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STUDY OF POLYPROPYLENE MORPHOLOGY OBTAINED FROM BLOWN AND CAST FILM PROCESSES: INITIAL MORPHOLOGY REQUIREMENTS FOR MAKING POROUS MEMBRANE BY STRETCHING*

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ABSTRACT: Four different polypropylene (PP) resins are extruded using the tubular blown and cast film processes. The resin morphology is observed by SEM and the effect of extrusion processing variables on the morphology is investigated. Melt rheological experiments are also carried out to characterize the polymer melts. It has been found that the molecular weight distribution and the chain structure as well as the processing conditions have important effects on the morphology. Efforts have been focused on developing a row-nucleated morphology from PPs through the control of processing conditions. The possibility of generating a porous membrane from the initial row-nucleated morphology using a stretching technique is evaluated. It has been found that the initial lamellae arrangement of the precursor films and the stretching conditions play a significant role in obtaining a porous structure.

KEY WORDS: polypropylene, orientation, porous membrane and stretching.

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INTRODUCTION

THE IMPORTANCE OF polymeric materials in separation applications can be easily illustrated from the progress made in polymeric porous membrane technology for the past three decades. One of the most widespread applications of this type is porous membrane from polymers.

There are different methods for making porous membranes from polymers. Among them, film stretching followed by voids creation has been in focus recently since it is inexpensive and more environmentally friendly [1,2]. The main idea of this process is the formation of a two-phase structure that, upon stretching, creates stress concentration at the interface, which results in cracks between the phases and thus, voids creation. The two-phase structure can be:

- two immiscible polymers
- a polymer containing an inorganic filler or
- a semicrystalline polymer whose crystalline morphology is arranged in a row-nucleated lamellar form.

A porous membrane obtained by stretching a neat unfilled polymer (possessing a row-nucleated structure) has a number of advantages when compared to other methods. The most important one is the use of a single material, thus eliminating the need for blending, compounding, dissolution, and solvent removal.

Generally, porous membrane formation from a semicrystalline polymer proceeds through three main stages:

- production of the precursor film having a row-nucleated morphology.
- annealing of the film to thicken the lamellae.
- film stretching at a low temperature to create voids and then at a higher temperature to enlarge the voids as illustrated in Figure 1 [2].

Understanding the mechanisms of micropore formation in the stretching process and the effect of the processing parameters on the pore size will guide us in developing membranes with different pore sizes, size distributions, and porosities. It has been shown that an initial row-nucleated lamellar morphology with high crystalline orientation function is necessary for making such a porous membrane [3]. Polyethylene and polypropylene (PP) are regarded as suitable materials because of their crystalline structure, low cost, and wide range of grades. Polypropylene is usually favored because of its higher temperature resistance and better mechanical properties, compared to polyethylene. In this study, the variation of PP morphology with processing conditions and resin properties are examined. A special emphasis is directed to

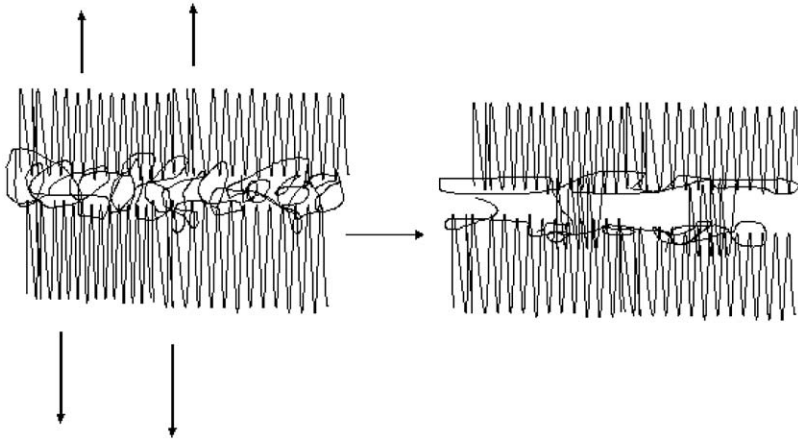


Figure 1. Schematic of pore creation by stretching; the applied stress will separate the blocks from each other.

conditions leading to row-nucleated lamellar structure formation and then morphology of porous membranes made by stretching.

The Role of Initial Morphology

Polypropylene morphology is defined as the final crystalline structure and amorphous phase in the solidified state. The arrangement, form, and crystalline phase content are controlled by the PP molecular structure and process conditions. There are three main crystalline structures possible for PP. The spherulitic structure is formed in a quiescent state (or low stresses) and will result in the arrangement of crystal blocks (lamellae) in a spherical shape leading to an isotropic configuration. The row-nucleated lamellar structure is formed when the polymer is crystallized under high or moderate stresses. The third one, the fibrillar structure, is formed when the material is highly stretched, in which case the crystalline blocks break and are embedded into a fibrillar structure in the stress direction. The first step in this study is to generate a row-nucleated lamellar morphology for PP. This involves understanding the relationship between the material, processing conditions, and morphology.

In the row-nucleated lamellar morphology development, the stresses applied during crystallization extend the polymer chains that act as initial nuclei and crystallization follows in lamellar blocks perpendicular to the extended chains [4]. Extended chains are necessary for the

achievement of a row-nucleated morphology, especially at the beginning of the crystallization process because they serve as nucleating sites. Nogales et al. [4] have studied the shear-induced crystallization of isotactic PPs having different molecular weight distributions. They found that the development of an oriented morphology in a resin having a high molecular weight is much faster than for those with a lower molecular weight. The presence of the long chains increases entanglements between the chains, which then increase the relaxation time; hence, the chance for the extended chains to relax before crystallizing decreases [5].

On the other hand, it has been shown that the PP microstructure, such as crystallinity, crystal size, and crystal type can be related to mechanical properties [6]. In fact, it has been shown that the yield strength is related to the degree of crystallinity, lamellar thickness, or both. A linear relationship between the tie molecule fraction and yield strength was developed in another study [7].

In this article, we are concerned with the understanding of the morphological requirements for isotactic alpha form PP films to obtain porous membranes.

EXPERIMENTAL

Materials

In this study, the four different polypropylene grades used are listed in Table 1.

PDC1280 is a general purpose grade and Pro-fax 6823 possesses the lowest MFI in the homopolypropylene list introduced by Basell Company. Pro-fax 6823 is a high molecular weight homopolypropylene of Basell. Pro-fax 814 (PF-814) is a branched PP with long chain branches. It was selected because of the effect of branched chains on its relaxation time. In this article, we refer to these materials as PP-1280,

Table 1. Polypropylene grades used in this study.

Resin code	Company	MFI 230°C/2.16 kg
PDC1280	Basell	1.2
Pro-fax 6823	Basell	0.5
Pro-fax 814	Basell	2.8
PP4292E1	ExxonMobil	2.0

PP-6823, and PF-814. PP4292E1 is a grade apparently used for producing oriented films according to the Company. They were all reported as homopolymers by the manufacturers and we could not verify this information. It is possible that in some cases, a small amount of ethylene is present.

Sample Preparation

Two processes were used to obtain the precursor films: film blowing and cast film extrusion. The controllable processing parameters for blown film were:

- cooling gap of the air ring, which allows for frost line (crystallization line) control through the cooling air,
- draw down ratio (DDR), which is the ratio of take-up speed to the average velocity of the extrudate at the exit of the die and represents the amount of stretching in the machine direction (MD),
- blow-up ratio (BUR), which is the ratio of the bubble diameter to the diameter of the die and represents the amount of stretching in the transverse direction (TD),
- die temperature, which affects the temperature profile of the film below the freezing height and consequently affects the melt relaxation mechanism.

The processing conditions are presented in Table 2. The die temperature was 230°C and the die gap was kept at 1.1 mm for all the blown film samples.

For the cast films, a slit die was used and the die temperature was fixed at 220°C. The key parameters were the chilled roll temperature and take-up roll speed. A fan was also installed to supply air to the film surface right at the exit of the die, to improve the cooling. The cast film samples were prepared at different draw ratios (roll speed to die exit velocity, thus implying different thicknesses) as shown in Table 3.

Table 2. Process conditions used for the blown films.

No.	Resin	DDR	BUR	Thickness (μm)	Cooling gap* (%)
1	PP-1280	22.0	1.9	50	37
2	PP-6823	22.0	1.9	50	50
3	PP-6823	45.3	1.7	25	100
4	PF-814	45.0	1.7	25	50
5	PP4292E1	44.0	1.7	25	60

*The maximum cooling gap considered as 100% when it was fully open.

Table 3. Summary of films produced by cast extrusion process.

No.	Sample	Draw ratio	Thickness (mm)
1	PP4292E1-1	21	92
2	PP4292E1-2	40	46
3	PP4292E1-3	83	25
4	PP-6823	80	26
5	PP-1280	82	25

Characterization

The linear viscoelastic behavior was obtained using a Rheometric Scientific SR-5000 stress controlled rheometer with 25-mm-diameter parallel plates. Stress sweep tests were performed to determine the linear region and the frequency sweep tests were carried out within the linear region at 230°C.

Field emission scanning electron microscope (FE-SEM-Hitachi S4700) was employed for observation of the films. The samples were etched with a solution of 60% H₃PO₄ and 40% H₂SO₄ mixed with ≈0.5 wt% potassium permanganate. The etching time was 25 min and the samples were rinsed off and washed with a dilute solution of H₂SO₄, H₂O₂, and distilled water.

Crystalline orientation measurements were carried out using a Bruker AXS X-ray goniometer equipped with a Hi-STAR two-dimensional area detector. The generator was set up at 40 kV and 40 mA and the copper CuK α radiation ($\lambda = 1.542 \text{ \AA}$) was selected using a graphite crystal monochromator. The sample to detector distance was fixed at 8 cm. Prior to measurements, careful sample preparation was required to get the maximum diffraction intensity. This consisted in stacking several film layers to obtain the optimum total thickness of about 2.5 mm.

A standard test method for the tear resistance of plastic films based on ASTM D1922 was used to obtain MD and transverse direction (TD) tear resistance. Permeability of the samples was also examined through measuring the water vapor transmission using a Mocon apparatus (PERMATRAN-W Model 101 K).

RESULTS AND DISCUSSION

The relaxation spectrum is particularly sensitive to the molecular structure and can likely be employed to investigate the presence of high molecular weight chains with long relaxation times, responsible for the

row-nucleated morphology. The complex viscosity curves and relaxation spectra are shown in Figures 2 and 3, respectively. The relaxation spectrum was calculated from the modulus data (G' , G'' , ω) using the NLREG (nonlinear-regularization) computer software [8]. The structure features of PP are influenced by the melt rheological characteristics. PP-1280 and PP4292E1 exhibit a very similar rheological behavior. PP-6823 shows a larger complex viscosity and the zero-shear viscosity plateau is not reached at the lowest frequencies (Figure 2). This is indicative of a higher average molecular weight. The complex viscosity curve of PF-814 is similar to that of PP-6823, but with much lower viscosity values and the shear thinning behavior for this resin is less pronounced than that of PP-6823. Its relaxation spectrum is skewed with respect to the spectra of the other PP resins and its peak shifted to a higher relaxation time (Figure 3), which could be due to the branched structure of PF-814.

To achieve a row-nucleated morphology by film blowing, it is important to approach a BUR of one. This improves the uniaxial orientation of chains at the beginning of crystallization. The minimum BUR that we obtained with our blown film line was 1.7 for PP. This limitation arises from the particular PP bubble shape because of PP's low melt strength in comparison to polyethylene. The processing parameters for the film

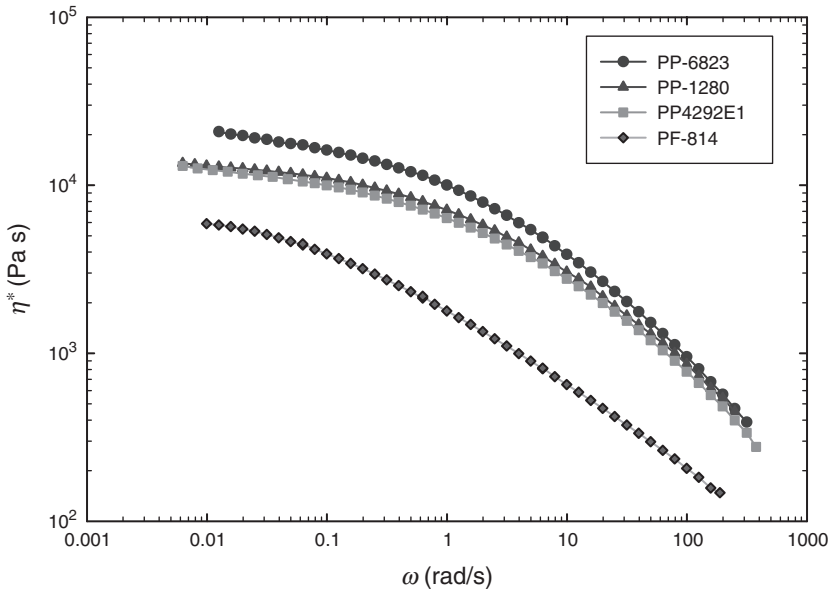


Figure 2. Frequency sweep tests for the resins at 230°C.

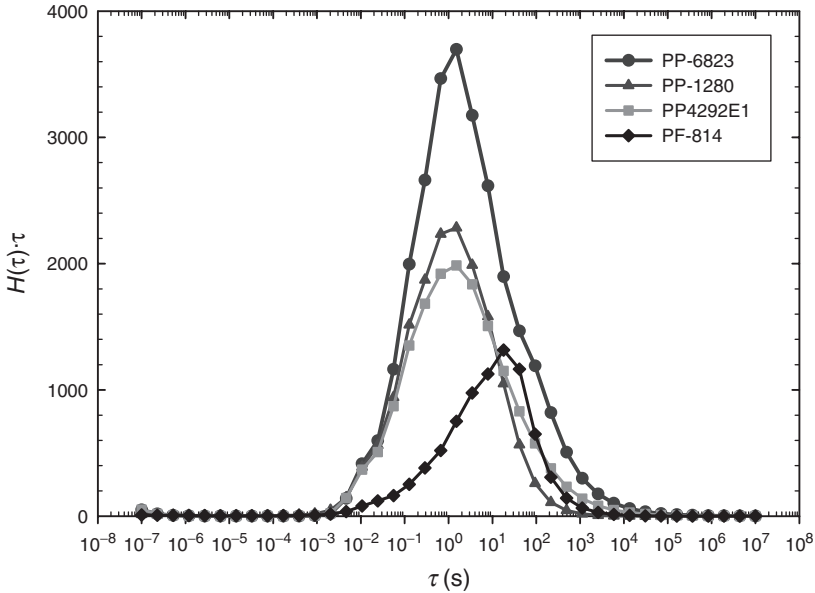


Figure 3. Relaxation spectra for the resins at 230°C.

blowing were selected in a way that promotes the conditions for lamellae formation. High draw ratios, BURs as close as possible to 1, and cooling rates as fast as possible are considered to be the desirable conditions.

Figure 4 shows the morphology of the films' surface. A spherulitic form with crosshatched structure is observed for PP-1280 in Figure 4(a). Figure 4(b) shows the spherulitic structure for PP-6823, the resin that possesses the largest zero shear viscosity. It was attempted to change the processing conditions to the highest draw ratio and cooling rate for this resin. Larger draw ratio will enhance the extension of the chains in the melt state and faster cooling will preserve the extended form of the chains for the crystallization before they relax [3]. Even for the extreme allowable processing conditions, a spherulitic structure was obtained for PP-6823 as shown in Figure 4(c). By contrast, a row-nucleated morphology was observed for PF-814 (Figure 4(d)). We believe this is due to the long branched chains, which increase the mean relaxation time. For PP4292E1, a disrupted lamellar structure with a tendency to spherulitic was observed, as shown in Figure 4(e).

The chain relaxation time is the key parameter that is controlled by the polymer structure and cooling process. Our results suggest the existence of a relationship between the draw ratio, drawing rate, and relaxation time.

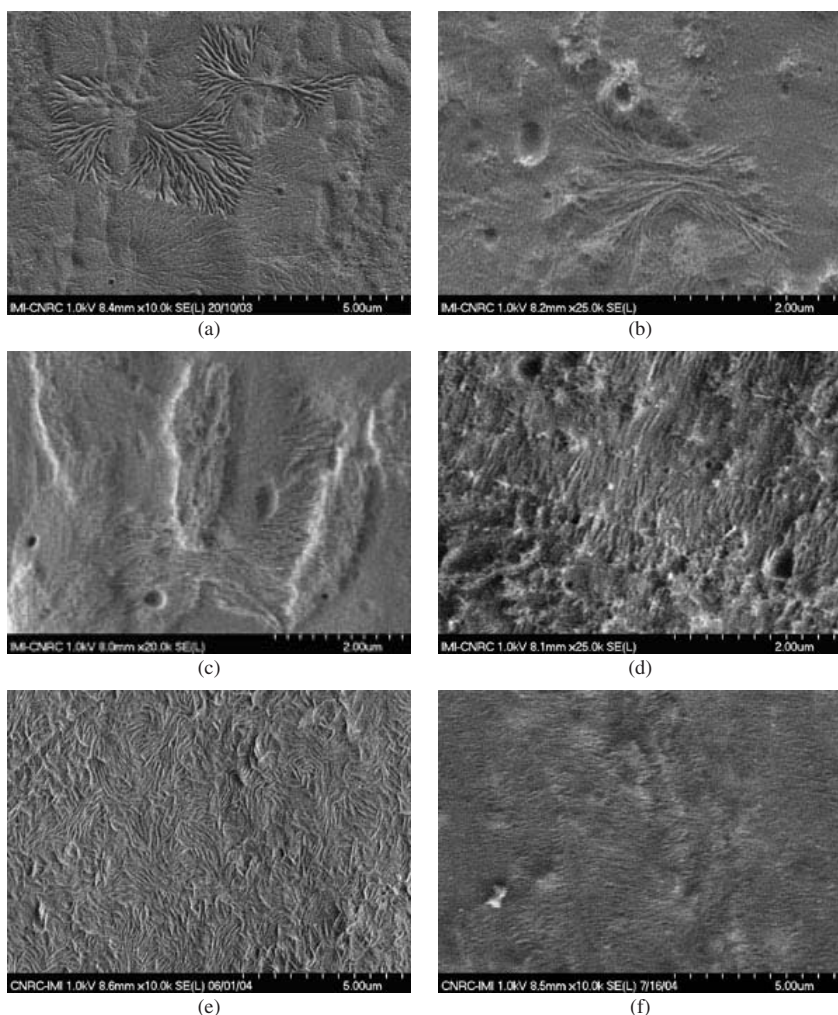


Figure 4. Scanning electron micrographs of the samples: (a) PP-1280 blown film Table 2 #1; (b) PP-6823 blown film Table 2 #2; (c) PP-6823 blown film Table 2 #3; (d) PF814 blown film Table 2 #4; (e) PP4292E1 blown film Table 2 #5; and (f) PP4292E1 cast film Table 3 #3.

The lamellar structure (lamellae thickness in the row-nucleated structure) and the mechanical properties of the final film obtained from PF-814 were very weak for post extrusion stretching. To obtain a row-nucleated morphology for all the resins, the process was changed to cast film. The advantage of cast film extrusion is the possibility of using

a higher cooling rate and also of achieving pure uniaxial deformation. Freezing of thin films (25 μm) could be achieved within 1.5 cm from the die exit. Figure 4(f) shows the row-nucleated morphology in the form of stacked lamellae obtained via this process for one of the PP samples (PP4292E1).

Tensile tests were carried out to reveal the response of the two different morphologies. Figure 5(a) shows the stress–strain curve for a PP4292E1 film obtained by film blowing. A yield point is clearly observed, and right after that, the spherulites break up through a shear-yielding mechanism. Strain hardening at the end of the curve is believed to result from the uniaxially oriented fiber network structure of the crystals. A different behavior is seen for PP4292E1 with a lamellar structure in Figure 5(b). Strain-hardening starts right after the yield point and this may be due to the orientation of the noncrystalline part between lamellae. Some voids are created at this stage and finally the larger lamellae separation ends up with the sample failure. The same behavior has been reported in the literature [9].

Figure 6 shows the orientation parameters for PP4292E1 films. Crystalline orientation functions were obtained from the pole figures of (110) and (040) reflection planes. Herman's orientation functions were derived from (110) and (040) pole figures using the Bruker analytical system software. The c -axis of the isotactic PP unit cell is parallel to the helical axis of the PP molecule in that cell while the three crystallographic axes in a monoclinic unit cell are not mutually perpendicular (the angle between α - and c -axes in isotactic PP is $99^\circ 20'$), so a modified coordinate system is utilized for the calculation of orientation parameters. Since the b -axis of unit cells is perpendicular to its (040) plane, its orientation relative to reference (stretch) direction can be measured directly, that is $\cos^2 \beta = \cos^2 \phi_{040}$, where β is the angle between the stretch direction (machine direction) and the b -axis of the crystal, this value ($\cos^2 \phi_{040}$) is measured experimentally and using Herman's equation [10], the orientation for the b -axis will be determined:

$$f_b = f_{040} = f = \frac{3 \cos^2 \phi_{040} - 1}{2} \quad (1)$$

On the other hand, f_c (orientation of the c -axis) with respect to MD is determined by the combination of data of two planes for the PP, which are (040) and (110) [11]:

$$\cos^2 \phi_c = 1 - 1.099(\cos^2 \phi_{110}) - 0.901(\cos^2 \phi_{040}) \quad (2)$$

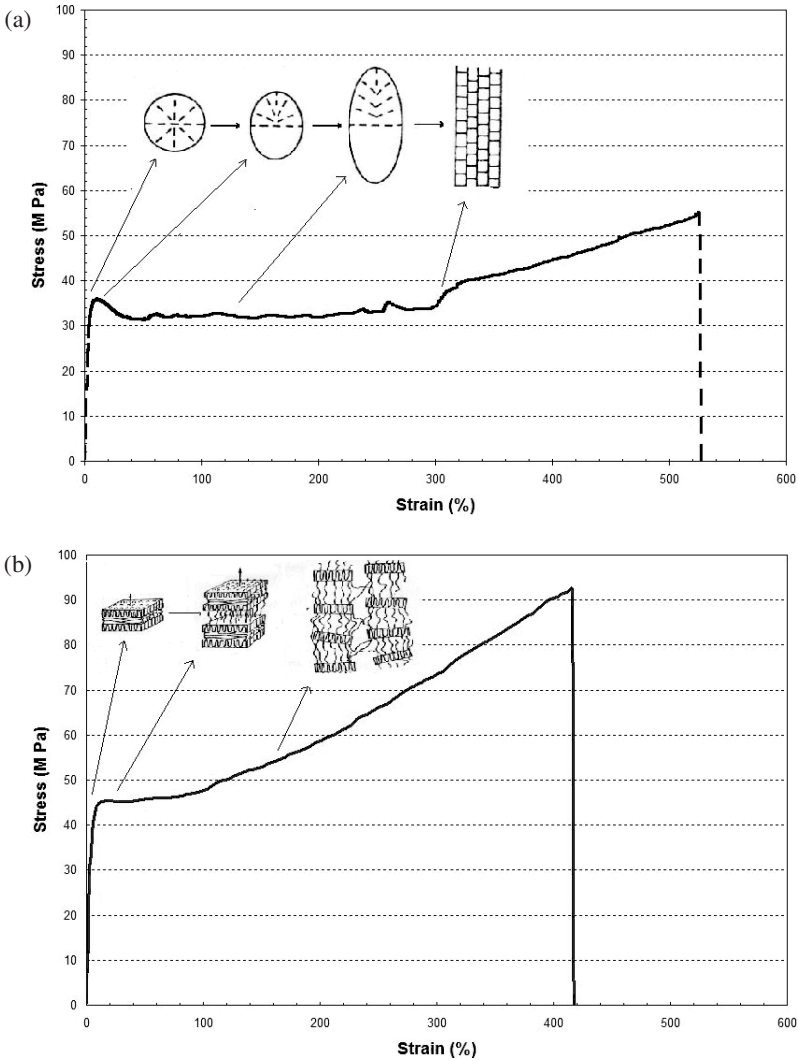


Figure 5. Tensile strength tests for PP4292E1 samples obtained by: (a) film blowing (spherulitic structure) and (b) cast film (lamellar structure).

Substituting this result in Herman’s equation, the orientation factor for the *c*-axis can also be obtained. The other orientation factor, (*a*-axis), can be calculated from the orthogonal relation:

$$\cos^2 \phi_a = 1 - (\cos^2 \phi_c) - (\cos^2 \phi_b) \quad (3)$$

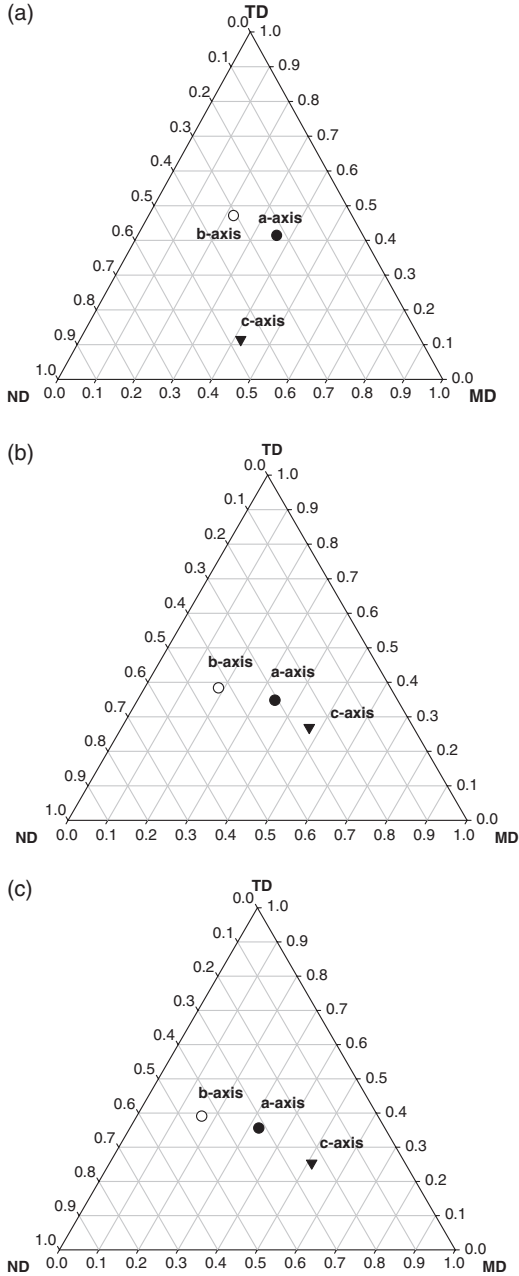


Figure 6. Orientation parameters $\cos^2 \bar{\phi}_a$, $\cos^2 \bar{\phi}_b$, and $\cos^2 \bar{\phi}_c$ for PP4292E1 sheets: (a) No. 1; (b) No. 2; and (c) No. 3 (as listed in Table 3).

As stated earlier, since the unit cell of PP is not orthogonal, the orientation factor calculated from the above equation is not really the same as that of the a -axis of the unit cells, but can be interpreted as an axis that has a small angle with the real a -axis and considered as a replacement for a -axis [10].

Figure 6 presents the orientation parameters $\cos^2(\phi_a)$, $\cos^2(\phi_b)$, and $\cos^2(\phi_c)$ for PP4292E1 samples prepared using the cast film process. It can be observed that the c -axis moves towards the MD as the draw ratio increases from 22 to 66 (Figure 6(a)–(c)), while the a - and b -axes take a position between the TD and the normal (ND) direction. The results for other resins showed the same trend. The orientation factor for the c -axis in the MD is almost a linear relation with the drawing ratio as seen in Figure 7. The higher orientation function of PP-6823 is likely to be attributed to its high molecular weight. It can be concluded that a correlation exists between the shear zero viscosity and orientation function of the c -axis along the MD. The different behavior of PF814 is attributed to its long chain branches.

It has been shown that a certain orientation is needed to develop lamellar structure for the precursor film in a way that can be used in the stretching process for making porous membranes from polyethylene [3].

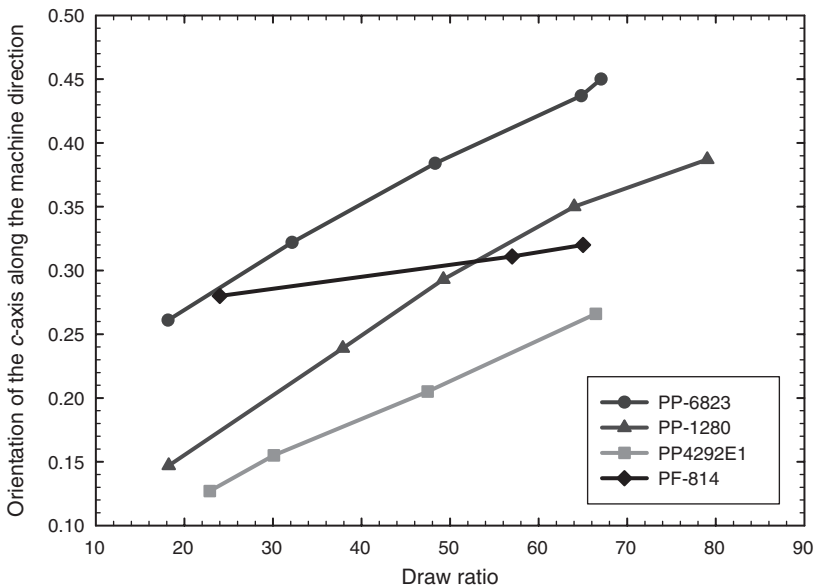


Figure 7. Orientation factor for samples produced by cast film process.

Based on our experiments, an orientation factor larger than 0.3 in the cast films was needed for PP.

The porous membranes were made from Samples 3–5, given in Table 3. The samples were initially annealed at 145°C resulting in a 3% extension of their initial length and then cold stretched for about 35% at room temperature. This was followed by a hot stretching of about 45% at 145°C. Figure 8 compares the structure obtained for three different polypropylene resins. The number of pores per unit area and the pore size are somewhat larger for PP4292E1. The permeability of the samples to water vapor is listed in Table 4. The permeability for PP-6823 is much larger than the two others. We believe that it is due to a better interconnection of the pores across the thickness. This can be also related to the higher orientation function of the *c*-axis of PP-6823 along the MD.

It should be noted that the crystallization kinetics and other parameters, such as isotacticity are important as well. It has been shown that for drawn PP samples, the characteristic length (thickness of lamella and amorphous part together) increases with draw ratio. Increases from 14.4 to 32.4 nm have been reported for samples uniaxially stretched five times [9]. At this stage, we believe that thinner samples that experienced larger draw ratio and faster cooling rate possess larger orientation function and thicker lamellar structure, and hence show a better lamellae separation configuration for the pore creation stage.

Table 5 shows the tear resistance for the same series of PP4292E1 films. The tear resistance is the most sensitive mechanical property to orientation. This is due to the arrangement of lamellae blocks along the MD, which increases the number of the tie chains in this direction. Arrangement of the tie chains in the MD causes a large resistance to tear in the TD compared to that in the MD.

CONCLUSIONS

A row-nucleated morphology for PP can be achieved if optimum processing conditions are used. The most important processing factors for blown films are fast cooling, large draw ratio, and a low BUR. Different responses to tensile tests were observed for samples with spherulitic and lamellar structures. For the cast film process, an almost linear relationship between the draw ratio and the orientation factor was observed for all the PP samples tested. For a large orientation factor (>0.3) a row-nucleated morphology suitable for porous membrane

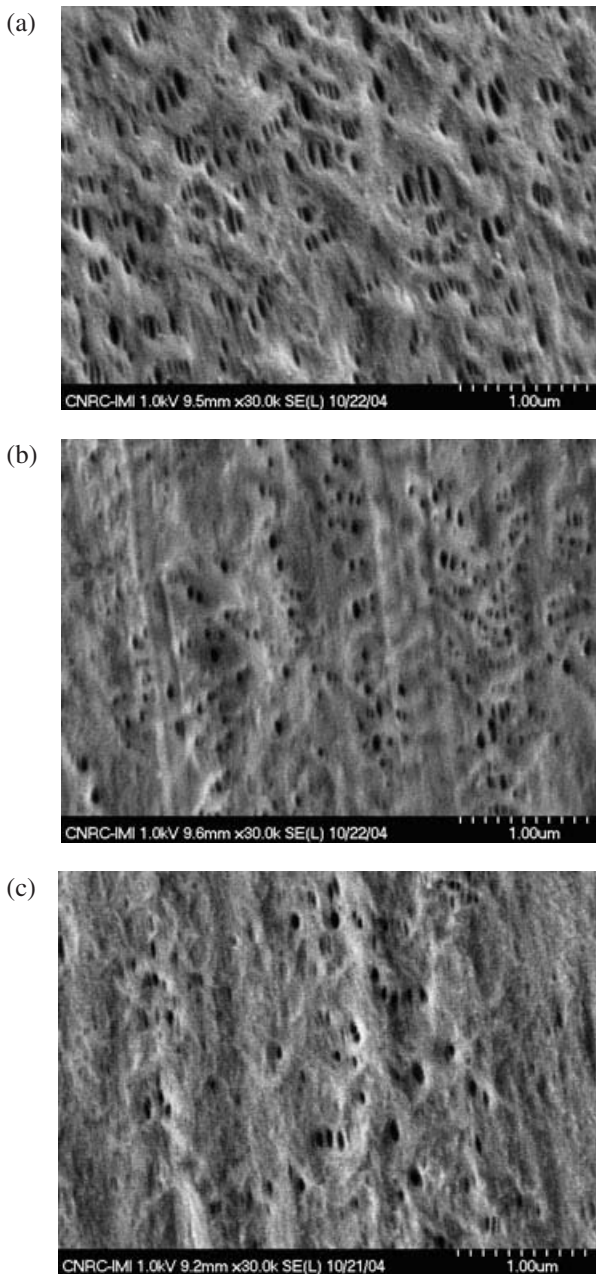


Figure 8. Scanning electron micrographs of membrane surfaces: (a) PP4292E1; (b) PP-1280; and (c) PP6823.

Table 4. Permeability of the samples as listed in Table 3 (the tests were performed at atmospheric pressure and 35°C).

Sample	Permeability (g/m ² day)
PP4292E1-3	590
PP-1280	2700
PP-6823	6000

Table 5. Tear resistance of the PP4292E1 samples, as listed in Table 3.

PP4292E1	Tear resistance MD (g/μm)	Tear resistance TD (g/μm)	Tear resistance TD/MD
1	0.33	14.8	44.4
2	0.44	23.5	53.3
3	0.24	18.1	76.4

formation was obtained. Tear resistance in the TD improved while it became worse in the MD as the orientation increased in the MD with increasing draw ratio. Porous membranes were obtained with precursor films having a row-nucleated morphology and an orientation factor of the *c*-axis in the MD > 0.3. Interconnection of pores is suspected to be an important factor in the permeability evaluation.

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BIOGRAPHIES

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Farhad Sadeghi obtained his MScA in Polymer Engineering from Tehran Polytechnique (Iran) and worked in a plastic film producer company for three years as a production engineer. He is currently a PhD student in his third year at Ecole Polytechnique of Montreal and his project is focusing on the structure of microporous membranes obtained from polypropylene films by stretching.

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Pierre J. Carreau

Pierre J. Carreau obtained his BScA and MScA in Chemical Engineering from Ecole Polytechnique of Montreal, and then a PhD in 1968 from the University of Wisconsin (Madison). He is professor of Chemical Engineering at Ecole Polytechnique of Montreal, where he is presently the director of the Center for Applied Research on Polymers and Composites, CREPEC. He has been highly active for over 35 years in research on the rheology and processing of polymeric systems, an area in which he has published over 200 articles in peer-reviewed scientific journals. He is the author and coauthor of two books.