

The Influence of Matrix Viscosity on Properties of Polypropylene/Polyaniline Composite Fibers—Rheological, Electrical, and Mechanical Characteristics

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ABSTRACT: Electrically conductive composites containing polypropylene (PP) and polyaniline (PANI) were prepared using PP with three different melt flow rates (MFRs) and a commercial PANI-complex in proportions of 80% by weight and 20%, respectively. Composite blends were melt-spun to fibers under different solid-state draw ratios. Rheological studies of dynamic viscosity, as well as the storage modulus and loss modulus showed that the prepared PANI-complex/PP blends exhibit different dynamic rheological behavior, depending on the PP used. This confirms the blends' morphological differences. PP

matrix viscosity was found to play an important role in the electrical properties of the prepared fibers. Fibers prepared using the matrix with the lowest viscosity, showed a larger dispersed phase size in the cross-sectional SEM micrographs, maximum conductivity observed at higher draw ratios and a more linear resistance–voltage relationship than those of the fibers prepared using the higher viscosity matrices. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 119: 2800–2807, 2011

Key words: fibers; blends; conducting polymers

INTRODUCTION

Polymer blends with electroconductive properties are materials that have several potential applications in different and novel areas. In recent years, polymer-polyaniline (PANI) conductive polymeric composites have attracted considerable attention because of their possible applications in biosensors, batteries, antistatic coatings, gas sensors, membranes, electrodes, and light-emitting diodes.^{1–5} One interesting and new approach is the use of PANI to prepare electrically-conductive fibers for innovative textile applications, such as sensors, actuators, and electrostatic discharge. Conductive textile fibers have different potential applications, such as electrotherapy,⁶ health monitoring shirts,⁷ dust and germ free clothing,⁸ heat generation,⁹ cooling systems,¹⁰ data transfer in clothing, electrochromic display, sensors and military applications like stealth technology.¹¹

Preparing conductive fibers using melt spinning process is of interest, as melt spinning is the most common process in the production of continuous thermoplastic textile fibers. Blending an insulating polymer with different conductive materials and

then spinning it into fibers using the melt spinning method was studied previously.^{12–16} Intrinsically conductive polymers (ICPs) have offered a new possibility to produce electroconductive polymeric blends with potential use in fiber preparation. Among ICPs, PANI is favored due to its environmental stability in the conductive form, ease of synthesis and a lower price than other conductive polymers.^{17,18} PANI emeraldine base can be easily doped using strong acids to prepare the conductive form. Melt processing of PANI is quite difficult, but it is possible to make it fusible and thermally processable^{19–21} by certain methods. This fusible compound, which is called PANI-complex, can be blended with thermoplastic polymers using common processing equipment.^{22–24}

Kim et al.¹⁵ prepared melt spun fiber from PANI salt/polypropylene (PP) blend, but the electrical conductivity was unsatisfactory because of homogeneity problems. WAXS and TEM characteristics of melt processable PANI/PP have been reported by Passiniemi et al.²⁵ According to previous studies, PANI/PP blend is a two-phase polymeric system, in which PP is the matrix phase.^{25,26} Properties of two-phase blends depend greatly on the morphology of the blend. On the other hand, the final morphology of a blend is determined by many factors, such as composition of materials, viscosity ratio, mixing and processing conditions and the adhesion between the dispersed phase and matrix. It is quite well known that the viscosity ratio of the dispersed phase over

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TABLE I
Properties of the Polypropylene Matrix

	PP grade	MFR (g/10 min)	Density (g/cm ³)
A	HF 350FB	19	0.9
C	HE 445FB	11	0.9
D	HB 120FB	2.2	0.9

MFR is measured under 2.16 kg at 230°C.

that of the matrix plays an important role in the morphology of a two-phase blend.^{27–29} It is reported that the morphology under shear is the result of the viscosity ratio, which determines the size of the droplet-phase.^{30,31} On the other hand, our previous study²⁶ showed that the droplet-phase size is a critical factor in the electrical conductivity of PANI-complex/PP fibers. This implies the importance of a viscosity study for such blends.

The viscosity ratio is the viscosity of the dispersed phase over the viscosity of the matrix, $\lambda = \eta_d/\eta_m$, where λ is the viscosity ratio and η is the viscosity. It is more common to study the viscosity ratio in two phase blends, but if one of the viscosities is a constant value (i.e., dispersed phase), the viscosity measurement of the other phase (i.e., matrix) is enough to determine the morphology changes. This is also applicable when a specified additive is added to the polymer, and the viscosity of polymer matrix or blends are studied.

In this study, the influence of matrix viscosity, PP, on the rheological properties of PANI/PP composite blends and the electrical, morphological, and mechanical properties of the fibers prepared from the above blends is investigated. Three PP grades with different viscosity levels (different melt flow rates (MFRs)) are mixed separately with 20 wt % of a commercial PANI-complex using a twin-screw extruder. The blends are then melt-spun to fiber filaments under different solid-state draw ratios. The rheological properties of the blends and the electrical and mechanical properties of the prepared composite fibers are studied, as well as the microscopic morphology using SEM.

EXPERIMENTAL

Materials, blending, and fiber spinning

Three fiber grade PPs from Borealis, that have different MFRs, which are listed in Table I, were melt-mixed separately with a PANI compound (Panipol CXL from Panipol, Finland) using a 15-cc twin-screw micro compounder (DSM Xplore, the Netherlands). The Panipol CXL compound is based on a proprietary technology using PANI doped by protonation with dodecylbenzoic sulfonic acid and toluene sulfonic acid, and plasticized with zinc sulfonate.

Blends of PP/Panipol CXL were prepared at 200°C and with a screw rotation rate of 70 rpm for 15 min. The P.CXL (Panipol CXL) in the main prepared blends was 20 wt % by weight. First, three main sample series, “20A,” “20C,” and “20D,” were prepared, reflecting the differing viscosities of the PP matrices used, with “A” representing PP with MFI = 19 (called low viscosity), “C” representing PP with MFI = 11 (called medium viscosity), and “D” representing PP with MFI = 2.2 (called high viscosity). Then, to ensure the influence of matrix viscosity and to see the influence of the Panipol content, the same sample series were prepared but using P.CXL at 25% by weight.

Fibers were melt-spun from the processed compounds through a 0.3-mm spinneret die attached to the micro compounder barrel. The fibers were then drawn over two Godet rolls (Fourné Polymertechnik, Germany), where the first Godet was heated to a temperature of 120°C and the second was kept at room temperature for cooling. The rotational speed of the Godet rolls was adjusted so that draw ratios of 2, 3, 4, 5, and 6 were obtained.

Rheological properties

The rheological properties of the pure PPs and their blends were measured at 210°C under a nitrogen atmosphere to minimize thermal degradation of the polymer, using a cone and plate rheometer, Bohlin Rheometer, BRS, to study the dynamic viscosities and the dynamic modulus measured under oscillatory shear deformations. The rheometer had a plate diameter of 25 mm, a cone angle of 5° and a gap of 50 μm , which is the shortest distance between the points of the cone and the plate. The frequency used was in the range of 0.01–30 Hz or 0.0628–188 rad/s.

Electrical conductivity and resistance–voltage relation

For constant-voltage measurements, the conductivity C ($\Omega^{-1}\text{cm}^{-1}$ or S cm^{-1}) of single fibers was calculated using the equation $C = L/RA$ where R is the measured resistance in ohms, L is the length of the fibers, and A is the cross section of the fibers. The resistance of the single fibers was measured at a constant voltage of 10 V using a Keithley 6487 resistance meter. The electrical current was applied between two clips holding 100 mm of fibers. For each sample, the mean value of conductivity for five single fibers is reported.

The resistance was also measured at various voltages ranging from 0.1 to 500 V. Bundles of 16 fibers were attached to clips at a distance of 100 mm. This was done, to see if the resistance would vary when the voltage is changed. All resistance measurements

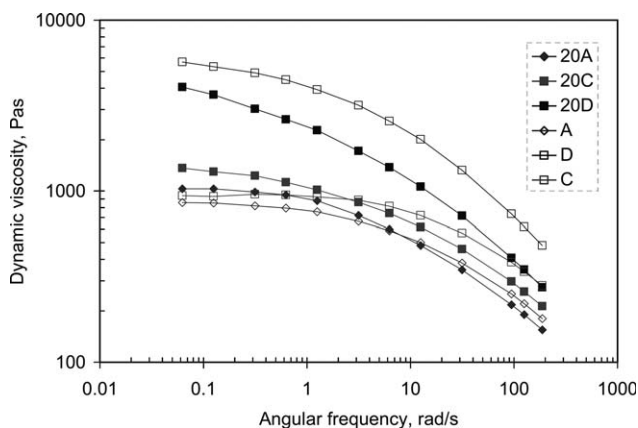


Figure 1 Dynamic viscosities of different grades of pure PP (A, C, and D) and 80% : 20% PP/Panipol CXL blends made using three different PPs. (20A, 20C, and 20D). Open symbols represent the data for pure PP and the filled symbols for the PP/P.CXL blends.

were carried out at ambient conditions. (temperature of 21°C, relative humidity of 12%).

Morphological and mechanical properties

Low vacuum scanning electron microscopy (SEM) was employed to analyze the fiber cross sections and fiber surfaces. Three different cross sections were studied for each sample using the same magnification of 100 μm . To evaluate mechanical properties, a Tinius-Olsen universal testing machine with a 250 N load cell and a test speed of 30 mm/min was used. A total of 10 fibers for each sample and gauge length of 30 mm were measured.

RESULTS AND DISCUSSION

Rheological properties

The rheological data appear in Figure 1 and Table II. Open symbols represent data for pure PPs (A, C, and D) and filled symbols for their blends (20A, 20C, and 20D) with P.CXL at 20 wt %. Figure 1 shows, as was expected considering the MFR data, that D has the maximum dynamic viscosity and A has the minimum dynamic viscosity of the three pure PPs. All samples in Figure 1 show pseudoplas-

tic behavior. The viscosity of a pseudoplastic fluid drops as force (or shear rate) is applied. The more that energy is applied, the more will viscosity reduction (thinning) occur.³² In fact, increasing the shear rate enhances the structural breakdown, leading to concomitant decrease in viscosity.³³

The comparison of the viscosity values of pure PPs and their blends in Table II shows that adding P.CXL has decreased the viscosity of "D" within the entire frequency range, while in "A" and "C," it has lead to increased viscosity at low frequencies and decreased amounts at high frequencies (indicated by * in the Table II). This shows that, initially, P.CXL has reduced the viscosity of "D" and increased the viscosity of "A" and "C," but, on the other hand, adding P.CXL to PP, has increased the slope of the shear thinning viscosity (Fig. 1). By increasing the angular frequency, each PP/P. CXL blend shows a greater viscosity reduction than the respective pure PP. This is attributed not only to the disentanglement of the chains, but also to the reduction in the domain size and the increase of the interfacial area in the blends, as also reported for polystyrene/poly(methyl methacrylate) two-phase blends before.³⁴ In addition, our previous study²⁶ showed that PP/P.CXL is a two-phase blend.

Finally, this drastic reduction in viscosity results in a lower viscosity of 20A and 20C blends than those of the pure PPs, A and C, at high frequencies. (There is also a lower viscosity for 20D than for D, but 20D has had a lower viscosity than "D" since the beginning).

In fact, each P.CXL/PP blend has shown a higher pseudoplastic (shear thinning) behavior than the constitutive pure PP.

The storage modulus and loss modulus appear in Figures 2 and 3, respectively. Polymers are viscoelastic materials. All viscoelastic materials behave more or less solid-like (elastic) and liquid-like (viscous), depending on the deformation rate.³⁵ The elastic behavior is related to the fact that stretched polymer chains tend to pull back into their original conformations. In viscoelastic solids, the storage modulus and loss modulus measure the stored energy of the elastic portion, and the energy

TABLE II
Viscosity Measurements of Three Pure PP and Their Blends with PANI-Complex

Sample	Material	Viscosity at 0.1 rad/S	Viscosity at 1 rad/S	Viscosity at 10 rad/S	Viscosity at 100 rad/S
A	PP1	849	757	501	251
20A	PP1/20 wt % P.CXL	1030	878	483*	217*
C	PP2	931	923	720	385
20C	PP2/20 wt % P.CXL	1300	1010	616*	296*
D	PP3	5330	3920	2000	735
20D	PP3/20 wt % P.CXL	3670	2270	1060	409

*Decreased viscosity at high frequencies compared to the relevant pure PP.

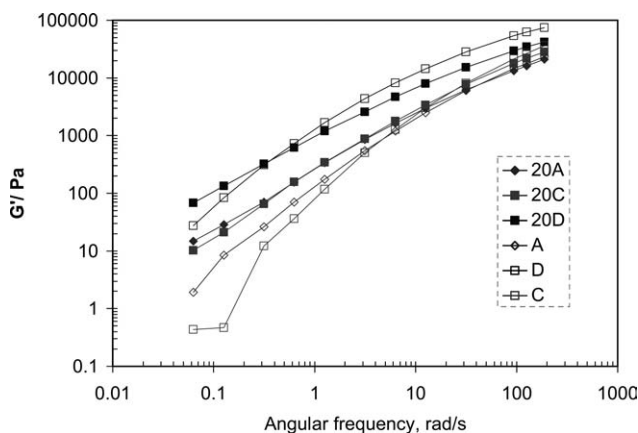


Figure 2 Storage modulus (G') of three different pure PPs (A, C, and D) and their respective 80% : 20% PP/P.CXL blends (20A, 20D, and 20C).

dissipated as heat of the viscous portion.³⁶ As Figure 2 shows, the storage modulus (G') of all samples increases as the frequency is increased, which means that all pure PPs and PP/P.CXL blends have a higher melt elasticity at high frequencies. Furthermore, at low frequencies, each blend (20A, 20C, or 20D) has a higher storage modulus than that of the respective pure PP (A, C, or D).

This shows that the dispersed P.CXL phase dissipates less of the energy than the continuous PP phase. Therefore, the total recoverable elastic energy would be greater in a two-phase PP/P.CXL system than in a one-phase PP system. This behavior is more evident in blends 20A and 20C, which are made from lower viscosity matrices, than that of the 20D. This may be because the high-viscosity PP used in the 20D blend consists of high molecular-weight polymer chains and such long chains have more molecular entanglements. These increase the recoverable stored energy and so the difference in energy dissipation between P.CXL and pure PP is decreased.

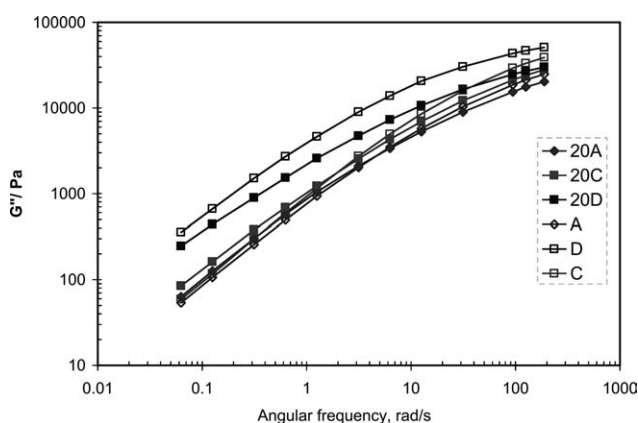


Figure 3 Loss modulus (G'') of three different pure PPs (A, C, and D) and their respective 80% : 20% PP/P.CXL blends (20A, 20D, and 20C).

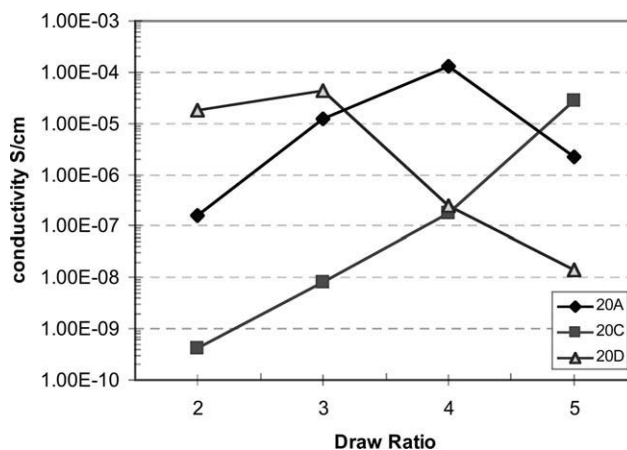


Figure 4 Conductivity (S/cm) versus fiber draw ratio for 80% : 20% PP/P.CXL, prepared using PP matrices with different viscosities (A: low, C: medium, and D: high viscosity).

The dependency of the loss modulus (G'') on frequency is shown in Figure 3. A comparison of the blends shows that a blend made using a higher viscosity matrix has a higher loss modulus, so that the G'' of 20D > 20C > 20A. Furthermore, blend 20D has a lower G'' value than the relevant pure PP, D, throughout the entire frequency range, while 20C and 20A in contrast to the pure PPs, C and A, show such a behavior only at high frequencies. Because the loss modulus G'' represents the viscous behavior (i.e., the amount of energy dissipated), the results show that, although the addition of P.CXL to the highest viscosity virgin PP, D, has reduced the energy dissipation of the blend, 20D, the latter still dissipates more energy (as heat) than the other two blends that are made of lower viscosity matrices (20A and 20C).

The rheological observations of dynamic viscosities and dynamic modulus, can imply how important matrix viscosity in PP/20 wt % P.CXL is in determining

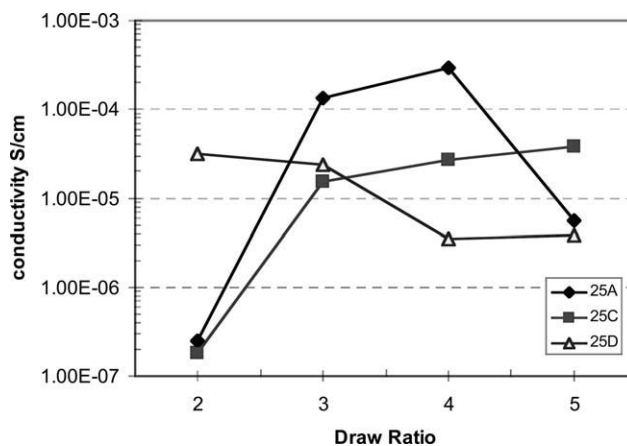


Figure 5 Conductivity (S/cm) versus fiber draw ratio for the 75% : 25% PP/P.CXL blend prepared using PP matrices with different viscosities (A: low, C: medium, and D: high viscosity).

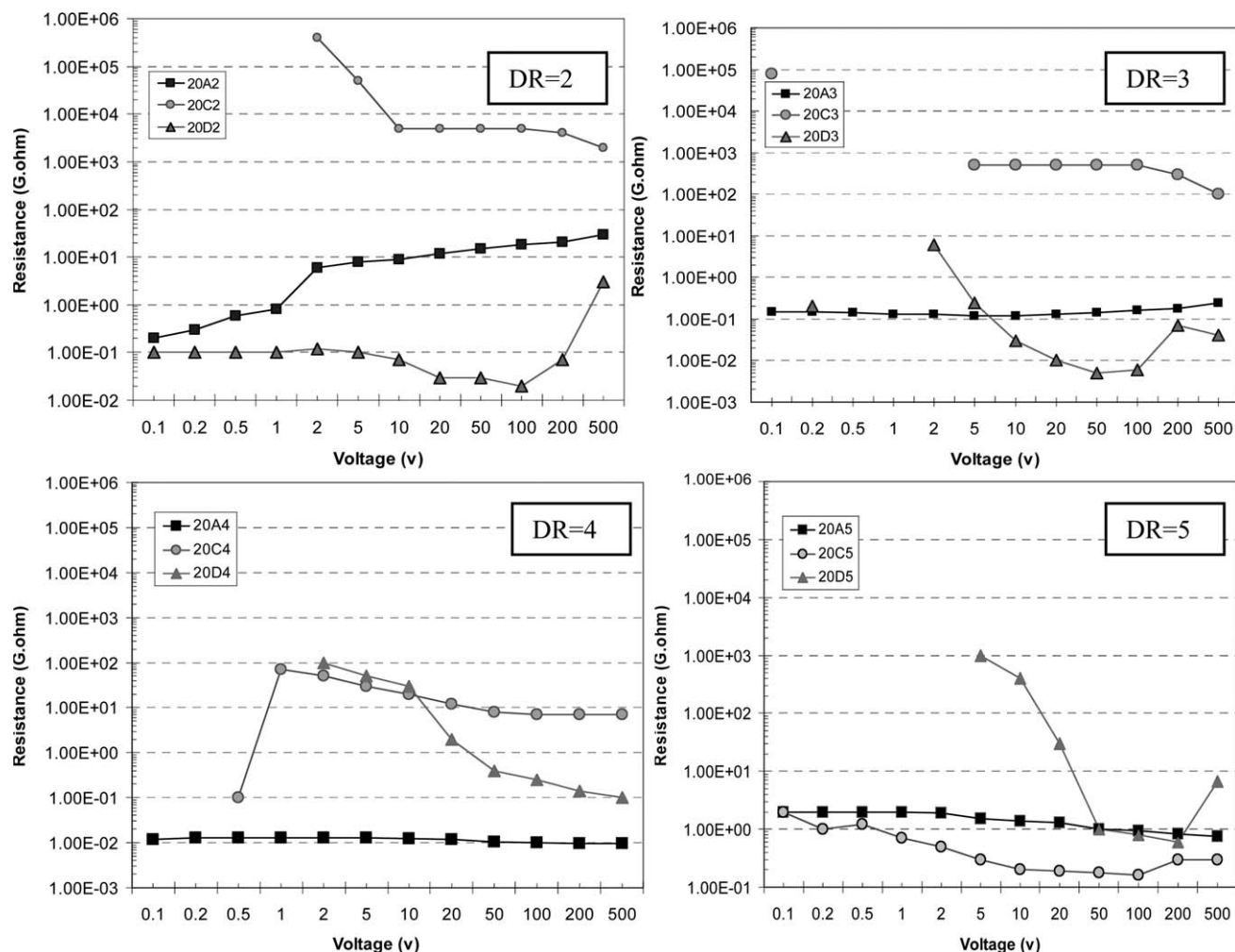


Figure 6 Resistance versus voltage for fibers made from 80% : 20% PP/P.CXL blends using different PP matrices (20A, 20C, and 20D) and different draw ratios: DR = 2, 3, 4, and 5.

the flow behavior, elasticity and heat generation of the melt blends under the applied deformation, especially in the melt blending and melt spinning methods in which the blends undergo great deformation during blending, extruding, and drawing. Moreover, flow behavior of a blend under deformation is a result of its morphology and consequently affects the other properties of the prepared blend and fiber.

Electrical properties

Conductivity measurement

Figure 4 shows the conductivity measurements for fibers made from 80 : 20% PP/P.CXL blends using different PP matrices and prepared using different draw ratios. The conductivity measurements show that the selection of the PP matrix has a great influence on how dependent the fiber's conductivity is on the fiber draw ratio. 20A and 20C, which contain lower viscosity matrices than 20D, show higher fiber's conductivity at higher draw ratios (draw ratio of 4 and 5, respectively). 20D, which is prepared using the matrix with the high-

est viscosity, shows the high conductivities in fibers made at low draw ratios (draw ratios of 2 and 3).

To ensure the influence of matrix viscosity and to see the influence of P.CXL content, the same sample series were prepared with A, C, and D, but using P.CXL in a proportion of 25 wt %, instead of 20 wt %. (Fig. 5). Although by using 25 wt % of P.CXL, higher conductivities were obtained than by using 20 wt % of P.CXL, the results of conductivity dependency on the draw ratio were the same; 25A and 25C showed higher conductivity at higher draw ratios (4 and 5 respectively), whereas 25D showed higher conductivity at the lowest draw ratio (draw ratio of 2). This observation proved the previous results regarding the influence of the PP matrix on conductivity where 20 wt % of P.CXL was used. The possible reasons will be discussed later in the morphology section.

Resistance–voltage relationship

Figure 6 shows the electrical resistance versus applied voltage. It is obvious that in fibers prepared

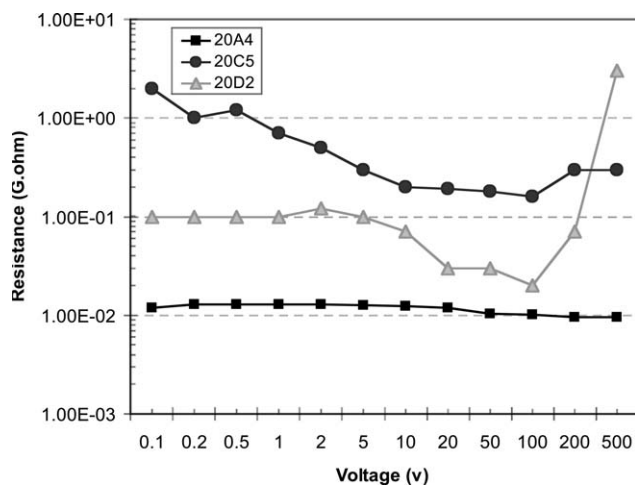


Figure 7 Resistance versus voltage for fibers made from 80% : 20% PP/P.CXL blends using differing PP matrices and optimum draw ratios for greatest conductivity (20A4, 20C5, and 20D2 are prepared at draw ratios of 4, 5, and 2 respectively).

under the same draw ratio (DR), Figure 6, DR of 2, 3, 4, or 5, the resistance–voltage linearity is affected by the matrix viscosity, because 20A, 20C, and 20D show different linearities. In Figure 6 for fibers made at DR = 3, 4, and 5, 20A3, 20A4, and 20A5 show a more linear curve than the other two fibers. However, we are not sure if the observed differences in R - V linearity in this comparison are due to the viscosity of the matrices or the fibers' conductivities, because we have shown before²⁶ that the most conductive fibers show the least dependency of resistance to voltage and exhibit plots that are more linear. With this in mind, the fibers with the best conductivity and prepared using different PP matrices (appearing in Fig. 1 as 20A4, 20C5, and 20D2, where the last digit of the sample name—4, 5, and 2—is the fiber draw ratio), are presented in Figure 7 to better compare the relationship between resistance–voltage linearity and matrix viscosity. Among the samples with the best conductivities in Figure 7, 20A4, which is the fiber made from the PP matrix with the lowest viscosity (A) and prepared using a draw-ratio of four, shows the most linear resistance–voltage relationship.

Morphology

Cross section SEM micrographs of the fibers containing different PP matrices, 20 wt % of P.CXL and prepared under draw-ratio of two are shown in Figure 8. The lighter area of the SEM images represents the dispersed PANI-complex phase and the darker area is the PP matrix. All images were taken at the same magnification (the same scale bar of 100 μm). A comparison of pictures (a), (b), and (c) shows that sample 20A2 represents the maximum

number of visible PANI domains in cross section, while 20D2 shows the minimum number (this result is based on the comparison of three pictures of each, although one of each is presented here). According

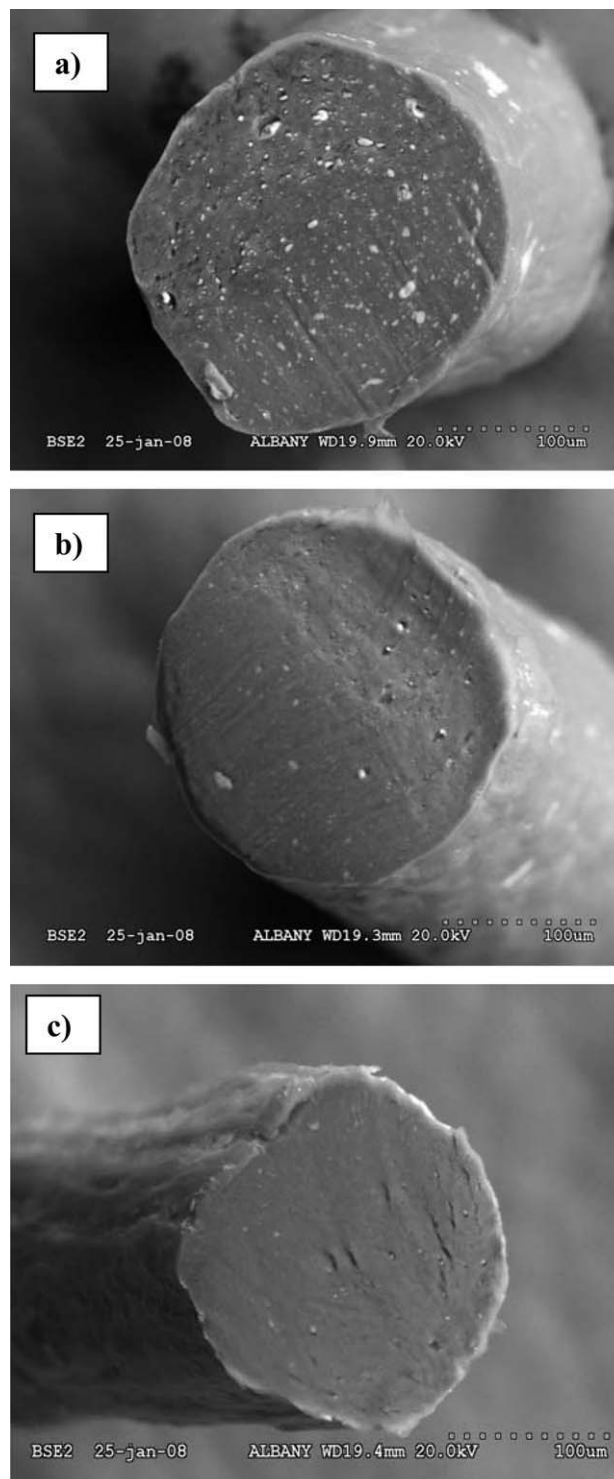


Figure 8 SEM micrographs, cross-sections of fibers made of 20 wt % P. CXL dispersed in three different polypropylene matrices, prepared with a draw ratio of 2.0. (a): 20A2, (b): 20C2, (c): 20D2 showing low, medium, and high viscosity PP matrices, respectively. (Label of SEM images: WD 21.1 mm, 20.0 kV, 100 μm).

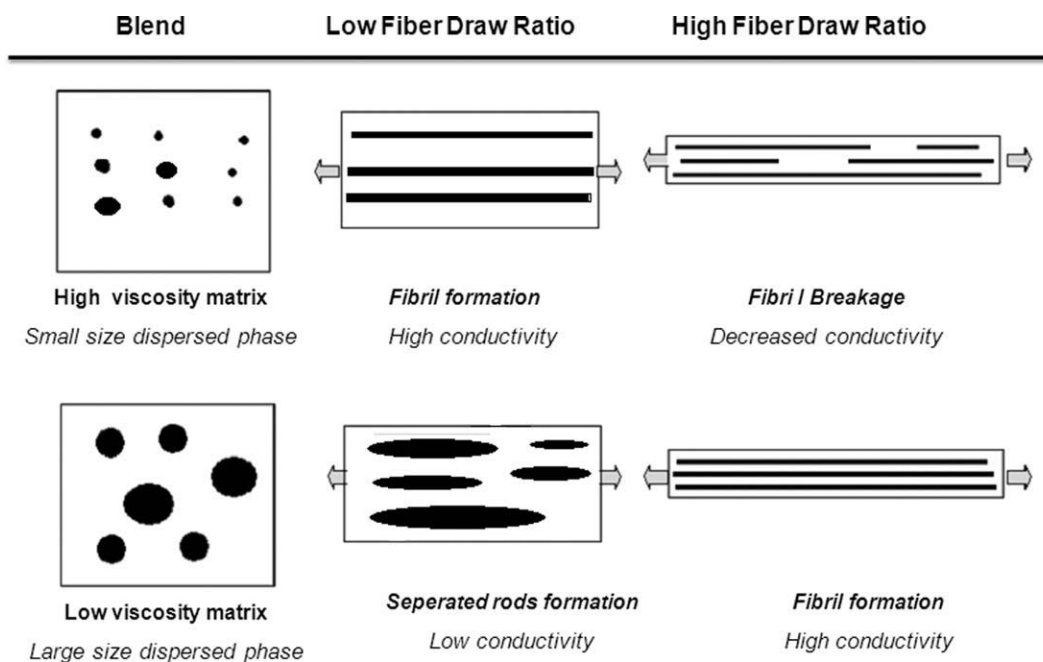


Figure 9 Comparative scheme of morphology and conductivity changes during fiber drawing for PP/P.CXL fibers prepared from blends made from high- and low-viscosity matrices.

to our previous study,²⁶ when the size of the dispersed phase is reduced, the number of visible domains in the fiber cross section is decreased when using the same magnification. Thus, it can be concluded that using a PP matrix with a higher viscosity can result in a dispersed PANI-complex phase with smaller sizes. This is in accordance with previous studies on the influence of viscosity ratio on the size of the dispersed phase.^{30,31} In 20D2, which contains PP with the maximum viscosity, the domains are very small and not visible using the applied magnification, leading to a more homogeneous structure in cross section.

It is now possible to explain the previously discussed observations of conductivity measurements for fibers made using matrices with different viscosities and different draw ratios. As our previous study of PP/P.CXL showed,²⁶ to have a better conductivity in a two-phase blend like PP/P.CXL, a continuous pathway of conductive phase is neces-

sary. This is dependent on both the initial dispersed phase size and the applied stress (fiber draw ratio). An optimum draw ratio (which depends on the initial dispersed phase size) is necessary to form conductive fibrils and balance the fibrils formation/breakage along the fiber axis. According to these results, it is now expected that increasing the draw ratio in the blend that has the small dispersed phase size, 20D, would increase fibrils breakage and decrease conductivity, while in 20A and 20C it would result in more fibrils formation and more conductivity until draw ratios of 4 and 5, respectively. However, draw ratios that exceed the latter could result in fibrils breakage and decreased conductivity. Considering the discussion earlier based on our previous study, a comparative scheme of morphological changes during fiber drawing is illustrated in Figure 9 to show the influence of polymer matrix on dependency of fiber's conductivity to the draw ratio.

TABLE III
Mechanical Properties of PP/PANI-Complex Composite Fibers Made from 20 wt % P.CXL and PP with Differing Viscosities (A, C, and D) and Draw Ratios of 2 and 4

Fiber	Matrix visco./ draw ratio	Elong at max %	Initial Mod. (cN/Tex)	St. dev. (%)	Tenacity (cN/Tex)	St. dev. (%)
20A2	Low/2	293.8	2.99	23	0.43	17
20C2	Medium/2	889.3	2.09	6	0.72	13
20D2	High/2	994.5	2.14	8	0.86	17
20A4	Low/4	312	4.36	12	1.30	5
20C4	Medium/4	481.8	3.05	30	1.53	22
20D4	High/4	312.9	2.52	7	2.08	11

Mechanical properties

The mechanical properties of PP/P.CXL blend fibers are presented in Table III for two draw ratio series, draw ratios of 2 and 4. Increasing the draw ratio from 2 to 4, has increased the tenacity and initial modulus of the 20A, 20C, and 20D fibers. To investigate the influence of matrix viscosity on mechanical properties, the fibers prepared with the same draw ratio should be compared. The results of each series (draw ratios of 2 and 4) show that using the PP matrix with a higher viscosity has increased the fiber tenacity and decreased the initial modulus. With regard to tenacity, this observation is more evident in fibers made using a draw ratio of 4 rather than 2.

CONCLUSIONS

The PP grades with different viscosity levels were melt mixed separately with a commercial PANI-complex (Panipol CXL) in proportions of 80% PP and 20% Panipol CXL. The blends were used to prepare conductive fibers using solid-state draw ratios of 2, 3, 4, 5, and 6 and the melt-spinning method. The rheological properties were studied regarding dynamic viscosity as well as storage and loss modulus (G' and G''). The dynamic viscosity study showed that adding PANI-complex increased the pseudoplastic behavior of PP in all of the blends. This could confirm the previous results that PP/P.CXL is a two-phase blend. Furthermore, the blend's dynamic rheological properties depended on the MFR of the PP used, which indicates the importance of matrix viscosity selection on the final morphology of the blends. A study of the electrical properties of the fibers revealed that matrix viscosity has a significant influence not only on the conductivity level of the fibers, but also on the conductivity's dependency on the fiber draw ratio and the linearity of the electrical resistance–voltage plots. In fibers prepared using the matrix with the lowest viscosity, SEM micrographs showed a PANI dispersed phase with a larger size in the fiber's cross section, the highest conductivity was observed in fibers prepared at higher draw ratios and such fibers showed a more linear resistance–voltage relationship than those made using higher viscosity matrices. The mechanical properties showed that, using a matrix with the maximum viscosity will increase the tenacity and decrease the initial modulus in the composite fibers.

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