The Effect of Reprocessing of Polypropylene on Fiber Spinning

C. CHRISTIANI,¹ C. KLASON,^{1,*} R. SHISHOO²

¹ Department of Polymeric Materials, Chalmers University of Technology, S-412 96 Gothenburg, Sweden

² The Swedish Institute for Fiber and Polymer Research, S-431 22 Mölndal, Sweden

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ABSTRACT: Because of the environmental considerations, recycling is becoming a very important factor in any production planning within the fiber industry and textile industrial sectors. One of the determining factors for recycling of fibrous assemblies into fiber is the spinnability of the incoming material. The hypothesis underlying the present work is that by carrying out rheological analysis of the polymer melt it should be possible to predict the state and level of degradation of the polymeric fibrous waste material—hence its spinnability. Our experimental studies show that a melt spinning grade polypropylene could be recycled five times without significant decreases in fiber properties. Afterward, the fiber production failed in the cold-drawing step before melt rupture occurred in the draw-down step. Rheological analysis of the melt in long-term stability experiments showed that unspinnable melts show an abrupt change in loss factor at a certain level of loss and storage modulus. The reasons for this behavior are associated to changes in molecular weight, viscosity, and melt strength. © 1999 John Wiley & Sons, Inc. J Appl Polym Sci 73: 1859–1867, 1999

Key words: polypropylene; fibers; spinnability; recycling

INTRODUCTION

Because of environmental considerations, recycling has become a very important factor in any production planning within the fiber industry and textile industrial sectors. Recycling of fibrous materials can be divided up into industrial waste recycling and consumer waste recycling, the difference being that industrial waste never reaches the consumer and can directly be put back into production. Significant differences in properties in consumer vs industrial waste exists since the industrial waste material does not get exposed to degradation in terms of oxidation, radiation, wear, etc. When discussing recycling of polymeric products and especially synthetic fibers into fibers using melt spinning, the demands on the quality on the incoming recyclable materials are extremely high. It would be costly to have interruptions in the fiber production or performance variations of the final product as the result of fiber breakage, caused by contamination of the melt or changes in the molecular weight of the polymer.

Several studies have been reported in the literature describing the results when a waste material has been added to a material stream in a polymer processing unit in order to study the effects on the final plastic product. Boldizar et al.¹ have shown that for injection molded and extruded polyethylene, large variations could occur between simulated industrial waste and simu-

 $[\]ast$ Professor Carl Klason died in July 1997, a great loss to the polymer science community.

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lated consumer waste. The latter, being exposed for thermo-oxidative aging and repeated processing, show greatly reduced mechanical properties after several recycling cycles. Repeated processing by itself or only aging will not decrease the properties of the product to such an extent.

Bernardo et al.² have developed an equation capable of predicting the properties of mixtures of virgin and waste polyolefines processed by injection molding. Their theoretical predictions agree well with experimental data and according to their results melt flow index, i.e., viscosity, is a suitable index method for evaluating the degradation of polyolefines after repeated processing.

A major problem in recycling is the reduction in stabilizer activity in processing. Horrocks et al.³ have studied that problem in the processing of polypropylene geotextile tapes by successively adding new stabilizer to the material before processing. They have found that up to 25% by weight of waste material can be added to the virgin material for achieving acceptable levels of tenacities. On the other hand, if stabilizers are added as master batch to the material, the level of waste material inclusion can be as high as 75% by weight.

Not much published literature is available related to the recycling of waste textile fiber into fiber form. Davies and Horrocks⁴ have studied the effect of addition of waste polypropylene fiber prior to extrusion on the final fiber properties. It is shown that about 20% by weight of waste material can be added with acceptable reductions in fiber properties. They argue that if stabilizers had been added, the level of added waste could have been increased into the material stream.

According to the cited articles, it has been possible to recycle waste fibers into fibers. A determining factor for recycling of fibrous assemblies into fiber is the spinnability of the incoming material. Spinnability is a very complex and poorly defined concept. The term *spinnability* refers to the possibility of the melt to be drawn into a continuous fiber. It is influenced by rheological and deformation properties through several factors such as molecular weight and architecture, additives, and processing conditions.

However, an important consideration in the melt spinning of a fiber into a final product is the necessity of the as-spun fiber to be postdrawn. Therefore, the requirement on the base polymer to be extruded into industrial fibers is that the fibers have to withstand both hot- drawing in the molten state and cold drawing in the solidified



Figure 1 Diagram of the melt spinning process. "HD" refers to hot drawing and "CD" refers to cold drawing.

state (Fig. 1). In the hot-drawing step, the polymer melt is extruded through small capillaries in the die. Due to shear forces, the polymer chains are oriented along the fiber axis. At the exit the chains relax and die swell occurs. The melt string is then subjected to extensive spin line stress and orientation is induced in the filament. The spin line stress level is determined both by the material and the process parameters such as the spinning speed, molecular weight, extrusion temperature, extrusion velocity, and cooling conditions. The solidified filament after drawing consists of crystalline and noncrystalline regions that coexist in complicated structures. The mechanical properties of the fibers depend on the relation between the two areas as well as the molecular orientation within each. In hot drawing, the orientation depends on the stretch rate and not the amount of stretch. In cold drawing, the amount of stretch is determining for the orientation. In the cold-drawing step, deformation and rearrangement of the crystalline region occurs. Eventually microfibrils are formed and these consist of blocks of chainfolded crystals and noncrystalline regions. The fiber deformations occurs mainly by two mechanisms; either uniformly or by necking. The drawing conditions determine which of the deformation routes is preferred.

Beyreuther⁶ has shown with modified polymer melts that a well-spinnable polymer has a loss factor, tan δ , greater than 10 and a nonspinnable melt has a loss factor less than 5. One of the conclusions drawn is that for modified polyamides



Figure 2 Illustration of the processing steps involved in the experimental setup. "E" refers to extrusion, "A" to analysis, and "S" to spinning.

and polyesters the ratio of the loss and storage modulus of the melt correlates with the spinnability.

Using this rheological approach to define spinnability, it might be possible to predict the spinnability of a given polymer waste, e.g., with or without the addition of recycled polymer. The hypothesis underlying the present work is that by carrying out rheological analysis of the polymer melt it should be possible to predict the state and level of degradation of the polymeric fibrous waste material hence the spinnability.

MATERIALS AND EXPERIMENTAL

A polypropylene melt spinning grade, HG235J, from Borealis was extruded 7 times through a Betol extruder in order to simulate melt spinning steps (see Fig. 2). The three extruder heating zones were adjusted to 205, 215, and 225°C, respectively. After each extrusion, a sample was taken for analysis and melt spinning. The equipment for melt spinning was a Lab spin II from ESL.

The heating zones in the spinning apparatus were set to 205, 215, and 225°C in the extruder and 225°C in the spinning head. The prepump pressure was 8.7 MPa. A spinneret with 55 circular holes of 0.35 mm in diameter and 1.4 mm capillary length was used. The extrusion rate was approximately 100 g/min. The take-up speed was 300 m/min and the fibers were hot drawn, draw ratio 25, and cold drawn, draw ratio 3.9. The homopolymer had a density of 903 kg/m³, a melt flow rate of 25 g/10 min, and a melt peak temperature of 162–165°C. The analyzed samples were given the code names according to the following system: virgin granules were denoted 0E and material once extruded was called 1E, twice extruded 2E, etc. Similarly, fiber produced from 0E material was called 0S, S referring to spinning, and fiber produced from 1E was called 1S, etc.

ANALYTICAL PROCEDURE

The granules were analyzed with regard to molecular weight, molecular weight distribution, rheological properties, and melt strength in order to study the influence of processing on the melt state. The melt-spun fibers were analyzed with regard to tensile properties, orientation and thermal properties.

Size Exclusion Chromatography

The effect of extrusion upon molecular weight and molecular weight distribution was analyzed using size exclusion chromatography. The equipment consisted of a Waters 150 CV system. The solvent was 1,2,4-trichlorobenzene and the dissolution time was 16 h at 135°C. The solution was filtered in 0.45 μ m metal filter and the concentration was approximately 2 g/dm³. Narrow PS standards and broad PE standards were used in analyzing the results. The results were then recalculated using universal calibration to fit PP.

Dynamic Mechanical Testing

The rheological properties of the melt were analyzed using a Dynamic Stress Rheometer from Rheometrics with parallel plates. A dynamic frequency sweep default test at 0.01-70 Hz, 180° C, and 250 Pa in stress was used to obtain a flow curve of the melt. The second experimental test was performed using a dynamic time sweep default test at a frequency of 0.1 Hz at 225°C. The storage and loss modulus, G' and G'' were analyzed for the above-mentioned tests.

Evaluation of Melt Strength

The melt strength was measured using a Ceast capillary viscometer with a capillary of 40/1 in length/diameter ratio. The extrusion speed was 3.04 mm/s and the up take reel had a constant acceleration of $3.3*10^{-3} \text{ mm/s}^2$. The equation for calculating the breaking stress, i.e., melt strength, is taken from the work of Becker and co-workers.⁷

$$\sigma_f = \frac{4FD\rho_c}{\pi\rho_f d_c^2} \tag{1}$$

$$D = \frac{\nu_f}{\nu_c} \tag{2}$$

where *D* is the draw ratio defined as in eq. (2), ρ_c and ρ_f are the densities of the melt and the fiber, *F* is the drag force applied to the fiber, and d_c is the capillary diameter. The v_f and v_c are the peripheral speed of the winder and the speed of the extrudate.

Differential Scanning Calorimetry

The differential scanning calorimetry (DSC) measurements were performed on a Perkin-Elmer Pyris 1. The experiments were run from 50 to 220° C in a scanning cycle. The granules were given the same thermal history by scanning from 50 to 220° C at 40° C/min, cooling at 40° C/ min to 50° C and then scanning at 10° C/minute to 220° C. The fibers were run in the following cycle: scanning from 60 to 200° C at a rate of 10° C/min to analyze the melt endotherm and then cooling at 10° C/min to evaluate the crystallization exotherm.

Tensile Testing

The fibers were tested in an Alwetron tensile tester. The fibers were heat treated taut at 120° C for 7 min before analysis. A 500 N load cell was used and the fibers were tested at a cross-head speed of 200 mm/min with a gauge length of 100 mm. A bundle of 55 fibers was tested. The calculation of the elastic modulus from the tensile measurements was done by fitting a 5th-order curve to the first 200 points of measurement. The derivative of that curve was then plotted against strain and the *y*-axis interception was taken as the elastic modulus. The average of 10 measurements was reported as the modulus.

IR Spectroscopy

The Fourier transform IR measurements were performed on a Perkin-Elmer 1720 X FT-IR spectrometer using a grid polarizer model IGP 228 from Cambridge Physical Sciences. The fibers were analyzed with regard to chain degradation and molecular orientation in the fiber. The molecular orientation in the fiber may well be characterized using plane polarized light.⁸ The crystalline orientation gives rise to several absorption peaks. The most suited to study, however, appears to be at 842 cm⁻¹. The orientation of the amorphous phase is more difficult to establish.⁹ The amorphous absorption bands coincide with some of the crystalline and these are difficult to separate. The band at 974 cm⁻¹ provides a good estimate of the general molecular orientation in the fiber. The orientation is defined as the ratio between absorption of plane polarized light parallel and perpendicular to the fiber axis. Degradation products of polypropylene contain carbonyl groups and double bonds,¹⁰ and the carbonyl index was calculated for the absorption peak at 1718 cm⁻¹. The absorbency was normalized with respect to the 974 cm⁻¹ peak.⁴

RESULTS AND DISCUSSION

The most notable result of the processability of the degraded material is that after six consecutive extrusions fiber breakage occurred in the cold-drawing step during spinning. After seven extrusions the fiber breakage was so severe that the melt was denoted unspinnable. The most important observation has been that the spinnability in the molten state at this low take-up speed is not affected by the thermal and mechanical degradation. Rather, the possibility to cold draw the fibers has changed dramatically. The effects of processing can be analyzed in four steps—viz. effects of molecular level, melt rheology, orientation, and solidification in the draw-down step and physical and mechanical properties of the fiber.

Effects on Molecular Level

Size exclusion chromatography (SEC) measurements made on the polymer after each extrusion step in the sequence 0E to 7E showed that there occurs a significant decrease in the molecular weight directly after the first extrusion. After further processing, no significant change occurs in the weight average molecular weight M_w and the number average molecular weight M_n , as found in Table I. The subsequent processing does not seem to affect the chain length. After the first processing step, there is a shift in the whole molecular weight distribution (MWD) because absolute values of M_n and M_w are significantly lowered. However, the MWD also become much wider. Further processing narrows the MWD but it still remains broader than the initial distribution.

The unusual behavior of the molecular weight during processing, especially the marked de-

Sample	$M_n\cdot 10^{-3}$	$M_w\cdot 10^{-3}$	M_w/M_n
$0\mathrm{E}$	52.0	205	3.9
$1\mathrm{E}$	22.2	135	6.8
$2\mathrm{E}$	22.0	133	6.0
3E	25.2	123	4.8
$4\mathrm{E}$	26.5	139	5.2
$5\mathrm{E}$	24.4	129	5.3
$6\mathrm{E}$	25.5	128	5.0
$7\mathrm{E}$	25.9	119	4.6

Table I Table of Results of the Molecular Weight Determination of Virgin Granules, 0E, and Material After Processing 1E–7E

crease after the first processing step, led us to undertake a comparative study of two other polypropylene materials. Sample HH 315 is a homopolymer for injection molding and BE 170 is a polypropylene block copolymer for injection molding, both from Borealis. In Figure 3(a,b), the three materials have been exposed to different treatments before SEC analysis-no pretreatment, remelting, and mild thermomechanical treatment. It is seen that remelting before analysis can induce the decrease in MN and Mw of HG 235. The other two materials do not behave in a similar manner to HG 235. Hence the drastic decrease after the first processing step in the original study cannot be attributed to an actual decrease in chain length.

Misra et al.¹¹ have found that a melt having a wide MWD is less spinnable than a narrow one. The suggested reason behind this is related to differences in elongational viscosity vs elongational rate, which will affect the drawing after solidification since the crystalline structure of the fibers is changed.

The stabilizer activity was tested by oxygen induction time on the degraded products. The results showed a very large scatter. The temperature of oxidation was also measured, and it was seen that the temperature increased the more processed the material became (see Table 2).

It is noteworthy that the unprocessed material has a lower temperature of oxidation than the material being processed seven times. This can be explained by the fact that processing enables dispersion of agglomerated stabilizer resulting in inactive stabilizer successively becoming active at the surface. One should be careful in drawing conclusions of stabilizer activity from the temperature of oxidation since it is both surface dependent and a diffusion-controlled process.¹²



Figure 3 Comparison of three different PP materials, each treated in three different ways before SEC analysis. (A) No treatment. (B) Remelting. (C) Mild thermomechanical treatment.

IR studies of degradation products showed an increase in carbonyl groups between virgin granules and material extruded seven times. The normalized absorption peak increased from 0.008 to 0.02. During drawing, these degraded regions may result in stress-hence fiber breakage. Regions of stress concentration may also be a result of gelation. However, no gel particles larger than 45 μ m were found in the SEC analysis. These degraded regions will accumulate in the noncrystalline areas and affect the drawability.

Table II	Results	of Stability	Measurement	by
Temperat	ture of O	Dxidation		

Sample	Temperature of Oxidation (°C)
Unstabilized HG 235 ^a	189.2
0E	198.2
7E	205.2

^a Supplied by Borealis Polymers Oy.

Sample	Melt Strength (MPa)	8	Max. DDR
0E	18.6	5, 2	430
1E	14, 5	2, 5	423
$3\mathrm{E}$	13, 1	2, 7	387
7E	9, 1	2, 0	340

Table IIIResults of the Melt StrengthEvaluation^a

^a The "s" refers to standard deviation.

Melt Rheology

A common method of evaluating spinnability is measurement of the melt strength. In our experiments it was found that under the chosen spinning conditions, the spinnability of the melt was enough to form a fiber. The results of the melt strength evaluation are shown in Table III. It is evident that the melt strength has decreased by approximately 50% of the initial strength. It can also be seen in Table III that the maximum drawdown ratio (DDR) decreases as a result of repeated processing. It is slightly erroneous to denote it "maximum draw-down ratio" as the fibers normally failed in the solidified phase and not in the melt at the exit of the capillary.

The melt string was subjected to an acceleration of $3.3 \cdot 10^{-3}$ mm/s² during the testing. It was found that a fiber subjected to an acceleration of $3.3 \cdot 10^{-2}$ mm/s² did not break even though the draw-down ratio, DDR, was approximately 490. One possible explanation for this behavior is a strain-hardening effect of the polypropylene melt string due to increased shear or the shear-thinning effect of PP.¹¹

It was found in the dynamic viscosity measurements that the viscosity increased after the first processing step (Table IV) despite the fact that M_n and M_w decreases. A similar phenomenon was observed earlier¹¹ where resins with the same Melt Flow Rate but different MWD were tested. Resins with broader MWD had generally higher



Figure 4 Melt viscosity vs time at constant stress. Designations 0–7E refers to the number of extrusions prior to analysis. The tests were run at 225°C.

dynamic viscosity than narrow MWD at low scanning frequencies. Whether the above holds true for the case where the molecular weight has decreased to such an extent as in our results is not clear.

It was found that granules having lower zeroshear viscosity than the virgin granules such as 5E, 6E, and 7E have poor processability. However, the observed melts did not have problems in fiber formation in the extrusion step and clearly managed the draw-down ratio applied to the melt string.

The rate of change in viscosity, at low frequency, is illustrated in Figure 4. This experiment gives an indication of the stabilizer activity of the material. It is seen that samples having lower viscosity than the virgin granules, 0E, are almost unspinnable.

The slope of the curves for samples 5E and 7E is larger at longer times compared to the others.

Beyreuther⁶ proposed that the polymer melt string can be classified from "not spinnable" to "well spinnable" by plotting the loss modulus vs the storage modulus. In our results (Fig. 5) this is not evident; the curves overlay despite the fact that they are ranging from well spinnable to not spinnable melts.

Table IV Zero-Shear Viscosity η and Cross-Over Modulus G_c , Dependence Upon Processing^a

Sample	$0\mathrm{E}$	$1\mathrm{E}$	$2\mathrm{E}$	3E	$4\mathrm{E}$	$5\mathrm{E}$	6E	$7\mathrm{E}$
η (Pa.s) $G_c \cdot 10^{-4}$ (Pa)	$\begin{array}{c} 1530\\ 2.5\end{array}$	$\begin{array}{c} 1820 \\ 2.5 \end{array}$	$\begin{array}{c} 1760 \\ 2.5 \end{array}$	$\begin{array}{c} 1740 \\ 2.6 \end{array}$	$\begin{array}{c} 1530\\ 2.5\end{array}$	$\begin{array}{c} 1430 \\ 2.7 \end{array}$	$\begin{array}{c} 1300\\ 3.0 \end{array}$	$\begin{array}{c} 1280\\ 3.2 \end{array}$

^a The measurements were made at 180°C.



Figure 5 The relationship between loss modulus G'' and storage modulus G' for some samples run at 180°C, in the frequency range of 0.1–70 Hz and with an applied stress of 250 Pa.

Study of the so-called long-term stability experiment, in Figure 4, in terms of loss modulus and storage modulus, gives interesting results. The experimental points for all the samples lie on the same line denoted A in Figure 6. The samples OE, 1E, and 3E follows that line for a period of 4.4 h. However, samples 6E and 7E follow that line for only a certain time, after which the loss modulus becomes constant and then line B is followed. The loss factor is constant for samples that are spinnable over several hours. For unspinnable material the loss factor changes abruptly after some time and then decreases with time eventually. It is expected that all samples



Figure 6 The relationship between loss modulus G'' and storage modulus G' for some samples run at 180°C at 0.1 Hz for 4.4 h. A and B are estimated curves describing the two different dependencies.



Figure 7 The relationship between the molecular weight distribution M_w/M_n and cross-over modulus G_e .

will have that behavior if they are analyzed for longer times.

From these results the following conclusion can be drawn. In order to be spinnable, a melt should preferably have an initial loss modulus G'' greater than 300 Pa and a storage modulus G' greater than 23 Pa. Below these limits the melt is unspinnable. This conclusion applies to polypropylene HG 235 and a similar approach could be used to test other materials.

The intersection of the storage and loss shear modulus vs angular frequency was studied. It has been stated by Ghijsels and De Clippeleir that the point of intersection, cross-over modulus, would indicate the width of the molecular weight distribution. In our results the cross-over modulus and cross-over frequency G_c and f_c varied to some extent. However, it was difficult to find a correlation between the MWD and G_c , as seen in Figure 7. A possible reason could be that the processing window is too small to be able to observe the correlation.

Effects on Orientation and Solidification

DSC runs of samples 0E–7E showed that the melt and crystallization enthalpy remained constant for all samples. The melt temperature also remained constant. However, a trend was found for the temperature of crystallization T_c . The more processed the material was the lower the T_c . For sample 0E, T_c was 118°C, and for 7E, T_c was 115°C. This would imply that crystallization and solidification occurs closer to the spinneret for virgin material and further away for the more processed material. The stretch rate would be

Sample	0S	1S	3S	5S
$D_{974} \\ D_{842}$	$2.7 \\ 12.3$	$\begin{array}{c} 3.2\\ 14.6\end{array}$	$\begin{array}{c} 3.1\\ 9.8\end{array}$	$3.6 \\ 17.6$

Table V Dichroic Ratio Measurement of the Overall Fiber Orientation D_{947} and Crystalline Phase D_{842} , in the Fibers

higher for the virgin material as the stretching occurs over a shorter distance and thus higher molecular orientation would be induced in the material. However, this theory was not confirmed by the dichroic ratio measurements.

The results of the molecular orientation measurements expressed as dichroic ratio are found in Table V. From the literature⁹ it is known that the narrow band at 1155 cm⁻¹ is most suitable to study for the orientation of the amorphous phase. However, in our experimental results the peak at 1155 cm⁻¹ was strongly overlapped—hence difficult to separate. From our results in Table V one can notice that the D₉₇₄, which have been shown to be a good estimate of the general orientation, clearly demonstrates increased overall fiber orientation as a result of repeated processing.

DSC measurements were carried out on the fibers and the results showed that the repeated processing did not influence either the melt enthalpy or the melting point.

Final Fiber properties

The results from tensile testing show a decrease in *E* modulus and yield stress σ_y with increasing processing steps, which is expected since the molecular weight decreases (Table VI).

It is not possible to judge from the tensile tests whether or not a subsequent recycling step will degrade the material to such an extent that processing is impossible.

DISCUSSION ON PROCESSABILITY AND SPINNABILITY

A very important consideration in context of the present work is how the spinnability can quantitatively be measured. It is seen from the analysis of the melt that the molecular weight significantly decreases as a result of the first extrusion but remains nearly unchanged after subsequent extrusions. The reasons for this are not clarified. However, it seems that there is a measurement problem rather than an actual decrease in molecular weight that is observed. The SEC method of molecular weight analysis has drawbacks especially in measuring the large and small molecular weight fractions. These fractions may have a determining role in fiber formation, especially the large molecular-weight fractions. The calorimetric measurements on melting point and melt enthalpy provide very little information on the changes of the melt properties. Only the temperature of crystallization changed with the number of processings. The melt strength did clearly decrease with increasing process cycles of the material. However, the problems in spinning were due not to melt fracture but to the decrease in drawability.

Beyreuther and Vogel⁶ has tried to explain spinnability in terms of the dynamic mechanical behavior and put forward a structural rheological model. In their results the complex modulus G^* is divided up in the real and imaginary part, G' and G''. Plotting these parameters against each other would then provide a spinnable and not spinnable range. In our work several tests have been run on the molten samples and some interesting results have been obtained, such as a melt should preferably have an initial loss modulus G'' greater than 300 Pa and a storage modulus G' greater than 23 Pa in order to be spinnable. Below these limits the melt is unspinnable.

The processability of a melt into a fiber is not only dependent on the spinnability in the molten state but also on the stretching performance in solid state. The repeated processing of the melt results in the accumulation of degradation byproducts. At the solidification stage, the material is crystallizing and degradation products are pushed in front of the growing crystalline phase. The concentration of degradation products will be largest in the noncrystalline regions. This is a

Table VI Table of Tensile Properties of the Fibers at a Cross-Head Speed of 200 mm/min^a

Sample	E (MPa)	s (MPa)	σ_y (MPa)	s (MPa)
0S	2490	88	287	0
1S	2500	67	280	14
3S	2450	90	276	0
5S	2330	60	255	3.5

^a The "s" refers to standard deviation.

problem since these regions get oriented during stretching. Degradation products as well as remaining spin finish would disturb that process.

CONCLUDING REMARKS

In this paper it has been demonstrated that a melt spinning grade polypropylene can be industrially recycled and spun into fibers five consecutive steps without any significant reduction in mechanical performance of the fibers. After the sixth extrusion the processability decreases rapidly and the melt is denoted unprocessable after the seventh extrusion. The decreased processability can be explained in terms of decreased molecular weight, lowered viscosity, and hence lower melt strength. It is concluded that a good index of spinnability of a polypropylene melt spinning grade is when the loss and storage modulus relationship is a straight line and when the values of these is greater than 300 and 23 Pa, respectively.

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REFERENCES

- Boldizar, A.; Gevert, T.; Markinger, M. Polym Rec 1996, 1(4).
- Bernardo, C. A.; Cunha, A. M.; Oliveira, M. J. Polym Eng Sci 1996, 36, 511–519.
- Horrocks, A. R.; Richards, A. F. Tex Res J 1995, 65, 601–606.
- Davies, A. R.; Horrocks, A. R. In Recycling 1997 Conference Proceedings. Eds. Barrage, A. and Edelmann, X. Geneva, II 10–II 15.
- Wishman, M.; Hagler, G. E. In Fiber Chemistry, Handbook of Fiber Science and Technology; Lewin, M., Pearce, E. M., Eds. Marcel Dekker Inc., New York, 1985.
- Beyreuther, R.; Vogel R. Inter Polym Process 1996, 6, 154–158.
- Becker, J.; Klason, C.; Kubát, J.; Sáha, P. Plastics Rubber Process Appl 1990, 14, 23–28.
- Jasse, B.; Koenig, J. L. J Macromol Sci Rev Macromol Chem 1979, C17, 61–135.
- 9. Kissing, Y. V. J Polym Sci 1983, 21, 2085-2096.
- Kelen, T. Polymer Degradation; Van Nostrand Reinhold: New York, 1983; 24–25.
- Misra, S.; Lu, F. M., Spruiell, J. E.; Richeson, G. C. J. Appl Polym Sci 1995, 56, 1761.
- 12. Holmström, A. The Swedish National Testing Research Institute. Personal comunication, 1997.
- Ghjsels, A.; Clippeleier, J. D. Inter Polym Proc 1994, 4(3).