Biodegradation of Natural Textile Materials in Soil

Abstract

World is facing numerous environmental challenges, one of them being the increasing pollution both in the atmosphere and landfills. After the goods have been used, they are either buried or burnt. Both ways of disposal are detrimental and hazardous to the environment. The term biodegradation is becoming more and more important, as it converts materials into water, carbon dioxide and biomass, which present no harm to the environment. Nowadays, a lot of research is performed on the development of biodegradable polymers, which can "vanish" from the Earth surface after being used. In this respect, this research work was conducted in order to study the biodegradation phenomenon of cellulosic and non-cellulosic textile materials when buried in soil, for them to be used in our daily lives with maximum efficiency and after their use, to be disposed of easily with no harmful effects to the environment. This research indicates the time span of the use life of various cellulosic and non-cellulosic materials such as cotton, jute, linen, flax, wool when used for the reinforcement of soil. The visual observations and applied microscopic methods revealed that the biodegradation of cellulose textile materials proceeded in a similar way as for nontcellulosic materials, the only difference being the time of biodegradation. The nontcellulosic textile material (wool) was relatively more resistant to microorganisms due to its molecular structure and surface.

Keywords: biodegradation, composting, natural textile materials, FT-IR

Izvleček

V svetu se soočamo z vse večjimi okoljskimi izzivi. Velik ekološki problem so onesnaženost ozračja in odlagališča odpadkov. Izdelek na koncu svojega življenjskega cikla pristane bodisi na odlagališču odpadkov bodisi ga sežgeamo sežigalnici. Oba načina odstranjevanja odpadkov sta zelo nevarna in tudi škodljiva za okolje. Izraz biorazgradnja je čedalje pomembnejši. Biorazgradljiv material je material, ki po naravni poti v relativno kratkem času razpadne v enostavne snovi, kot so voda, ogljikov dioksid in biomasa, ki ne pomenijo nikakršne škode za okolje. V današnjem času je veliko raziskav usmerjenih v razvoj biorazgradljivih polimerov, ki bi po uporabi lahko preprosto »izginili«. Biorazgradljivost celuloznih in neceluloznih tekstilnih materialov smo študirali tako, da smo jih zakopali v zemljo. Takšne biorazgradljive tekstilne materiale je mogoče z maksimalno učinkovitostjo uporabiti v vsakdanjem življenju in jih lahko po uporabi brez težav in brez škodljivih vplivov na okolje zavržemo. Proučevali smo, kako s časom prihaja do razgradnje različnih celuloznih in neceluloznih tekstilnih materialov, kadar se le-ti uporabljajo za utjemanje tal. Taka mikroskopska metoda kot tudi metoda vizualnega opazovanja biorazgradljivosti celuloznih tekstilnih materialov kaže podoben potek razgradnje teh materialov, edina razlika je v času biorazgradljivosti, medtem ko so necelulozni tekstilni materiali (volna) zaradi njene molekularne strukture in površine precej bolj odporni proti mikroorganizmom.

Ključne besede: biorazgradnja, kompostiranje, naravni tekstilni materiali, FT-IR
1 Introduction

The disposal of fabric materials used in textiles is a serious challenge to waste management. The most common waste management options for textile materials are used clothing for markets (second-hand clothing), conversion to new products, wiping and polishing cloths, landfill and incineration for energy [1]. Addition to these traditional processing routes, the cellulosic waste decrease can also be achieved using composting.

Composting is a method of waste disposal that allows organic materials to be converted into a product that can be used as a valuable soil amendment. In the broad sense, biodegradation is the biologically catalyzed conversion in the complexity of chemicals [2, 3]. A material is defined as “biodegradable” if it is able to broken down into simpler substances by naturally occurring decomposers. It must be non-toxic and able to be decomposed in a relatively short period of time [2]. The biodegradation of material takes place in three steps:
- biodeterioration
- biofragmentation
- assimilation.

Biodeterioration of materials is a combined result of lots of degradative factors like mechanical degradation, thermal degradation and degradation due to the presence of moisture, oxygen, ultra violet light and environmental pollutants. Due to the result of these mentioned factors, a huge amount of microorganisms stick onto the surface of materials. Biofragmentation is a process in which microorganisms increase their population and secrete enzymes and free radicals, which break down macromolecules to oligomers, dimers and monomers. In the step of assimilation, energy, new biomass and various metabolites used by microorganisms are produced and simple gaseous molecules and mineral salts are released into the environment [4].

The aerobic biodegradation of materials depend upon the polymers chemical composition and the environment to which they are exposed. Some of the important factors that directly influence the rate of biodegradation are as follows [5]:
- presence of microorganisms
- availability of oxygen
- amount of water available
- temperature
- chemical environment (pH, electrolytes, etc).

For a material to be biodegraded, first microorganisms as a “biodestructor source” are required. Microorganisms are present in atmosphere and in soil as well. In fact soil is very rich in microorganisms and its layer from 5 to 15 cm deep is most saturated with microorganisms; one gram of soil can contain up to $10^8$ different microorganisms [6].

Microorganisms attack material surface according to the following steps [4]:
- microorganisms stick onto the surface of a material either by adhesion or aggregation
- proliferation of attached microbial cells
- production of enzymes
- biodegradation of material (reduction of degree of polymerization of the material polymers; production of degradable products).

The biodegradation of cellulose and cellulosic textile substrates such as fibers and fabrics has been extensively studied over the last decades [7−11] and a book including biodegradable and sustainable fibres with essential references was published [12]. Biopolymers represent the most abundant compounds in the biosphere and constitute the class of polymers that are renewable, sustainable and biodegradable. Biopolymers are polymers produced by living organisms. Cellulose, starch and chitin, proteins and peptides, and DNA and RNA are all examples of biopolymers, in which the monomeric units, respectively, are sugars, amino acids, and nucleotides [13]. Therefore, the biopolymers and the fibres that can be produced from them are very attractive at the market because of the positive human perception about what the term biodegradability means and further such materials also offer suitable solution connected with waste disposal problem. These polymers can be degraded by microorganism into biomass and can be used as alternative to synthetic polymers which are produced from non-renewable energy source.

The most common biopolymer in the biosphere and the main component of most of the natural fibres like cotton, linen, jute etc, is cellulose. Products produced from biopolymers including cellulose are very susceptible for microbial growth which can leads to many aesthetic, functional problems and even infection. But on the other hand this phenomenon can be used as an advantage by implementing cellulose containing materials into the biodegradable products. The degradation rate of cellulose and cellulosic textile substrates mostly depends on microorganisms used.
Bacteria and fungi are the two main groups of microorganisms responsible for enzymatic degradation of cellulose. In the presence of bacteria the degradation of cellulose fabrics proceeds from the surface towards the inside, in the presence of fungi, after the revival of the cuticle, the organisms penetrate through the secondary wall into a lumen where they grow [14]. The main function of the enzymes is to decrease the degree of polymerization, resulting in damaging the structure of the fibres and the fibres losses their strength. The rate of degradation of cellulose is directly related to its degree of crystallinity. Hence the amorphous cellulose is more susceptible for enzymatic degradation than the crystalline one. The degradation rate also depends on other parameters like degree of orientation, degree of substitution and presence of non-cellulosic substances [14]. Biodegradation of natural fibres and textiles is a widely explored area; in this paper data of cellulosic textile materials composting abilities buried in soil are presented. The main aim of our research was to study the stability of natural fibres (mainly cellulosic fibres and wool) against the microorganisms in the soil. The addition of non-degradable fibres (in our study the PET fibres were used) towards the reducing of biodegradation ability of the textile system as a whole was studied. The results of our research could be applied in geotextiles made of natural fibres. The major use of natural fibre geotextiles is in the erosion control. Because the main natural fibres are relatively quickly biodegradable (exception is the chitosan fibre), they are ideally suited for the initial establishment of vegetation that in turn provides a natural erosion prevention facility. By the time natural vegetation has become well established (12 months) the textile materials have started to rot/ degrade and disappear without polluting the land.

2 Materials and methods

2.1 Materials

Table 1 presents the used materials which were standard treated. The unit mass of each material before experimentation was determined by Zweigle apparatus. The average diameter of fibres was measured by using Axiotech microscope (numbers of readings were 200 for each of analyzed fibres). Cotton, jute, linen and wool were in woven form and flax was in non-woven form.

<table>
<thead>
<tr>
<th>Materials</th>
<th>Mass per unit area (g/m²)</th>
<th>Diameter of fibres</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Average (µm)</td>
</tr>
<tr>
<td>Cotton</td>
<td>182</td>
<td>17.16</td>
</tr>
<tr>
<td>Jute</td>
<td>263</td>
<td>68.00</td>
</tr>
<tr>
<td>Linen</td>
<td>211</td>
<td>24.38</td>
</tr>
<tr>
<td>Flax</td>
<td>413</td>
<td>70.64</td>
</tr>
<tr>
<td>Wool</td>
<td>198</td>
<td>23.04</td>
</tr>
</tbody>
</table>

2.2 Analyses and measurements

Soil burial test

The biodegradation of fabrics was done by burying the samples in the soil for different time. Cellulosic fabrics were exposed to the test soil according to standard ISO 11721-1:2001, Part 1 [15] and ISO 11721-2:2003 Part 2 [16]. The samples were cut into the square shape of dimensions of 5x5cm² and buried in soil into the beakers of capacity 1000 ml. The soil used was stabilized and matured compost obtained by organic fractions of communal waste (Kompostarna Ptuj), 2 to 4 months old with the characteristic:
- particle size: 0.5 to 1 cm
- content of dry substances: 50 to 55%
- content of volatile compounds: 15% (according to wet mass) or 30% (according to dry mass).

The water content of test soil was 55–65% of the maximum moisture retention capacity and the pH of the test soil was in the range of 4.0 to 7.5. The beakers containing the buried samples were then placed into the climatic chamber KK-105 CH for varying periods of time (3–4 weeks for samples in direct contact with soil and 3–4 months for sampled sawn in bags). Incubation of the soil burial samples was carried out at 95 to 100% relative air humidity and 29 °C. After the defined burial time the samples were removed from the soil and rinsed in ethanol/water (70/30 vol.%) solution for approximately 10 min before drying at room temperature.

Samples in direct contact with soil

In this method samples all types of fabrics were cut into square pieces of 5x5 cm² and four samples were taken from each kind of fabric and they were buried in soil according to the ISO 11721-1:2001 and ISO
11721-2:2003 standards. All four pieces of one type of fabric were buried in the same beaker of 1000 mL, so that different materials may not mix with each other. After every week soil from all beakers was taken out and moisturized with distilled water, after that soil with samples was again put back in the beakers and one piece of fabric from every kind of textile material was kept out to study the effect of microorganisms. These samples were first rinsed in ethanol/water (70%/30% volume fraction) solution for approximately 10 min before drying at room temperature and after that further experiment were conducted.

No direct contact of samples with soil
It is not possible to obtain data on the exact decrease in mass after a specific time because of the direct contact of soil with the fabrics, therefore all fabrics were first defibrillated into fibres and then sewn into more hydrophilic bags (nylon knitted textile material, mass of 25 g/m²) and into more hydrophobic bags (polypropylene/polyethylene blend in 50/50 wt.% woven textile materials, mass of 22 g/m²). The concept behind usage of bags is to be able to follow the reduction of natural fibre mass in soil due to biodegradation. We are aware that the time of degradation when textile material was directly buried in the soil is much shorter than degradation time of textile material sewn in the bag. The main two reasons are in the fact that the bag will resists the penetration of microorganisms and hinder the contact of microorganisms with the textile material. Of course the form of textile materials influences the time of degradation as well. Defibrillated textile fibres sewn in the bags are the only form of textile which can be used to study the reduction of material mass according to time.

Four samples of each type of textile material are sewn into hydrophilic bags and two in hydrophobic bags. There are seven different textile materials, so we prepare forty-two (42) samples and to keep them separate every sample is coded. After the preparation of bags they were put into the soil for three months by following the ISO 11721-1:2001 and ISO 11721-2:2003 standards.

After every month the samples were taken out and dried at room temperature for one day, and then heated four hour at 105 ºC to remove the moisture completely from the samples. Samples cooled down in a desiccator for one hour. After that all samples were weighted and again put into the soil. The reduction in mass percentage of the bags due to the degradation process is calculated by the formula as:

\[
\text{Weight loss (\%)} = \frac{M_b - M_a}{M_b} \cdot 100
\]

where weight loss (\%) is the percent weight loss after degradation, \(M_b\) is the weight of the sample before degradation and \(M_a\) is the weight of the sample after degradation.

Axiotech 25 HD (+POL) microscope (ZEISS)
Axiotech 25 HD (+pol) microscope (ZEISS) equipped with AxioCam MRc (D) high-resolution camera and KS 300 Rel. 3.0 image analysis software were used for fibres morphology studies. The measurements were performed according to a pre-defined macro, which ensured that all samples were analyzed in the same way and under the same conditions. All of the measurements were performed in light transmission mode with a halogen lamp as the light source. The illuminating power of the lamp was adjusted using a potentiometer.

Scanning electron microscope (TS 5130)
TESCAN Vega TS 5130 high vacuum electron microscope with maximum resolution of 3 nm was used to investigate the morphological changes during the biodegradation. Textile materials were defibrillated prior the preparation of samples.

ATR FT Infrared spectroscopy (Perkin Elmer)
IR spectroscopy was carried out with a Perkin-Elmer Fourier Transform infrared (FTIR) spectrophotometer with a Golden Gate attenuated total reflection (ATR) attachment with a diamante crystal.

Thermalgravimetric analysis (TGA Q500)
Thermogravimetrical analyses were carried out with TGA Q500. The sample that is to be run on this machine is heated at constant rate (10°C/min), while change in mass of sample is recorded as function of temperature. The weighing of the sample is done by a thermo-balance in the furnace.
3 Results and discussion

3.1 Direct contact with soil

Cotton

Cotton samples before experimentation and taken out from soil after seven, fourteen and twenty-one days of composting have been analyzed visually (the day light) and with the help of Axiotech microscope and Scanning electron microscope. The findings are pictorially represented in Figure 1.

The biodegradation of the fabric is not uniform, because of the non-homogeneity of the textile fibres (amorphous/crystalline region, surface porosity and fibre diameter, some damages etc). In cotton, the cellulosic polymers have a high degree of polymerization (≥ 7,000 – regarding the glucose remains) [17], highly reactive hydroxyl (–OH) groups, and the ability to support hydrogen bonding with its 70% crystalline area. The remaining 30% of the fibre is amorphous [18]. The structural deformation (such as destroyed surfaces, damage of
individual fibres), can be easily observed after the first week by naked eye and by both types of microscope. After two weeks the fabric was highly degraded and the structure of the fibres is almost collapsed. After three weeks the cotton fabric was so much degraded as it is clear in the photos that it was very difficult to separate it from the soil.

It should be mentioned that the band at 1638 cm$^{-1}$ increases and the new band appeared at 1542 cm$^{-1}$ after degradation. These bands are characteristic for amide groups and are in agreement with reference [19]. They observed increase in absorbance at 1650 cm$^{-1}$ and new band at 1540 cm$^{-1}$ when acetylated cellulose fibres were examined after 13 days of exposure with a cellulolytic bacterial strain. They surmised that these bands are characteristic for amide group and their appearance after degradation suggesting that the proteins are bound to the residuals fibres. Furthermore investigations [10, 11, 16, 18, 20, 21] confirmed presence of the bands at 1640 and 1548 cm$^{-1}$ belonging to the Amide I and II and are result of protein produced by microbial growth.

According to reference [22] the spectra of cellulose show decrease of bands particularly at 1372 cm$^{-1}$, 1336 cm$^{-1}$, 1313 cm$^{-1}$, 1280 cm$^{-1}$,1160 cm$^{-1}$ and 1105 cm$^{-1}$ when moving from high crystalline to amorphous cellulose, which indicates apart from chemical changes mentioned above that the samples are degraded.

In thermogravimetric analysis for cotton the maximum temperature is set to 500 °C and the ramp rate is set to 10 °C per minute. The weight of the sample taken should be very small, in the range of 5 mg to 10 mg. The reduction in weight percentage versus increase in temperature plot for cotton samples is shown in Figure 3. All curves indicate the loss of water (around 10%) at the beginning of heating. In the temperature interval 250–390 °C the curve for non-degraded cotton sample starts to decrease at higher temperature compare to samples exposed to soil for 2 and 3 weeks. This could be due to the fact that partly biodegraded samples contain more short length polymers compare to original samples. It is clear from Figure 3 that for the cotton sample exposed to soil for two and three weeks the final mass reduced significantly due to microorganisms activities and due to contamination of samples by the soil.

Figure 3: TGA analysis of biodegraded cotton samples

**Jute**

Figure 4 shows morphology of samples of jute fabric. After four weeks the jute fibres are highly degraded as it is clear from the microscopic view but from the naked eye it seems to be less degraded. The reason for this can be in higher mass per unit area and larger fibre diameter; in addition the fabric structure of jute was very compact. So these factors can hinder the attack of microorganisms to some extent. The amount of lignin present in jute is the highest among all other cellulose fibres used in our research and lignin behaves as a retarding agent for swelling and thus results in the limitation of intra-crystalline swelling so absorbance of moisture is also limited [23].

As jute mainly consists of cellulose (about 60%) so its degradational behaviour when studied by FT-IR spectroscopy seems not very different from that of cotton. The presence of an absorption band near 1730 cm$^{-1}$ in the FT-IR spectra (Figure 5) is due to
C=O stretching of the carboxyl groups. The sharp bands at 1595 and 1505 cm$^{-1}$ show the presence of aromatic rings in jute fibre. The spectra of lignin show sharp bands in these regions, due to the stretching modes of the benzene ring. The bands near 1250 and 1235 cm$^{-1}$ are possibly due to C–O–C bond in the cellulose chain and OH deformation respectively [24].

<table>
<thead>
<tr>
<th>Composting</th>
<th>Visually</th>
<th>Axiotech Microscope</th>
<th>SEM</th>
</tr>
</thead>
<tbody>
<tr>
<td>Before</td>
<td><img src="image1" alt="Before" /></td>
<td><img src="image2" alt="Before" /></td>
<td><img src="image3" alt="Before" /></td>
</tr>
<tr>
<td>1 Week</td>
<td><img src="image4" alt="1 Week" /></td>
<td><img src="image5" alt="1 Week" /></td>
<td><img src="image6" alt="1 Week" /></td>
</tr>
<tr>
<td>2 Weeks</td>
<td><img src="image7" alt="2 Weeks" /></td>
<td><img src="image8" alt="2 Weeks" /></td>
<td><img src="image9" alt="2 Weeks" /></td>
</tr>
<tr>
<td>3 Weeks</td>
<td><img src="image10" alt="3 Weeks" /></td>
<td><img src="image11" alt="3 Weeks" /></td>
<td><img src="image12" alt="3 Weeks" /></td>
</tr>
<tr>
<td>4 Weeks</td>
<td><img src="image13" alt="4 Weeks" /></td>
<td><img src="image14" alt="4 Weeks" /></td>
<td><img src="image15" alt="4 Weeks" /></td>
</tr>
</tbody>
</table>

*Figure 4: Juta samples*
Figure 5: FT-IR spectra of biodegraded jute samples

Figure 6 represents TGA analysis of jute samples. TGA of samples of jute expose to soil for two and four weeks shows significant final mass reduction due to microorganisms activities. Further, as it is possible to see on images in Figure 4, after biodegradation samples are contaminated by soil.

**Linen**

The biodegradation of linen fabric was fast compared to other cellulosic fibres and after two weeks it was extremely difficult to separate linen fabric from the
soil. Fast biodegradation effects are linked with the structure of the linen fabric. Linen fabric, as the fibres were not tightly twisted in the yarns. Pictorial representation of linen fabric is shown by the Figure 7.

The FT-IR spectra (Figure 8) of linen fabric are almost the same as for cotton, because the major portion of linen consists of cellulose.

![Figure 8: FT-IR spectra of biodegraded linen samples](image)

The TGA analysis (Figure 9) shows that the weight loss percentage for fabric taken out of soil after two weeks is much less as compared to the fabric that has no contact with the soil. These are the clear signs that sample has been biodegraded after two weeks.

![Figure 9: TGA analysis of biodegraded linen samples](image)

**Flax**

For experimentation non-woven fabric sample of flax/PET blend is taken, so polyester fibres are used to hold the matrix of flax fibres together. Flax fibres before and after degradation period have been analyzed visually, by Axiotech and scanning electron microscope (Figure 10).

Visually analysis indicates minor change in the samples. This is because the mass per unit area of the fabric is high and secondly the fabric is blended with polyester fibres which show no effect of degradation. Microscopic observation indicated that the major portion of cellulose have been degraded by the microorganisms.

The FT-IR spectra of flax fibres (Figure 11) shows intensive absorption in the region 1600–1720 cm⁻¹ which is caused by stretching vibrations of carbonyl groups which arise from polyester present in the blend. Two intensive bands at 2850 and 2918 cm⁻¹ are attributed to deformation vibrations of C–H groups in methyl and methylene groups (\(\text{CH}_3, \text{CH}_2, \text{CH}_2=\text{OH}\)) belonging to cellulose as well as to lignin. The shape of this band is not typical of cellulose, which usually exhibits three-shoulder band at 2900 cm⁻¹ in this region. Moreover, the band at 2900 cm⁻¹ exhibits typical cellulose shape [25].

![Figure 11: FT-IR spectra of biodegraded flax samples](image)
<table>
<thead>
<tr>
<th>Composting</th>
<th>Visually</th>
<th>Axiotech Microscope</th>
<th>SEM</th>
</tr>
</thead>
<tbody>
<tr>
<td>Before</td>
<td><img src="127.png" alt="Image" /></td>
<td><img src="127.png" alt="Image" /></td>
<td><img src="127.png" alt="Image" /></td>
</tr>
<tr>
<td>1 Week</td>
<td><img src="127.png" alt="Image" /></td>
<td><img src="127.png" alt="Image" /></td>
<td><img src="127.png" alt="Image" /></td>
</tr>
<tr>
<td>2 Weeks</td>
<td><img src="127.png" alt="Image" /></td>
<td><img src="127.png" alt="Image" /></td>
<td><img src="127.png" alt="Image" /></td>
</tr>
<tr>
<td>3 Weeks</td>
<td><img src="127.png" alt="Image" /></td>
<td><img src="127.png" alt="Image" /></td>
<td><img src="127.png" alt="Image" /></td>
</tr>
<tr>
<td>4 Weeks</td>
<td><img src="127.png" alt="Image" /></td>
<td><img src="127.png" alt="Image" /></td>
<td><img src="127.png" alt="Image" /></td>
</tr>
</tbody>
</table>

*Figure 10: Flax samples*
<table>
<thead>
<tr>
<th>Composting</th>
<th>Visually</th>
<th>Axiotech Microscope</th>
<th>SEM</th>
</tr>
</thead>
<tbody>
<tr>
<td>Before</td>
<td><img src="image1.jpg" alt="Image" /></td>
<td><img src="image2.jpg" alt="Image" /></td>
<td><img src="image3.jpg" alt="Image" /></td>
</tr>
<tr>
<td>1 Week</td>
<td><img src="image4.jpg" alt="Image" /></td>
<td><img src="image5.jpg" alt="Image" /></td>
<td><img src="image6.jpg" alt="Image" /></td>
</tr>
<tr>
<td>2 Weeks</td>
<td><img src="image7.jpg" alt="Image" /></td>
<td><img src="image8.jpg" alt="Image" /></td>
<td><img src="image9.jpg" alt="Image" /></td>
</tr>
<tr>
<td>3 Weeks</td>
<td><img src="image10.jpg" alt="Image" /></td>
<td><img src="image11.jpg" alt="Image" /></td>
<td><img src="image12.jpg" alt="Image" /></td>
</tr>
<tr>
<td>4 Weeks</td>
<td><img src="image13.jpg" alt="Image" /></td>
<td><img src="image14.jpg" alt="Image" /></td>
<td><img src="image15.jpg" alt="Image" /></td>
</tr>
</tbody>
</table>

*Figure 13: Wool samples*
It is clear from the graph that the cellulose portion of the sample has been degraded at about 350 °C and the polyester one part at 450 °C.

FT-IR spectra (Figure 14) of the wool samples indicated no change in the material after the first week and degradation in material starts after that. The peak at 3067 cm\(^{-1}\) shows the presence of amides, peak at 1631 cm\(^{-1}\) is due to the (stretching of \(\text{CH}_2\text{−NH}_2\)) primary amines. The spectra of first week to fourth week samples show that with the increase in degradation time the representative functional groups of wool start to degrade and convert into biomass, that’s why their absorbance of infrared decreases.

The thermogravimetric analysis of biodegraded wool samples is shown in Figure 15. This analysis shows that wool is very much resistant to the attack of microorganisms and after four weeks the samples are not biodegraded too much.

**Figure 12: TGA analysis of flax samples**

**Wool**

Figure 13 shows wool fabric examined visually, by Axiotech and scanning electron microscope.

There are not prominent changes in the wool fabric samples buried for one to two weeks due to resistance of the wool to the attack of microorganisms (the presence of hydrophobic substances such wool grease). After two week composting the samples start to degrade and at the end of the fourth week the degradation in the fabric is very prominent.

**Figure 14: FT-IR spectra of biodegraded wool samples**

**Figure 15: TGA analysis of biodegraded wool samples**

**3.2 No direct contact with soil**

**Cotton**

The average weight reduction (%w/w) of cotton samples in more hydrophilic and in more hydrophobic bags is represented in Figure 16.

The results after three months show that there is not a great difference in the degradational behaviour of cotton whether it is placed in hydrophilic or hydrophobic bags. Graph shows that during the first month the loss in fabric mass is much higher compared to the second and the third month. This data indicates that biodegradation is faster at first and reach a plato towards the end of reaction/biodegradation.

*Tekstilec, 2014, letn. 57(2), str. 118–132*
Biodegradation of Natural Textile Materials in Soil

Figure 16: Weight loss of cotton fibres

Jute
The results of three month soil burial experiments on jute fibres are represented graphically in Figure 17. The loss in weight of jute fibres is irrespective of the nature or type of the bags in which the fibres are sealed. As most of the portion of jute fibres consist of cellulose, so it follows the same pattern as cotton but has more reduction in mass than cotton during the first month.

Figure 17: Weight loss of jute fibres

Linen
The weight loss percentage of linen fibres after three months is shown in Figure 18. The graph shows that cellulose is attacked by microorganisms in the very first month and weight loss is much higher as compared to the remaining two months.

Figure 18: Weight loss of linen fibres

Flax
The weight loss of flax fibres is indicated (Figure 19), but PET fibres remain intact.

Figure 19: Weight loss of flax fibres

Wool
The degradation behaviour of wool fibres for three months was measured and results are shown in Figure 20. According to Figure 20 wool fibres are much more resistant to attack of microorganisms in hydrophobic bags. The weight loss in negative digits means no weight loss but gain in weight due to the attachment of micro particles of soil. As it is reported before, wool is relatively resisted to microorganisms due to the wax content. In our experiment, the burial time was too short when hydrophobic bags were used. Hydrophobic bags additionally hindered the contact of microorganisms with textile surface. Additional explanations have been found in literature [26]. Wool contains proteins keratin which has some resistance to biodegradation because of the
two reasons. The first reason is the highly cross linked structure of keratin, which has high concentration of sulphur crosslinks; the second reason is that the surface of wool is covered by water repelling membrane and stops the penetration of microorganisms and enzymes into the fibre.

![Figure 20: Weight loss of wool fibres](image)

4 Conclusions

The biodegradation of natural fabric samples (cotton, jute, linen, wool) under the attack of microorganisms present in soil was studied by using standard burial method where textile materials were directly buried and indirect method (not standard method) where textile materials were sawn in bags and exposed to the soil. Visual observations and microscopic methods used reveal that the biodegradation of fibres containing cellulose precede in similar way, the only difference is the time of biodegradation. The fastest biodegradation effects were linked with the structure of the linen fabric, as the fibres were not tightly twisted in the yarns, which lead to better accessibility of material to the microorganisms in soil. The lowest degree of biodegradation occurred when flax/PET blend material was exposed to the conditions in the soil, which is again linked to the structure of the material as from all cellulose materials the mass per unit area of the fabric is the highest and secondly the fabric is blended with polyester fibres which show no effect of degradation. Microscopic observation, FTIR, TGA analysis, indicated that the major portion of cellulose have been degraded by the microorganisms, while PET fibres stayed undamaged. Wool is rather resistant to the attack of microorganisms because of the molecular structure and its surface. These two factors make it quite difficult for the microorganisms present in the soil to penetrate into the structure of wool and biodegrade it.

References

10. TOMŠIČ, Briga, SIMONČIČ, Barbara, OREL, Boris, VILČNIK, Aljaž, SPREIZER, Helena.
1. Tekstilec, 2014, letn. 57(2), str. 118–132


