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# Polyolefin Fibres with Waste Raw Material Content

## Abstract

*When companies process waste-drinkbottles, after the poly(ethylene terephthalate) flakes have been isolated, a waste mass remains which includes mainly the polymers of bottle caps manufactured from polyolefins, such as high density polyethylene (HDPE) and polypropylene (PP). This article presents the results of research aiming at checking the ability of using HDPE/PP polyolefin waste for spinning fibres. We have indicated that such material, after appropriate preparation, might be added to fibre-grade PP with a melt flow index (MFI) within the range of 25 to 35 g/10 minutes. Polyolefin staple fibres were spun with a 40% content of HDPE/PP waste raw material. The fibres with a linear density of 15 dtex were characterised by a tenacity of 17 cN/tex, elongation at break of 235%, a high degree of elasticity  $ER_{5\%} = 99\%$ , as well as of resistance to the influence of acid media similar to that of PP fibres, and only a little worse to the influence of alkalis.*

**Key words:** polyolefin fibres, polypropylene fibres, polyolefin wastes, polyolefin recycling, poly(ethylene terephthalate).

full-quality polymers which are easy to isolate in a pure form. Poly(ethylene terephthalate) – PET – waste from bottles remaining after drinks is an example of such waste. More than 50% of this waste is processed into staple fibres, whereas the remains are used to manufacture other products, such as bottles and thick films [1, 2]. Recently in Poland, the production of staple fibres from PET waste was started by the company IMP Comfort in Świdnica, which uses PET flakes offered by company IMP Polowat in Bielsko-Biała. The IMP enterprise has also been developing its hitherto production in the field of various kinds of nonwovens, including waste raw material.

In enterprises which process bottles dedicated to drinks, such as the IMP Polowat Company, after the isolation of PET flakes, a waste mass remains which contains polymer from the caps and bottle necks. After cutting the waste and preliminary washing with water, a polyolefin matter is obtained which contains minimum 95% of a high-density polyethylene (HDPE) mixed with polypropylene (PP). So far this waste has been offered to different consumers of plastics as thermoplastic material dedicated to mould products. Both enterprises, IMP Polowat and IMP Comfort, declared their interest in using the HDPE/PP waste product obtained from the caps of PET bottles to manufacture products for textile applications. The fibres produced from such a raw material could be applied to the thermal bonding the webs of nonwovens manufactured from waste raw material. So far PP staple fibres with linear density of 15 dtex and staple length of 60 mm have been used by IMP Com-

fort for this purpose. Fibres which fulfil the part of glue need not be characterised by good tensile strength properties, and therefore it is justified to use a recycled-polymer for its production.

The use of waste-polymers to manufacture nonwovens is a difficult problem and requires a detailed analysis of the undertaking's profitability. Fibre-grade polymers are characterised by special properties, considering their chemical structure, and physical-chemical characteristics at molecular, supermolecular, and morphological levels, as well as their homogeneity and purity [3]. As opposed to the chemical homogeneous PET flakes from bottles cut to pieces, essential difficulties in obtaining recycled products of good quality occur in the case of polyolefin waste, caused by the great material-differentiation of this group of polymers. We dispose of low- and high-density polyethylene (LDPE and HDPE), which are differentiated by their chemical structure (including the presence and amount of branches), by their density, and what is most important, by their melt viscosity measured by the melt flow index (MFI) [4]. Polyolefin waste material is firstly processed with the aim of obtaining mould products. Disposable products, such as seedling plant pots, make up an important part of them. Textile waste, which is selected, pure and homogeneous - considering the raw material used, is most often used in the enterprise where they were manufactured. Modern equipment devoted to manufacturing spun-bonded and melt-blown nonwovens enables the immediate return of a part of PP waste products directly to the extruder feeding

## Introduction

Waste plastic is a great burden for the environment. Economical and ecological aspects demand the use of methods allowing the elimination of polymer waste, which would be more and more efficient, and at the same time enable the obtaining of useful material of the best possible quality. Some polymer waste includes

the manufacturing lines [5]. PP re-granulates of good quality may be added to the initial raw material [6]. Recently, great interest has been awoken by the possibility of using waste polymers devoted to fibres used for manufacturing geotextiles. Considering the low price and good resistance against the influence of acid and alkali environment, PP is the main raw material for these fibres. Research carried out at the Centre for Innovative Materials, Bolton Institute (UK), indicated that an addition of maximum 20% of HDPE waste to PP is available in the case of manufacturing fibres devoted to geotextiles [7].

This article presents the results of research aimed at checking the usefulness of HDPE/PP polyolefin waste obtained from bottle caps to manufacture fibres.

## Materials and investigation methods

### Polymers:

- HDPE/PP waste polymer from PET bottle caps, supplied by IMP Polowat. The company offers this polymer with the following specification:
  - HDPE/PP content - minimum 95%
  - PET content - 0 – 5.0%
  - other contaminations (PVC, paper, metal foil) - 0 – 50 ppm
  - moisture - 5 – 15%
  - apparent density - 0.350 – 0.430 g/cm<sup>3</sup>
  - dust content - 0 – 0.1%
- polypropylene (PP) – Malen P, S 901 type, MFI = 25 – 35 g/min, and S 702 type, MFI = 11 – 16 g/10 min., produced by PKN Orlen Poland;
- polyethylene (HDPE) – Eraclene, MFI = 0.8 g/10 min., produced by Eni Chem Company, Italy.

### HDPE/PP waste treatment for processing fibres

The PET remains of HDPE/PP waste were rejected by the process of static flotation in water with an addition of surfactants. The polyolefin fraction collected was filtered off in sieves for 8 hours, and next dried for 6 hours at a temperature of 80 °C, at a decreased pressure of 10 mm Hg. The HDPE/PP polyolefine flakes were melted by a re-granulation line equipped with a sieve filter of 16,000 meshes/cm<sup>2</sup>, at a temperature of 280 °C. Re-granulates of waste-matter not processed by preliminary purification, whose melt was not filtered by a

sieve filter, were also prepared for our investigation. Pure PP (Malen PS 901) was added to a part of the waste-material during melting. In this way, independent of the purified re-granulate of the waste-material (100% of HDPE/PP), the following re-granulates were prepared:

- 20% PP + 80% of HDPE/PP waste-mater,
- 40% PP + 60% of HDPE/PP waste-mater,
- 60% PP + 40% of HDPE/PP waste-mater,
- 80% PP + 20% of HDPE/PP waste-mater.

### Manufacturing fibres

Laboratory spinning tests of fibres spun from blends of different shares of the HDPE/PP waste material and pure PP were carried out with the use of a test spinning installation in IBWCh. The process was conducted within the temperature range of 260 °C – 300 °C, with the use of a six-hole spinneret, at an efficiency of 16 g/minute and take-up velocity of 500 m/minute. The fibres were drawn with the use of a J6/2a drawing frame from Rieter, heated by a godet at a temperature of 110 °C, at drawing ratios of 2.5 to 3.7 and a speed of 267 m/minute. The fibre filaments obtained in this way were wound on bobbins, and next cut into segments of 60 mm length, in preparation for further investigation.

Staple fibres with linear density of 15 dtex and staple length of 60 mm were manufactured with the use of an industrial installation, by a single-stage process, which included spinning and drawing at a temperature of 220 °C, extruding efficiency of 100 kg/hour, and a final feeding speed of the tow at the input of the crimping machine of 23 m/minute.

### Methods of property estimation of waste-polymers and fibres

- Ash content estimated in accordance with standard PN-EN ISO 3451-1.
- Estimating the PE-HDPE, PP, and PET

content by the method of selective dissolving in xylene according to [8].

- Solid contaminations estimated by chemical methods elaborated in IBWCh.
- Melt viscosity by assessing the melt flow index (MFI) with the use of method A in accordance with standard PN 93/C89069 (with a spinneret hole of 2 mm, at a temperature of 200 °C), and method B developed in IBWCh (using a spinneret hole of 0.5 mm within the temperature range of 200 °C to 300 °C).
- Melt filterability on the basis of determining the mass- and the time-filterability coefficients ( $F_m$  and  $F_t$  respectively) with the use of a Plasticorder apparatus from Brabender, in accordance with standard EN 13900-5.
- Fibres' physical-mechanical properties assessed in accordance with standards PN-EN ISO 5079 and PN ISO 1973.
- Fibres' surface estimated with the use of QUANTA SEM, from FEI.
- Resistance of fibres against acids and bases by determining the tensile breaking force and elongation at break of fibres treated with a 1% sodium hydroxide solution and a 1% sulphuric acid solution for 1,000, 4,200, and 9,000 hours, at a temperature of  $19 \pm 1$  °C.

### Results of investigation

In order to determine the processing properties of the HDPE/PP waste-matter, 4 lots (P-1, P-2, P-3, and P-4), each of 100 kg of broken-up PET bottle caps, were prepared. The samples were taken at different time intervals over one year, from March 2004 to March 2005, from IMP Polowat's production line in Bielsko-Biala.

On the basis of the physical-chemical analysis carried out, the raw material content and the purity (ash and content of solid & not melting contaminations) of each of the HDPE/PP waste-material lots were estimated.

Table 1. Estimation of selected lots of polyolefin wastes.

HDPE/PP waste-mater	Raw material content, %			Ash, %	Cellulose, %	Other solid impurities, %
	PET	PP	HDPE			
P-1	1.0	36.2	61.2	0.51	0.09	1.00
P-2	18.1	24.2	55.1	0.55	0.19	1.86
P-3	3.4	16.0	76.6	0.57	0.04	3.39
P-4	3.0	27.4	69.0	0.60	-	-

The results listed in Table 1 indicate that when considering the impurities, a filtration process must purify the waste polymer devoted to spinning fibres. Chalk and pigments, which are introduced into the HDPE and PP injection mould together with the bottle caps, are part of the solid contamination.

It was stated that the particular lots were differentiated by the raw material content. However, according to the producer's specification, the PET content should not exceed 5%; in reality lot P-2 was characterised by 18% of this polymer. The PP content in the waste was within the range of 16% for lot P-3 to 36% for lot P-1, whereas the HDPE polyethylene fraction content was from 55% for lot P-2 to 77% for lot P-3.

The re-granulates were prepared from samples taken from lots P-1, P-2 and P-3. We stated that the MFI determined by method A for the re-granulates P-1/R, P-2/R and P-3/R had values within the range of 2 to 3 g/10 minutes. These are values characteristic for polyolefin devoted to injection processing, whereas PP granulates with MFI of 11 to 35 g/10 min. are devoted to textile manufacturing. Only to very thick fibres, above 50 dtex, is PP of MFI = 9 g/10 minutes applied.

The values of the filterability coefficients,  $F_m = 140 \text{ MPa} - 230 \text{ MPa}$ , and  $F_t = 0.8 \text{ MPa} - 3.9 \text{ MPa}$ , determined for the re-granulates P-1/R, P-2/R, and P-3/R, testify the high content of impurities in the waste-polymer. In the case of tests of a re-granulate from lot P-2, which contains 18% of PET, we observed several piercings of the filtration meshes in the Plasticorder measuring apparatus.

Our own experience until now indicates that the presence of PET in the polyolefin matter dedicated to obtaining fibres, even an amount of 1 – 3%, results in many disturbances of the spinning process, which are caused by the lack of the components' mixing ability and significant differences in their melting temperatures.

After rejecting the PET fraction from each of the lots (P-1, P-2, P-3, and P-4) a mixture was prepared of equal weight proportions. According to theoretical calculations, the mixture included 28.4% of PP and 71.6% of HDPE.

Table 2 presents test results estimating the filterability of the re-granulate ob-

tained from the HDPE/PP waste polymer mixture. For comparison, the results of filterability estimation of the Eraclene polyethylene and Malen polypropylene are also included in Table 2. However, during the separation of the polyolefin fraction from the PET fraction and rejection of solid impurities by the static flotation process in which the mixture was several times washed, the filterability coefficients determined for the HDPE/PP waste polymer several times exceeded the filterability coefficient values of polyolefin polymers used for manufacturing fibres.

The results of estimating the HDPE/PP waste polymer obtained from the bottle-caps indicate that the material demands a special preparation procedure in order

to obtain the correct purity and viscosity, which would guarantee the proper procedure of spinning the fibres.

Solid impurities were rejected from the HDPE/PP waste polymer by filtration during the melting of the polymer flakes in a two-screw extruder equipped with an exchangeable mesh filter. After filtration, the MFI and the purity of the waste polymer devoted to fibres were determined. Figure 1 presents the dependence of the MFI, determined by method B, on temperature for the following different polyolefin polymers:

- HDPE/PP waste polymer,
- blends of this polymer with PP (with MFI = 26 g/10 minutes), and
- pure PP fibre-grade granulates (Malen P – type S 901 and type S 702).

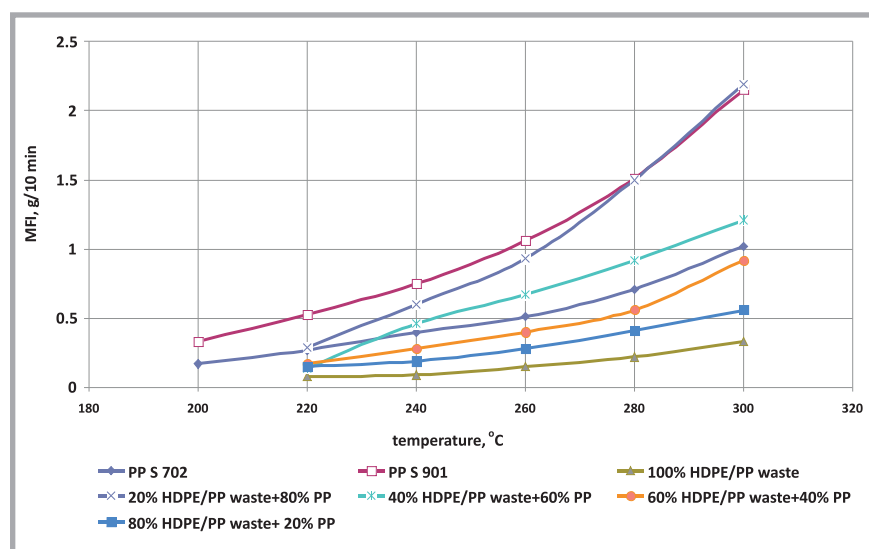


Figure 1. Dependence of the melt flow index (MFI) on temperature for different polyolefin polymers.

Table 2. Estimation results of the filterability of the HDPE/PP waste polymer and pure polyolefin granulates;  $F_t$  – time filterability coefficient,  $F_m$  – time filterability coefficient.

Rodzaj polimeru	Initial pressure, MPa	Final pressure, MPa	$\Delta p$ , MPa	Polymer mass, g	Process time, min	$F_t$ MPa/min.	$F_m$ MPa/kg
HDPE/PP wastes	40	280	240	121	13	18.6	1990
HDPE Eraclene	49	119	70	1297	125	0.57	54.0
PP S 702	14	70	56	1920	260	0.002	29.1
PP S 901	14	49	35	2205	200	0.175	15.9

Table 3. Comparison of properties of purified re-granulates from waste polymers (PR) prepared for fibre formation and fibre-grade PP granulates.

Parameter	Re-granulate from HDPE/PP wastes		Fibre-grade PP		
	100 % PR	40%PR +60% PP	S 901	S 702	
Ash content, %	0.68	0.28	0.011	0.01	
Chalk content, %	0.27	0.18	-	-	
MFI $\phi = 2 \text{ mm}$ , 230 °C, g/10 min	3.6	15.3	26.0	14	
Filterability coefficients $F_t$ and $F_m$	MPa/min.	8.3	5.0	0.18	0.002
	MPa/kg	760	445	15.9	29.1

The very low MFI values of the HDPE/PP waste polymer verify the fact that the melt viscosity of this polymer is too high for its processing into fibres. In order to improve the spinnability of the HDPE/PP waste polymer, it is necessary to mix an addition of polypropylene with low viscosity to an amount above 40%.

This was confirmed by fibre spinning tests carried out in laboratory-scale. A correct procedure of the process stages of fibre forming by means of spinning and drawing was observed in the case of the PP blends with MFI = 26 g/10 min., mixed with waste material with proportions of 80%/20% and 60%/40%. From blends, which contained 80% of the waste polymer, it was not possible to spin fibres. From blends, which contained 60% and 50% of the waste polymer, it was possible to spin and take-up raw fibres, but many breakages occurred during the drawing process.

Table 3 presents the results of estimation of the purified re-granulates from the HDPE/PP waste polymer devoted to fibres. For comparison, the estimation of

**Table 4.** Comparison of physical – mechanical properties of polyolefin fibres with a content of HDPE/PP waste polymers and PP fibres.

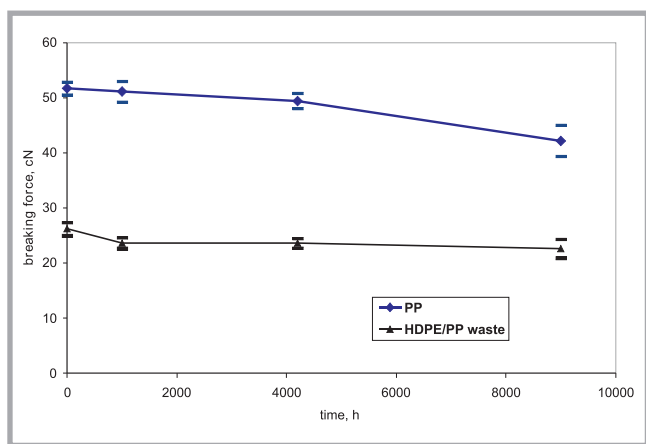
Parameter	Unit	Fibres with a content of HDPE/PP waste polymers	PP fibres made in Belgium
Linear density	dtex	15.3	14.6
	CV%	2.58	3.7
Breaking force	cN	26.2	51.7
	CV%	11.2	5.38
Tenacity	cN/tex	17.1	35.4
Elongation at break	%	235	220
	CV%	71.7	39.3
Loop breaking force	cN	45.0	105
	CV%	15.4	7.65
Loop tenacity	cN/tex	14.7	35.8
Relative loop tensile strength	%	86	100.0
Elastic recovery at elongation 5%	%	99	88.0
	CV%	3.02	5.99
Initial tensile strength modulus	cN/tex	127	226.0
	CV%	19.1	23.6

the S 901 and S 702 fibre-grade PPs were also included.

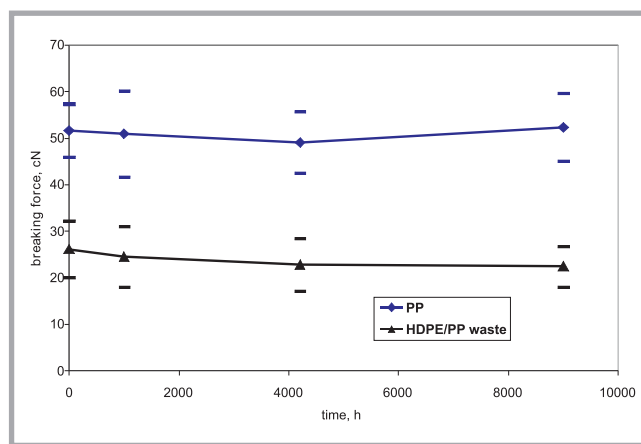
From the blends of 60% PP and 40% of the HDPE/PP waste polymer a batch of staple fibres was manufactured in industrial conditions. The process proceeded without any disturbances. However, the fibres absorbed significantly larger

amounts of the spin finish (preparation), and required additional drying before entering the cutting machine by the tow.

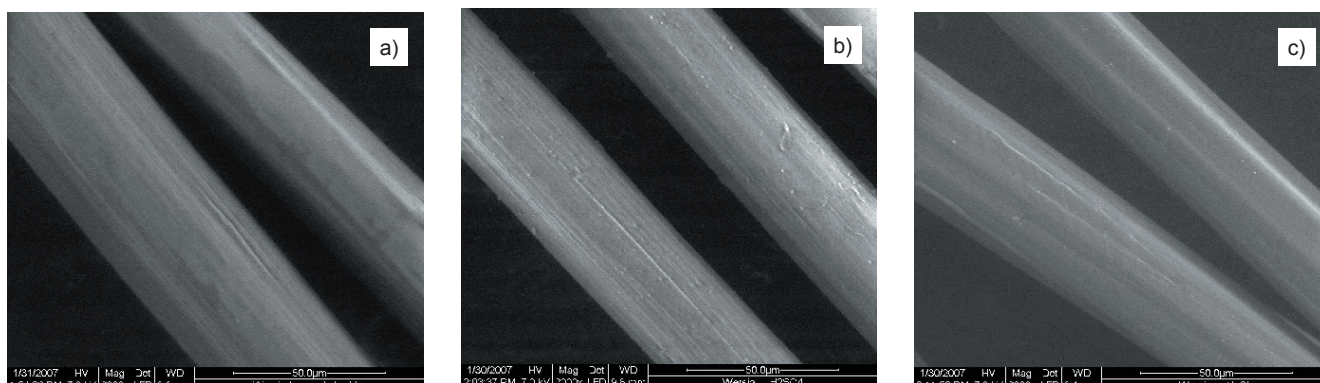
Table 4 presents a comparison of the properties of polyolefin fibres manufactured from a blend containing 40% of the HDPE/PP waste polymer with those produced from pure PP, using PP



**Figure 2.** Changes of the breaking force of polyolefin fibres treated by an acid medium.



**Figure 3.** Changes of the breaking force of polyolefin fibres treated by an alkali medium.



**Figure 4.** Surfaces of polyolefin fibres with a content of HDPE/PP wastes: a) not processed, b) after a 9,000 hours treatment by acid medium, c) after a 9,000 hours treatment by alkali medium.

fibres made in Belgium and used in IMP Comfort, Świdnica for thermal bonding of nonwoven webs obtained from waste raw material.

Fibres with a content of 40% HDPE/PP waste polymer with a linear density of 15 dtex have a tenacity of about 17 cN/tex, which means half the tenacity of PP fibres. The elongation at break of air conditioned fibres has similar values for both samples tested, but the dispersion of the results of this parameter for the particular measuring tests is significantly higher for those fibres which contain waste. The elastic recovery at 5% elongation ( $ER_{5\%}$ ) of fibres with waste polymer content was higher than the  $ER_{5\%}$  of PP fibres. The values were 99% and 88%, respectively. A distinct difference was observed in the initial drawing. For PP fibres we assessed a value of 226 cN/tex of this modulus, which was nearly twice that assessed for fibres with a waste content (126 cN/tex).

Figures 2 and 3 present changes of the breaking force of polyolefin fibres with a content of HDPE/PP waste which was treated by acid and alkali mediums. The curves are marked by the average values of 25 measurements and the average confidence interval. For comparison, the results of PP fibres are presented in the same way. The fibres with waste content lose about 10% of its tensile strength after 1,000 hours of acid medium impact. A further decrease in tensile strength is small, and only after 9,000 hours the drop amounts to 15%. In the case of PP fibres, the factors are equal to 1% and 18%, respectively. In an alkali medium the fibres from polymer waste lose about 14%, whereas the PP fibres are totally resistant to alkalis.

Figures 4.a – 4.c present the surfaces of polyolefin fibres with HDPE/PP content, estimated with the use of SEM, at magnification  $\times 2,000$ . No changes are visible on the surfaces of fibres submitted to the action of alkali and acid mediums over 9,000 hours. The pictures prove the fibres' resistance to the action of acid and alkali mediums.

## Summary

The ability of using HDPE/PP polyolefin waste material obtained from PET bottle caps to produce staple fibres was checked by us. We stated that after appropriate

preparation, which means rejecting PET residues and filtrating the polymer melt, such material may be added to a fibre-grade PP with MFI of 25 g/10 minutes to 35 g/minutes to an amount of up to 40%. Polyolefin staple fibres were manufactured with the use of 40% HDPE/PP waste raw material. The fibres with linear density of 15 dtex had a tenacity of 17 cN/tex, elongation at break of 235%, and high elastic recovery of  $ER_{5\%} = 99\%$ . The fibres are characterised by resistance to the action of acid medium, similar to that of PP fibres, and only a little worse resistance to the action of alkalis.

Fibres with a content of HDPE/PP waste material were applied to the thermal bonding of nonwoven webs made from textile waste raw material. The results of these experimental investigations will be published in the foreseeable future. The manufacturing of fibres with a content of polyolefin waste will be implemented by IMP Comfort with the use of a new production line manufacturing synthetic staple fibres from waste raw material.

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