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Biodegradation properties of natural fibres from renewable resources

Yenilenebilir kaynaklardan elde edilen doğal liflerin biyodegradasyon özellikleri

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Biodegradation Properties of Natural Fibres from Renewable Resources

Highlights

- *Natural Fibres from Renewable Resources*
- *Better biodegradation properties of PLA fibres*
- *A minimum of 11 days is recommended for testing natural fibre biodegradability*
- *The importance of the obtained knowledge about the biodegradation of natural fibres*

Graphical Abstract

The combination of biodegradation conditions and microbial populations in the farmland soil contributed to the chemical hydrolysis of PLA fibres (molecular weight of PLA decreases) increasing the amount of fungal and bacterial population that degrades the fibre and decreasing the tenacity by 37.2%.

Table. The mass loss and mechanical properties

	Jute		PLA	
	Control	11 days	Control	11 days
Mass loss $\bar{x}$ [%]	/	24.84	/	4.00
Finesess				
$\bar{x}$ [dtex]	31.02	26.14	6.84	6.75
xmin [dtex]	13.68	13.35	6.86	5.87
xmax [dtex]	80.50	47.16	6.92	7.82
s [dtex]	12.16	8.27	0.65	0.46
CV [%]	39.19	31.62	9.57	6.74
pgg [%]	7.68	6.20	2.65	1.32
Δ [%]	/	15.73	/	14.61
Tenacity				
$\bar{x}$ [cN/tex]	43.94	33.39	17.86	11.21
xmin [dtex]	16.37	11.65	14.00	7.55
xmax [dtex]	67.92	55.82	25.93	14.02
s [dtex]	12.44	10.82	5.36	1.53
pgg [%]	28.30	32.41	30.04	13.66
CV [%]	5.55	6.35	8.33	2.68
Δ [%]	/	24.01	/	37.23

Aim

The aim of the presented work is to investigate the biodegradation behaviour of Jute and PLA fibres.

Design & Methodology

After 11 days of burying Jute and PLA fibres in the soil, a standard analysis involves assessing the mechanical properties, mass loss, and morphological analysis to characterize buried fibres.

Originality

The limited scientific literature on fibre biodegradability emphasizes the importance of obtained knowledge for the selection of suitable fibres for the production of environmentally friendly products.

Findings

The study findings highlight better biodegradation properties in PLA fibres compared to Jute fibres, offering valuable insights for further exploration in this field.

Conclusion

The results of the conducted research indicate the fact that PLA fibres show better biodegradation properties compared to Jute fibres. This provides significant guidance for conducting further research in this wide area.

Declaration of Ethical Standards

The authors of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

Biodegradation Properties of Natural Fibres from Renewable Resources

Araştırma Makalesi / Research Article

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ABSTRACT

The interest in natural fibres in non – textile applications has increased as a result of the search for new renewable materials. Especially attractive for environmental safety demands are biodegradable and renewable fibres such as lignocellulose fibres and biopolymers such as PLA.

The analysis of their biodegradation is often taken as a standard measure for environmentally friendly textile materials. Therefore, the aim of this paper is to investigate the biodegradation properties of Jute and PLA fibres by soil burial test. The fibres were exposed to the farmland soil for 11 days. The efficiency of the biodegradability was determined by comparison of mass loss, mechanical properties (finesses and tenacity) and morphological analysis by SEM microscope. With the purpose of a better understanding of biodegradation, the number of total fungi and bacteria in the soil is also determined.

Keywords: Biodegradability, natural fibres, properties, soil burial test.

Yenilenebilir Kaynaklardan Elde Edilen Doğal Liflerin Biyodegradasyon Özellikleri

ÖZ

Yeni yenilenebilir malzeme arayışları sonucunda tekstil dışı uygulamalarda doğal liflere olan ilgi artmıştır. Çevresel güvenlik talepleri için özellikle cazip olan, lignoselüloz lifleri ve PLA gibi biyopolimerler gibi biyolojik olarak parçalanabilen ve yenilenebilir liflerdir. Biyobozunmalarının analizi genellikle çevre dostu tekstil malzemeleri için standart bir ölçü olarak alınır.

Bu nedenle, bu makalenin amacı Jüt ve PLA liflerinin biyolojik bozunma özelliklerini toprağa gömme testi ile araştırmaktır. Lifler, 11 gün boyunca tarım arazisi toprağına maruz bırakıldı. Biyobozunurluğun etkinliği, SEM mikroskobu ile kütle kaybı, mekanik özellikler (incelik ve sağlamlık) ve morfolojik analizin karşılaştırılmasıyla belirlendi. Biyobozunmanın daha iyi anlaşılması amacıyla topraktaki toplam mantar ve bakteri sayısı da belirlenir..

Anahtar Kelimeler: Biyolojik olarak parçalanabilirlik, doğal lifler, özellikler, toprağa gömme testi.

1. INTRODUCTION

With the huge increase in the global population and advances in technology, synthetic polymers play a major role in everyday life, primarily due to the wide range of properties that enable a wide variety of products and areas of use. Growing global environmental concerns and awareness of renewable green resources are constantly increasing the demand for environmentally friendly, sustainable and biodegradable fibre which synthetic polymers are not primarily due to their long-term decomposition process, which has a harmful effect on the environment. Synthetic polymers are characteristically inert and resistant to microbial attack and therefore they remain in nature without any deformation for a very long time. Increased pollution and inadequate disposal of non-biodegradable materials into nature are increasingly endangering the environment. Using natural fibres as a substitute for non-biodegradable polymers in the production of biomaterials is considered a viable alternative in many industrial sectors, with the aim of reducing the impact on the environment [1-4].

Many potential applications for biodegradable materials lie in direct use in soil. Examples are primarily agricultural products etc., but also other products such as road construction aids (for slopes), body bags, etc. The biodegradation behaviour in the soil is therefore an important question and cannot always be deducted from other biodegradation tests. In many instances, the soil will turn out to be a much more biologically aggressive environment than water because of the high fungal activity.

Many different degradation mechanisms combine synergistically in nature to degrade polymers. Biodegradation is usually primarily caused by the action of enzymes or by-products (such as acids and peroxides) secreted by microorganisms (bacteria, yeasts, fungi, etc), although often non-biotic effects such as irradiation,

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thermal degradation or chemical hydrolysis contribute to the degradation process.

Two key steps occur in the microbial polymer degradation process: first, a depolymerisation or chain cleavage step, and second, mineralization. In the first step, the long polymer chain is converted into oligomeric fragments small enough to be assimilated. Hydrolysis and/or oxidation and extracellular enzymes may be responsible for this step. The second step occurs inside the cell where small-size oligomers are converted into biomass, minerals and salts, water, and gases such as C02, CH4, N2 and H2. Many variations of this general view of the biodegradation process can occur, depending on the polymer, the organisms, and the environment. Nevertheless, there will always be, at one stage or another, the involvement of enzymes. Enzymes are biological catalysts that can induce enormous (108-1020 times) increases in reaction rates in an environment that is otherwise unfavourable for chemical reactions. [5].

The activity of microorganisms is closely connected to the presence of water. The supply of nutrients to the microorganisms and the transportation of excreted enzymes and metabolic products takes place by diffusion in an aqueous environment surrounding the cells. However, in an environment such as soil or compost, microorganisms can also be active as long as the aquatic microenvironment allows the transport processes necessary for biological activity. For example, microbial life occurs in the thin water films between the particles or in water-filled cavities in the soil components. A soil humidity of around 50-60% is optimal for aerobic biological processes [3, 4].

The biodegradability of fibres depends on many factors, not only on the physical and chemical properties of the material but also on the conditions under which biodegradation is considered. Thus, the conditions of biodegradation vary between composting, soil biodegradation, marine biodegradation, sewage sludge biodegradation, anaerobic biodegradation, landfill biodegradation and others. Under controlled conditions, it is possible to lead the biodegradation process to the desired result by controlling the biodegradation conditions. Biodegradation in real conditions is much more complex considering that many factors from the environment can affect the biodegradation process which cannot be influenced or managed.

Natural fibres are favourable resources of raw materials for industrial application, as they are environmentally efficient with high tenacity, biodegradability, renewability and sustainability. Due to their biodegradable nature, natural fibres are gradually replacing synthetic fibres in the field of non-woven agrotextiles [1, 2]. Natural fibres are constituted of cellulose, hemicellulose, lignin, waxes and some water-soluble compounds whose percentage depends on the type of natural fibre. Furthermore, they differ in chemical structure, such as the proportion of chemical composition, the size of macromolecules, the proportion of crystallinity, and also in the method of fibre production. All these differences affect the degradation behaviour, which results in different biodegradability [3, 4].

Jute fibres, as a lignocellulosic fibre, are biodegradable and recyclable, i.e. environmentally friendly fibres. The use of Jute fibres is also justified by their low cost and high availability, low density, easy biodegradability, nontoxicity, better physical and mechanical properties than other natural fibres and no energy consumption during fibre production. The chemical composition of Jute fibre includes cellulose (64.4%), hemicellulose (12%), pectin (0.2%), lignin (11.8%), water-soluble (1.1%), wax (0.5%), and water (10%) [5-7]. Like any other natural fibre, the performance of the Jute fibre varies due to the natural variability in surface and internal microstructural characteristics, which can be influenced by a number of factors including growing conditions (i.e., temperature, humidity, soil condition), retting (water, dew or enzymatic) and fibre extraction processes, fibre length and diameter, chemical constituents and their proportional amounts [8-11].

Today, biopolymers derived from starch are gaining more and more importance due to their availability, low cost and their biodegradability. One of the most widely used eco-friendly polymers is PLA biopolymer whose monomer (lactic acid) is derived from natural sources using bacterial fermentation of corn, sugarcane, potatoes, and other biomass [12, 13]. PLA is renewable and biodegradable, with good mechanical properties, with considerable stiffness and its composites can be fabricated by traditional manufacturing methods. Under the right conditions, PLA biopolymer dissolves quickly through the hydrolysis process [14, 15]. PLA is a very useful material to be used as a replacement for petroleum-based polymers because of its good mechanical properties and good processability. Its application comprises several industries, such as packaging, textile, biomedical, automotive and agriculture. PLA biopolymers have positive characteristics such as controlled crimp, smooth surface and low moisture regain. One unique property in comparison is that it is the only melt-processable fibre from annually renewable natural resources [16-18]. PLA is known to be entirely biodegradable. Through hydrolysis, microorganisms convert PLA (lactic acids) to smaller oligomers which can be converted into water and carbon monoxide. Degradation mainly depends on the molecular weight and other factors such as the temperature, time, impurities and residual catalyst concretisation which increases the rate of PLA biodegradation [16].

With the aim of supporting more intensive use of biodegradable and renewable sources of raw materials for use in various industries and with a general attitude of greater responsibility towards the environment and the need to protect natural resources, natural fibres are becoming important again in the strategy of economic development. The interest in natural fibres in non –

textile applications has increased as a result of the search for new renewable materials. Especially attractive for environmental safety demands are biodegradable and renewable fibres such as lignocellulose fibres and biopolymers such as PLA [18, 19].

2. MATERIAL AND METHOD

The investigation of the biodegradation properties of natural fibers from renewable resources was performed on of Jute (supplied by Derotex) and PLA fibres (supplied by NatureWorks LLC Company) by a simple soil burial test (ISO 11721) conducted under controlled conditions in the laboratory.

Considering a small number of studies based on fibre biodegradability and the absence of standardized testing methods, it was estimated that 11 days would be appropriate to test fibre biodegradability. For the optimal activity of microorganisms, soil moisture was 60 ± 5% and soil pH was 6.8. To prevent fibres from mixing or overlapping, samples of each fibre type were buried in separate containers of unglazed pottery, at a depth of 150 mm. Pots with fibres were placed in an incubator with a constant air humidity of 95 ± 5 % and a temperature of 29 ± 1 °C. The fibres were removed from the soil after a defined time period of 11 days, cleaned and washed in an ethanol/water solution (70/30 vol. %) for approximately 40 minutes, and afterwards dried at room temperature. The samples were then subjected to relevant tests to determine the time and rate of fibre biodegradation.

Typical analysis performed after burial in the soil is the evaluation of the mechanical properties, mass loss and fibres surfaces microscopy has been applied to characterise polymeric samples after degradation in soil (Table 1).

With the purpose of a better understanding of biodegradation, the number of total fungi and bacteria in the soil is also determined. The total number of fungi and bacteria in the soil by classical microbiological methods was tested. The percentage of dry matter for all soil samples was determined after drying the samples. Soil samples in a sterile saline solution were homogenized for 5 min. After that, a series of dilutions were made, followed by the smear of relevant sample volume on solid nutrient media. After the cultures had grown on nutrient media, the colonies were counted, and the CFU value of individual groups of microorganisms per gram of soil was determined.

The protocol of investigation is shown in Figure 1.

3. RESULTS AND DISCUSSION

Analysis of the biodegradation properties of jute and PLA fibres, i.e. testing of mass loss and mechanical properties obtained after burying the fibres in the soil are shown in Table 2.

One of the typical and very simple analyses for testing the fibre's biodegradation is the evaluation of the mass loss after burial in the soil. Mass loss results of samples after burial in the soil are shown in Table 5, expressed as a percentage compared to control samples. Considering that fineness and tenacity are tested using the method of individual measurement, Table 1 also shows statistical indicators of variability - arithmetic mean value ( ), min and max value of tested properties, coefficient of variation, CV, standard deviation (s), and practical error limit (pgg).

As expected, the mass loss of jute fibres is greater compared to PLA fibres, due to the greater ability of Jute fibres to absorb moisture, thus creating better conditions for the development of microorganisms responsible for biodegradation.

Table 1. Mechanical properties

Fibres properties	Methods	Adjustment of methods
Mass loss, %	Gravimetrical methods	
Fineness	ISO 1973:2021 Vibroscop 400	*cogged steel clamp gauge length: 5 mm testing speed: 3 mm/min pre-tension: 1500 mg number of measurements: 100
Tenacity	ISO 5079:2020 Vibrodyn 400	
*Due to the non-homogeneity of jute fibres used standards and regulations are adapted to testing flax fibres. The number of measurements was increased and determined according to statistical indications of the degree of reliability with 95%. The fineness and tenacity of the PLA fibres were determined according to the above standards without any modification of the test. Measurements of tested properties were performed on conditioned fibres.		

Biodegradation properties of Jute and PLA fibres are also analysed by using a scanning electron microscope (FE-SEM//Mira, Tescan) at a magnification of 2000x. SEM images were obtained with an accelerating voltage of 5kV. Before the SEM investigation, samples were steamed with chrome for 180 seconds to increase their electrical conductivity.

The analysis of the obtained results of the mechanical properties of Jute fibres shows a high coefficient of variation, which is expected considering that, the method of individual measurement was used and that jute fibres are characterized by a large non-homogeneity of morphology. Fineness analysis showed that the decrease in fineness of PLA fibers is slightly less (14.61%) than of Jute fibers (15.73%). Analyses of the results show that the decrease in PLA fibre tenacity (37.23%) is higher than in Jute fibres (24.01%). The obtained results are as expected because jute fibres consist of 60% cellulose and about 12% lignin, which preserve fibre from degradation. Lignin is one of the main components with the main purpose to protect the cellulose fibres from microorganism attacks by creating a protective layer preventing the fibre's internal structure from degrading. In addition, due to its insolubility in water, it acts as a glue for cellulose and hemicellulose, thus preserving the fibres.

The biodegradation of PLA fibres is much slower than that of cellulose fibres. PLA, as a biopolymer, has high crystallinity, brittleness and biodegrades slowly in soil conditions. Multiple factors, such as the polymer's characteristics, chemical structure, molecular weight, glass transition temperature, melting point, polydispersion, and crystallinity influence its biodegradation behaviour. In addition, under conditions of high temperature and high humidity, PLA will degrade quickly especially if it is in the form of fibres that are free and without the protection of the structural parameters characteristic of woven or non-woven materials as is in this research. In addition, the combination of biodegradation conditions and microbial populations in the farmland soil contributed to the chemical hydrolysis of PLA fibres (molecular weight of PLA decreases) increasing the amount of fungal and bacterial population that degrades the fibre and decreasing the tenacity by 37.2%.

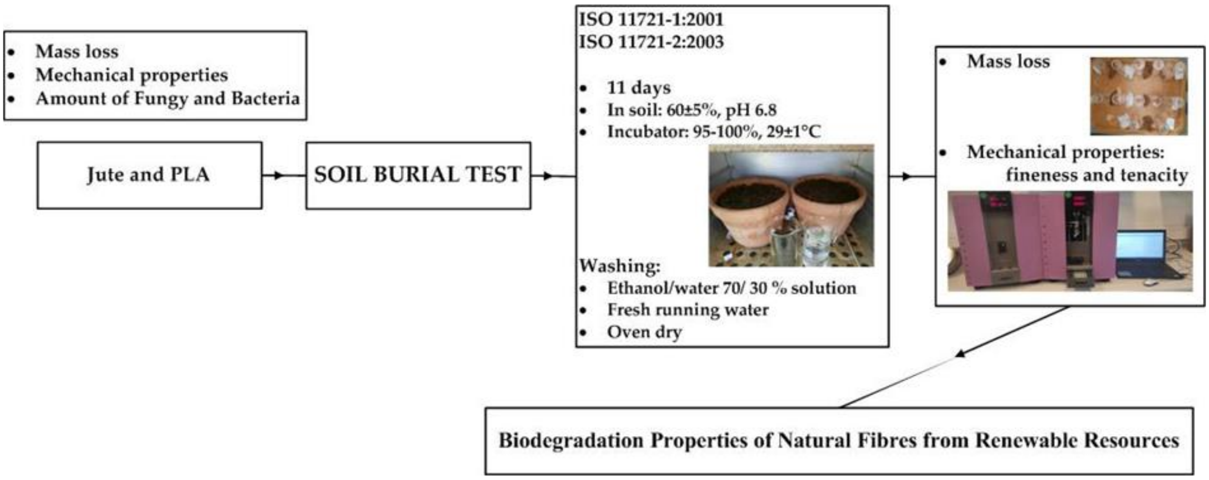


Figure 1. The protocol of investigation

Table 2. The mass loss and mechanical properties.

	Jute		PLA	
	Control	11 days	Control	11 days
Mass loss $\overline{x}$ [%]	/	24.84	/	4.00
Finesess				
$\overline{x}$ [dtex]	31.02	26.14	6.84	6.75
xmin [dtex]	13.68	13.35	6.86	5.87
xmax [dtex]	80.50	47.16	6.92	7.82
s [dtex]	12.16	8.27	0.65	0.46
CV [%]	39.19	31.62	9.57	6.74
pgg [%]	7.68	6.20	2.65	1.32
Δ [%]	/	15.73	/	14.61
Tenacity				
$\overline{x}$ [cN/tex]	43.94	33.39	17.86	11.21
xmin [dtex]	16.37	11.65	14.00	7.55
xmax [dtex]	67.92	55.82	25.93	14.02
s [dtex]	12.44	10.82	5.36	1.53
pgg [%]	28.30	32.41	30.04	13.66
CV [%]	5.55	6.35	8.33	2.68
Δ [%]	/	24.01	/	37.23

The obtained results were confirmed with surface microscopic images before and after the biodegradation processes (Table 3). Analysing and comparison of the fibre's surface microscopic images before and after biodegradation processes in the soil show a high attack of the microorganisms on the samples after 11 days buried in the soil. As seen in the pictures, buried fibres have a damaged and rougher surface compared to the control samples.

Compared to the control soil, the highest amount of Fungi and Bacteria (Table 4) was determined in the soil in which the PLA fibres were buried and these results follow the obtained results of a tenacity decrease of buried PLA fibres. The degradation mechanism of PLA polymer is different from natural cellulosic fibres such as Jute. The microorganisms can degrade PLA only after high molecular weight PLA, after the hydrolysis process, the molecular weight of PLA decrease. In addition, the biodegradation properties of PLA biopolymer depend on the polymer's degree of crystallinity and biodegradation conditions such as temperature and humidity.

4. CONCLUSION

The results of the conducted research indicate the fact that PLA fibres show better biodegradation properties compared to Jute fibres. This provides significant guidance for conducting further research in this wide area. A review of available scientific literature revealed

Table 3. Morphological analysis

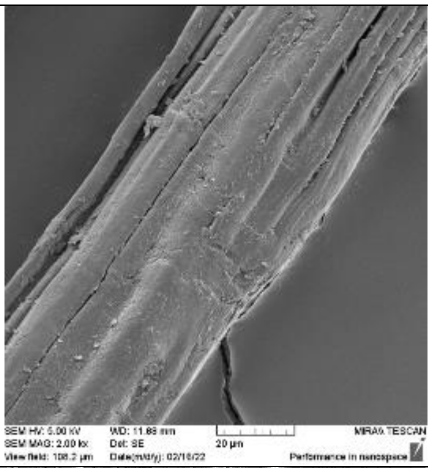
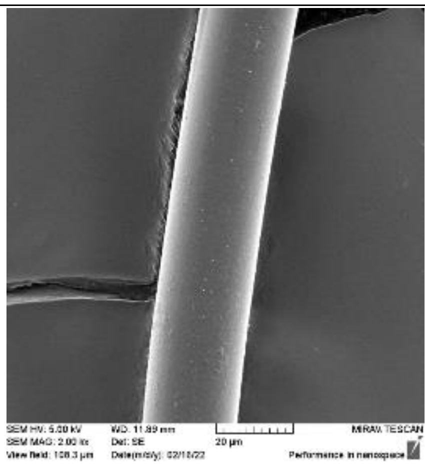
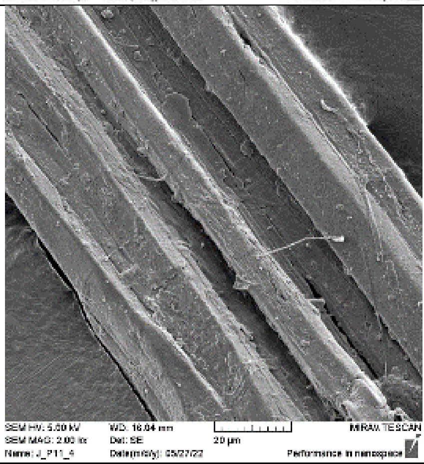
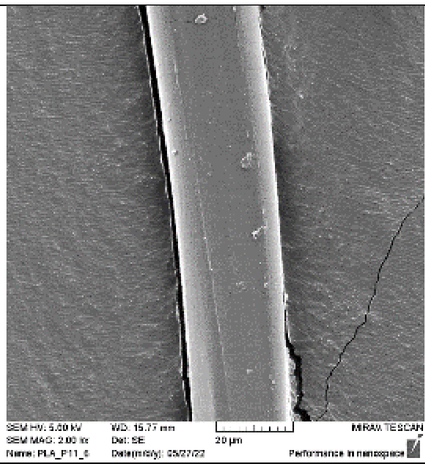
	Jute	PLA
Control		
11 days		

Table 4. Total number of fungi and bacteria in the soil.

	Jute		PLA	
	Control	11 days	Control	11 days
Fungi $\bar{x}$ [CFU/ ml] stDEV	8.50E+04 7.55E+03	1.03E+05 3.21E+03	8.50E+04 7.55E+03	1.10E+05 9.85E+03
Bacteria $\bar{x}$ [CFU/ ml] stDEV	1.28E+07 7.64E+05	1.42E+07 4.36E+05	1.28E+07 7.64E+05	1.66E+07 1.97E+06

a lack of research on fibre biodegradability. Therefore, the obtained knowledge on fibre biodegradability is highly valuable for the selection of suitable fibres for the production of environmentally friendly products.

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DECLARATION OF ETHICAL STANDARDS

The author(s) of this article declares that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

AUTHORS' CONTRIBUTIONS

Ruzica Brunsek: Performed the experiments, methodology, writing- review and editing, and supervision.

Ivana Schwarz: Writing- review and editing, visualisation

Dragana Kopitar: Writing- review and editing, visualisation

Paula Marasovic: Performed the experiments and analysed the results.

CONFLICT OF INTEREST

There is no conflict of interest in this study.

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