.33	154.19	235.20	521.28
Pure PLA 6	3.24	12.36	151.22	220.27	482.97
Average Value			157.01	235.51	489.3

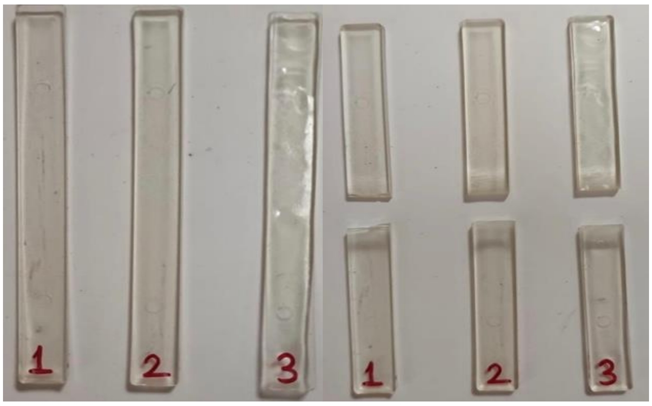

Figure 9: Images of Pure PLA Samples Before and after three Point Bending Test

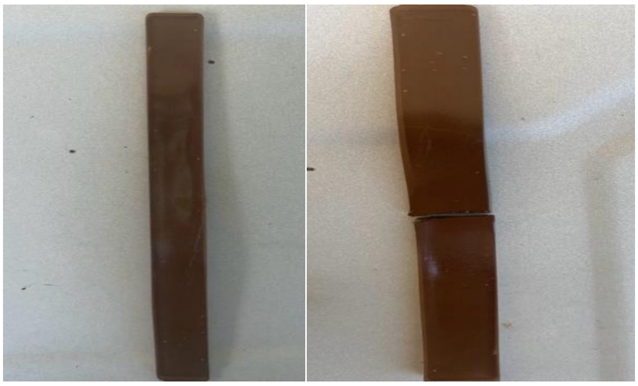

Figure 10: Images of Biocomposite Samples Before and After 3 Point Bending Test

Table 5 Three Point Bending Test Results of Biocomposite Samples

Sample	Sample thickness (mm)	Samples Width (mm)	Maximum Force (Mpa)	Flexural Strength (Mpa)	Bending Modul (Mpa)
%25 KK + %75 PLA 1	3.12	12.25	320.19	803.47	1302,25
%25 KK + %75PLA 2	3.23	12.33	332.54	828.43	1286.38
%25 KK + %75 PLA 3	3.15	12.27	328.17	825.36	1329.75
%25 KK + %75 PLA 4	3.18	12.36	338.42	827.23	1244.56
%25 KK + %75PLA 5	3.14	12.37	345.8	834.20	1402.07
%25 KK + %75PLA 6	3.16	12.29	328.16	810.52	1272.24
Average Value			342.29	821.53	1306.20

4. Results

Within the scope of this study, the mechanical behaviors of pure PLA and chestnut shell reinforced PLA biocomposites were examined. Tensile and 3-point bending tests were carried out to reach mechanical values and as a result; Tensile strength, tensile modulus (modulus of elasticity), bending strength and bending modulus data were obtained.

- ✓ First, pure PLA was molded by injection and the results obtained concluded that pure PLA showed low mechanical properties and could not compete with synthetic polymers in industrial sectors. In order to improve the mechanical properties of PLA, a natural additive that would not lose its biodegradability was researched and it was decided to use chestnut shell.
- ✓ Tensile and 3-point bending tests were also performed on the chestnut shell reinforced PLA biocomposite. According to the results obtained, chestnut shell increased the mechanical strength of PLA.
- ✓ The force applied to the biocomposite samples was increased, but a decrease in the amount of deformation of the sample was observed. 25% chestnut shell reinforcement increased the tensile strength of PLA by approximately 3 times and the modulus of elasticity by approximately 4 times.
- ✓ According to the data obtained as a result of the three-point bending test, chestnut reinforcement increased the bending strength of PLA by approximately 3.5 times and the flexural modulus by approximately 2.5 times.
- ✓ During the test, the shape change of the material was observed and it was concluded that PLA and chestnut reinforced PLA composite were ductile due to their high deformation amounts.

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Author Contributions

This publication was produced from Busra Akduman's master thesis under the supervision of Hulya Demiroren.

Statements of Conflicts of Interest

The author has no conflict of interest regarding this article.

Statement of Research and Publication Ethics

The author declares that this study complies with Research and Publication Ethics.

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