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Investigation of Usage Possibilities of Mushroom Production Waste Fibers in Polycaprolactone (PCL) Based Biocomposite Material Production

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Abstract

Aim of study: The possibilities of using oak and beech wood wastes used in the cultivation of Lentinus edodes fungus in the production of pcl biocomposite film material were investigated.

Material and methods: Lentinus edodes mushroom was subjected to 2 harvest periods in both lignocellulosic waste types and equal degradation times were obtained. Chemical contents of degraded fibers were determined. Then, the effect of pcl biocomposite film produced using 15% and 30% raw and degraded fibers on mechanical properties was found. The water uptake and swelling values of the composite material were determined.

Main results: As a result of lentinus edodes fungus degradation of Oak and Beech wood, it was determined that holocellulose and lignin contents decreased, while cellulose and alpha cellulose contents increased. It was determined that the density of the film produced by using oak and beech waste increased. The highest increase was obtained from the films produced by adding 30% fiber. It was determined that the tensile strength and elongation at break values of pcl composite film decreased and the modulus of elasticity increased. In general, it was found that the water uptake of the film increased compared to the control sample.

Highlights: To investigate the suitability of mushroom waste for polymer material production.

Keywords: Oak and Beech Wood, PCL Biocomposite, Chemical and Mechanical Properties.

Mantar Üretimi Atık Liflerinin Polikaprolakton (Pcl) Esaslı Biyokompozit Malzeme Üretiminde Kullanım Olanaklarının Araştırılması

Öz

Çalışmanın amacı: Lentinus edodes mantarının yetiştirilmesinde kullanılan meşe ve kayın odunu atıklarının pcl biyokompozit film malzemenin üretiminde kullanım olanakları araştırılmıştır.

Materyal ve yöntem: Lentinus edodes mantarı her iki lignoselülozik atık türünde 2 hasat periyoduna tabi tutulmuş ve eşit bozunma süreleri elde edilmiştir. Bozunan liflerin kimyasal içerikleri belirlenmiştir. Ardından %15 ve %30 ham ve bozunmuş lifler kullanılarak üretilen pcl biyokompozit filmin mekanik özelliklere etkisi bulunmuştur. Kompozit malzemenin su alma ve kalınlığına şişme değerleri bulunmuştur.

Temel sonuçlar: Meşe ve Kayın odununun Lentinus edodes mantarı bozunumu sonucunda, holoselüloz ve lignin içeriğinin azaldığı, selüloz ve alfa selüloz içeriğinin arttığı tespit edilmiştir. Meşe ve kayın atığı kullanılarak üretilen filmin yoğunluğunun arttığı belirlenmiştir. En yüksek artış %30 lif ilave edilerek üretilen filmlerden elde edilmiştir. Pcl kompozit filmin çekme mukavemeti ve kopma uzama değerlerinin azaldığı ve elastisite modülü değerinin arttığı tespit edilmiştir. Genel olarak, kontrol örneğine göre filmin su alma miktarının arttığı bulunmuştur.

Araştırma vurguları: Mantar atıklarının, polimer malzeme üretimine uygunluğunu araştırmak.

Anahtar Kelimeler: Meşe ve Kayın Odunu, PCL Biyokompozit, Kimyasal ve Mekanik Özellikler.

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Introduction

Traditional polymer composite material production in the world serves a wide range of uses that the polymers are non recyclable and not degradable in nature and and as a result cause environmental problems (Dhakal et al., 2013). In particular, the polymers used in the packaging of the materials are large and most of them are produced from petroleum-based polymers (Zinoviadou et al., 2016; Reddy et al., 2013). In recent years, environmentally friendly bio-based polymers that are degradable in nature replace traditional polymers. Bio-polymers can usually be degraded by living organisms and these are: PLAs, polyglycolic acid, poly-β-hydroxyalkanoates, which are thermoplastics, polycaprolactone, which is a thermoplastic (Mishra et al., 2018). Polycaprolactone (PCL) is a biopolymer that has been studied a lot and especially food packaging, biodegradable bottles, compostable bags, various devices for medicinal (Garcia et al., 2014).

The use of polymers by combining them with natural fibers is also called biocomposite and this is called natural fiber-polymer composites (NFPCs) (Shinoj et al., 2011). NFPC material renewable and usable materials that is embedded in polymer matrix and a lot of research has been done on NFPCs (Mengeloğlu and Karakus, 2008; Cavdar et al., 2019; Vaisanen et al., 2016). The addition of natural fibers into the polymers provides many advantages such as the reduction of the amount of polymer used in the composite material, as a result of which the material is cheaper, degradable and produced at a lower density (Vaisanen et al., 2016). Many natural fiber usage such as sisal, hemp, jüte, palm etc. and different wood fiber have been investigated in polymer composites (Dhakal et al., 2007; Bledzki and Gassan, 1998; Bourmaud et al., 2017; Wibowo et al., 2004; Kaymakci and Ayrilmis, 2014).

In addition to lignocellulosic fibers, there are studies on the use of bacterial fibers, fungal mycelium fibers, insect larvae and fungi from polymer composite material obtained from many living organisms (Cecchini, 2017; Ayrilmis et al., 2015). Fungi have advantage because ıt has both missel fiber production and degraded fibers. Fungi are classified according to their mechanism of action on cellulosic cell structure and Lentinus edodes (shitake) mushroom is white rot fungi. Enzymes of this fungus primarily degrade the lignin compound found in the lignocellulosic cell structure (Gaitan-Hernandez et al., 2006; Mester et al., 2004).

The aim of this study was to investigate the effect of different compost fibers and PCL proportions on the chosen properties of composites. Selective media feature and mushroom cultivation time on compost fiber were determined and chemical analyzes were made on the compost fibers. Physical properties such as density, water absorption and thickness swelling and mechanical properties such as tensile strength, modulus of elasticity and elongation at break were determined.

Materials and Methods

Materials

In this research, the thermoplastic matrix used was Poly (ε-caprolactone) (CAPA, 6500) and it was supplied as a solid, sharp melting point of 58-60C. PCL has 50.000 molecular weight linear thermoplastic polycaprolactone diol polymer and obtained from Perstorp UK. Beech and oak sawdusts were obtained from timber factorys in the Düzce region in Türkiye and granulated into flour form using Willey mill. The obtained flours were screened to 40, 60, 80, 100 and 200 mesh-size and oven dryied (105Cº) and flours were used in this research are between 74-400 micron and oven dried.

Methods

Lentinus edodes mushroom micelles was inoculated on antibiotic malt-agar and then waited in incubator at 25Cº degree for three weeks. Oak and beech sawdust were wetted for three weeks until it reaches to suitable moisture level (75 ± 5). The samples were put in heat resistant polyproplene bags (1 kilogram) and then sterilization process was applied to the filled bags. Process was applied at 90Cº temperature and 90 minutes in autoclave. After the completed process, the bags were cooled and then Lentinus edodes micelle was innoculated on oak and beech samples and inoculated samples are placed in the room (room conditions: temperature 25 Cº, relative humidity 90%) for mycelial development. After micelle development is completed, micelled samples were removed from the bags and put in the room at 18±2Cº degrees, 90% relative humudity to get the product. After two harvest periods mushrooms are obtained on oak and beech sawdusts. Completed the harvesting, mushroom wastes were refined on specimens, washed and samples were dried.

The chemical combinations (holocellulose, hemicellulose, cellulose, ɑ-cellulose and lignin) of lignocellulosic materials to be used in PCL composite production, before and after degredation, were analyzed. The amount of holocellulose of the ingredients was determined by the chlorite method (Wise, 1962), the cellulose content by the analysis method of the Kuscher and Hoffer (1969), hemicellulose were calculated from holocellulose-cellulose, the ɑ-cellulose content by the TAPPI T 203 (om-88) and the lignin content by the TAPPİ T 222 (os-71) standards.

Before the polimer composites production, beech sawdusts, degrade beech sawdust, oak sawdusts and degrade oak sawdusts were dryied until 1% humidity. According to combination, lignocellulosic wastes were mixed with PCL to be homogeneous in the mixer (Table 1). And then, mixed materials fed into the hopper of a single screw extruder. The screw extruder speed is 40 rpm and temperature of extruder is 100˚C. The extruded materials were cooled in a water bath and pelletized. These pellets were dried until the oven dried weight prior to film manufacturing. To produce pcl films, granulated pellets were placed 0.5 mm thickness, 160 mm width, 160 length molds in certain quantities and pressing them at 150 bar pressure and 120C˚ temperature for 10 minutes. The mechanical properties of manufactured PCL composites films were conducted in according to ASTM standards. Tensile stregth, modulus of elasticity and elongation at break tests were conducted in according to ASTM 638 standards, using Zwick 10 KN test machine. The results of tests were evaluated by analysis of variance (ANOVA) and Duncan tests to populate homogenity groups that showed significant differences at the 95% confidence level.

Table 1. Combination parameters of biocomposites

Groups	Wood Wastes	Ratio (%)	Polimer	Ratio (%)
Control			p-caprolactone	100
B1	Beech sawdust	15	p-caprolactone	85
B2	Degraded Beech sawdust	15	p-caprolactone	85
B3	Beech sawdust	30	p-caprolactone	70
B4	Degraded Beech sawdust	30	p-caprolactone	70
O1	Oak sawdust	15	p-caprolactone	85
O2	Degraded Oak sawdust	15	p-caprolactone	85
O3	Oak sawdust	30	p-caprolactone	70
O4	Degraded Oak sawdust	30	p-caprolactone	70

Results and Discussion

It can be seen from Table 2 that Ratio, moisture, pH, degradation times of compost wastes. Before the lentinus edodes mushroom was inoculated, 100% oak sawdust and beech sawdust were soaked to approximately 75% humidity and the pH of the substrates was determined as 6.54 in oak and 6.34 in beech. After inoculating the mushroom mycelium in both substrates, the spawn run times were almost the same. Two harvests were obtained in both substrates and total degradation times were determined as 80 days. Moisture content of the substrates is a very important factor in fungal mycelium development (Sözbir et al., 2015). In a study, it was reported that the cultivation of the lentinus edodes mushroom showed easier growth in acidic environments (Özçelik and Pekşen, 2007). Many studies have been carried out on the cultivation of this mushroom, and different results have been obtained in the spawn run time and harvest times depending on the variety of the substrat used (Morais et al., 2000; Ozcelik and Peksen, 2007; Philoppousis et al., 2007; Ashrafuzzaman et al., 2009).

Table 2. Ratio, moisture, pH, degradation times of compost wastes

Compost Wastes	Ratio	Moisture	pH	Spawn run time (days)	I. Harvest time (days)	II. Harvest time (days)	Total time (days)
Oak Sawdust	100%	75 ± 5	6.54 ± 1	41	20	19	80
Beech Sawdust	100%	75 ± 5	6.34 ± 1	40	22	18	80

Table 3 shows the chemical composition (Holocellulose, Hemicellulose, Cellulose, α-Cellulose, Lignin) of oak sawdust, decomposed-oak sawdust, beech sawdust and decomposed-beech sawdust. The holocellulose amounts of oak sawdust decomposed-oak sawdust, beech sawdust, decomposed-beech sawdust among lignocellulosic contents were determined as 74, 72, 74, 72% respectively. The highest hemi-cellulose ratio was found from beech sawdust (27%). Hemi-cellulose amount of decomposed sawdust decreased, which is due to fungal degredation. It has been determined that the cellulose and alpha-cellulose content increases and lignin content decreases due to degradation. It has been determined that the chemical content of non-decomposed beech wood and oak wood is proportional to other studies (Bodîrlău et al., 2007, 2008). In a study, the lentinus edodes mushroom was inoculated onto oak wood of different types and its effects on its chemical composition were investigated at certain time intervals. They found different increases and decreases in the chemical composition of both types depending on the degradation time. Cellulose is responsible for the resistance properties of the fiber (Ayrılmış et al., 2015), and according to the results of the study, the resistance values of the biocomposite were higher because the cellulose value of the fiber in the degre was higher than the raw fiber.

Table 3. Chemical ingredients of compost wastes

Content	Holo-cellulose (%)	Hemi-cellulose (%)	Cellulose (%)	Alpha-Cellulose (%)	Lignin (%)
Oak Sawdust	74.36	26.59	47.77	40.46	25.57
Decomposed-Oak Sawdust	71.97	23.18	48.79	42.22	23.06
Beech Sawdust	74.44	28.54	45.90	36.01	23.55
Decomposed-Beech Sawdust	72.40	25.33	47.07	42.86	21.44

The density and tensile strength, modulus of elasticity and alongation at break of biocomposite film produced using the determined ratios of wastes are shown in the Table 4. There was statistically significant difference in density between parameters ($p \leq 0.05$). It has been found that degradation reduces the density of the biocomposite when compared to the contents added at the same rate in general. It was determined that the density increased in all combinations compared to the control sample. Statistically, significant differences in tensile strength values between groups were determined ($p<0.01$). In the biocomposite material produced, it has been determined that as the amount of lignocellulosic additive increases and the ratio of degraded fiber increases, the tensile strength value decreases (excep group O3). The slight increase in group O3 is statistically insignificant. In a study by Karakus and Mengeloglu, they determined that the tensile strength value of PCL composite material produced with wheat straw also decreased and stated that this was due to the incompatibility between polymer and lignocellulosic filler (Karakus and Mengeloğlu, 2016). SEM images support the lack of compatibility between polymer and lignocellulosic materials (Figure 1 and 2). In the SEM images, visible hollow structures between polymer and lignocellulosic material are clearly seen. Connection between polymer and wood flour weakness and detachments were also observed in Figure.1-2. Sözbir (2021) supported this incompatibility between lignocellulosic material and polymer in her study.

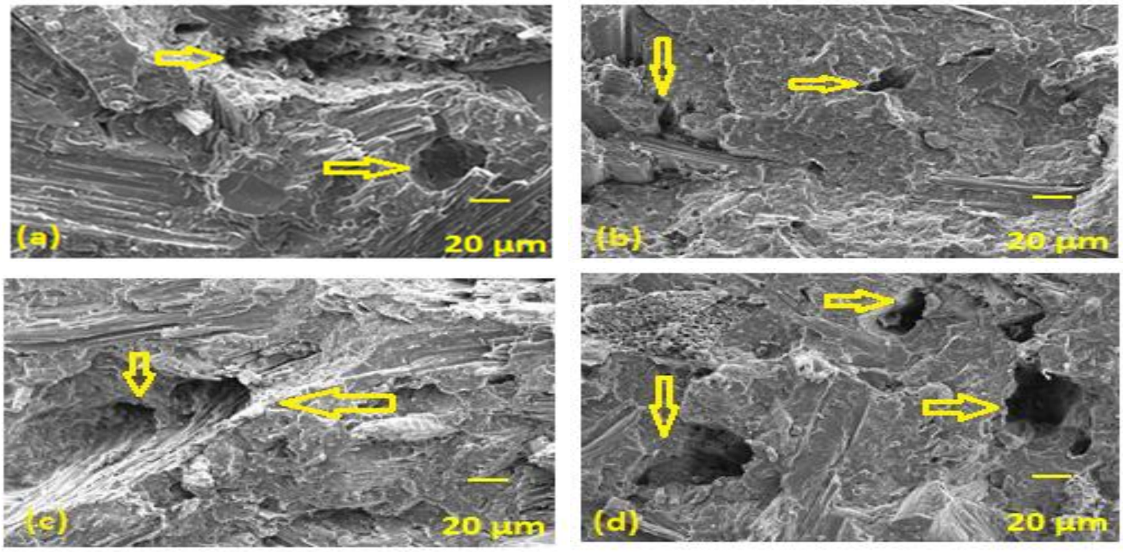

Figure 1. SEM images of fillers and polymer composites: a) B1; b) B2; c) B3; and d) B4

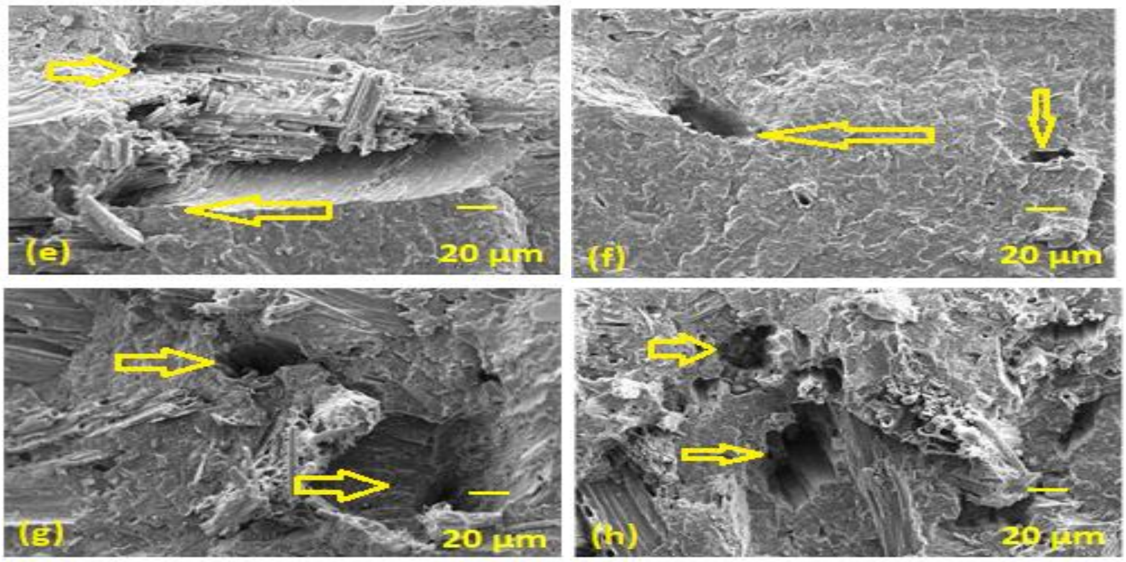

Figure 2. SEM images of fillers and polymer composites: e) O1; f) O2; g) O3; and h) O4

It was found that the modulus of elasticity value of all combinations is higher than the control sample compared to the control sample ($p<0.01$). Among all combinations, the highest values were obtained from biocomposites with 30% lignocellulosic additive. These results showed similar properties with the result that it increased the tensile modulus value in pcl composite material produced using 19, 26, 30, 38% hemp fiber in another study (Dhakal et al., 2018).

Statistically, significant differences in elongation at break values between groups were determined ($p<0.01$). In the production of pcl biocomposite, it was determined that the polymer material produced by adding 15% and 30% fiber significantly reduced the elongation at break values and statistically, there was no difference between the contents (B1, B2, B3, B4, O1, O2, O3, O4). In their study by Garcia et al., (2014) they produced bio material using 10, 20, 30% almond skin as filler in the PCL composite and as a result, they determined that as the amount of filling increases, the elongation at break value decreases due to the interaction between the polymer and filler.

Table 4. Density in water and mechanical properties PCL biocomposites

Combinations	Density	Tensile Strength (Mpa)	Modulus of Elasticity (Gpa)	Modulus of Elasticity (Gpa)
Control	1.131 a (0.02)*	15.62 c (1.13)*	548 a (209.26)*	334.55 b (64.46)*
Group B1	1.150 abc (0.01)*	10.71 b (1.57)*	695 b (82.8)*	8.48 a (5.71)*
Group B2	1.145 ab (0.05)*	10.48 b (0.7)*	563 a (45.01)*	5.63 a (2.41)*
Group B3	1.211 bc (0.1)*	8.51 a (0.75)*	937 d (81.69)*	6.27 a (6.49)*
Group B4	1.200 abc (0.04)*	7.92 a (0.84)*	921 d (69.17)*	7.07 a (2.07)*
Group O1	1.140 ab (0.01)*	10.07 b (1.18)*	750 bc (131.99)*	6.51 a (2.11)*
Group O2	1.150 abc (0.02)*	9.86 b (1.3)*	835 cd (253.61)*	10.28 a (4.14)*
Group O3	1.219 c (0.16)*	10.55 b (1.21)*	1108 e (92.08)*	3.78 a (3.04)*
Group O4	1.165 abc (0.05)*	7.91 a (0.85)*	927.86 d (120.44)*	5.28 a (1.37)*
Sign.	$p \leq 0.05$	$p<0.01$	$p<0.01$	$p<0.01$

*The value in parentheses indicate the standard deviation

It was determined that the amount of water absorption of fiber reinforced biocomposites produced increased compared to the control sample (Figure 3). The highest amount of water absorption was obtained from combinations with 30% additives, and it was found that the degreed fiber reinforced composite materials absorb more water end of time (at 720 hours). In a study, it was determined that the water absorption value of the pcl composite produced by using 10, 20, 30% filler material increases with the amount of water absorption as the amount of filler in the material increases (Garcia et al., 2014).

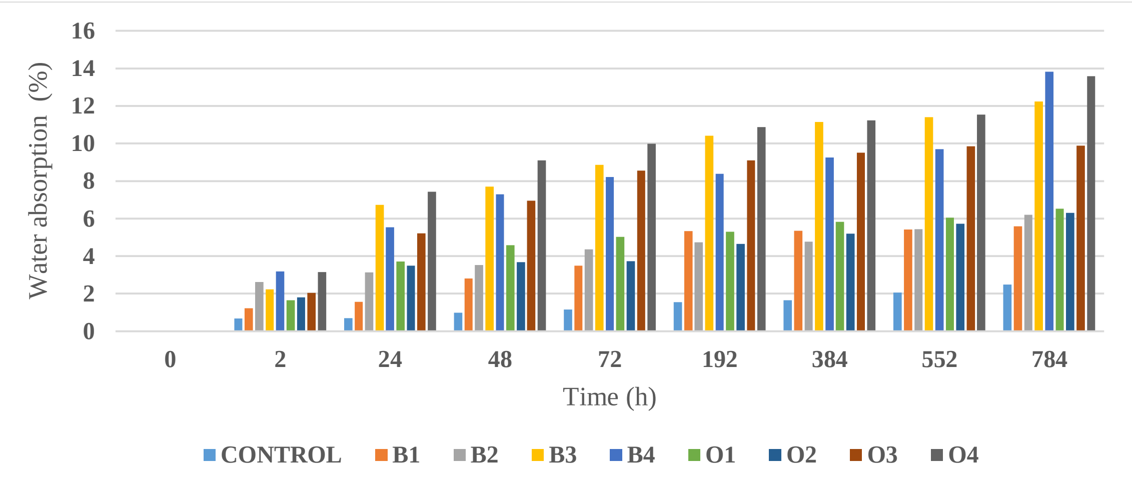

Figure 3. Water absorption (%) of Biocomposites materials

Figure 4 shows the thickness swelling of biocomposites materials. While there was no swelling to its thickness in the first 48 hours in the control sample, it was observed that swelling to its thickness began from the first hour in all other combinations. It has been determined that 15% fiber-reinforced composites show less swelling than 30% fiber-reinforced composites.

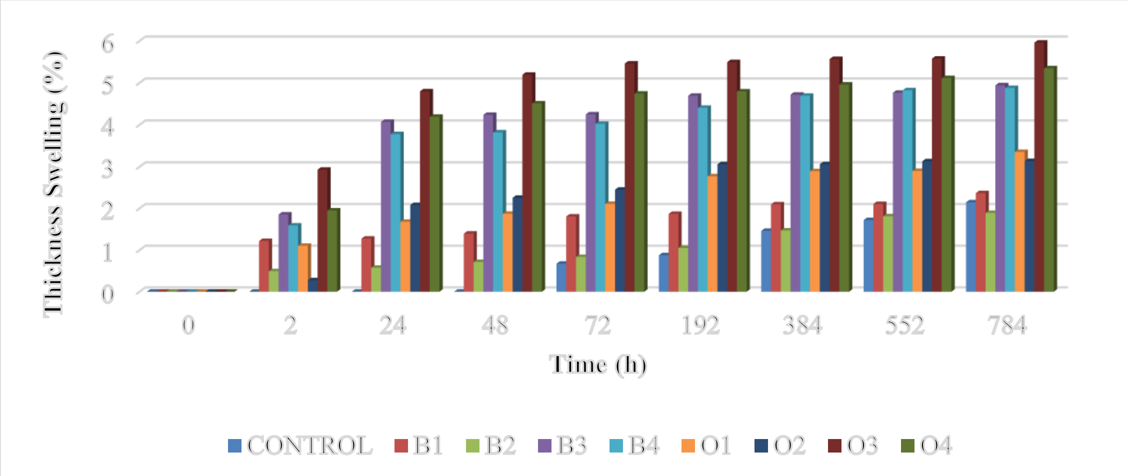

Figure 4. Thickness swelling of Biocomposites materials

Conclusions

The usability of mushroom compost wastes has been determined, especially in the production of pcl biocomposite film material used in the packaging industry. The degradation time that lignocellulosic compost wastes were exposed to during a two harvest in the cultivation of lentinus edodes was obtained and the changes in their chemical composition were revealed. It has been found that the tensile resistance of the produced material decreases as the fiber amount increases, and also the gradient fibers decrease the tensile resistance more. The modulus of elasticity value increased as the amount of fiber increased. It was found that the modulus of elasticity value of the material produced by adding oak sawdust increased more compared to beech. The elongation at break value caused a huge decrease by adding fiber to the pcl composite. In general, compared to the control sample, the amount of water intake increased and the highest increase was found in materials containing 30% fiber in both waste types (oak and beech). It has been determined that films produced with degreed fibers take up more water compared to raw fibers.

Ethics Committee Approval

N/A

Peer-review

Externally peer-reviewed.

Author Contributions

Conceptualization: G.D.S.; Investigation: G.D.S., Ç.A.; Material and Methodology: G.D.S., F.M., M.Y.; Supervision: G.D.S., F.M.; Visualization: G.D.S., K.K.; Writing-Original Draft: G.D.S.; Writing-review & Editing: G.D.S.; Other: All authors have read and agreed to the published version of manuscript.

Conflict of Interest

The authors have no conflicts of interest to declare.

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