Influence of forming conditions of a spun-bonded nonwoven from Bionolle polymer on their properties

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INTRODUCTION

Spunbonding is a single step process for converting plastic pellets into nonwoven fabrics. Almost all thermoplastic resins can be processed on spunbonding equipment [1]. One of them are biodegradable aliphatic polyesters of the trade name Bionolle, produced by Showa Highpolymer. Bionolle is manufactured through a polycondensation of glycols with aliphatic dicarboxylic acids and additional chemical reactions. We used the following two grades of Bionolle: poly(butylene) succinate (PBS) and poly(butylene) succinate/adipate (PBSA), the copolymer of 1,4-butandiol and succinic acid/adipic acid, also known as 1000 series and 3000 series, respectively [2,3].

In this study, we investigated the influence of forming conditions of spun-bonded nonwovens on their properties, analysing their physical and mechanical parameters, sorption rate, crystallinity, compost biodegradation rate and morphological structure.

MATERIALS AND METHODS

MATERIALS

Polymer: Bionolle (granules type #3001), polybutylene succinate adipate copolymer by Showa Highpolymer co.LDT (Japan).

Drying of polymers: Polymers were dried in a dryer made by Piovan. Drying temperature was 55°C and dew point - 30°C. Drying process was carried out until the water content in polymer was ≤ 50 ppm. Time of drying was 4 hours.

Forming of nonwoven:

Equipment: Laboratory scale spun-bonding technological line used at IBWCh was constructed by POLMATEX-CENARO. Spinning conditions: processing temperature 232-248 °C, throughput 0,09 g/min/hole.

ANALITYCAL METHODS

Differential Scanning Calorimetry (DSC) using Diamond (Perkin Elmer)

where: T_g- Glass temperature [°C],T_m - Melting point [°C],

Thermal Gravimetric Analysis (TGA)

where: T_{max} - Temperature of maximum weight loss speed [°C], W_{loss} -Weight loss at temperature of 238 °C [%].

Melt flow index (MFI) of polymers - according to method A in accordance with DYNISCO Polymer Test standard (with a spinneret hole of 2 mm, at a temperature of 190 °C, and method B developed at IBWCh for fibre - forming polymers (using a spinneret hole of 0.5 mm, within the temperature range of 230 to 270°C).

Viscosity was measured in chloroform at 25°C using viscometer Ubbelohde`a with capillary 0Ak=0.00498. The inherent viscosity was calculated from:

$$\eta_{inh} = \ln \eta_{rel} / c \qquad \text{Eq. (1)}$$

where: η_{rel} – relative viscosity, c – polymer concentration 0,1 g/dl.

Ash content in polymer – gravimetric method. Ash content in polymer was calculated from:

$$x=(a \cdot 100)/W \qquad \text{Eq. (2)}$$

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

Moisture content in polymer - coulometric Karl Fischer method with DL39X apparatus by Mettler Toledo.

Degree of crystallinity –WAXS (wide-angle X-ray scattering) – polymer and nonwoven were analysed using X'Pert Pro X-ray diffractometer (from PANalytical). The diffraction patterns were obtained byCu Kα (λ=0.154 nm) X-ray source operating at 30 kV and 30 mA. The samples were studied in the powder form. A degree of crystallinity was estimated by the use of WAXSFIT software [4] according to Hindeleh and Johnson's method and the following equation:

$$Xc=Ac/Ac+Aa \qquad \text{Eq.(3)}$$

where: A_a and A_c are calculated area under amorphous and crystalline curves of decnvoluted X-ray pattern, respectively.

Molecular weight and polydispersity – size-exclusion chromatography (SEC) coupled with Multiangle Laser Light Scattering (MALLS) detection. SEC-MALLS - was composed of an 1100 Agilent isocratic pump, autosampler, degasser, thermostatic box for columns, a photometer MALLS DAWN EOS (Wyatt Technology Corporation, Santa Barbara, CA), and differential refractometer Optilab Rex. ASTRA 4.90.07 Software (Wyatt Technology Corporation) was used for data collecting and processing.

Scanning Electron Microscope (SEM) Quanta 200 by EI. The research was performed in low vacuum, in a natural state without coating.

Elongation at break in two directions (lengthwise and crosswise) - PN-EN 29073-3:1994 , **tear resistance**-PN-EN ISO 9073-4:2002. Nonwoven were measured using Instron 5540 apparatus, according to Polish-ISO standards.

Sorption rate – research was carried out with a system to evaluate the liquid sorption. For tests we used distilled water. The sorption rate was calculated from:

$$d=(S_{max} \cdot 10)/M_p \qquad \text{Eq. (4)}$$

where: S_{max} - sorption capacity [μl/cm²], M_p – surface mass [g/cm²].

Biodegradation - determination of adegree of disintegration of nonwoven under simulated composting conditions by a laboratory scale test (at 58±2°C and a humidity of 50±2%). Studies based on the methodology developed in the accredited Biodegradation Laboratory IBWCh according to standards: PN-EN 14045: 2005, PN-EN 14806: 2010, PN-EN/ISO 20200:2007. A method for determining the weight loss.

CONCLUSIONS

Analysis of spun-bonded nonwoven produced from Bionolle #3001 polymer at different processing temperatures, indicates that temperature has an influence on properties of these fabrics.

All of the obtained samples are characterised by a similar crystallinity degree of approximately 43,5%. Change in forming temperature within the range of 232-248ºC has an insignificant influence on the structure of PBSA. The values of crosswise and lengthwise elongation changed from 73 to 97% for the crosswise direction within the range of temperature: 234-238ºC, and for the lengthwise direction the elongation decreased above 238ºC (Fig.4). In Fig.3 sorption rate is the highest at temperature 238ºC. Weight - average molecular weight (Fig.7) and tear resistance (Fig. 5) shows an influence of processing temperature on the degradation of polymer. With the highest temperature, molecular weight and tear resistance are the lowest. In Fig.6 we can see that temperature has an influence on the time of biodegradation.

Nonwovens made at 234ºC were fully biodegradable after 8 weeks in a compost environment, while the biodegradation of nonwovens produced at higher temperatures fully degraded after 4 weeks.

With an increase in temperature, the properties of nonwovens are worse and the weight loss (in biodegradation process) is the highest. The optimum temperature to produce nonwoven with good mechanical properties is 238ºC.

RESULTS

Table 1. Selected physico- chemical properties of Bionolle #3001 polymer.

Polymer	SEC/MALLS			x [%]	Xc [%]	η_{inh} [dl/g]	DSC		TGA	
	$\bar{M}_n$ [g/mol]	$\bar{M}_w$ [g/mol]	M_w/M_n				T_g [ºC]	T_m [ºC]	W_{loss} [%]	T_{max} [ºC]
Bionolle #3001	76500	157000	2,08	0,03	33	1,47	-44,0	90,0	0	408

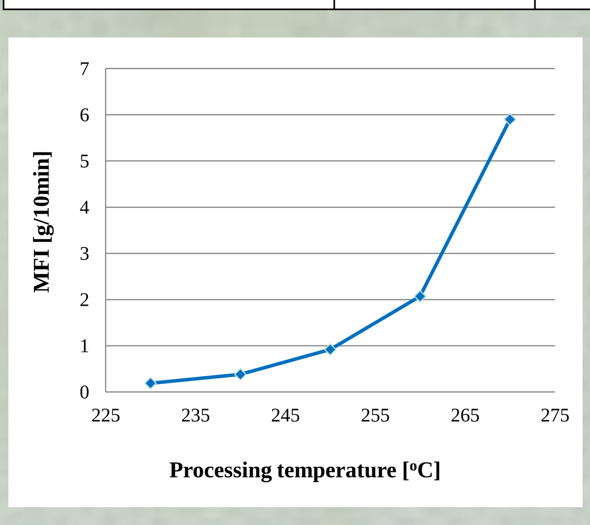


Figure 1. Changes in the Melt Flow Index of Bionolle #3001 as a function of temperature. Method B.

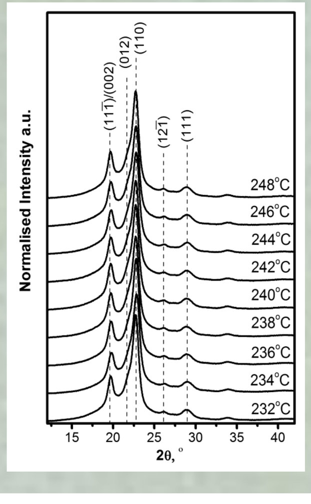


Figure 2. Comparison of X-ray diffraction patterns obtained for different forming temperatures.

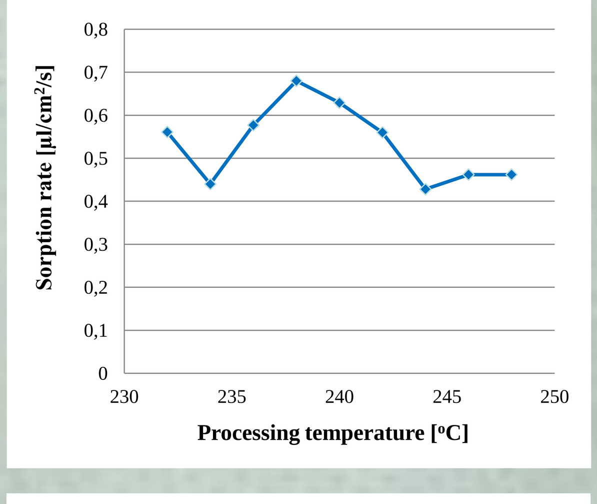


Figure 3. Influence of the processing temperature on the sorption rate of nonwoven fabrics.

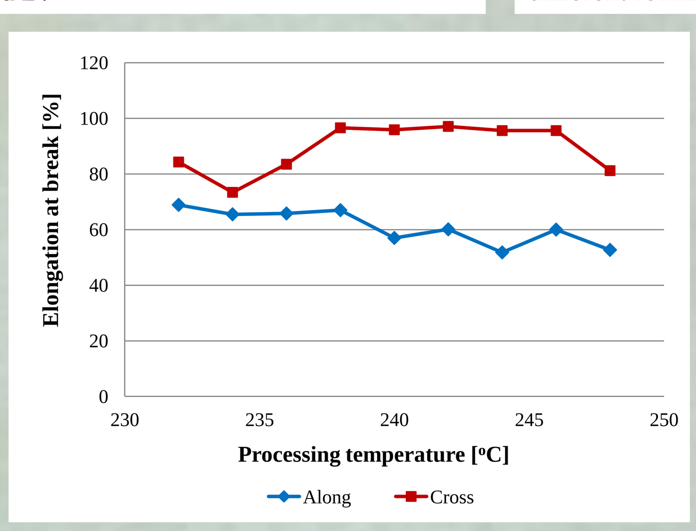


Figure 4. Influence of the processing temperature on the elongation at break of nonwoven fabrics.

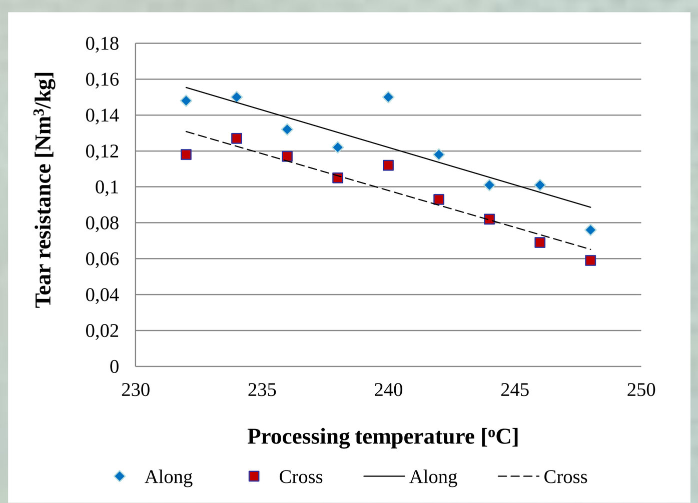


Figure 5. Influence of the processing temperature on the tear resistance of nonwoven fabrics.

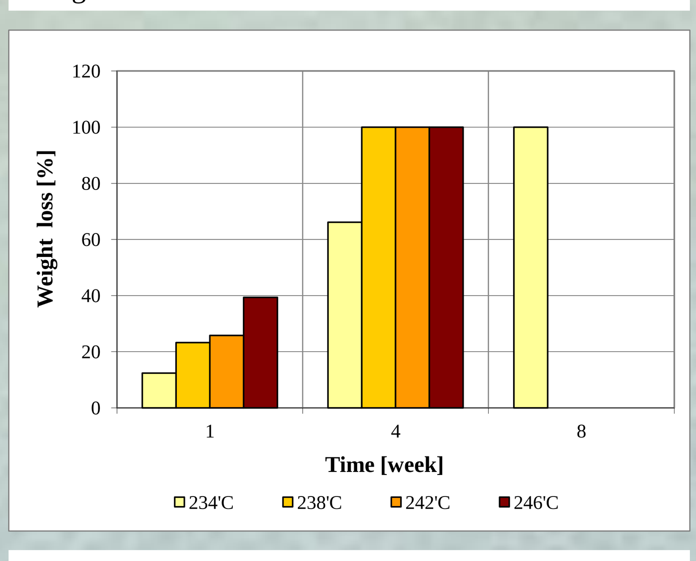


Figure 6. Influence of the processing temperature on the biodegradation of spun-bonded nonwoven fabric in a compost environment.

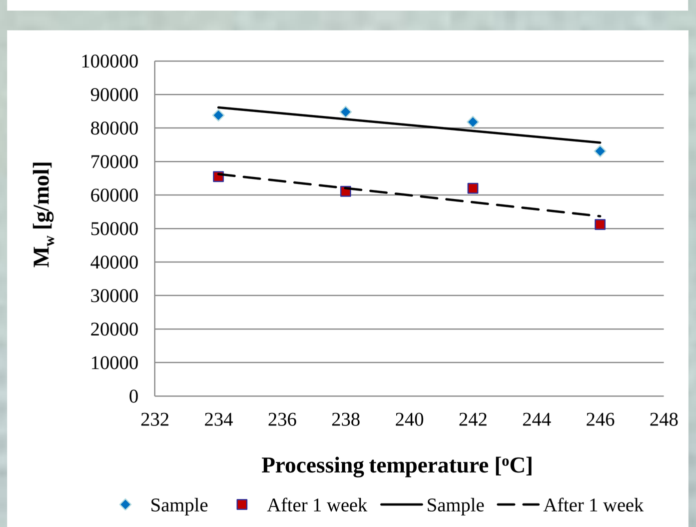
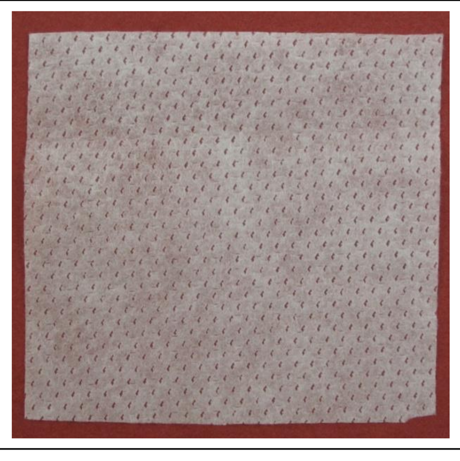
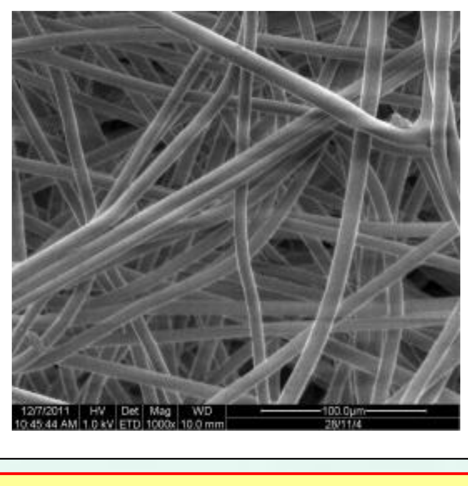


Figure 7. Influence of the processing temperature on change in the weight-average molecular weight spun-bonded nonwoven before and after 1 week of biodegradation in a compost environment.

Table 2. Photographic documentation and SEM images of spunbonded nonwovens manufactured at 238ºC under simulated composting conditions.

Week of degradation	After 0 weeks	After 1 week
Photographic documentation		
SEM images		

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