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Investigations into the Impact of the Formation Temperature on the Properties of Spun-Bonded Nonwovens Manufactured from PBSA

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Abstract
This article presents the influence of the formation temperature of nonwovens from biodegradable aliphatic polyesters (trade name Bionolle) on their structure and mechanical properties. Nonwovens were produced by spun-bonded technique at a laboratory installation at IBWCh, changing one of the process parameters i.e. the temperature of the polymer. Basic parameters of the polymer were evaluated in order to determine the spun-bonded process condition. Phase transition temperature - glass transition temperature (Tg), -melting point (Tm) and mass flow index (MFI)were assessed. The effect of manufacturing conditions on the properties of the nonwovens obtained was identified on the basis of the analysis of their mechanical parameters, the sorption rate, crystallinity and compost biodegradation rate. It has been shown that 238 °C is the best formation temperature to produce nonwovens with good mechanical properties and at the same time accessible biodegrability.

Key words: *nonwovens, spun-bonded, bionolle, biodegradation, mechanical properties, formation temperature.*

Introduction

Spun-bonded nonwovens have been known since the 1950s. The production of spun-bonded webs is described and illustrated in many patents [1 - 5]. This process is the most productive of all the non-conventional methods of textile fabric formation. Almost all thermoplastic resins can be processed on spunbonding equipment. Typical polymers widely used for manufacturing nonwovens are polypropylene, polyester, polyethylene, and polyamide [6 - 8, 18, 19].

However, for environmental reasons, eco-friendly polymers are becoming increasingly important [7]. One of them is biodegradable aliphatic polyester by the trade name Bionolle, produced by Showa Highpolymer Co., Ltd. Bionolle is polymerized from glycols and aliphatic dicarboxylic acids. We have two grades of Bionolle: poly(butylene succinate) (PBS) and poly(butylene succinate adipate) (PBSA), the copolymer of 1,4-butandiol succinic acid and adipic acid, respectively. We call PBS the 1000 series, and PBSA the 3000 series [9 - 11]. According to the producer PBSA polymer can be used on conventional forming equipment for polyolefin or with minor modification. Bionolle is suitable for some applications such as production of fibres, spun-bonded nonwovens, melt-blown nonwovens, multifilament and staple fibre [11, 12].

An overview of patents indicates that a known method of forming spun-bonded nonwovens from PBSA [17] and its blends with other polymers such as polybutylene succinate (PBS) exists. The blends (Bionolle 1020 and 3020) have been modified by the addition of adipic acid and a wetting agent (ethoxylated alcohol) in order to improve wettability [13]. Another patent informs that PLA and PBS were placed separately in two extruders, each heated and melted at 230 °C and formed into nonwovens [14] A different patent informs that PLA was combined with PBSA (Bionolle 3020) in order to obtain a web of fibres composed from a core (Bionolle) and the sheath (PLA). PLA was extruded at a final formation temperature of about 230 °C. Bionolle was extruded at a formation temperature of about 200 °C [16]. The invention presented in patent [17] relates to nonwovens with excellent heat stability, mechanical strength and biodegradability. This patent refers to the research into the impact of nonwovens' surface mass on the mechanical properties of the nonwovens obtained from polymers PBSA, PBS and polyester synthesized by using ethylene glycol, succinic acid and citric acid. Unfortunately in all the patents mentioned above the influence of the formation temperature on the properties of the nonwovens obtained was not cleared. There are no tests to determine the effect of formation temperature on the susceptibility to degradation of nonwovens. On the other hand an information was given that after 5 months of biodegradation in ground nonwovens no longer existed [17].

Aim of the study

The aim of the study was to investigate the influence of the formation temperature on the structural and mechanical properties of spun-bonded nonwovens produced from PBSA [poly(butylene succinate adipate)] as well as their susceptibility to biodegradation.

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Experimental

Materials and Technology

Materials

Bionolle (type #3001), a poly(butylene succinate adipate) (PBSA) copolymer was supplied by Showa Highpolymer Co.Ltd (Japan). All Bionolle polymers of the series 3000 are characterised by low modules and very good biodegradability. The melt flow index (MFI) of Bionolle #3001, measured at 190 °C and a load of 2.16 kg is 1.4 g/10min [9]; average number molecular mass Mn = 52 000 g/mol and weight average molecular mass Mw = 112 300 g/mol [15].

The molecular structure of Bionolle 3000 series is expressed by the following formula.

$$-O-(CH_2)_n-O-CO-(CH_2)_m-CO- \quad [9]$$

n – n=4 (1,4-butanediol)
m – m=2 succinic acis, m=4 adipatic acid

The polymer was dried in a dryer made by Piovan (USA). The drying temperature was 55 °C and the dew point - 30 °C. The drying process was carried out until the water content in polymer was ≤ 50 ppm. The drying time was 4 hours.

The spun-bonded nonwovens were produced using a laboratory scale spunbonding technological line used at IBWCh(1). A spinneret of 467 holes was used. The formation temperatures varied between 232 and 248 °C ± 0.5 °C, the temperature of the cooling air was 20 °C. The throughput per hole was constant of 0.09 g/min, the speed of fibre formation was 1792 m/min. The fleece was thermobonded by means of a temperature of a colander at 65 °C and the take up speed of the nonwovens obtained of 2.4 m/min.

Analytical methods

- Thermal analysis was carried out by means of *Diferential Scanning Calorimetry (DSC*) using Diamond (Perkin Elmer, Germany). The first and second heating scan and the cooling scan for the polymer were performed at a temperature range of -70 ÷ 170 °C. The samples were 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 the HI-REST TGA 2950 Thermogravimtric analyzer under nitrogen atmosphere with a heating rate of 20 °C/min at a temperature range of 20 - 600 °C.
- *Ash content in the polymer* was measured by the gravimetric method in a muffle furnace (Techmozbyt, Poland) holding sample of the polymer of 5 g for 12 hours at 800 °C then ash content calculated from:

$$x = (a \cdot 100)/W$$

where: x - ash content in polymer in %, a – mass of ashin g, W – weight of sample in g.

- *Degree of crystallinity* of polymer and the fibres was analysed by means of Wide-Angle X-ray Diffractometer (WAXD) using the X'Pert Pro System (PANalytical, Netherland). The diffraction patterns were obtained using Cu Kα (λ = 0.154 nm) X-ray source operating at 30 kV and 30 mA. The samples were examined using the powder form. The degree of crystallinity was estimated using WAXSFIT software [20] according to Hindeleh and Johnson's method and the following equation:

$$X_C = \frac{A_C}{A_C + A_A}$$

where: A_A and A_C are the calculated area under amorphous and crystalline curves of deconvoluted X-ray pattern, respectively.

- Additionally, approximated theoretical curves allow calculating crystalline size using the Scherrer equation:

$$L_{(hkl)} = \frac{K\lambda}{B \cdot \cos\theta}$$

where: $L_{(hkl)}$ – average crystalline size in orthogonal direction to the plane (hkl), θ – Bragg angle, λ – X-ray wavelength, B – FWHM of diffraction peak for plane (hkl), K – Scherrer constant, for polymers K is equal to 0.9.

- Distribution of *molecular weight and polydispersity* of polymer and fibres were analyzed using size-exclusion chromatography (SEC) – coupled with Multiangle Laser Light Scattering (MALLS) detection. The SEC-MALLS apparatus used composed of an 1100 Agilent isocratic pump, autosampler, degasser, thermostatic box for columns, a MALLS DAWN EOS photometer (Wyatt Technology Corporation, Santa Barbara, USA), and differential refractometer Optilab Rex. ASTRA 4.90.07 Software (Wyatt Technology Corporation) used for data collecting and pressing.
- *Melt flow index (MFI)* - was assessed according to method A and B, determined in accordance to standard PN 93/C89069 using the DYNISCO Polymer Test Standard apparatus (USA), with a spinneret hole of 2 mm, at a temperature of 190 °C (method A) and spinneret hole of 0.5 mm at a temperature range of 230 ÷ 270 °C (method B) [21] .Changes in the MFI of the polymer under the influence of the residence time of 6 - 36 minutes in the molten state were evaluated at a tempeature range of 230 - 270 °C.
- *The water content in polymer* was determined by the coulometric Karl Fischer method using DL39X apparatus produced by Mettler Toledo (USA).
- *Scanning Electron Microscopy (SEM)* investigations of the nonwovens were carried out using Quanta 200 (FEI, USA). The research was performed in low vacuum, in a natural state without coating.
- Mechanical properties of nonwovens were measured using an Instron 5544 (England) tensile tester, according to Polish-ISO standards: *elongation at break and strength,* in two directions (in the machine direction and crosswise) according to PN-EN 29073-3:1994 as well as *tear resistance* according to PN-EN ISO 9073-4:2002.
- *The Mass per unit area was evaluated* according to PN-EN 29073-1:1994*, the thickness using* PILMED-64 (Poland) according to PN-EN ISO 9073-2:200 and the *fibre diameter using a* LANAMETR MP 2 (Poland) according to PN-86/P-04761.08.
- *Sorption rate* – was assessed with a system designed to evaluate the liquid sorption (Kontech, Poland). For tests we used distilled water. The sorption rate was calculated from:

$$V_{max} = S_{max}/t$$

where: S_{max} - sorption capacity (the value determined by the apparatus) in μl/cm^2, t – time in s.

- *Biodegradation rate* - Studies were based on the methodology according to standards: PN-EN 14045: 2005, PN-EN 14806: 2010, PN-EN/ISO 20200:2007. The determination of the mass loss was carried out under simulated composting conditions at a laboratory scale, at a temperature of 58 ± 2 °C and a humidity of 55 ± 2%.

Results and discussion

Investigations on the PBSA - polymer characteristics

In order to determine the best suitable process parameters, the features of the polymer raw material and the polymer of the fibres obtained at different formation temperatures were the following features were assessed: the molecular weight ($\bar{M}_n$ number-average molecular weight, $\bar{M}_w$ weight-average molecular weight, $\bar{M}_w / \bar{M}_n$ polydispersity) and degree of crystallinity (*Xc*). Ash content (x) to determine the purity of the polymer was also measured. The melt flow index (MFI-method A in 190 °C) was used as a quality control parameter for the initial selection of the polymer raw material. The measurement of the melt flow index is an important step in the verification of the polymer spin ability.

Thermal behaviour of poly (butylene succinate adipate) was also evaluated by Differential Scanning Calorimetry (in order to obtain the glass temperature and the melting point - T_m) as well as the thermogravimetric analysis (in order to obtain weight loss at temperature at 238 °C) W_{loss}, - temperature of maximum speed of weight loss - T_{max}). The results are shown in ***Figures 1*** & ***2*** and ***Table 1***.

Table 1. *Some properties of PBSA (Bionolle #3001) polymer.*

Polymer (trade name)	SEC/MALLS			x, %	Xc, %	MFI, g/10 min	TGA	
	$\bar{M}_n$, g/mol	$\bar{M}_w$, g/mol	$\bar{M}_w / \bar{M}_n$				W_{loss} in 238°C, %	T_{max}, °C
Bionolle #3001	76500	157000	2,08	0.03	33	0.99	0	408

Figure 1 shows DSC scans of PBSA (Bionolle #3001) raw material. The first heating indicates glass transition of -44.0 °C, the melting enthalpy of 53.2 J/g and melting point of 90.0 °C. On the DSC scan of cooling one exothermic peak at 60.9 °C and a glass transition at – 46.8 °C are visible. The thermogram of the second heating is similar to the first one indicating the glass transitions temperature of -46.3 melting enthalpy of 50.6 J/g and melting point at 90.6 °C.

The characteristics of the polymer described above allowed selecting the optimum drying temperature of 55 °C set below the temperature of any noticeable melting effects.

The study of MFI allows us to observe the influence of the temperature on the polymer and to initially determine the formation temperature. From the point of view of processing, the residence time of the polymer in the molten state is also important (***Figure 4***).

Figure 4 shows the effect of residence time of the polymer in molten state at different formation temperature. It is visible that the viscosity of the polymer decreases which may be caused by the degradation of the polymer. As can be seen, the higher the temperature, the melt flow index of the polymer is higher. It can also be noted that, with a long residence time

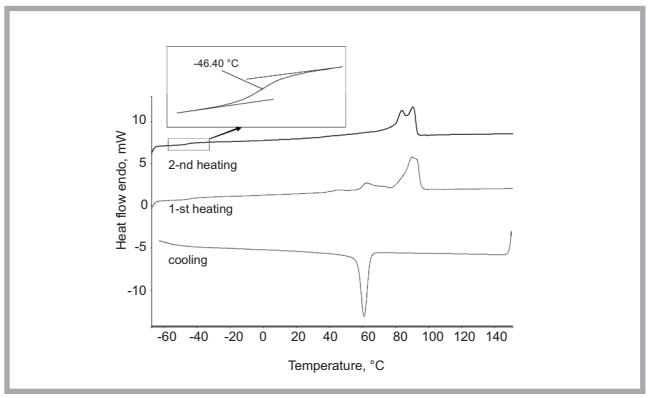

Figure 1. *DSC scans of first and second heating and cooling.*

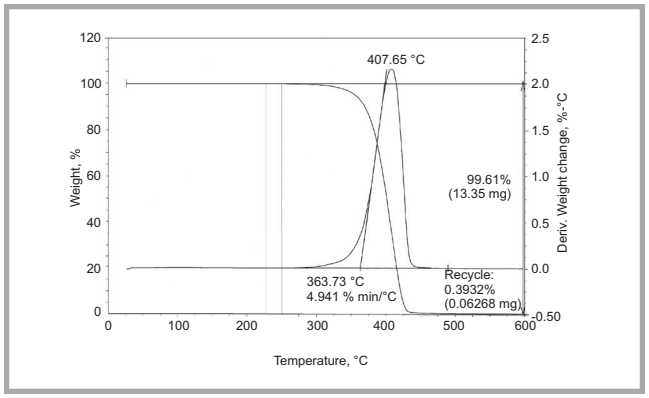

Figure 2. *Thermogravimetric analysis of PBSA (Bionolle #3001).*

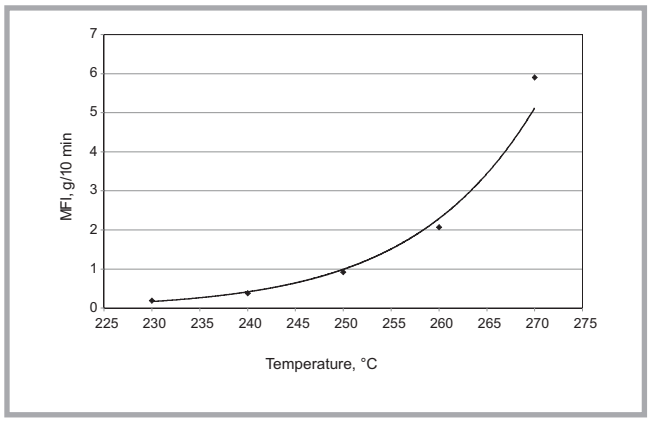

Figure 3. *Changes of the melt flow index of PBSA (Bionolle #3001) polymer in function of temperature. Method B.*

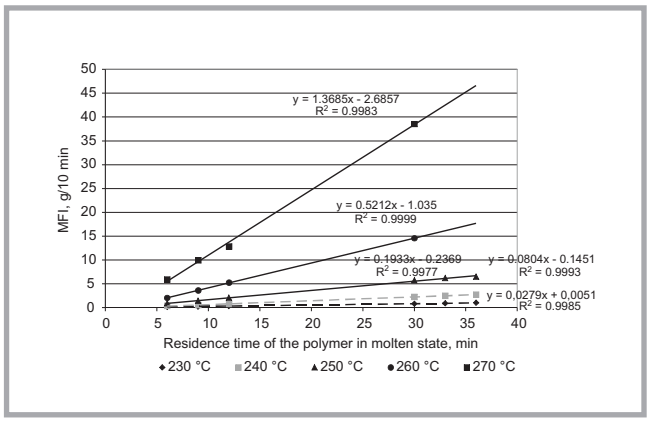

Figure 4. *Changes of the MFI of PBSA (Bionolle #3001) in function of residence time of the polymer in molten state.*

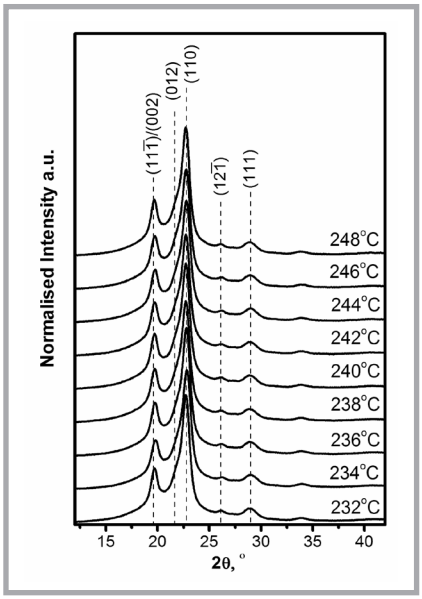

Figure 5. *Comparison of X-ray diffraction patterns obtained for different formation temperature.*

of the polymer in the molten state the viscosity decreases faster.

In ***Figures 3*** and ***4*** it can be observed that the MFI increases with increasing temperature, which is connected with a decrease in viscosity of the polymer. This means that if the nonwovens would be formed at high temperatures, it is necessary to reduce the residence time of the polymer in the molten state – which means the manufacturing at high throughput. This is important because of the nature of the polymer (it can be assumed that the biodegradability depends of the formation temperature), and also for economic reasons.

Based on ***Figure 4***, it can be initially stated that the formation temperature should be in the range of 230 ÷ 250 °C, as in this range no weight loss of the polymer (***Figure 2***) was observed.

Study on the impact of formation temperature on the molecular and morphological structure and physical-mechanical properties of the nonwovens

All of the tests described above were necessary to determine all processing parameters, such as temperature of the heating zones of the extruder, temperature of the spinning pump, temperature of the molten polymer at the output (formation temperature), extruder speed, metering pump rotational speed, fibre take-up speed, temperature of quenching air etc. All these parameters, although very important, are not the subject of this paper (except the formation temperature).

In ***Figure 5*** the comparison of WAXD patterns obtained for all studied nonwovens is presented. The X-ray diffraction peaks at 2θ = 19.7°, 22.1°, 22.8°, 26.2° and 29.1°, corresponding to (11$\bar{1}$)/(002), (012), (110), (12$\bar{1}$) and (111) planes of poly(butylene succinate) monoclinic crystal lattice are visible. In the diffraction patterns there is a lack of information about poly(butylene adipate) crystalline structure. The strongest diffraction peaks which according to literature should be visible at 2θ = 17.6° and 21.7° corresponding to (002) and (110) planes of PBA were not detectable. This important result clearly shows that the content of poly(butylene adipate) in the studied copolymer is probably very low (<25% wt) [22]. Additionally, the X-ray diffraction patterns obtained for the spun-bonded nonwovens studied show only insignificant differences.

A detailed structural analysis of the samples investigated was obtained by the deconvolution of the patterns into the crystalline peaks and amorphous halo. An example of the deconvolution of the diffraction pattern recorded for the sample forming at 238 °C is presented in ***Figure 6.***

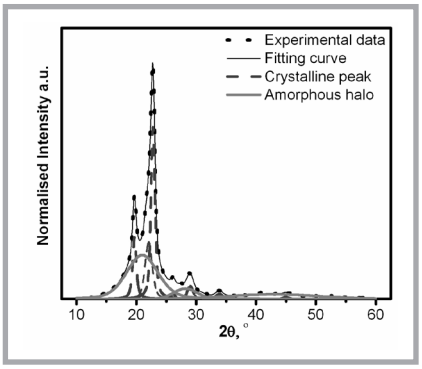

Figure 6. *Deconvolution of X-ray pattern obtained for 238 °C.*

The results of calculations of the crystallinity degree (X_C) and crystalline size ($L_{(hkl)}$) are presented in ***Table 2***.

All of the samples obtained are characterised by a similar crystallinity degree of approximately 43.5%. The degree of crystallinity of the polymer after processing, increased by approximately 10% relative to the polymer where the degree of crystallinity was 33%. According to the Scherrer equation the estimated crystalline size confirmed the similarity of the crystalline structure of the PBSA spun-bonded nonwovens studied. The crystalline size in the (11$\bar{1}$)/(002) plane direction is about 12 nm, in the (012) is about

Table 2. *Crystallinity degree and crystalline size of investigated PBSA nonwovens calculated by WAXSFIT software.*

Formation temperature, °C	X_C, %	$L_{(11\bar{1})/(002)}$, nm	$L_{(012)}$, nm	$L_{(110)}$, nm
232	44.2	12.5	6.6	11.6
234	42.2	11.4	7.5	10.6
236	43.0	12.3	7.9	11.0
238	43.1	12.0	6.4	11.4
240	44.8	12.1	6.5	11.4
242	42.8	12.0	7.2	11.0
244	42.8	11.8	7.8	10.7
246	43.0	11.7	7.6	10.8
248	44.6	12.8	7.7	11.2

Table 3. *Selected physical-mechanical properties of spun-bonded nonwovens.*

Formation temperature, °C	Fibre diameter, µm	Mass per unit area, g/m^2	Nonwoven thickness, mm	Sorption rate, µl/cm^2/s
232	7.36 ± 0.24	56.0 ± 6.0	0.23 ± 0.01	0.56
234	6.40 ± 0.22	53.1 ± 2.5	0.23 ± 0.01	0.44
236	7.84 ± 0.27	48.3 ± 1.4	0.21 ± 0.01	0.58
238	7.09 ± 0.24	48.6 ± 1.2	0.22 ± 0.01	0.68
240	7.54 ± 0.20	47.9 ± 1.8	0.21 ± 0.01	0.63
242	7.57 ± 0.18	46.9 ± 0.5	0.21 ± 0.01	0.56
244	7.02 ± 0.19	45.6 ± 1.2	0.20 ± 0.01	0.43
246	7.21 ± 0.20	43.5 ± 1.5	0.20 ± 0.01	0.46
248	7.58 ± 0.18	43.7 ± 3.8	0.20 ± 0.01	0.46

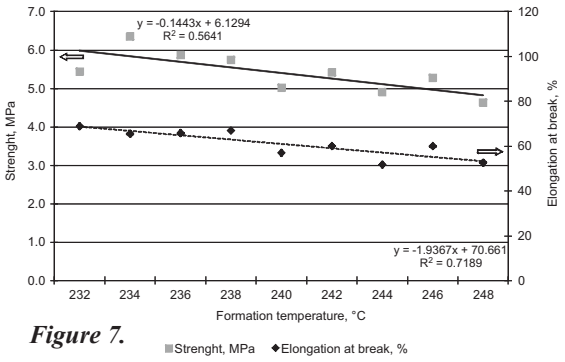

Figure 7.

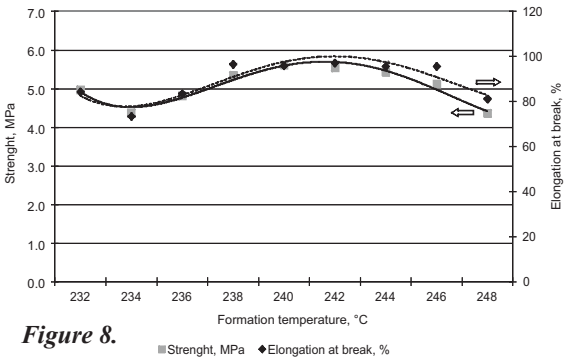

Figure 8.

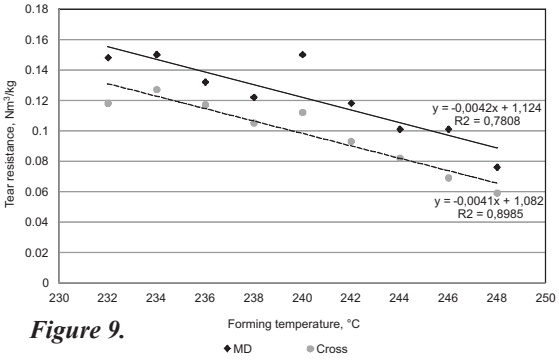

Figure 9.

Figure 7. *Relationship between formation temperature and mechanical properties of nonwovens along the structure.*

Figure 8. *Relationship between formation temperature and mechanical properties of nonwovens cross the structure.*

Figure 9. *Influence of the formation temperature on the tear resistance of nonwovens.*

7 nm, and in the (010) planes is about 11 nm. The WAXD results of structural studies obtained demonstrate the lack of effect of formation temperature on the qualitative and quantitative supramolecular structure of materials studied, which is important from the point of view of the processing technology.

In ***Table 3*** and in ***Figures 7, 8*** & ***9***, we have shown the influence of formation temperature on selected physical-mechanical properties of nonwovens.

We can observe that with increasing temperature, the strength, elongation at break in machine direction and tear resistance in both directions of the nonwovens decreases (***Figures 7, 8, 9***). As can be seen in ***Table 3*** the sorption is the highest at the temperature of 238 °C.

In ***Figure 7*** the effect of temperature on the changes in nonwovens strength can be observed. With increasing formation temperature the structure in machine direction- strength and elongation at break of the nonwovens decrease. This may be related to the thermal degradation of the polymer, resulting in the reduction in viscosity of the molten polymer, which was confirmed by a pressure drop in the formation zone after the metering pomp (1830 hPa at 232 °C to 920 hPa at 248 °C) which could be the reason of worsening of the nonwovens obtained. The fibres were also more easily stretched after the spinneret holes, which in turn could influence the reduction of the surface mass and thickness of nonwovens with an increase in formation temperature (***Table 3***) which is confirmed by literature [7]. However, due to the high degree of fibre fusion, a stretch of fibres during sampling can occur. In the future it is advisable to sample fibres from the web before bonding by the calander.

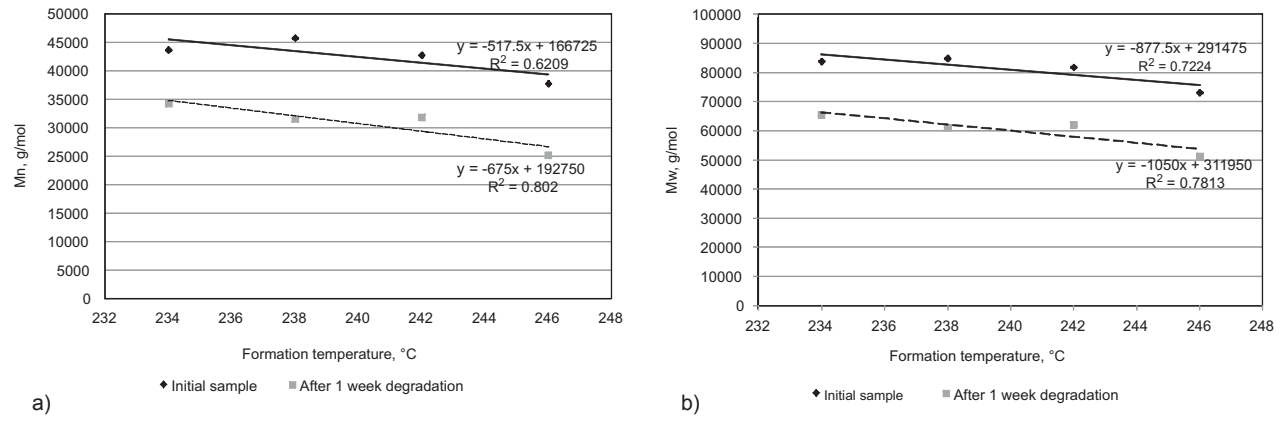

Figure 10. *Influence of the formation temperature on the changes in the number-average (a) and weight-average (b) molecular weight of spun-bonded nonwovens before and after 1 week of biodegradation in a compost environment.*

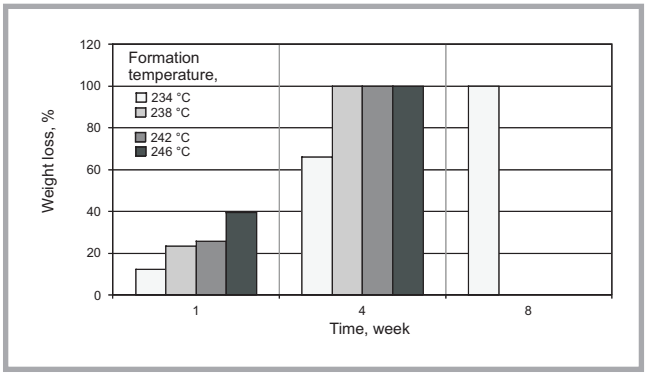

Figure 11. *Influence of the formation temperature on the biodegradation spun-bonded nonwovens fabric in compost environment.*

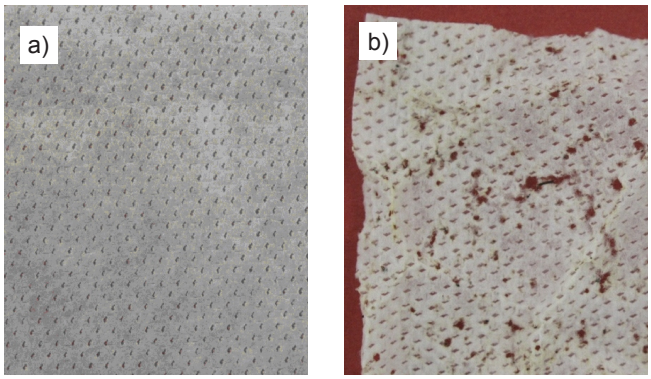

Figure 12. *Photographic documentation of spun-bonded nonwovens manufactured at 238 °C (a) and under simulated composting conditions (b).*

However, the results in ***Table 3*** carry a relative error of not more than 5%.

Analyzing the mechanical properties of spun-bonded nonwovens in the cross direction (***Figure 8***), it can be seen that the nature of the changes in strength and elongation at break are similar taking into account the relation to the formation temperature. At a temperature of 234 °C, these parameters are the lowest, while with the increase in the formation temperature, an increase in elongation and strength are visible. An important observation is also the fact that in the range of 238 - 244 °C nonwovens' properties are the best, whereas a further temperature increase worsens them. Comparing ***Figure 7*** and ***8*** also shows variations in the nonwovens elongation in the machine and cross directions. The nonwovens investigated in the machine direction have the elongation at break in the range of 50 - 70% and in the cross direction much higher of 73 - 97%. The effect of the formation temperature is also seen in the changes of tear resistance (***Figure 9***), namely the increase in the formation temperature results in weakening of the nonwovens, and in that the strength in the cross direction is lower than in the machine direction. This difference should be cleared by further studies. Due to the uneven structure of the nonwovens' the uncertainty of strength measurement is very high even ±20%. Taking into account the great differences between the particular measurements, only the trend of the changes can be established.

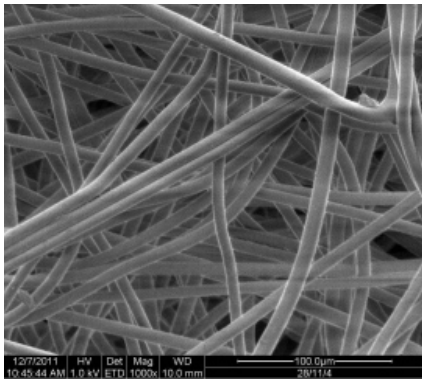

Figure 13. *SEM images (magnification 1000×) of spun-bonded nonwovens manufactured at 238 °C.*

The results of metrological research indicate that the best temperature for nonwovens formation is the intermediate temperature within the range of 238 - 244 °C.

For some nonwovens, which were subjected to biodegradation test, the molecular mass was determined. The results are shown in ***Figure 10***.

It can be concluded that with an increase in the formation temperature, the weight-average and number –average molecular mass decrease.

The weight-average and number-average molecular mass (***Figure 10***) shows an influence of the formation temperature on the polymer degradation. The number average molecular mass of the polymer after processing has decreased in relation to the initial number-average molecular mass of about 43 - 51% where as the weight average molecular mass decreases of about 47 - 53% depending on the formation temperature. This data indicates that at high molecular mass the formation of nonwovens is preferred at lower temperatures. The lowest molecular weight is observed at the highest temperature. The effect of the formation temperature on the susceptibility to biodegradation of nonwovens in a compost environment was also studied. The ***Figure 10*** also show the results of the molecular mass of the nonwovens after one week biodegradation at compost environment. The effect of formation temperature can also be seen. Namely, in the process of biodegradation (after one week degradation) of the samples produced at a temperature of 234 °C the loss of weight- average and number-average molecular mass was about 20% whereas for the nonwovens produced at 246 °C the loss was as high as 30%. However, this difference may also be caused by the influence of reducing the weight of nonwovens surface mass with increasing of the formation temperature.

In ***Figure 11*** we can see that the formation temperature has an influence on time of biodegradation.

Nonwovens produced at 234 °C were fully biodegradable after 8 weeks at compost, while the biodegradation of nonwo-

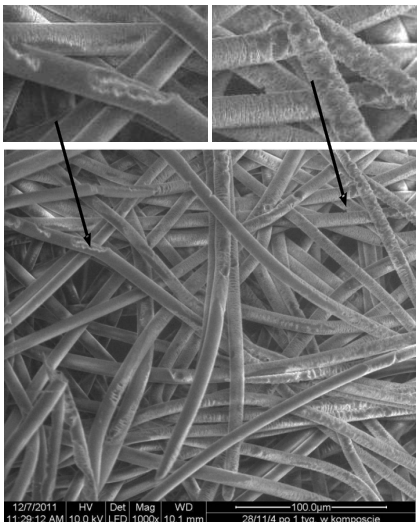

Figure 14. *SEM images (magnification 1000×) of spun-bonded nonwovens manufactured at 238 °C under simulated composting conditions.*

vens produced at higher temperature of 238 °C degraded fully after 4 weeks. After the first week of degradation the loss of the nonwovens mass is distinctly visible with increasing formation temperature. No influence of the sorption rate on the nonwovens biodegradability was observed, but a correlation between the loss of molecular mass and susceptibility to biodegradation was noted. With a decrease in the molecular mass, an increased ability for biodegradation was observed.

The spun-bonded nonwovens formed at 238 °C are shown in the photographic documentation in ***Figure 12*** (see page 75) and SEM images in ***Figures 13*** & ***14*** (see page 75). It can be observed that the impact of the composting environment on the nonwovens after one week of biodegradation is visible in changes in the morphological structure.

Conclusions

- The analysis of spun-bonded nonwovens produced from poly(butylene succinate adipate) (Bionolle #3001) at different formation temperatures, indicates that however all of the samples obtained are characterised by a similar degree of crystallinity of approximately 43.5% and insignificant changes in the cristaline structure. In general, the nonwovens are characterized by properties depending on the formation temperature within the range of 232 - 248 ºC.
- Most of the mechanical properties decrease with an increase in the formation temperature. Contrary to them the strength and elongation at break of the nonwovens tested in the cross direction increase in the temperature range of 238 - 244 °C. Considering the increase in strength in this range and the highest sorption rate of the spunbonded nonwovens, the temperature of 238 °C can be selected and accepted as the best formation temperature .
- With the increase in formation temperature the weight-average and number-average molecular mass decrease, influencing the degradation of the polymer due to the increases in the rate of biodegradation in a compost environment.

The formation temperature has an influence on the time of biodegradation. Nonwovens made at 234 ºC were fully biodegradable after 8 weeks in a compost environment, while nonwovens produced at higher temperatures fully degraded after 4 weeks . Due to the influence of the formation temperature on the degradation time we can use this fact to select the temperature in order to obtain the needed time of biodegradation of the final product suitable for the assumed agriculture applications.

Editorial notes

1) *The spun-bonded technological line used was constructed by the Research and Development Centre of Textile Machinery "POLMATEX-CENARO" (Poland).*
2) *Some of the results were presented at:*
   - *The 7th Central European Conference (Cec 2012), Fibre-Grade Polymers, Chemical Fibres And Special Textiles organized by the University of Maribor, Faculty of Mechanical Engineering, Department of Textile Material and Design. September 2012, Portorose, Slovenia.*
   - Scientific Conference "Changes in the structure and properties of biodegradable polymers in the processing and biodegradation" *organized by the Lodz University of Technology, Faculty of Material Technologies and Textile Design, Department of Fibre Physics and Textile Metrology. October 2012, Lodz, Poland.*

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