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In-Compost Biodegradation of PLA Nonwovens

Abstract

Presented in this work are the results of an investigation into the biodegradation of selected nonwoven materials made of commercial poly[(DL)-lactide] – PLA 6252D supplied by Nature Works® LLC, USA. The biodegradation was examined under laboratory conditions, simulating composting by the mass-loss method at constant process parameters: temperature $t = 58 \pm 2$ °C, pH 7 and inoculum humidity $W = 52.6\%$. The nonwovens examined, with a surface density of about 60 g/m², revealed a varied crystallinity degree in the range of 10.4 - 35.6% and fibre diameter of 8.2 - 10.9 μm. The nonwovens were formed by the spun-bond method from a melt at 211 - 213 °C on a laboratory stand. The commercial PLA 6252D was also examined. The polymer PLA 6252D and nonwoven materials made thereof are entirely biodegradable in the research environment applied, and after 16 weeks of biodegradation weight loss in the materials reaches 100%.

Key words: PLA, nonwovens, biodegradation, composting.

Introduction

Extensive investigations of new environmentally-friendly materials proceed along with the quick marketing and commercialisation of biodegradable polymers and composites, all of which may add to ecologists' concept of "green chemistry" [1]. Thanks to unique properties, biodegradable resins are suited to composting, which is a natural low-energy utilisation process without hazardous emissions. In the course of biological processing, the biodegradable polymers are disintegrated and mineralised under the action of such factors as temperature, humidity and microorganisms (bacteria and fungi). Carbon dioxide, methane, water and biomass are amongst the degradation products. The manufacture of resins that combine the properties of typical classical thermoplastics with biodegradability fits very well to the concept of a consistent development [2].

The growing interest shown in biodegradable materials triggered an investigation aimed at defining the speed and degree of their biodegradation in the environment under the action of microorganisms like bacteria, protozoans, fungi and algae. In the environment, the biodegradation process is influenced by several factors, both internal and external [3 - 6], which may act synergistically, either accelerating or slowing down the polymer decomposition. Examples are a temperature too high or too low for the growth of microorganisms, inadequate pH and a content of water adequate to promote the migra-

tion of microorganisms appearing in the inoculum used [7].

In recent years there has been growing interest in biodegradable materials that, unlike crude-derived synthetic materials, undergo decomposition in the environment [6 - 10]. These are primarily natural materials like poly[(D,L)-lactide] – PLA, which show good physical-mechanical and physical-chemical properties as well as susceptibility to biodegradation. A chance exists to tailor their lifetime by modifying the structure of the polymer chain. Apart from the polymer structure, the biodegradation process is also influenced by other factors like micro-flora appearing in the testing medium, process conditions and the parameters at which the materials were made [11, 12].

Poly(lactide) (PLA) is amongst the main entirely biodegradable materials, comprising a 40% share of all commercial biodegradable polymers. It is a linear aliphatic polyester derived from lactic acid, often labelled "double green" since it is both biodegradable and is made from renewable resources.

Thanks to its thermoplasticity, good mechanical strength, biodegradability, and both the non-toxicity of the resin itself and its decomposition products, PLA has found a wide avenue of uses. Advances in the manufacturing of PLA leading to products with properties resembling classical resins like poly(ethylene terephthalate) (PET) or polystyrene (PS) foster its use in applications in which the biodegradation susceptibility can be exploited

in a rational way. This is the reason why the estimation and testing of the PLA biodegradation ability is of profound importance [13 - 15].

The aim of the present work was to assess the susceptibility of textile materials made of the 6252D PLA to biological decomposition induced by aerobic microorganisms, for which the compost medium is an abode. Examined were nonwoven fabrics with a surface density of about 60 g/m², crystallinity degree varying in the range of 10.4 to 35.6% and a fibre diameter from 8.2 to 10.9 μm.

Modern polymeric materials used in agriculture are mainly polyethylene film and polypropylene nonwoven applied in plantations. The durability of materials enhanced by photo-stabilisers, which protect against UV radiation, is a challenge. With a usefulness of 1 - 3 years, the materials degrade under moderate climatic condition over many years. In contrast, new materials made of biodegradable polymers degrade much faster in soil without its contamination. A low surface density but with high tenacity is expected of the materials. The textiles for agriculture use investigated are modern materials with high mechanical properties, and are non-hazardous to the environment.

The biodegradation process was performed under laboratory conditions simulating aerobic composting, during which the mass loss of the samples was measured in accordance with the testing procedure prepared. Constant process

parameters were applied: temperature 58 ± 2 °C, pH 7 and humidity of the medium $W = 52.6\%$.

Materials and methods

Characteristic of the PLA and nonwoven fabrics subjected to in-compost decomposition

Poly(D,L-lactide) – PLA marked 6252D, supplied by Nature Works® LLC (USA), and textile materials made thereof were tested in respect of biodegradation. 6252D polymer is a thermoplastic fibre-grade resin derived primarily from annually renewable resources. Available in pellet form, PLA 6252D is designed for spun-bond products using conventional spun-bond equipment. PLA 6252 can be converted into a broad range of fibre products. Potential applica-

tions for PLA polymer 6252D include wipes, geotextiles, hospital garments, absorbent pad liners, personal hygiene products and agriculture/horticultural products. The polymer was dried in a dryer made by Piovan (USA) at a temperature of 80°C and dew point of 50 °C. The drying was carried out until the water content in the polymer dropped below 50 ppm. The time of the drying was 6 hours.

Table 2 presents the results of differential scanning calorimetry with the following parameters: the glass transition temperature (T_g), the temperature of cold crystallisation (T_{cc}), the temperature of crystallisation from the melt (T_c), the melting temperature (T_m), the specific heat (ΔC_p), the enthalpy of cold crystal-

lisation (ΔH_{cc}) and the enthalpy of melting (ΔH_m).

A rather strong endothermic effect of the melting appeared in the first heating, which indicates the polymer's ability to crystallize. The polymer does not crystallise from the quenched melt. Only slight thermal effects appear during the second heating related with cold crystallisation and melting, which is an indication of low crystallisation kinetics. A glass transition temperature in the first heating higher than that observed during quenching as well as in the second heating is related to the remarkable crystallinity of the initial sample and its nearly amorphous condition in the remaining phases. The low crystallisation kinetics of the polymer observed do not imply that the polymer is going to crystallise equally slowly when subjected to a mechanical force (orientation crystallization) or when in contact with solvents. The crystallinity of the initial sample may result from the thermal pre-processing at an elevated temperature and adequately prolonged time or from long storage at a temperature above the glass transition point.

The PLA 6252D polymer was also analysed chromatographically (GPC/SEC) to estimate its molecular mass $M_w = 45,800$ g/mol, $M_n = 59,120$ g/mol and polydispersity $M_w/M_n = 1.29$, and in respect of its physical-chemical properties such as the inherent viscosity (1.04 dl/g), ash content (0.03% wt.) and acidic group content (24.5 mval/kg).

The nonwoven fabrics marked: 37/12/1, 37/12/3, 37/12/5 and 37/12/8 were also analysed by GPC/SEC and DSC and tested in respect of their physical-mechanical properties. **Table 3** presents results of the physical-mechanical testing of the textile materials examined.

Table 4 shows the average molecular mass M_w , M_n and polydispersity M_w/M_n of the initial PLA nonwoven fabrics of defined surface density and crystallinity degree in the range of 8.9 - 31.2%.

Table 5 presents results of the examination of the thermal properties of the nonwoven fabrics.

Four nonwoven fabrics marked 37/12/1, 37/12/3, 37/12/5 and 37/12/8, with a surface mass of 58.2, 59.7, 55.2 and 58.4 g/m², were selected for the assessment of in-compost biodegradability.

Table 1. Characteristic of polymer PLA 6252D (as quoted by the producer).

Polymer	Density, g/cm ³	Content of isomer D, %	Melt flow index MFI, g/10 min	Melting temperature, °C	Glass transition temperature, °C
PLA 6252 D	1.08	1.4	70 - 85	160 - 170	55 - 60

Table 2. Thermal properties of polymer PLA 6252D.

Polymer		Heating					Quenching				
		T_g , °C	ΔC_p , J/gK	T_{cc} , °C	ΔH_{cc} , J/g	T_m , °C	ΔH_m , J/g	T_g , °C	ΔC_p , J/gK	T_c , °C	ΔH_c , J/g
PLA 6252D	I heating	71.0	0.175	-	-	172.4	49.2	-	-	-	-
	II heating	59.8	0.465	140.1	2.07	166.7	2.10	54.0	0.482	-	-

Table 3. Physical-mechanical properties of the nonwoven PLA fabrics.

Symbol of nonwoven	Surface mass, g/m ²	Thickness, mm	Fibre diameter, µm	Breaking force, N		Tearing resistance, N		Elongation at break, %	
				along.	across.	along.	across.	along.	across.
37/12/1	58.2	0.32	10.9	23.5	22.2	24.4	16.6	1.63	2.67
37/12/3	59.7	0.33	9.46	38.9	22.9	21.2	20.4	2.04	1.73
37/12/5	55.2	0.30	8.38	39.4	17.8	16.4	19.4	1.97	1.35
37/12/8	58.4	0.32	8.18	31.9	16.0	12.1	13.2	1.55	1.04

Table 4. PLA nonwoven fabrics with varied surface mass and crystallinity degree.

Symbol of nonwoven	Surface mass, g/m ²	Crystallinity degree, %	Value of M_n , M_w in PS units		
			M_n , g/mol	M_w , g/mol	M_w/M_n
37/12/1	58.2	8.9	64,200	110,100	1.7
37/12/3	59.7	13.0	62,700	112,100	1.8
37/12/5	55.2	23.5	61,300	105,700	1.7
37/12/8	58.4	31.2	62,100	110,300	1.8

Table 5. Thermal properties of nonwoven fabrics made of polymer PLA6252D.

Symbol of nonwoven	Heating					
	T_g , °C	ΔC_p , J/gK	T_{cc} , °C	ΔH_{cc} , J/g	T_m , °C	ΔH_m , J/g
37/12/1	60.8	0.46	94.4	30.7	170.7	40.2
37/12/3	60.3	0.39	89.7	27.2	170.4	41.0
37/12/5	59.6	0.25	83.3	20.2	171.2	45.1
37/12/8	58.5	0.19	75.9	15.5	166.5	48.6

The thickness of the fabrics was about 0.3 mm and the diameter of the fibres contained therein was in the range of 8.18 - 10.9 μm . The values of the surface mass that characterise the textile materials ensue from the specific conditions of spun-bond forming of the nonwovens. The nonwovens were prepared at a calendar temperature of 66 - 105 °C and are characterised by a varying crystallinity degree in the range of 8.9 to 31.2%. They were made by the Team of Synthetic Fibres at IBWCh.

Spinning of the nonwoven fabrics

The nonwoven fabrics were made from commercial PLA 6252D by the melt spun-bond technique using a laboratory stand constructed by "Polmatex-Cenaro", Łódź, Poland.

Manufacturing parameters:

- temperature of the melt in the spinning head: 212 ± 1 °C
- take-up speed: 2.9 - 6.8 m/min
- extrusion through-put: 42.4 - 101.8 g/min
- temperature of the calendar: 60 - 105 °C
- pressure of air in the feeding chambers: 1500 - 2000 Pa
- vacuum in the soaking chamber: from -548 to -334 Pa.

Examination of the biodecomposition degree of the PLA polymer and nonwoven fabrics

The biodegradation of the PLA 6252D polymer and spun-bond fabrics was tested at the accredited Laboratory of Biodegradation at IBWCh¹ according to the testing procedure "Determination of the degree of disintegration of plastic materials and textiles under simulated composting conditions in a laboratory-scale test". The procedure is based on PN-EN/ISO Standards [16 - 18]. Examination of the nonwoven fabrics' biodegradation by the method of mass loss was accomplished on a laboratory scale under aerobic conditions simulating intensive composting. Compost after effervescent ripening originating from an industrial compost prism (Municipal Services Company of the city of Łódź) was used as inoculum. The quality of the compost satisfied national requirements concerning microbiology, parasitology and physical-chemical parameters. Microbiological activity of the compost used was determined, revealing a value of 1.6×10^8 cfu/cm³. The amount of mi-

croorganisms in the medium should not fall below 10^6 cfu/cm³. Prior to the composting, the samples tested were photographed, weighted and put into a test reactor, and then incubated at constant temperature (58 ± 2 °C), with pH 7 of the inoculum and 52.6% moisture of the medium. The parameters of the biodegradation process were controlled at defined time intervals (1, 4, 8, 12, 16, 20 & 24 weeks). Moisture was supplemented with water to the initial value. After a defined incubation time, the samples were dried to constant weight and the mass loss was estimated. For each of the samples, testing was made in 3 repetitions. The resulting dispersion of the biodegradation test was below 10%.

Analytical methods

Assessment of biological activity (total number of microorganisms) – estimation of the total number of microorganisms in the inoculum used, was done at the accredited Laboratory of Microbiology at IBWCh¹ according to the testing procedure: "Estimation of total number of microorganisms in compost and soil" [19].

Physical-mechanical properties were estimated at the accredited Laboratory of Metrology at IBWCh¹ using Instron 5544 (England) apparatus, according to Polish-ISO Standards. Elongation at break, tenacity, in two directions (along and crosswise) - PN-EN 29073-3:1994, tear resistance - PN-EN ISO 9073-4:2002, mass per unit area PN-EN 29073-1:1994, thickness (PILMED-64, Poland) - PN-EN ISO 9073-2:200, Fibre diameter by LANAMETR MP 2 (Poland) - PN-86/P-04761.08.

Rheological properties of polymer, melt flow index (MFI) - according to method A in accordance with the DYNISCO (USA) Polymer Test standard (with a spinneret hole of 2 mm, at a temperature of 190°C).

The water content in the polymer was determined by the coulometric Karl Fischer method with DL39X apparatus, made by Mettler Toledo.

The crystallinity degree of the textile materials was estimated by the method of differential scanning calorimetry (DSC). The thermographic assessment of fibre crystallinity consists, in general, in measurement of the melting heat and calculation of the crystallinity degree from the formula:

$$\kappa = \frac{\Delta H_m}{\Delta H^0}$$

where:

ΔH_m - melting enthalpy of the fibre,
 ΔH^0 - melting enthalpy estimated by extrapolation for fibre material with 100% crystallinity.

$$\kappa_{PLA} = \frac{\Delta H_m - \Delta H_c - \Delta H_c}{\Delta H^0_{PLA} \cdot x_{PLA}}$$

where:

ΔH_{cc} - enthalpy of "cold crystallization" of the fibre material,
 ΔH_{rc} - enthalpy of the re-crystallization of the fibre material,
 ΔH^0_{PLA} - melting enthalpy estimated by extrapolation for 100% crystalline PLA, with the adoption of the value of 106 J/g,

x_{PLA} - mass fraction of PLA in the fibre material.

$$\kappa_{iPP} = \frac{\Delta H_m - \Delta H_c}{\Delta H^0_{iPP} \cdot x_{iPP}}$$

where:

ΔH^0_{iPP} - melting enthalpy estimated by extrapolation for 100% crystalline iPP, with the adoption of the value of 209 J/g

x_{iPP} - mass fraction of iPP in the fibre material.

Estimation of the crystallinity degree by measurement of the melting heat is a quick and accurate method. The precondition of obtaining reliable results is the adoption of methodical assumptions adequate to the microstructure of the fibre tested. Thermal analysis was carried out by means of differential scanning calorimetry (DSC) using Diamond (Perkin Elmer, Germany) apparatus. The first and second heating scan and the first cooling scan for the polymer were performed in the temperature range of -70 - 170 °C. The sample was scanned at a heating rate of 10 °C/min. The thermal stability of the polymer was investigated by means of thermal gravimetric analysis (TGA) using a HI-REST TGA 2950 Thermogravimetric Analyser in nitrogen atmosphere, with a heating rate of 20 K/min in the temperature range of 20 - 600 °C.

The distribution of molecular weight and polydispersity of the polymer and nonwoven fabrics were analysed by GPC/SEC (gel permeation chromatography/size exclusion chromatography).

The gel chromatography system is equipped with an isocratic pump - HP 1050 (Hewlett-Packard), a differential

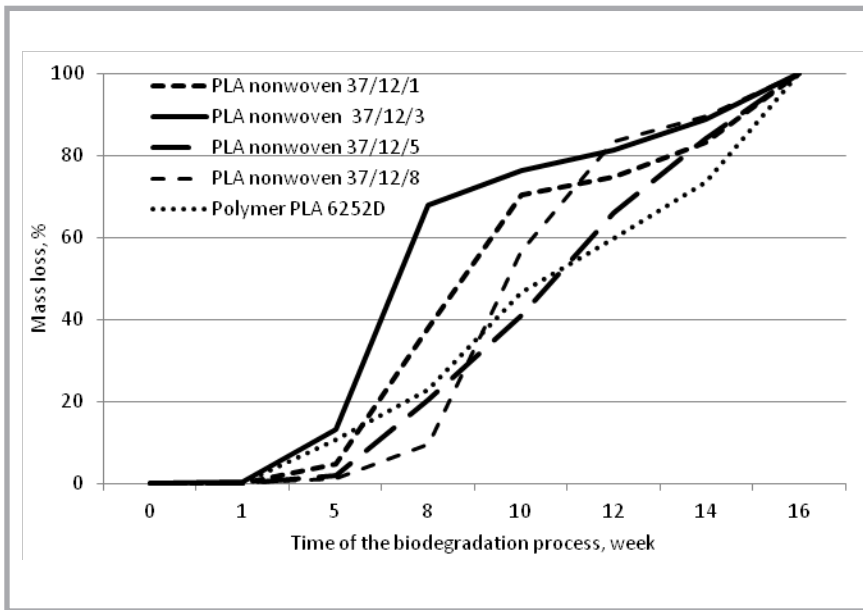


Figure 1. Mass loss in the polymer PLA 6252D and PLA nonwovens marked 37/12/1-37/12/8 during the biodegradation process in a compost medium.

refractometric detector - HP 1047 (Hewlett-Packard), an autosampler, a degasser of the phase tested - DG-700 (Viscolec), and a thermostat. The test results are analysed by specialised software - PL Caliber GPC Software 9 Polymer Laboratories Ltd.).

Parameters of the GPC/SEC analysis:

- solvent (mobile phase): chloroform
- column: Plgel Mixed C, 300 mm, 5 µm (Polymer Laboratories Ltd.)
- column temperature: 35 °C
- flow speed: 0.7 ml/min
- injection volume: 100 µl
- calibration standards: polystyrene with a molecular mass from 580 to 3993000 and a polydispersity in the range of 1.04 to 1.14 (Polymer Laboratories).

The average molecular mass (Mw/Mn) was calculated by the method of universal calibration with the following constants in the Mark Houwink equation: for PS: $a = 0.794$, $K = 0.0049$ [21], and for PLA: $a = 0.759$, $K = 0.0153$ [22].

Spectrophotometric analysis in infrared FTIR

The tested preparations were used in form of tablets in KBr with concentration: 1mg of the tested material in 300mg of KBr. Reading accuracy of the wave numbers of the characteristic bands was $\pm 1 \text{ cm}^{-1}$.

The spectrophotometric IR spectrum was drawn by the use of the apparatus Genesis Series FTIR™, Unicam Co. The device is equipped with the specialized software WinFIRST of ATI Mattson Co USA. Operational parameters of the device: measurement range - 4000 - 500 cm^{-1} , resolution - 4.0, number of scans collected from background and spectrum - 16.

Observation of the nonwoven by scanning electron microscopy (SEM) - Quanta 200 (FEI, USA). The observation was made in a low vacuum, in a natural state without spraying at an electron-accelerating voltage of 5 KV and magnification of 500×.

Results and comments

biodegradation of polymer PLA 6252D and nonwoven fabrics made thereof

Presented herein are the results of biodegradation tests of the polymer PLA 6252D and spun-bond nonwovens made thereof. Decomposition was accomplished in aerobic compost. The total amount of microorganisms in the medium was first determined, revealing a value of $1.6 \times 10^8 \text{ cfu/cm}^3$.

The dependence of mass loss in the PLA 6252D polymer and nonwovens on the time of the biodegradation process is presented in **Figure 1**.

From the results of the decomposition testing, it ensures that the polymer PLA 6252D and nonwoven fabrics made thereof examined undergo complete biodegradation in a compost medium in the presence of aerobic microorganisms.

The biodegradation of the samples tested lasted no longer than 16 weeks, after which time a 100% mass loss was found. Molecular properties of the polymer PLA 6252D and nonwoven fabrics were analysed to determine the average molecular masses Mn and Mw (weight average and number average molecular mass). The molecular characteristic is compiled in **Table 6**.

The results in the above Table show that the average molar mass of the materials declines significantly along with the proceeding biodegradation. Prior to biodegradation, the average molar mass Mw was 115500 g/mol for PLA and in the range of 105700 to 112100 g/mol for the nonwovens. After 10 weeks of biodegradation, the lowest values appeared in nonwoven 37/12/5 (Mw = 1600 g/mol), with 23.5% crystallinity.

The molecular characteristic of the polymer and nonwoven fabrics is presented in

Table 6. Change in the average molecular mass of polymer PLA 6252D and nonwoven fabrics made thereof.

Symbol of nonwoven	Before biodegradation			Time of biodegradation, week								
				1			5			10		
Value of Mn, Mw in PS units	Mn	Mw	Mw/Mn	Mn	Mw	Mw/Mn	Mn	Mw	Mw/Mn	Mn	Mw	Mw/Mn
Polymer PLA 6252D	66,800	115,500	1.7	33,900	65,100	1.9	4,400	7,000	1.6	2,700	4,300	1.6
Nonwoven 37/12/1	64,200	110,100	1.7	50,200	92,100	1.8	12,600	27,300	2.2	2,200	3,500	1.6
Nonwoven 37/12/3	62,700	112,100	1.8	50,600	90,400	1.8	19,400	40,800	2.1	1,200	2,400	1.9
Nonwoven 37/12/5	61,300	105,700	1.7	48,900	93,000	1.9	21,800	46,300	2.1	400	1,600	3.8
Nonwoven 37/12/8	62,100	110,300	1.8	48,700	92,100	1.9	22,300	50,400	2.3	600	2,900	5.2

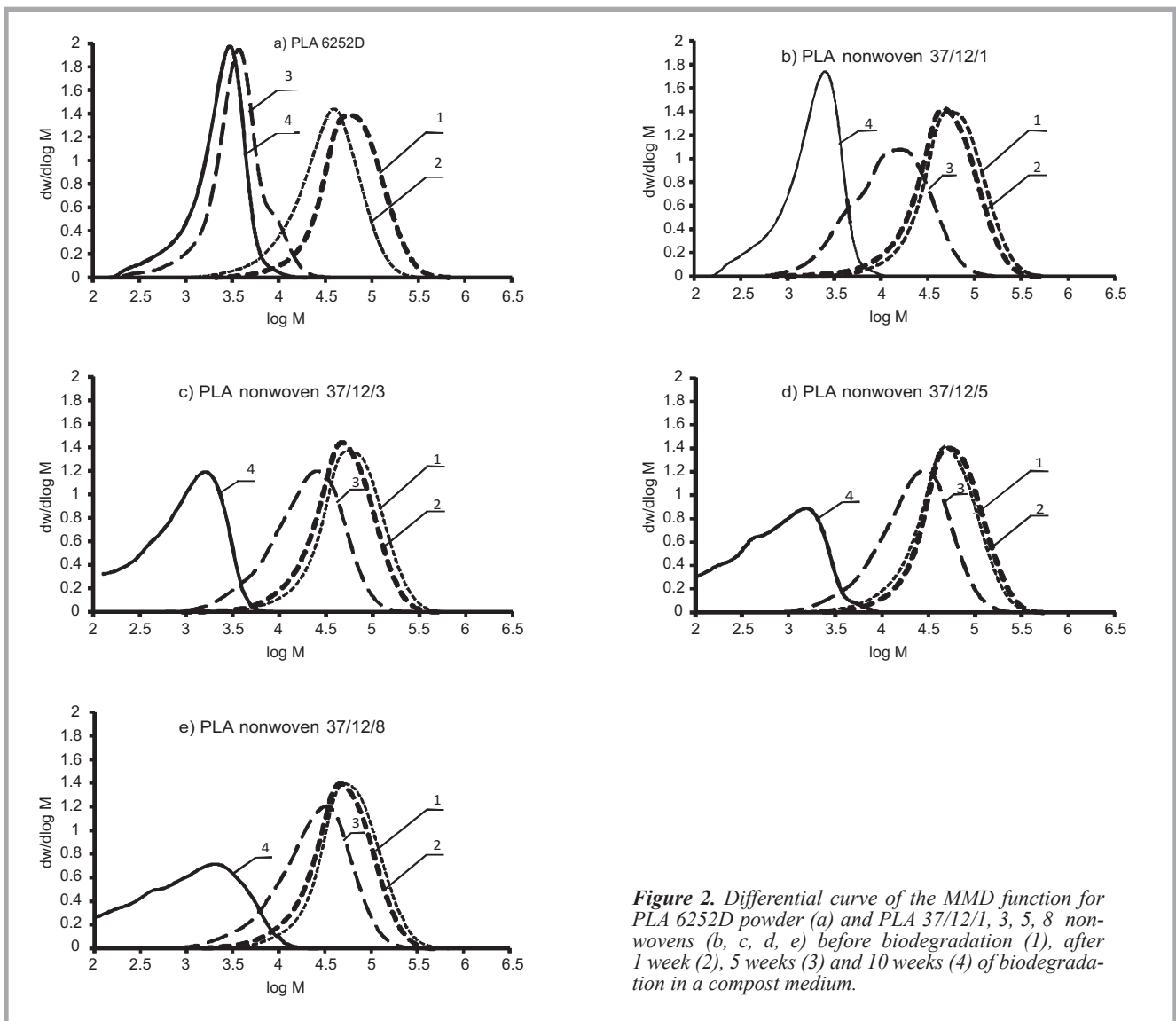


Figure 2. Differential curve of the MMD function for PLA 6252D powder (a) and PLA 37/12/1, 3, 5, 8 nonwovens (b, c, d, e) before biodegradation (1), after 1 week (2), 5 weeks (3) and 10 weeks (4) of biodegradation in a compost medium.

Figure 2 as differential function MMD (molecular mass distribution).

After 1 week of biodegradation, the average molecular mass M_w dropped the most in the initial polymer PLA 6252D to a value of 65,100 g/mol, while the nonwovens were much less prone to biodegradation, expressed by the reduction of M_w from ca. 110,000 g/mol to ca. 90,000 g/mol at the same time. This is illustrated by the remarkable difference in MMD curves No. 1 and No. 2 for PLA 6252D and the slight deviation between the same curves for the nonwoven, which implies that the crystalline structure of the nonwoven effectively retards the biodegradation process. The impact of the crystallinity degree upon the run of the biodegradation can be best seen after 5 weeks of the process: M_w for the PLA 6252D polymers is down at 7,000 g/mol, while for the nonwovens with 8.9 to

31.2% of the crystallinity degree it drops to only 27,300 g/mol and 50,400 g/mol, respectively.

Figures 2.a and **2.b** present the remarkable reduction in the M_w value of the PLA 6252D polymer and 37/12/1 nonwoven with the lowest crystallinity degree; the MMD curves are clearly shifted from position 2 to 3.

After 10 weeks of biodegradation, the average molecular mass M_w of both the PLA 6252D and nonwovens appears in the range of a few thousands of g/mol.

In that phase of the process, it could be observed that the polydispersity (M_w/M_n) rises from 1.6 to 5.2 with the higher crystallinity degree, which can be seen in **Figure 2**, where the shape of MMD curve No. 4 changes along with an increase in the crystallinity degree. The amount of

shorter macromolecules increases with an increase in the distribution of the molecule size.

Polymer PLA 6252D and nonwoven fabrics made thereof were analysed by IR spectrophotometry, which enabled the determination of the characteristic functional groups appearing in the samples analysed. Structural changes proceeding in the samples examined were followed in the course of long-lasting in-compost biodegradation at $58 \pm 2^\circ\text{C}$.

Figure 3 present structural changes revealed by IR analysis that proceed in the polymer PLA 6252D and nonwoven fabrics after the biodegradation in comparison with the initial condition.

Two characteristic regions of transmission can be seen in the FTIR spectra of the PLA polymer and nonwoven fabrics:

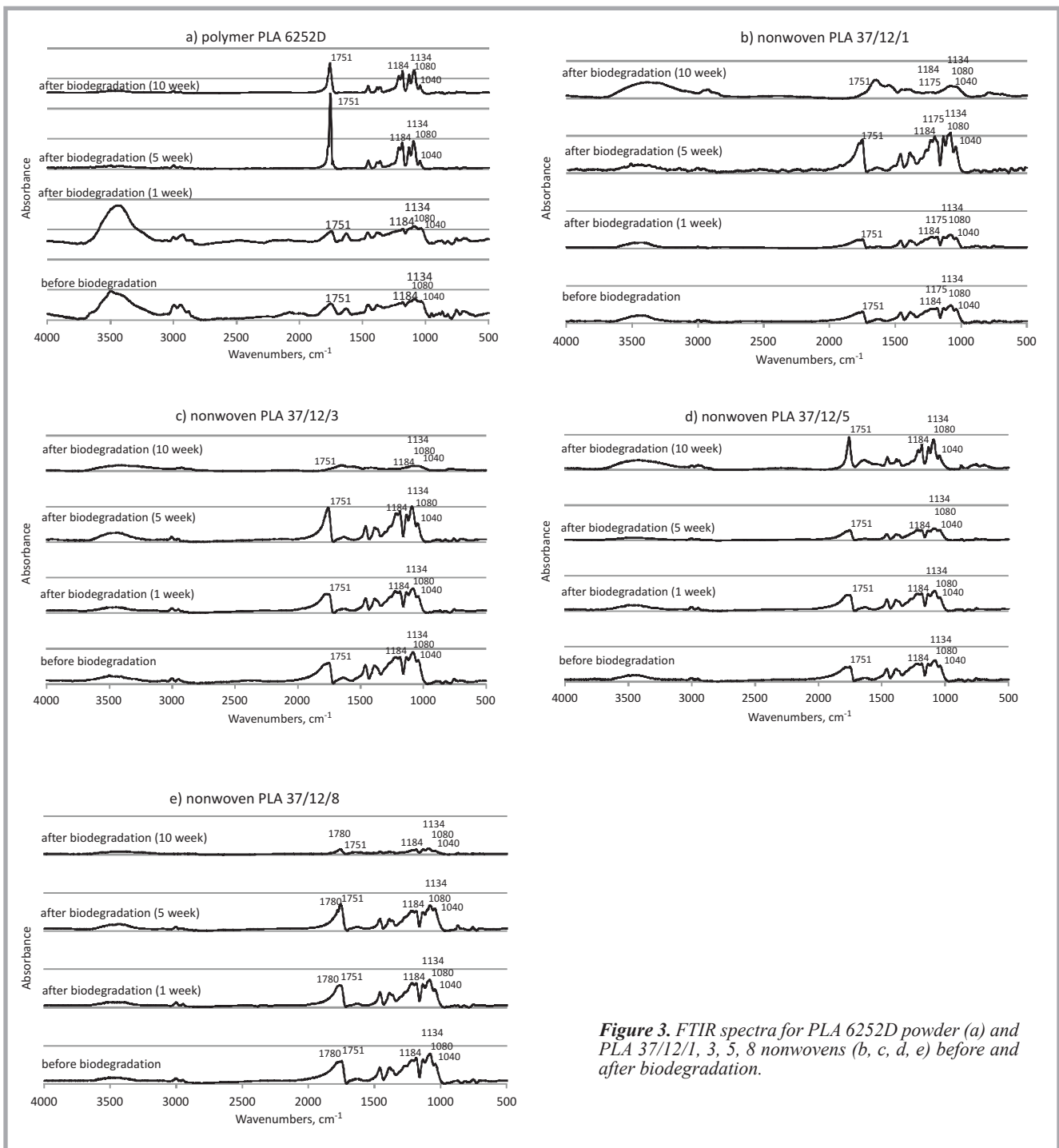


Figure 3. FTIR spectra for PLA 6252D powder (a) and PLA 37/12/1, 3, 5, 8 nonwovens (b, c, d, e) before and after biodegradation.

- A band of extension vibration for the carbonyl group C=O in the wave length region of $1751 \pm 1 \text{ cm}^{-1}$
- A band of extension vibration of the C–O bond at wave lengths of 1184, 1134, 1080, $1040 \pm 1 \text{ cm}^{-1}$.

Significant changes could not be seen in the arrangement of the peaks in the bands of characteristic transmission regions after the compost biodegradation of the materials.


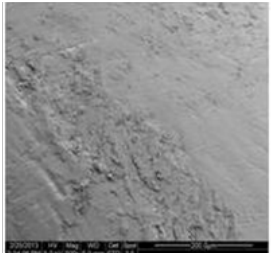

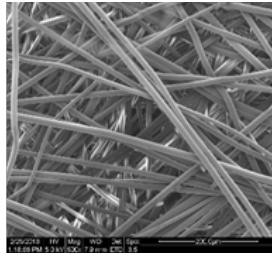

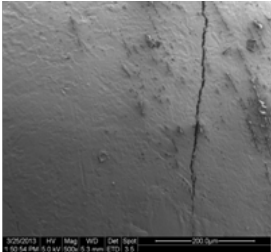
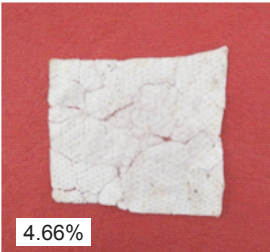
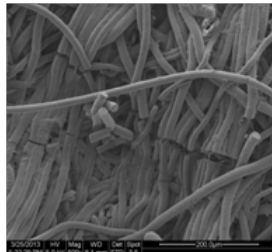
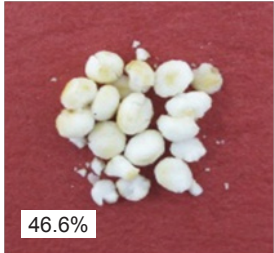
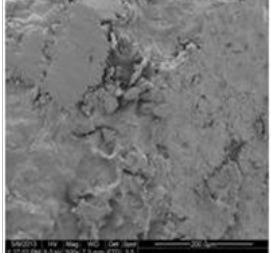

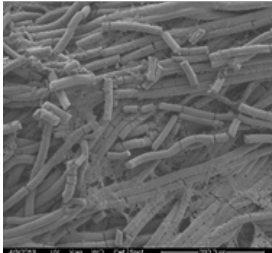
After 1 week of the biodegradation, the band of extension vibration for the carbonyl group C=O at a wave length of $\lambda = 1752 \text{ cm}^{-1}$ appears in all the fabrics and polymer examined. The band of vibration of the C–O bond is situated at wave lengths of 1185, 1134, 1082 and 1042 cm^{-1} , remaining within the limit of error.

After 5 weeks of biodegradation, the peaks characteristic of the carbonyl group C=O and the C–O bond appear in the same place, that is at 1750, 1181, 1134

and 1078 cm^{-1} . However, in some of the spectra of the nonwoven fabrics examined, peaks emerge characteristic of carboxylic acids formed by hydrolysis (band for wave length $\lambda = 1175 \text{ cm}^{-1}$ in nonwoven 37/12/1 and that for wave length $\lambda = 1780 \text{ cm}^{-1}$ for nonwoven 37/12/8).

After 10 weeks of decomposition some displacement of the peaks can be seen in the spectra of both the polymer and nonwoven fabrics, which may be the result of implicit structural changes proceeding in the materials tested.

Table 7. Change in the appearance and morphology structure of the polymer PLA 6252D and 37/12/1 nonwoven before and after compost biodegradation (SEM photos at a 500× magnification).

	Polymer PLA 6252D		Nonwoven PLA 37/12/1	
Initial sample				
After 5 weeks of biodegradation				
After 10 weeks of biodegradation				

For the polymer PLA 6252D a SEM assessment was made of its appearance and morphology structure. **Table 7** presents changes in the appearance and morphology structure of polymer PLA 6252D before and after the biodegradation.

The appearance and morphology structure of the nonwoven fabrics made of polymer PLA 6252D was also assessed. For example, **Table 7** shows also photos illustrating changes proceeding in the appearance and morphology structure of the 37/12/1 material in the course of compost degradation. A similar process of biodegradation was observed in other nonwoven materials.

Summary and conclusions

Polymer PLA 6252D and the nonwoven fabrics marked: 37/12/1, 37/12/3, 37/12/5 and 37/12/8 are characterised by a susceptibility to a complete biodegradation under simulated laboratory composting. The maximum mass loss determined for both polymer PLA 6252D and the nonwoven fabrics amounted to $U_m = 100\%$ after 16 weeks of incubation. The susceptibility to complete biodecomposition

was observed and confirmed by visual observation and SEM photos.

Biodegradable nonwovens of PLA characterised by low surface mass and sufficient strength can be useful in agriculture as modern mulch and cover material. The PLA agro-nonwovens exert no negative impact upon the environment.

The crystallinity degree $X_c = 8.9 - 31.2\%$ of the nonwoven samples tested was of no influence upon the run of the biodegradation process.

For the polymer and nonwoven PLA a molecular characteristic was prepared by GPC/SEC measurements before and after the biodegradation. Average molecular masses and polydispersity were determined, showing a distinct decrease along with progressing biodegradation.

In the IR spectra of polymer PLA 6252D and of the PLA nonwoven fabrics before biodegradation, two characteristic bands of extension vibration can be seen: for the carbonyl group C=O at wave number $1751 \pm 1 \text{ cm}^{-1}$ and extension vibration for the C–O bond at wave numbers 1184, 1134, 1080 and $1040 \pm 1 \text{ cm}^{-1}$.

The results of the IR investigation are proof that the biodegradation process exerts an impact upon the structure of the polymer and fabrics examined.

Observation of the morphology structure by scanning electron microscopy (SEM) reveals a heterogeneous and coarse microstructure of the surface of the polymer and fabrics tested, evidence of progressive biodegradation.

The research on the assessment of the biodegradability of the spun-bond nonwoven made of the PLA 6252D polymer underpins the intended use of the material in agriculture as a cover for winter plants and as soil mulch. The application calls for biodegradable materials.

Editorial note

¹ Certificate of Accreditation AB 388.

Acknowledgements

The research was carried out within the project „Biodegradable fibrous products“ POIG.01.03.01-00-007/08, sponsored by European Union in the frame of the IE OP financed from the ERDF.

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Received 08.11.2013 Reviewed 12.06.2014



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The **Laboratory** is active in testing fibres, yarns, textiles and medical products. The usability and physico-mechanical properties of textiles and medical products are tested in accordance with European EN, International ISO and Polish PN standards.

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