Biodegradation Behaviour of Different Textile Fibres: Visual, Morphological, Structural Properties and Soil Analyses

DOI: 10.5604/01.3001.0012.7751

Abstract
The biodegradation of fabrics of various types of fibres: cotton (CO), viscose (CV), Modal (CMD), Tencel (CLY), polylactic acid (PLA), polyethylene terephthalate (PET) and polycrylonitrile (PAN) under the attack of microorganisms were studied using the soil burial method for two different burial intervals (1 month and 4 months). As opposed to previous studies, all analyses were simultaneously conducted for both of the buried fabrics and soil samples so as to examine the biodegradation and environmental effect as a whole in the same study. Visual observations, weight losses, Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) were used to examine the biodegradation behaviour. The total organic carbon (TOC), the total number of bacteria and the total number of fungi in the soil samples were studied to understand the soil content during the degradation of the fibres. The study revealed that the cellulose fibres samples changed both physically and chemically even after 1 month. Among the cellulosic fibres, weight losses of modal, cotton, and viscose fibres were close to 90%, showing high degradation, whereas Tencel fibre had the lowest with 60% for a 4 month burial interval. Within the synthetic fabrics, only PLA fabric lost weight.

Key words: biodegradation, textile, fibre, SEM, FTIR, soil, bacteria, fungi.

Introduction
Textile wastes can be classified as either pre-consumer or post-consumer waste. Pre-consumer waste, coming from the textile, fibre and cotton industries, is re-manufactured for the automotive, aeronautic, home building, furniture, mattress, coarse yarn, home furnishings, paper, apparel and other industries [1]. Post-consumer waste is defined as any type of garment or household article made from manufactured textiles that the owner no longer needs and decides to discard. These articles are discarded either because they are worn out, damaged, outgrown, or have gone out of fashion [1]. The U.S. EPA estimates that textile waste occupies nearly 5% of all landfill space [2]. A discarded textile item may be a reason for environmental pollution. Therefore the concept of biodegradation has started to be used widely because of the increasing efforts to prevent environmental pollution.

Biodegradation can be visualised as the method used by nature to recycle waste and to break down organic materials into compounds which can be used as nutrients by other organisms [3]. During biodegradation, numerous microorganisms such as fungi, bacteria, worms, and many other species attack the materials. Through this biodegradation process it is possible to see positive and negative effects together, such as cleaning up wastes, providing nutrients for new life, producing the energy necessary for various biological processes, and also probably harmful waste in nature and the environment.

Biodegradability is significant for materials particularly utilised in any part of daily life [4]. Accordingly it becomes essential to assess their biodegradability and possible damage to the environment. The biodegradability behaviour of polymers depends on some physical and chemical properties. The hydrophilic character, crystalline and amorphous structures, the linearity or branching of polymers, molecular compositions and chemical bonds are determinative elements for the biodegradability of polymeric materials [3, 6, 7]. Due to their primary ease of processing, as well as their high weathering resistance and strength, the production and consumption mass of synthetic fibres is increasing steadily [5]. However, these fibres exhibit high resistance to microbial degradation.

In the related literature, there are some researches on the biodegradation of textile materials, such as cotton, jute, linen, wool, viscose, polyester and the recent polylactic acid fibres [3, 6, 8-12, 22]. Biodegradation was especially evaluated via visual observation, determination of mass loss, and characterising the chemical structure and surface morphology. In some of these biodegradation studies, the microbiological content in soil was evaluated for cotton, acetate, rayon and linen fibres with the total organic carbon values after 28 days’ soil burial [6]. In this research, the biodegradation of fabrics composed of various different raw materials (i.e. cellulose based and synthetic) was examined using the soil burial test. Besides this, the impact of the soil buried test on the microbiological content of soil was also taken into consideration. Several studies on this topic have been already conducted in the last decade or so [7]. However, there has not been a simultaneous study taking into consideration both textile biodegradation and the environmental effect of textile decomposition on soil microflora by determining the amount of the total organic carbon, and the total number of bacteria and fungi together in the same study. The test fabrics were visually observed after two different soil burial intervals, and the weight losses were calculated. The biodegradation tendency of the fabrics was examined by Fourier transform infrared spectroscopy and scanning electron microscopy. As opposed to previous related studies on fibres [3, 6, 8-12, 22], the total organic carbon (TOC), and the total number of bacteria and fungi in the soil samples after burying were quantified in order to analyse the environmental effect of the textile biodegradation.

Experimental
Materials
Seven knitted fabrics of different fibre content and similar structural parameters...
were utilised in this study. No finishing nor dyeing treatment were applied to the fabrics before testing and analysis to better understand the effect of fibre type on biodegradation. Table 1 lists the basic structural parameters of the test fabrics. The mass per unit area values of the fabric samples were determined in accordance with ASTM D3776 [13] before and after each soil burial interval. The fabric thickness was measured using a James Heal Rx:B Cloth Thickness Tester under a 5 g/cm² pressure according to ASTM D1777 [14]. Fabric setting values were determined in accordance with the related standard – ASTM D8007 [15].

Methods

Soil burial testing

The soil used in the experiments had white peat, black peat, water retainer, and a pH (CaCl₂) value of 5.5–6.5. According to ISO 1172 [12], the pH value of the soil must be between 4.0 and 7.5. Test equipment with boxes was prepared for soil burial testing. Two different burial periods simulating a short and comparatively longer interval were selected, such as “1 month and 4 months”. The 4 month interval can be considered as a longer measurement interval based on some of the previous studies. The unburied fabric samples of each fabric were called “control fabrics” and were used for comparison. Three replicates were conducted for each fabric type and burial interval.

Test samples were cut in a square shape (5 cm x 5 cm) for each fabric type and weighed under standard atmosphere conditions (20±2 °C temperature, 65±4% relative humidity). Then the test samples were placed into plexiglass boxes of 1000 ml volume, designed in accordance with ISO 11721-1:2001, and presented in Figure 1 [16]. As seen in Figure 1, small flags were used to arrange the different fabric types, replicates and burial intervals. Figure 2 shows test equipment after burying the fabric samples. Three replicates for each fabric type and burial interval were buried in different boxes, and the surface of the fabric was placed parallel to the top of the soil surface during the burying process.

The soil burial test lasted for 4 months, from June 2017 to October 2017. The temperature and relative humidity in the soil were regularly controlled and kept constant during the experiment by spraying with water. The average temperature and relative humidity values in soil during the tests were 25±5 °C and 95±5%, respectively.

The fabric samples were removed from the soil after 1 month and 4 months. Besides the fabric samples, soil samples in which fabrics were buried were also taken and stored carefully for soil analyses. The fabric samples removed from the soil were lightly rinsed twice with 30/70% v/v ethanol/ water solution, filtered and then dried at ambient temperature. Then weight losses were determined and characterisation analyses conducted. Table 2 lists the sample codes of the test fabrics.

Determination of weight loss after soil burial

After rinsing with ethanol and drying at ambient temperature, the test fabrics were conditioned for 24 hours at standard atmosphere conditions and then weighed. The masses of the buried and control fabrics were compared and the mass loss percentage calculated via the following formula to evaluate biodegradation:

\[
\text{Weight Loss} (\%) = \frac{W_1 - W_2}{W_1} \times 100
\]

Where, W1 and W2 correspond to the initial weight and that after being buried in soil, respectively [16].

Table 1. Basic structural parameters of the fabrics.

| Fabric code | Raw material                  | Courses, cm | Wales, cm | Mass per unit area, g/m² | Fabric thickness (pressure:5 g/cm²), mm |
|-------------|-------------------------------|-------------|-----------|--------------------------|----------------------------------------|
| CMD         | 100% Modac                   | 12.0        | 8.5       | 118.9                    | 0.65                                   |
| CV          | 100% Viscose                 | 12.0        | 9.0       | 156.9                    | 0.61                                   |
| CO          | 100% Cotton                  | 15.0        | 18.0      | 155.6                    | 0.60                                   |
| CLY         | 100% Tencel                  | 12.0        | 8.5       | 118.2                    | 0.77                                   |
| PLA         | 100% Poly (lactic acid)      | 12.0        | 9.0       | 139.6                    | 0.88                                   |
| PET         | 100% Polyethylene terephthalate | 12.0   | 9.0       | 132.8                    | 0.76                                   |
| PAN         | 100% Poly acrylonitrile      | 12.0        | 9.0       | 81.4                     | 0.64                                   |
**Figure 3.** Photographs and SEM micrographs (x500) of cotton and regenerated cellulose fabrics for different burial intervals.

**Morphology study**
The control and buried samples were viewed with a JEOL-JJM 6060 model scanning electron microscope (SEM). SEM images were obtained with an accelerating voltage of 5kV and magnification of 500X. The test samples were prepared by coating gold.

**FTIR analysis**
FTIR spectra of the fabric samples were recorded using a Fourier Transform Infrared Spectrometer (Perkin Elmer Spectrum BX). Spectra of the samples were obtained from 25 scans. Each spectrum was recorded in the range of 600-4000 cm⁻¹ with a resolution of 2 cm⁻¹.

**Determination of total organic carbon in soil**
The total organic carbon (TOC) of the soil samples was measured using TOC-V SSM-5000A in accordance with SM 5310B. In this test method, firstly the soil is dried at 40 °C. The dried soil is then sieved with a 200 mesh sieve and weighed up to
| COTTON FABRIC |  |  |
|---------------|---|---|
| Cotton fabric, control sample | Cotton fabric, 1 month | Cotton fabric, 4 months |
|  |  |  |
| TENCEL FABRIC |  |  |
| Tencel fabric, control sample | Tencel fabric, 1 month | Tencel fabric, 4 months |

**Figure 3.** Photographs and SEM micrographs (X500) of cotton and regenerated cellulose fabrics for different burial intervals (cont.)

30 mg of the total carbon from the sample under the sieve. The total carbon fraction of the device is first burned at 900 °C by the catalytic oxidation method and weighed up to 20 mg of inorganic carbon from the sample. Phosphoric acid is added to the inorganic carbon part of the device and next burned at 200 °C. The total organic carbon content is determined by subtracting the inorganic carbon content from the total carbon content. In the analysis conducted with TOC-V SSM-5000A, CO₂ formed in the total carbon and inorganic carbon part is compared with the generated calibration curve and NDIR (Non Dispersive Infrared Detection) method.

**Determination of the number of total fungi and total bacteria in the soil**

The number of total bacteria and fungi of the soil samples were determined according to ISO 6222 and SM 9610 A-C-D, respectively. These test methods are summarised below.
Figure 4. Photographs and SEM micrographs (X500) of synthetic fabrics for different soil burial intervals.

Table 2. Sample codes of the test fabrics.

| Fabric code | Control sample (not buried, 0 month) | Buried sample for 1 month | Buried sample for 4 months |
|-------------|--------------------------------------|---------------------------|---------------------------|
| CMD         | Modal                                | CMD0                      | CMD1                      | CMD4                      |
| CV          | Viscose                              | CV0                       | CV1                       | CV4                       |
| CO          | Cotton                               | CO0                       | CO1                       | CO4                       |
| CLY         | Tencel                               | CLY0                      | CLY1                      | CLY4                      |
| PLA         | Poly (lactic acid)                   | PLA0                      | PLA1                      | PLA4                      |
| PET         | Polyethylene terephthalate           | PET0                      | PET1                      | PET4                      |
| PAN         | Poly acrylonitrile                   | PAN0                      | PAN1                      | PAN4                      |

Total Bacteria Count (ISO 6222, by Membrane Filtration Method) 10 g soil samples are mixed with 90 ml of 85% sterile NaCl solution (w/v) into 250 ml aliquots and stirred at 200 rpm for 30 min at 21 °C. 1 ml of the eluant is taken, which then undergoes serial dilution in a 9 ml tube containing 0.9% saline (NaCl). After appropriate dilutions, the sample is passed through a membrane filter of 0.45 μm pore diameter, cultured
in a Tryptic Soy Broth medium, and then incubated at 22 °C for 48 hours. Colonies formed as a result of incubation are counted.

- **Total Fungi Count (SM 9610, by Membrane Filtration Method)**
  Serial dilution is handled according to the same procedure as for the total bacteria count. After that stage, by undertaking appropriate dilution, the sample is passed through a membrane filter of 0.45 μm pore diameter, and aureomycin-rose bengal glucose peptone is added to the agar medium, and the sample is incubated at 30 °C for 5 days. Colonies formed as a result of incubation are counted.

**Results and discussion**
The results and discussion part contains two main sections: fabric and soil results. Visual observations, weight losses, SEM micrographs and FTIR analyses are given in the first section and the results of soil analyses are placed in the second.

**Fabric results**

*Visual observation of the fabrics*
The biodegradation of materials such as fibres, films or textiles can be assessed firstly by their physical appearance. Changes in the physical appearance can be observed by the naked eyes. The appearance of fabrics before and after soil degradation are exhibited in Figures 3 and 4 by photographs and SEM micrographs. The changes determined from the visual appearance and handling the fabric samples are listed in Table 3. Slight degradation even after one month was observed for the fabrics made of cotton and regenerated cellulose fibres. This result is in agreement with the findings of Arshad et al. determined for four weeks’ degradation [12]. Colour change and disintegration were determined, where the buried fabrics were evaluated as thinner and more brittle than the control fabrics. A prominent change was recorded for Tencel fabrics after a 1-month burial interval (Figure 5). After 4 months, the fabrics made of cotton and regenerated cellulose fibres were found highly degraded, where the structure and integrity of the fabrics were damaged. In the case of synthetic fabric samples (Figure 6), slight colour changes were recorded, probably due to slight changes in the chemical and morphological structure of the fabric samples. This result is also in accordance with weight losses of the samples, as given in Figure 7.

SEM images show the changes of the test fabrics. After the 4 month soil burial test, the fabrics noticeably degraded, and the integrity and structure of the fabrics nearly collapsed. Fibre breakages of the cotton and Tencel fabrics are prominent, and it is noteworthy that the Modal fibres seem to be inseparable from the soil after the 4 month burial period. It is interesting to note that colonization of the microorganisms existed in the viscose fibre after the 4 month burial period.

**Table 3. Visual observations determined for the fabrics after different soil periods.**

| Fabric code | Observations of fabrics after soil burial period (in comparison with control samples) |
|-------------|-------------------------------------------------------------------------------------|
|             | After 1 month interval | After 4 month interval |
| CMD         | thin                   | small fabric pieces in soil |
| CV          | thin and brittle       | small fabric pieces in soil |
| CO          | brittle and colour change | small fabric pieces in soil |
| CLY         | brittle, colour change, small hole | colour change, brittle |
| PLA         | no change              | slight colour change by taking moisture |
| PET         | no change              | slight colour change with soil particles |
| PAN         | no change              | slight lightening of colour |
Microorganisms in the soil caused a reduction in the weight of the fabrics [12]. Furthermore the breakage and deterioration of the samples facilitate the colonisation of microorganisms into the fabric. For a 1-month burial period, the weight loss in Tencel fabric was highest amongst the other types, meaning that the biodegradation of Tencel fabric is faster for a 1 month burial. Besides this, the lowest weight loss value was determined again for Tencel fabrics amongst the cellulose fabrics for 4 months’ burial, which may be the cause of the lowest biodegradability of Tencel fibres amongst cellulose fibres. At the end of 4 months, it is hard to see Modal, viscose and cotton fabric particles in the soil. Especially Modal and viscose fabrics undergo nearly complete biodegradation after 4 months’ soil burial. Modal is about 50% more hygroscopic, or water-absorbent, per unit volume than cotton [20]. Modal, viscose, cotton and Tencel fibres have cellulose structures because of the different crystallinitis, polymer chains and hygroscopic behaviours, which may be the main reasons for several biodegradation behaviours.

Due to its aromatic structure, PET fibre has excellent physical and mechanical properties compared to aliphatic structures; however, their strong resistance to bacterial or fungal attack results in low degradability under environmental conditions [21]. No weight loss was determined after a 4 month burial period, which is in accordance with the findings of Chen et al. When PLA fibre was examined, although there was a little degradation (1.2% weight loss) after a 4 month burial period, it was clear that biodegradation would not be easy under the soil burial conditions applied in the study. This result is in accordance with the review of Karamanlioglu [22], explaining that no degradation was determined from the weight loss of PLA film when it was buried in soil for 120 days at 25 °C. PAN fibre is highly resistant to damage for months as well as to chemical substances, and no degradation was expected for this fibre. Only a slight lightening in colour was observed after 4 months’ burial.

When the variance analysis results given in Table 4 are examined, it can be seen that the effect of fibre type and the burial interval on weight loss is statistically significant at a 95% confidence level (p<0.05). If the weight loss results are reviewed according to the burial interval, then it is observed that they are divid-
ed into 3 and 4 subgroups according to burial intervals for different fiber types (Table 5). After 1 month’s soil burial, the weight loss results were divided into three subgroups, such as synthetic fibers, viscose and Modal fibres in the second group, and cotton and Tencel fibres in the third group. However, four subgroups were determined for weight loss values after a 4 month burial interval, as explained in detail for Tencel fibres.

**FTIR analysis**

FTIR analysis was performed to examine the effect of the burial test on the chemical structure of the fabrics. FTIR spectra of the control and buried cotton, viscose, Tencel, Modal, PLA, PET and PAN fabric samples are presented in Figures 6-12.

For cotton and regenerated cellulose fibres (viscose, Tencel and Modal), the main peaks at approx. 3330 and 2900 cm\(^{-1}\) absorption bands can be attributed to –OH stretching and C-H stretching vibration of cellulose, respectively [23]. The OH of water absorbed from cellulose is centered at around 1640 cm\(^{-1}\) [24]. The band at 1370 cm\(^{-1}\) corresponds to CH bending. The strong absorption peak at 1020 cm\(^{-1}\) can be indicative of the C-O stretching of cellulose. The smaller peaks at 1200, 1155 and 895 cm\(^{-1}\) absorption bands can be assigned to –OH in plane bending, C-O-C asymmetric stretching and C1 group frequency, respectively [25].

For the cotton fabrics, the intensity of the functional groups decreased in the 4 month burial test. Especially the decrease in the intensities of the peaks at 3300 and 2900 cm\(^{-1}\) is as a result of the rupture of the OH bonds and methyl and methylene of cellulose which occurs due to the attack of the organisms on the cellulose chain [9]. The change in functional groups is basically similar for Tencel fibres. However, C-O stretching at C3 (1053 cm\(^{-1}\) ), C-O stretching at C6 and C-C stretching (1030 cm\(^{-1}\) ) decreased after composting. Additionally the absorption bands at 1105 and 1160 cm\(^{-1}\) decreased due to enzyme degradation [9]. However, for the FTIR spectra of viscose and modal fibres, the intensities of functional groups increased after soil burial testing. Moreover the absorption peaks at 1720, 1645 and 1540 cm\(^{-1}\) became prominent after soil burial testing which can be ascribed to carbonyl groups, absorbed water and amide II, respectively [26-30].
ly [9, 25, 32]. The peak at 1408 cm\(^{-1}\) is attributed to the aromatic ring, which is a stable group, and is a characteristic absorption of PET [9]. The peaks centred at 1505 and 1175 cm\(^{-1}\) can be attributed to the bending vibrations of C-H bonds and C-O groups, respectively [33-34]. After soil burial testing, the FTIR spectra of the PET fabrics were similar to each other, which may be due to the high resistance of these polymers based on their chemical composition.

FTIR spectra of the control and buried PAN fibres are given in Figure 12. FTIR spectra of the control acrylic fabric sample shows many absorption peaks at 2921, 2242, 1737, 1453, 1368 & 1233 cm\(^{-1}\), which can be related to C-H bonds, nitrile bonds, C=O bonds and aliphatic CH group vibrations of different modes in CH, CH\(_2\) and CH\(_3\), respectively [36-37]. The peak indicating nitrile groups showed a noticeable increase after burial testing. However, no significant change was observed in the surface morphology and weight loss values of PAN fabrics after burial testing, as depicted in Figure 7. This may be due to the organic matter or residues of fungi and bacteria on PAN fabrics.

Soil results

Total organic carbon (TOC) analysis

TOC analysis shows the total amount of organic carbon in the soil. Carbon is used as an energy source for anaerobic bacteria and fungi species [34]. Microorganisms are excreted extracellular enzymes that depolymerise polymers because polymers have structures which are long molecules and do not dissolve in water. Thus residues in the degrading material migrate to the soil and cause increments in the total amount of carbons when biodegradation occurs [38]. Carbon containing compounds consumed by bacteria or fungi cause decrements in the total amount of organic carbon in the soil. As seen in Figure 13, TOC values increased after the 1-month burial test, which can be due to the increasing amount of bacteria in the soil, which may contribute to TOC as a result of their being organic matter. After 4 months, The TOC values of soil samples in which Modal and viscose fibres were buried decreased as a result of biodegradation.

For Modal fibres, an increment in the number of total fungi shows that fungi have an important effect on this fibre, be-
sides that of bacteria. The population of bacteria remains constant for the 4 month interval, and it can be said that the maximum level of the amount of bacteria was reached. For viscose fibres, the bacteria population decreased after reaching the maximum level during biodegradation, and after this stage fungi provided biodegradation. However, TOC values did not change noticeably for PLA, PET and PAN fibres, confirming other results that there was no biodegradation for these fibres. Therefore longer soil burial testing will be necessary to evaluate TOC for these fabrics.

**Total number of bacteria and total number of fungi**

The application of organic matter to the soil increases the microbial population, microbial activity and enzyme activity [39]. Free-living bacteria are dependent on soil organic matter as a food source, and their activities increase under favourable conditions. However, some bacteria can secrete enzymes that inhibit the development of fungi. Accordingly the amount of the total bacteria and fungi of the soil samples was examined to determine the biodegradation behaviours of the textile materials. The total bacteria and fungi of the soils are exhibited in Figures 14 and 15. The amount of the total bacteria colony distinctly varies from regenerated cellulose fibres to synthetic fibres (Figure 14), except viscose fibres. This may be due to the chemical substances released during biodegradation. Therefore the chemical substances can generate unfavourable conditions for several types of bacteria to increase in amount. Especially viscose fabrics degraded dramatically and so fast that even less total bacteria and fungi were determined in the soil in comparison with the other fibres. Viscose fibres having the lowest crystallinity amongst other cellulose fibres may be one of the reasons for the highest biodegradation with a lower amount of bacteria and fungi.

Although the total amount of bacteria is less in comparison with the other cellulose fibres, the amount is enough to biodegrade viscose fabric. The highest amount of microorganisms was also determined for Tencel and Modal fibres. A general observation of the total amount of bacteria and fungi in the soil samples shows that there is an increasing tendency in the data after 1 month burial testing. Examined in detail, biodegradation existed extremely quickly, especially after the 1st month, for Modal fibres and the total bacteria increased dramatically after 4 months. Additionally the total fungi increased significantly after 1 month, but remained similar after a 4 month burial period. This may be due to the fact that fungi reached the maximum level of degrading. Additionally fungi may not have found any element in the target fabric buried in soil.

For synthetic fibres, no significant change was recorded in the total amount of bacteria for both the 1 and 4 month burial periods. The total amount of fungi increased for all of the synthetic buried samples after a 1 month burial interval. On the other hand, due to their chemical structure, PAN and PET samples could be easily biodegraded by the bacteria and fungi existing in the soil samples.
Conclusions

In this study, the biodegradability behaviours of knitted fabrics consisting of similar structural parameters and different fibre types, such as cotton, viscose, Modal, Tencel, poly(lactic acid) (PLA), polyethylene terephthalate (PET) and polyacrylonitrile (PAN) were studied in soil burial tests for two different burial intervals. Six common fibre types and one biodegradable synthetic fibre (PLA) were compared by evaluating the changes in the fabrics and soil samples simultaneously.

Morphological characterisation, visual observations and soil analyses indicated that a major portion of biodegradation can be attributed to cotton and regenerated cellulosic fibres, while synthetic fibres stayed generally undamaged for 1 and 4 month burial intervals. The colour of the synthetic fabric samples slightly lightened, possibly due to the enzymes of bacteria. In the case of cellulosic fibres, the fabric colours darkened, especially for cotton and Tencel fabrics because of the residues of the vital bacteria. Morphological observations revealed that the cellulosic fibres began to disintegrate in varying portions, whereas the synthetic fibres kept their original status at the end of the 4 month burial interval. Especially viscose fabrics degraded dramatically, and so fast that even less total bacteria and fungi was determined in the soil in comparison with the other fibres. The biodegradation results of cellulosic fibres showed that it is possible to dispose of these materials by burying because of their high biodegradation.

When the synthetic fibres were examined, it was seen that only slight colour changes were determined for PET, PAN and PLA fibres under the same soil burial conditions as for cellulosic fibres. According to FT-IR and SEM results, no significant degradation in PET, PAN and PLA fabric samples was observed, despite the increase in the amount of bacteria and fungi in the soil samples in which these fibres were buried. Besides this, TOC values did not change noticeably for PLA, PET and PAN fibres, confirming the results that there was no biodegradation for these fibres. Therefore it is concluded that a 4 month burial interval is enough to evaluate the biodegradation behaviour of cellulosic fibres, and that longer soil burial intervals than 4 months are necessary to evaluate and make good comparisons of synthetic fibres.

The production technologies of fibres, the amount of chemicals used and pollution occurring during fibre production are also as important as the level of biodegradation. The results obtained can inspire to make a comparison between the biodegradation level and production to optimise the conditions of supplying environmentally friendly polymers.

Acknowledgements

The authors are grateful to Dokuz Eylul University for supporting this research as a scientific project (2017.KB.FEN.034), Dr. Yasemin SEKI for her assistance during tests, and to Prof. Dr. Delia Sponza for her invaluable help and suggestions during soil analyses.

References

1. Yogita Agrawal, Shyam Barhanpurkar and Ajay Joshi, Recycle Textiles Waste, p:1-9. https://static.fibre2fashion.com/ArticleResources/PdfFiles/68/6798.pdf, June 2018
2. Council for Textile Recycling, www.weardonaterecycle.org/index.html, June 2018
3. Ranu S, Pichandi S, Parveen S, Fanguero R. Biodegradation Studies of Textiles and Clothing Products, Roadmap to Sustainable Textiles and Clothing, edited by Muthu, S.S., Textile Science and Clothing Technology 2014; DOI: 10.1007/978-981-287-110-7-4.
4. Tokwara Y, Calabia BP, Ugwu CU, Aiba S. Biodegradability of Plastics. International Journal of Molecular Sciences 2009; 10(9): 3722-3742.
5. Kelsey DR, Scardino BM, Grebowicz JS, Chuah HH. High Impact, Amorphous Terephthalate Copolyesters of Rigid 2,2,4,4-Tetramethyl-1,3-cyclobutenediol with Flexible Diols. Macromolecules 2000; 33(16): 5810-5818.
6. Chung Hee Park, Yun Kyung Kang Seung Soon Im. Biodegradability of Cellulose Fabrics, Journal of Applied Polymer Science 2004; 94: 248-253.
7. Ledin M. Accumulation of metals by microorganisms – processes and importance for soil systems. Earth-Science Reviews 2000; 51(1-4): 1-31.
8. Arutculiev J, Sudhakar M, Arkatkar A et al. Biodegradation of polyethylene and polypropylene. Indian J Biotechnol 2008; 7: 9-22.
9. Li L, Frey M, Browning KJ. Biodegradability Study on Cotton and Polyester Fabrics. Journal of Engineered Fibers and Fabrics 2010; 5(4): 42-53.
10. Solazzo C, Dyer JM, Clerens S, Plowman J, Peacock EE, Collins MJ. Protemic evaluation of the biodegradation of wool fabrics in experimental burials. International Biodeterioration & Biodegradation 2013; 80: 48-59.
11. Weng YK, Jin YJ, Meng QY, Wang L, Zhang M, Wang YZ. Biodegradation behaviour of poly(butylene adipate-co-terephthalate) (PBAT), poly(lactic acid) (PLA), and their blend under soil conditions. Polymer Testing 2013; 32(5): 918-926.
12. Arshad K, Skifvars M, Vera V, Julija V. Biodegradation of natural textile materials in soil. Tekstilac 2014; 57(2): 118-132.
13. ASTM D3776/D3776M-09a. Standard Test Methods for Mass Per Unit Area (Weight) of Fabric, 2017.
14. ASTM D1777 – 96. Standard Test Method for Thickness of Textile Materials, 2015.
15. ASTM D8007-15. Standard Test Method for Wale and Course Count of Welt Knitted Fabrics.
16. ISO 11721-1:2001. Textiles – Determination of resistance of cellulose-containing textiles to micro-organisms – Soil burial test – Part 1: Assessment of rot- retardant finishing.
17. Yaacob ND, Ismail H, Ting SS. Soil Burial of Poly(lactic acid)/Paddy straw powder biocomposite. Bioresources 2016; 11(1): 1255-1269.
18. Luckachan GE, Pillai CKS. Biodegradable Polymers – A Review on Recent Trends and Emerging Perspectives. Journal of Polymers and the Environment 2011; 19(3): 637-676.
19. http://www.lenzing-fibers.com/en/tencel/hygienic/, 03 March 2018.
20. Jeyakodi Moses J, Gnanapriya K. Properties of Modal Fabric after Formic Acid Treatment. JECET, March 2016, Sec. A; 5 No.2, 31-44.
21. Yiwong Chen, Lichang Tan, Lie Chen; Yan Yang, Xiaofeng Wang. Study on biodegradable aromatic/aliphatic copolyesters. Braz. J. Chem. Eng. 2008; 25 2 São Paulo, http://dx.doi.org/10.1590/S0104-66322008000200011.
22. Karamanlioglu M, Prestosi R, Robson GD. Abiotic and biotic environmental degradation of the bioplastic polymer poly (lactic acid): A review. 2017; 37: 122-130.
23. Kılıç AÇ, Atagür M, Ozdemir O, Sen I, Kupçukoğan N, Sever K, Sarıkanat M, Seki Y. Manufacturing and characterization of vine stem reinforced high density polyethylene composites. Composites Part B 2016; 91: 267-274.
24. Carrillo F, Colom X, Sunol JJ, Saurino J. Structural FTIR analysis and thermal characterisation of lyocell and viscose-type fibres. European Polymer Journal 2004; 40(9): 2229-2234.
25. Seki Y,Seki Y, Sarıkanat M, Sever K, Durmuşkaya C, Bozaci E. Evaluation of linen fibre as a potential reinforce material for polymer composites. Journal of Industrial Textiles 2016; 45(6): 1221-1238.
26. Oh SY, Yoo DI, Shin Y, Seo G. FTIR analysis of cellulose treated with sodium hydroxide and carbon dioxide. Carbohydrate Research 2005; 340(3): 417-428.
27. Zhou D, Zhang L, Guo S. Mechanisms of lead biosorption on cellulose/chitin beads. *Water Research* 2005; 39(16): 3755-3762.

28. Yang G, Zhang L, Liu Y. Structure and microporous formation of cellulose/silk fibroin blend membranes: I. Effect of coagulants. *Journal of Membrane Science* 2000; 177(1-2), 153-161.

29. Tomsic B, Simoncic B, Orel B, Vilcnik A, Spreizer H. Biodegradability of cellulose fabric modified by imidazolidinone, Carbohydrate polymers 2007; 69(3): 478-488.

30. Tomsic B, Klemencic D, Simoncic B, Orel B. Influence of antimicrobial finishes on the biodeterioration of cotton and cotton/polyester fabrics: Leaching versus bio-barrier formation. *Polymer Degradation and Stability* 2011; 96(7): 1286-1296.

31. Xiao X, Chen F, Wei Q, Wu N. Surface modification of polyester nonwoven fabrics by Al₂O₃ sol–gel coating. *Journal of Coatings Technology and Research* 2009; 6: 537.

32. Donelli I, Taddei P, Smet PF, Poelman D, Nierstrasz VA, Freddi G. Enzymatic Surface Modification and Functionalization of PET. A Water Contact Angle, FTIR, and Fluorescence Spectroscopy Study. *Biotechnology and Bioengineering* 2009; 103 (5): 845-856.

33. Huang W, Jang J. Hydrophilic Modification of PET Fabric via Continuous Photografting of Acrylic Acid (AA) and Hydroxyethyl Methacrylate (HEMA). *Fibers and Polymers* 2009, 10 (1), 27-33.

34. Kamel MM, El Zawahry MM, Helmy H, Eid MA. Improvements in the dyeability of polyester fabrics by atmospheric pressure oxygen plasma treatment. *The Journal of the Textile Institute* 2011; 102 (3): 220-231.

35. Pamula E, Blazewicz M, Paluszkiewicz C, Dobrzynski P. FTIR study of degradation products of aliphatic polymers–carbon fibres composites. *Journal of Molecular Structure* 2001; 596(1-3): 69-76.

36. Wangxi Z, Jie L, Gang W. Evolution of structure and properties of PAN precursors during their conversion to carbon fibers. *Carbon* 2003, 41(14): 2805-2812.

37. Farsani RE, Raissi S, Shokuhfar A, Sedghi A. FT-IR Study of Stabilized PAN Fibers for Fabrication of Carbon Fibers. *International Journal of Mechanical and Mechatronics Engineering* 2009; 3(2): 161-164.

38. Alshehrei F. Biodegradation of Synthetic and Natural Plastic by Microorganisms. *Journal of Applied & Environmental Microbiology* 2017; 5(1): 8-19.

39. Fontaine S, Mariotti A, Abbadie L. The priming effect of organic matter: A question of microbial competition. *Soil Biology and Biochemistry* 2003; 35(8): 837-843.

Received 09.10.2018 Reviewed 06.12.2018