E. Wojciechowska, J. Fabia, Cz. Ślusarczyk, A. Gawłowski, M. Wysocki*, T. Graczyk

Institute of Textile Engineering and Polymer Materials, University of Bielsko-Biala, 43-309 Bielsko-Biala, ul. Willowa 2, Poland

*Research and Development Centre (BOSMAL), Laboratory of Physicochemical Analysis, 43-300 Bielsko-Biała, Sarni Stok 93, Poland

Processing and Supermolecular Structure of New iPP/PLA Fibres

Abstract

The increasing application of polypropylene fibres forces the need for searching after new methods of utikisation wastes with PP fibre content. The additions of biodegradable compositions to polymers, which are biodegradable resistant, is a very important technological problem in fibre manufacturing. On the basis of thermal investigations carried out with the system's components, a method of iPP/PLA fibre melt-spinning was developed. By selecting optimum processing temperatures, blend fibres were obtained of good quality within the whole range of component concentrations. This paper presents the research results into manufacturing and the structure of polypropylene (iPP) blends with fibre-grade poly(L-lactide). The supermolecular structure of the fibres obtained was investigated by wide-angle and small-angle X-ray scattering methods. We stated that the structure is dominated by the smectic phase of iPP. The long period of this structure decreases as the PLA content in the mixture increases. The paper presented is a preliminary stage of studies on the biogegradability of the iPP/PLA polymer system.

Key words: *polypropylene-blends, polylactide, melt-spinning, utilisation, biodegradability, WAXS, SAXS.*

Introduction

Polypropylene fibres are among the most important synthetic fibres used in the textile industry [1]. Their main advantages are low density, good stretching and abrasion resistance, resistance to chemical and biological agents, good heat-insulating properties and – last but by no means least – their low manufacturing cost [2]. The fast flow of humidity through polypropylene fibres recommends them for wide use in hygienic and sanitary products.

Apart from their undoubted usefulness, textile fibres also have many drawbacks, which restrict their use. From the environmental point of view, their chief disadvantage is their absolute non-biodegradability. Because of this, attempts have been made to obtain modified polypropylene fibres that would be at least partly biodegradable [3].
Obtaining such fibres turned out to be possible when a melt of polypropylene (iPP) and polylactide (PLA) is used for spinning [4]. The main advantage of PLA as a component of these fibres is its full biodegradability. It is also thermoplastic, fibre-forming and produced from recyclable materials. Apart from providing polypropylene fibres with at least partial biodegradability, adding PLA may result in changing their other properties, including improved dyeability [5, 6].

Certainly, the new properties of polypropylene fibres should increase the range and efficiency of their use. So far, there have been no reports of any fibrous material being obtained from a biostable polymer, such as polypropylene, by introducing bioresorbable polylactide. Thus our attempt to obtain such compound fibres is a pioneering work.

Experimental

Materials

The investigation was carried out on iPP/PLA fibres formed by means of a laboratory spinning machine. Slovnaft isotactic polypropylene TATREN TI 922 with MFI 30g/10min (230°C/2,16kg), and poly (L-lactide) RESOMER® L 207 produced by Boehringer Ingelheim Pharma GmbH were used.

Fibres were extruded from the melt with a temperature, which enabled good miscibility of components in the melt, and were spun with a take up velocity of 500m/min.

Analytical methods

Calorimetric investigations were carried out with a TA Instruments Thermal Analysis System 5100 equipped with a Differential Scanning Calorimeter model 2920. The linear function of temperature increase was used. The samples of fibres were heated at a rate of 10°C/min from –60°C to 300°C (atmosphere N_2; flow 40ml/min). The heats and temperatures of transitions were calculated by means of the Universal V2.6D TA Instruments computer program.

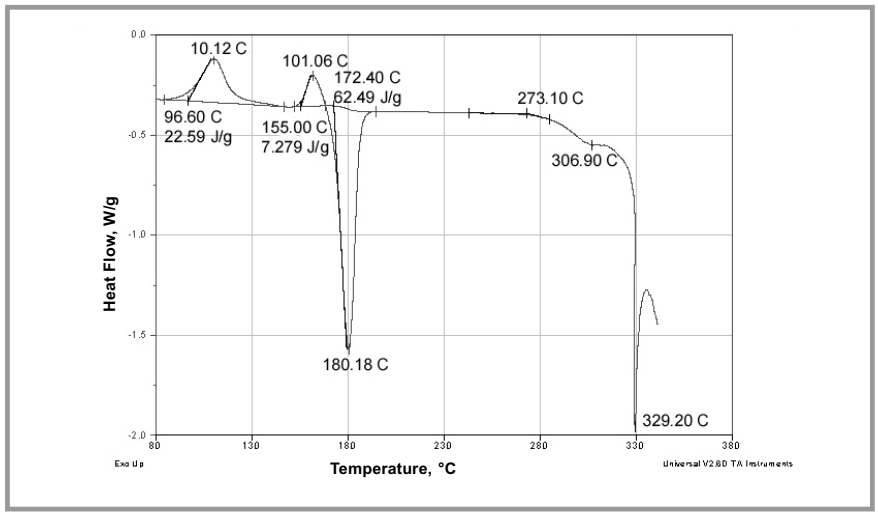


Figure 1. *DSC curve for poly (L-lactide) RESOMER® L 207 sample. The analysis of cold crystallisation, recrystallisation, melting and thermal decomposition regions. The curve registered for sample reheating after the previous controlled supercooling from the melt (235°C).*

The temperature of transition start ($T_{ON\ SET}$) was determined with the standard method as the intersection point of tangent to the front of heat at the point of inflection with the basic line.

Wide-angle X-ray scattering (WAXS) investigations were carried out with an HZG-4 Seifert diffractometer. CuK_α radiation was used at 40kV and 30mA. Monochromatisation of the beam was obtained by means of a nickel filter and a pulse-height analyser. A scintillation counter was used as a detector. Investigations were performed over the range of angles 5° to 40° with a step of 0.1°. Each diffraction curve was corrected for polarisation, the Lorentz factor, and incoherent scattering.

Small-angle X-ray scattering (SAXS) investigations were performed with an MBraun camera which utilises the conventional Kratky collimation system. The front of the camera was directly mounted on the top of the tube shield of a stabilised Philips PW 1830 X-ray generator. The X-ray tube was operated at a power of 1.5kW. CuK_α radiation was used; monochromatisation was performed by a Ni β filter and a pulse-height discrimination. The entrance slit was adjusted to 50mm. Scattered radiation was recorded over an acquisition time of 900s by means of a MBraun linear position-sensitive detector, model PSD 50. The detector had 1024 channels with a channel-to-channel distance of 52μm.

An IR spectroscopic analysis was performed using the FTIR-1720 X Perkin/Elmer Fourier spectrophotometer (resolution 2cm^{-1}).

Results of the research

Based on the thermal analysis (DSC) of iPP and polylactide (Figure 1.), the temperature range of thermal processing was established, and then the complete method of the forming of iPP/PLA filaments from a melt was developed. Blended fibres with good quality were obtained for the entire range of component concentrations.

The wide-angle X-ray diffraction patterns obtained for the investigated fibres (Figure 2.), show that the supermolecular structure of iPP/PLA filament after the formation process ("green fibres") is created in a rather similar manner to classical iPP fibres spun with a low take-up velocity. The distinct decrease of the intensity of all iPP crystalline peaks on the WAXS curve is observed. These changes prove that a special type of order-mesophase exists in the supermolecular structure of our fibres [7]. The mesophase structure called smectic or paracrystalline is characterised by a partial long range ordering of macromolecules, and is determined by diffraction reflexes at 2Θ of 14.8° and 21.2° respectively.

By comparison of the WAXS curves obtained for iPP fibres with different PLA content (Figure 2), we can conclude, that the increase of the modifier concentration

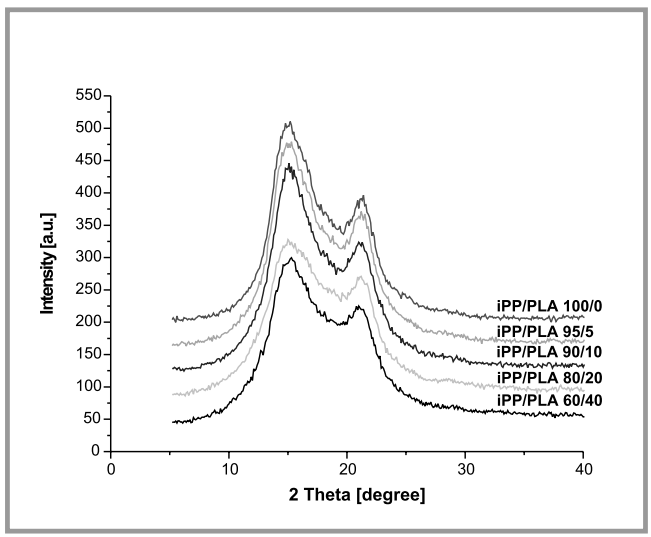


Figure 2. *WAXS curves for fibres formed from iPP/PLA blends with different PLA content. Structure created during the formation process.*

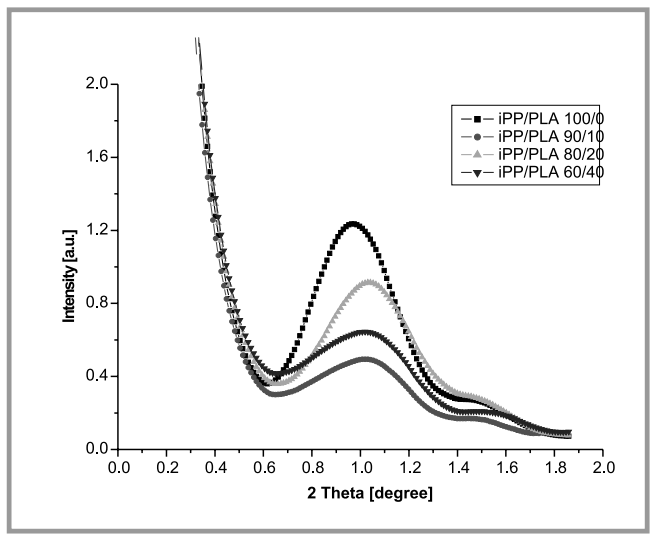
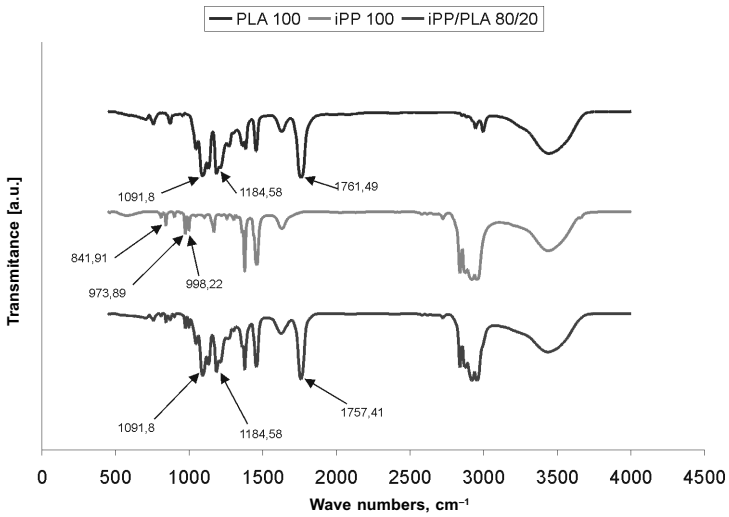


Figure 3. *SAXS curves (in the direction parallel to the fibre axes) for modified iPP/PLA fibres. Structure created during the formation process.*



Infrared frequency, cm^{-1}	Assignments
1754,40	νC=O
1761,49	νC=O
1184,58	ν_sC–O–C + $\tau_{as}CH_3$
1091,80	νC–O–C
998,22	ρCH_3, δCH, ωCH_2
973,89	ρCH_3, νCC_b
841,91	ρCH_2, νCC_b, νC–CH_3, ρCH_3

Denotations: ν – stretching, τ – twisting, ρ – rocking, δ – bending, ω – wagging, $_b$ – backbone, $_s$ – symmetric, $_{as}$ – asymmetric.

Figure 4. *The comparison of infrared spectra for samples of fibres obtained from poly (L-lactide), isotactic polypropylene and blend of iPP/PLA(80/20).*

changes the shape of these curves. The intensity of the peaks decreases. The changes prove that the index of order calculated in respect to the total amount of investigated samples decreases.

The results of the small-angle X-ray diffraction investigations in direction parallel to the fibres axes are presented in Figure 3.

The comparison of SAXS curves obtained for the different iPP/PLA filaments show the existence of the lamellar structure of iPP matrix [8] in all samples. The long period of this structure (calculated from Bragg's law) corresponds to the maximum varies from 9.1nm to 8.4nm for the fibres containing 100wt% of iPP and 40wt% of PLA respectively.

Based on FTIR analysis (Figure 4), the absence of chemical interactions between iPP and PLA was found. The components of the polymeric system investigated, without any compatibilisers, are probably only a physical polyblend.

Conclusions

The supermolecular structure of the fibres after the formation process was examined by means of X-ray methods. It was found that the crystalline structure as determined by WAXS was dominated by the smectic phase of iPP. The long period of this structure, as estimated by SAXS, decreases (from 9.1nm to 8.4nm) as the PLA content in the mixture increases.

The presented paper is a preliminary stage of studies on the biodegradability of the iPP/PLA polymer system.

❑

References

1. *Artzt, P.: Melliand Textilberichte, 79 (1998) pp. 125*
2. *Urbańczyk, G.: Physics of the fibre. Physical properties of fibres, WNT, Warszawa 1989*
3. *Karger-Kocsis, J. (ed.): Polypropylene. An A–Z reference, Kluwer Academic Publishers, Dordrecht 1999*
4. *Kister, G., Cassanas, G., and Vert, M.: Polymer, 39 (2003) p. 267*
5. *Jamshidi, K., Hyon, S.-H., and Ilkada, Y.: Polymer, 29 (1988) p. 2229*
6. *Zhang, J., Tsuji, H., Noda, I., Ozaki, Y.: Macromolecules, 37 (2004) p. 6433*
7. *Yang, R.D., Mather, R.R., Fotheringham, F.: Int. Polym. Processing, 14 (1999) p. 60*
8. *Włochowicz, A., Garbarczyk, J.: Polym. Int., 34 (1994) p. 253*

❑ ***Received 08.12.2004 Reviewed 10.02.2005***