



Reactive Extrusion and Barrier Properties of PP/PET Films

Hassan Ebadi, Ali Akbar Yousefi*, and Abdolrasoul Oromiehie

Iran Polymer and Petrochemical Institute, P.O. Box: 14965/159, Tehran, Iran

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ABSTRACT

The barrier polypropylene (PP) films were prepared via reactive blending of PP with polyethylene terephthalate (PET) in an intermeshed co-rotating twin-screw extruder. Films were prepared via extrusion through a flat die followed by post-extrusion stretching. The effects of compatibilizer, PP-*g*-MA, content and draw ratio were investigated. SEM micrographs showed that the dispersed phase inclusion size decreases significantly upon the addition of compatibilizer. Morphological studies of the films have shown that laminar morphology was developed in the machine direction rather than in transverse direction. The optimum morphology was observed at 5% compatibilizer concentration. Tensile tests showed that melt stretching has resulted to the inferior properties except modulus. Mechanical properties of blends decreased significantly upon the addition of PET and increased as the compatibilizer content was raised to 10%. Higher contents, however, showed no improvement, as it just improves the physical properties of the blends. Blending results in 200% improvement in the oxygen barrier properties. Permeability of the stretched films and compression moulded films showed 25-35% improvement in the oxygen barrier properties. The maximum barrier property of the extruded films was observed at 5% compatibilizer and draw ratio of 2. Higher contents (up to 15%) resulted in decreased oxygen barrier properties.

Key Words:

PP;
PET;
barrier properties;
oxygen permeability;
film extrusion;
draw ratio.

INTRODUCTION

Controlled permeation of gases and liquids through plastic films, membranes, and other articles is of prime concern in several packaging and industrial applications, such as maintaining fresh foods with maximum quality, preventing losses from containers of fuels and chem-

icals, pipes and tubes in automobiles, in air conditioning, and other industries [1]. The general approach in preparing high-performance barriers is to use materials having low diffusivity and low solubility towards the permeants.

Preparation of barrier films in

(*) To whom correspondence to be addressed.

E-mail: a.yousefi@ippi.ac.ir

the packaging industries is accomplished via different techniques such as multi-layer co-extrusion, extrusion coating, and adhesive lamination [1]. However, there are some drawbacks such as complexity and high cost of these techniques and non-recyclability of the final products. There are tendencies for replacing them with facile processes [1,2].

Blending of commodity polymers with barrier polymers such as polyethylene-vinyl alcohol (EVOH), polyvinylidene chloride (PVdC), and polyethylene terephthalate (PET) and liquid crystals are the alternatives [3]. Addition of a small quantity of barrier polymer into a low-cost matrix can lead to a low-cost product with much improved barrier properties. Blending with barrier materials followed by orientation and morphology development during post-extrusion film stretching process offers a unique, simple, and economical approach.

Morphology development is a complex process influenced by several parameters. A critical parameter influencing the dispersed-phase morphology is the interfacial tension between the matrix and the dispersed polymer. Rigorous calculations are carried out on interfacial tension in the flow of two polymers in a blend and to predict criteria for droplet and fibre formation and stratification of ribbons [1].

It has been established that high shear rate, viscosity ratio close and yet not equal to unity, and low interfacial tension are favourable for increasing the degree of deformation and breaking up the dispersed-phase droplets [4-19]. Thus, the rheological studies are crucially important in developing certain morphologies [1,2].

Polypropylene (PP) is a commodity polymer with good mechanical properties and good barrier to H₂O, but its poor barrier towards O₂ limits its applications [20]. PP semi-crystalline structure is an ideal material for development of oriented products [21].

In this work, PP, PET, PP-g-MA, and para-toluene sulphonic acid (PTSA) are used as matrix, dispersed phase, compatibilizer, and trans-esterification catalyst, respectively [22]. The effects of compatibilizer content and draw ratio on the different properties of PP/PET blends related to morphology, mechanical, and barrier properties are discussed. As the biaxial orientation machines are too expensive, therefore, the reactive blending using para-toluene sulphonic acid as

catalyst and post extrusion of monoaxial stretching of PP/PET blends with improved interfacial adhesion is a novel idea being introduced in this work.

THEORY

The presence of an impermeable dispersed phase increases the tortuosity of the path that a molecule must traverse for permeating through a film. It is useful to discuss this concept in terms of a tortuosity factor τ , which is an effective path length divided by actual thickness of the film. Maxwell derived an expression for τ considering the conductivity of a system in which a conducting phase contains a volume fraction ϕ_d of spherical non-conducting particles [1]:

$$\tau = 1 + \frac{\phi_d}{2} \quad (1)$$

The tortuosity is used to calculate the permeability of composites [1] in the following expression:

$$\frac{P_c}{P_m} = \frac{\phi_m}{\tau} \quad (2)$$

where, P_c , P_m , and ϕ_m are the permeability of the composite, the permeability of the matrix, and the volume fraction of matrix, respectively. Robeson [1] has extended Maxwell's work by applying it to blends for which both components are permeable. Derived by Robeson, P_c is given by the following equations where polymeric components are denoted by 1 and 2 subscripts and ϕ_i stands for volume fraction of the i th phase.

Series laminate (layers normal to permeant flow):

$$P_c = \frac{P_1 P_2}{(\Phi_1 P_2 + \Phi_2 P_1)} \quad (3)$$

Parallel laminate (layers parallel to permeant flow):

$$P_c = \Phi_1 P_1 + \Phi_2 P_2 \quad (4)$$

Semi-logarithmic additivity rule for blends is defined as follows [1]:

$$\ln P = \phi_1 \ln P_1 + \phi_2 \ln P_2 + \dots \quad (5)$$

where, ϕ_i is the volume fraction of the *i*th component.

EXPERIMENTAL

Materials

PP used in this study is a random copolymer of propylene and ethylene (R2400 grade from Yuhwa-Korewa Petrochemical Co.) with 8% of ethylene monomer and MFI=0.246 (210°C/5kg) and $T_g=13.04^\circ\text{C}$ (DMTA measurement), PET (Eastlon PET CB-602 grade, Taiwan) IV=0.8 (ASTM D240) and $T_g=85^\circ\text{C}$ (DMTA measurement), maleic anhydride (MA) from Fluka (Switzerland), antioxidants (Irganox 1010, Irganox 1076 from Ciba-Giegy Co.), dicumyl peroxide (DCP) as initiator and other materials such as acetone and xylene were all received from Merck Co. (Germany).

Procedures

Preparation of compatibilizer and blends was carried out in an intermeshed co-rotating twin-screw extruder (Collin ESC-T10 model) with screw diameter of 50 mm and L/D ratio of 15. Film samples were prepared in a single screw extruder (Haake rheomix system 90) with screw diameter of 19 mm and L/D ratio of 24 via flat die. SEM micrographs were obtained using a Cambridge scanning electron microscope (Stereoscan 360 model). Mechanical properties of samples were measured in an Instron tensile machine (MTS-10/M model). Gas permeability of samples was measured on a Davenport permeability apparatus (468/103 model) at room temperature.

PP-g-MA as compatibilizer was prepared via melt grafting process. MA and DCP were dissolved in acetone and mixed with PP in a turbo mixer at 800 rpm for 10 min (Table 1) in order to improve the adsorption of MA and DCP onto PP. Then, the mixture was dried and melt grafted in twin-screw extruder at

Table 1. Composition of PP and antioxidants mixture.

Materials	Composition (phr)
PP	100
Irganox 1010	0.1
Irganox 1076	0.1

Table 2. Composition for melt grafting of PP.

Materials	Composition (phr)
PP	100
MAH	3
DCP	0.15

30 rpm. The extruder had 6 heating zones from the feeding zone to the die and temperature profiles in these zones were $T_1=180$, $T_2=190$, $T_3=195$, $T_4=200$, $T_5=200$, and $T_{die}=190^\circ\text{C}$. The melt was cooled in a water bath and then cut into granules. The grafting efficiency was found to be 90% by titration method [23] and the melt flow index (MFI) of the grafted PP was 0.284 g/10 min, 5kg at 210°C.

The grafting percentage was found by titration method [22,23]. In this method a sample of prepared grafted PP was dissolved in the boiling xylene and then sedimented by methanol in order to remove unreacted maleic anhydride and dicumyl peroxide. Thus, the sample was not a mixture of maleic anhydride and PP resin but a pure PP-g-MA copolymer. This purified sample was titrated by alcoholic KOH in presence of phenolphthalein as titration indicator. IR Spectroscopy of the purified film sample confirmed MA grafts onto PP (peak in 1670-1720 cm^{-1}).

In order to prevent PP degradation during the blending process an antioxidant was added in the twin-screw extruder at 60 rpm with a composition given in Table 2. The temperature profiles were $T_1=185$, $T_2=220$, $T_3=235$, $T_4=230$, $T_5=220$, and $T_{die}=190^\circ\text{C}$. The melt was cooled to room temperature in a water bath and then cut into granules. In order to prevent hydrolysis of PET during blending, prior to mixing, polymeric materials were dried in a

Table 3. Composition of the blends.

Sample No.	PP	PP-g-MA+ 0.01%PTSA	PET
Blend 1	80	-	20
Blend 2	75	5	20
Blend 3	70	10	20
Blend 4	65	15	20

vacuum oven ($P=40$ torr and air atmosphere) for 12 h at 80°C and 120°C for PP and PET, respectively.

Para-toluene sulphonic acid (PTSA) with a concentration of 0.01% by weight was used as catalyst in trans-esterification reaction of the anhydride groups of PP-g-MA and the ester groups of PET. Then all

components were premixed according to the composition in Table 3 and fed into the twin-screw extruder at 30 rpm and the working temperature profiles were $T_1=210$, $T_2=220$, $T_3=240$, $T_4=250$, $T_5=240$, and $T_{\text{die}}=230^{\circ}\text{C}$. The blend melts were cooled in a water bath at the exit of die and cut into granules.

Table 4. Mechanical properties of original polymers and their blends.

Sample	Draw ratio (λ)	Direction	Break stress (MPa)	Standard deviation (MPa)	Modulus (MPa)	Standard deviation (MPa)	Strain-at-break (%)	Standard deviation (%)
PP	1	MD	18.4	1.20	853	12.5	320.4	24.50
		TD	8.9	2.10	657	25.9	320.1	45.10
	2	MD	21.7	2.50	468	14.53	310.8	8.71
		TD	6.3	3.40	520	12.30	316.1	15.30
PET	1	MD	52.4	7.20	2290	72.30	4.3	1.10
		TD	38.9	3.70	1175	27.30	4.9	0.50
	2	MD	63.1	1.30	1627	11.20	3.9	1.00
		TD	28.5	1.50	1102	57.23	4.5	0.30
Blend 1	1	MD	13.1	0.52	1399	28.50	311.8	1.96
		TD	9.2	0.70	1120	44.20	301.1	0.10
	2	MD	8.1	1.19	484	12.50	116.0	5.80
		TD	5.6	2.60	345	27.80	290.0	7.43
Blend 2	1	MD	15.3	0.80	1470	32.10	150.4	2.56
		TD	6.6	1.30	980	21.63	175.7	2.90
	2	MD	17.8	1.83	1390	27.50	146.0	6.10
		TD	6.5	1.23	967	15.50	155.6	6.70
Blend 3	1	MD	17.5	2.10	1563	12.67	170.1	8.43
		TD	10.2	0.30	1120	11.60	180.5	1.30
	2	MD	17.9	0.12	1430	18.90	163.2	11.50
		TD	10.0	1.12	1050	12.10	167.7	1.75
Blend 4	1	MD	17.9	1.90	1560	11.20	171.5	6.30
		TD	10.5	2.00	1156	17.10	177.2	2.30
	2	MD	18.1	0.20	1167	6.70	168.0	15.50
		TD	10.1	1.00	1059	9.60	173.6	12.50

Films were prepared via flat film extrusion (T die with 15 cm wide designed for biaxial melt tension) followed by post-extrusion stretching process using a Haake single screw extruder at 30 rpm and temperature profiles were $T_1=200$, $T_2=250$, $T_3=260$, and $T_{4(\text{die})}=220^\circ\text{C}$. The extruded films were drawn at draw ratios $\lambda=1$ and $\lambda=2$. Draw ratio is the ratio of linear rate of stretched film to that of melt at the exit of die (without stretching).

Films of the original polymers and their blends were drawn for tensile tests in two directions (MD and TD) for at least five samples. The samples (25×1.5 cm strips) were clamped by pneumatic grips. Then the films were tested at constant cross-head speed 0.33 mms^{-1} using a 490 N load cell according to ASTM D882-88 method.

Morphological analysis was carried out on blend films. The films were cryo-fractured in liquid nitrogen at MD and TD. Then fracture surfaces were coated with gold under vacuum. The permeability to oxygen was measured three times using Davenport permeability apparatus (using 100 microns flat-die extruded films). This standard equipment operation is based on pressure differences between the permeation cells. The permeation cells are divided into two chambers separated by a test sample. The test results were calculated in $(\text{cm}^3/\text{m}^2 \cdot 24\text{h} \cdot \text{bar})$ unit. Melt rheology and titration results and FTIR spectroscopy are reported elsewhere [22].

RESULTS AND DISCUSSION

Mechanical Properties

Effect of PP-g-MA as compatibilizer on mechanical properties of PP/PET (80/20) blends was examined. Tracing the variation of mechanical properties can be used as a method to evaluate the compatibility qualitatively [1]. Table 4 shows the data for mechanical properties of the samples at MD and TD directions. As expected stress-at-break for all samples at $\lambda=2$ is more than $\lambda=1$ except in blend No.1 with no compatibilizer. Increasing the compatibilizer content up to 10% resulted in an increased tensile strength of the blend films, attributed to the enhancement of interfacial adhesion between the PP and PET phases. Addition of more compatibilizer, however, has no sig-

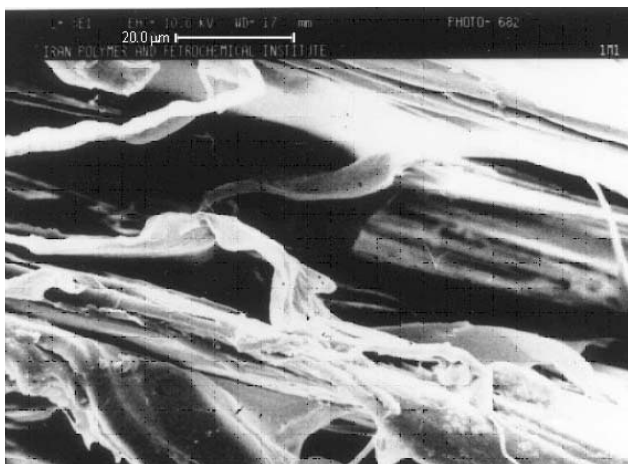
nificant effect, as there is an optimum content for any blend that it may saturate the interface region. Improvement of stress-at-break in TD showed similar pattern. As it was observed the modulus and strain-at-break were decreased by stretching, which could be due to the fact that the film drawing is performed in molten state, leading to a decrease in intermolecular interactions. It seems that drawing in presence of PET domain phase results in an increase in crystallinity and consequently the modulus [22]. In the case of strain-at-break increasing the compatibilizer content has a reverse effect (Table 4), which may be attributed to the differences in structure, physical, and thermal properties of PP and PET and those of PP employed.

The glass transition temperature (T_g) of PET was 85°C (DMTA method). Therefore, the amorphous phase of PET is in glassy state at the test temperature (28°C). Consequently, strain-at-break of PET is very low. In the case of PP, T_g is 13.04°C (DMTA method). Thus, its amorphous phase is in rubbery state at the test temperature. Moreover, the PP grade used in this study showed a high value of strain-at-break because of its random copolymer structure. Thus, compatibilization has resulted in a decrease in the value of strain-at-break.

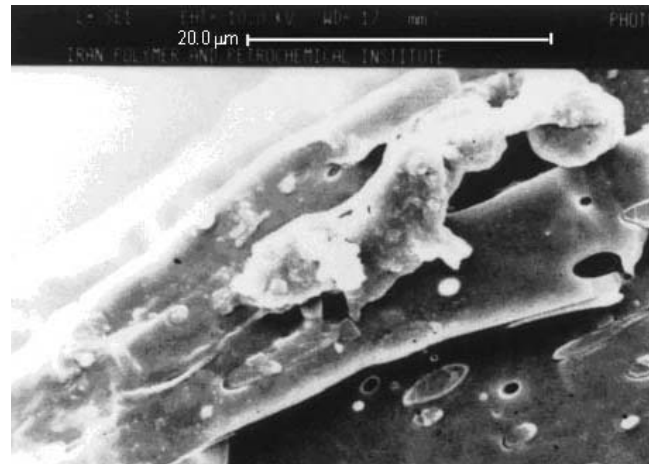
Morphology

In this study, the catalyzed compatibilizer (PP-g-MA+PTSA) was used to enhance the interfacial adhesion between the dispersed and continuous phases and therefore, to obtain a stable morphology in immiscible binary blends of PP and PET. Furthermore, it is known that the addition of a compatibilizer to immiscible blends prevents the coalescence of dispersed particles during the melt flow of two processes, i.e., melt blending and flat film casting. The film casting process via flat die has a limitation in enhancing barrier property of blend film because there is no sufficient biaxial stretching resulting in laminar structure of the dispersed domains with a large area. The effect of compatibilizer content and processing variables, i.e., draw (stretch) ratio on the oxygen permeability and morphology of blend films was investigated.

In SEM micrographs the dark areas are PP matrices and light areas are PET dispersed phases. Figures 1a - 8a show SEM micrographs at MD and Figures 1b - 8b are at TD. As it is evident in these micrographs



(a)

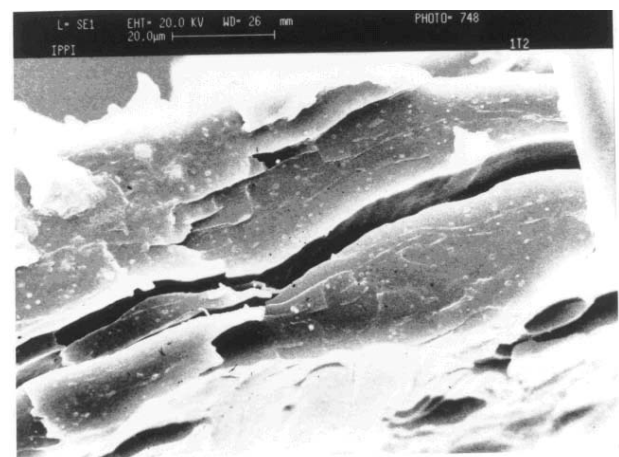


(b)

Figure 1. SEM Micrographs of uncompatibilized PP/PET blend films at: (a) MD and (b) TD at $\lambda=1$.

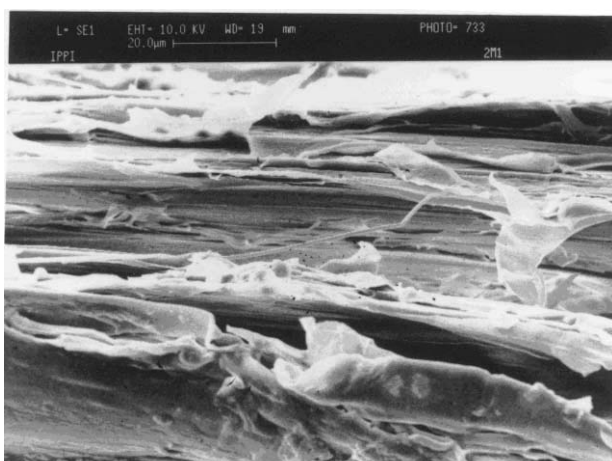


(a)



(b)

Figure 2. SEM Micrographs of uncompatibilized PP/PET blend films at: (a) MD and (b) TD at $\lambda=2$.

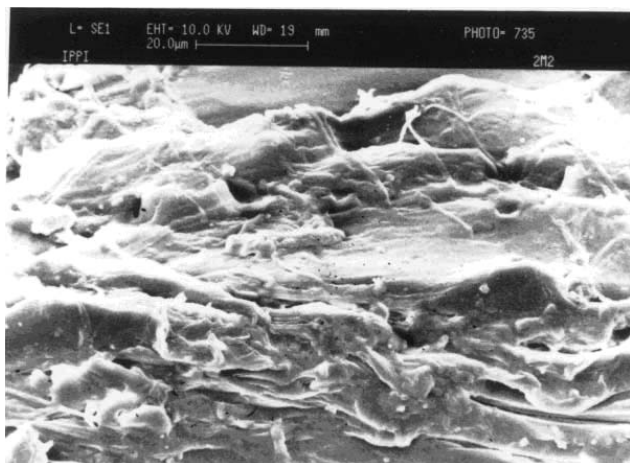


(a)

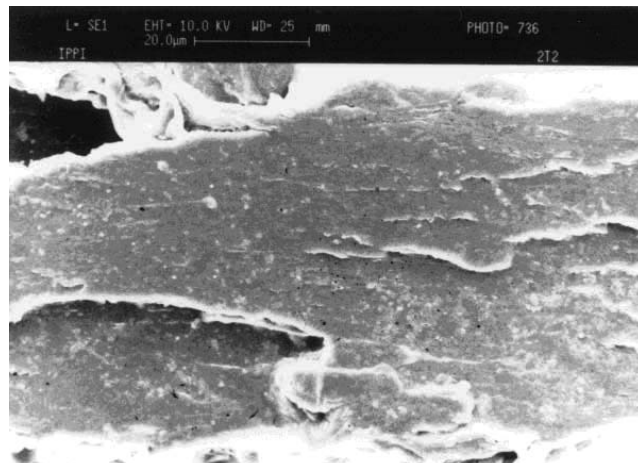


(b)

Figure 3. SEM Micrographs of PP/PET blend films with 5% compatibilizer at (a) MD and (b) TD at $\lambda=1$.

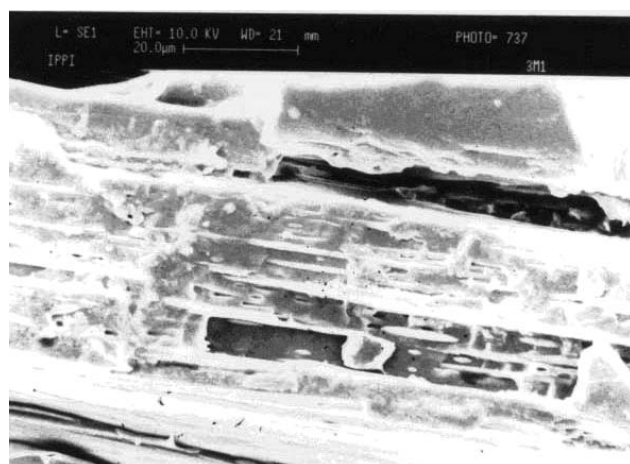


(a)



(b)

Figure 4. SEM Micrographs of PP/PET blend films with 5% compatibilizer at (a) MD and (b) TD at $\lambda=2$.

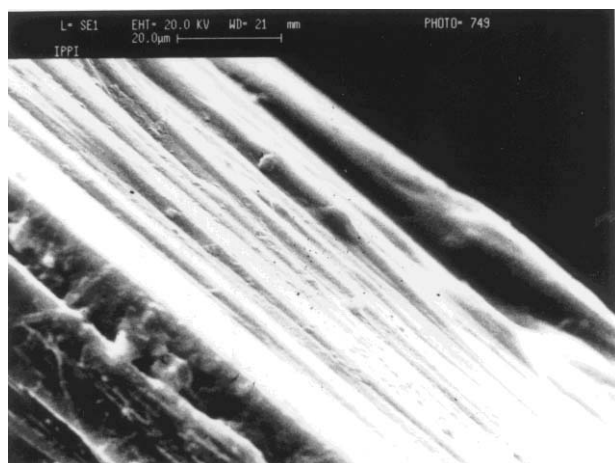


(a)



(b)

Figure 5. SEM Micrographs of PP/PET blend films with 10% compatibilizer at (a) MD and (b) TD at $\lambda=1$.

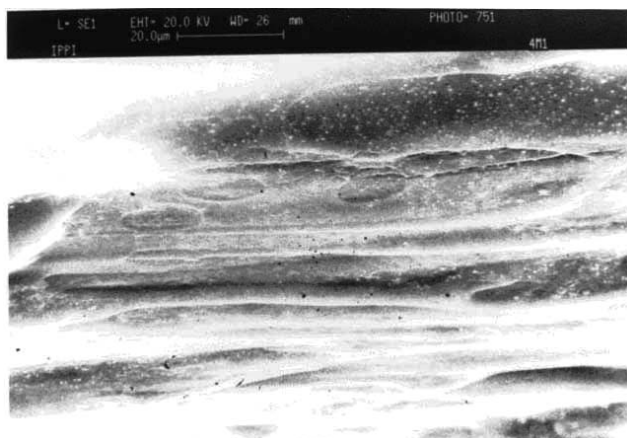


(a)

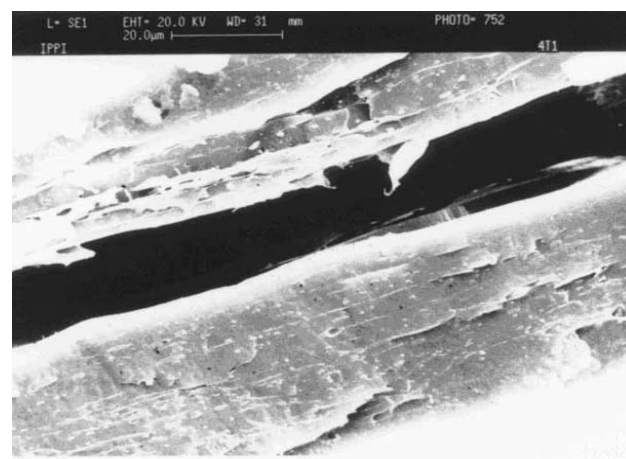


(b)

Figure 6. SEM Micrographs of PP/PET blend films with 10% compatibilizer at (a) MD and (b) TD at $\lambda=2$.



(a)

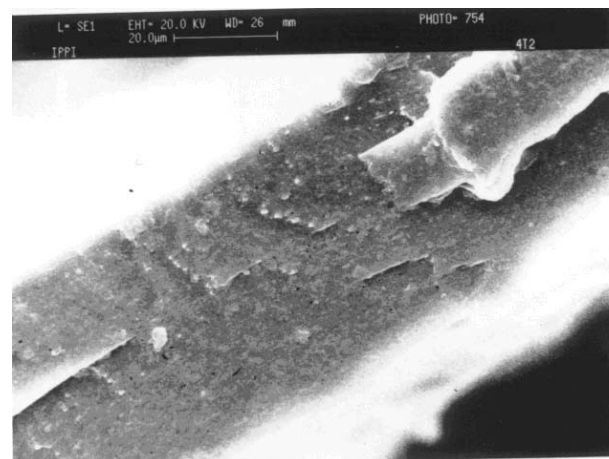


(b)

Figure 7. SEM Micrographs of PP/PET blend films with 15% compatibilizer at (a) MD and (b) TD at $\lambda=1$.



(a)



(b)

Figure 8. SEM Micrographs of PP/PET blend films with 15% compatibilizer at (a) MD and (b) TD at $\lambda=2$.

the lamellar morphology is more developed in MD compared to the TD. Compatibilization reaction between the hydroxyl groups of PET and maleic anhydrides has been repeatedly reported to give a finer and more homogeneous dispersion rather than in the non-compatibilized blends [21-37]. Polar PP-g-MA can also affect oxygen permeability because of the non-polar nature of oxygen [1]. The SEM micrographs of the films at MD (Figures 1a-8a) show the effect of compatibilizer content and draw ratio on the morphology and subsequent permeability as well.

As the Figures 1a and 2a of the blends show, there is no improvement, however, on the morphology in the absence of compatibilizer. The best lamellar morphology is produced by 5% compatibilizer and $\lambda=2$ (Table 5). With inclusion of more compatibilizer not

only a reverse effect is noticed but the fibril structures are appeared as well which is shown in Figures 5a-8a, contrary to Figure 4a in which the lamellar morphology is predominant. Studies on barrier properties have confirmed the above observations. Higher stretching values indicate considerable effects on the films morphology with lamellar morphology being formed at $\lambda=2$ rather than $\lambda=1$. The SEM micrographs also show that in presence of compatibilizer (up to 15%) thinning of PET domains has occurred.

Barrier Properties

The measured permeability, tortuosity factor (τ), permeability data according to Robeson model (series and parallel) and semi-logarithmic additivity rule for film samples are reported in Table 5 [1]. As can be

Table 5. Permeability data for original polymers and their blends.

Sample	Draw ratio (λ)	Tortuosity (τ)	Permeability (cm ³ /m ² .24h.bar)			
			Measured	Robeson (series)	Robeson (parallel)	Semi-logarithmic additivity rule
PP	1	-	362±2	-	-	-
	2	-	295±1	-	-	-
PET	1	-	7±1.2	-	-	-
	2	-	5±0.5	-	-	-
Blend1	1	1.61	193±1.6	44.7	312	208
	2	-	-	44.7	312	208
Blend2	1	1.55	187±2	32.5	291	164
	2	1.69	171±1.4	32.5	291	164
Blend3	1	0.98	278±5.6	26.5	273	135
	2	1.03	263±3.2	26.5	273	135
Blend4	1	0.79	315±11.5	21.6	252	107
	2	0.83	3.2±2.7	21.6	252	107

seen in Table 5, the oxygen permeability is initially reduced with increase in compatibilizer content, which may be explained by the fact that the addition of compatibilizer up to a certain level results in an increase in the number and frontal areas of thinner PET layers due to efficient stress transfer from the matrix (PP) to the dispersed phase (PET) during film stretching process.

In the case of uncompatibilized film blends as compared with the compatibilized films, a lower barrier property is observed, because oxygen molecules can diffuse through the micro-voids formed at the interface during stretching process. This may be due to poor interfacial adhesion between PP and PET. In addition, the increased permeability may be attributed to a reduction in number of PET domains resulting from coalescence of dispersed particles in the absence of compatibilizer. Poor barrier properties were also obtained at high levels of compatibilizer content. It is clear that by exceeding its optimum content (5% in this study) the size of PET is extremely reduced. This can be confirmed by morphological

variation with compatibilizer content in the oriented blend films, as shown in SEM micrographs. These results confirm that there exists an optimum level of compatibilizer content for obtaining enhanced barrier properties with a well-developed laminar structure (Figures 3a and 4a). At draw ratio 1, the films are not stretched. Therefore, the permeability data at $\lambda=1$ is lower compared to $\lambda=2$. This is also true for the original polymers.

The decrease in permeability of the original PP and PE may be attributed to an increase in the degree of crystallinity resulting from the film stretching. For 5% compatibilizer content and $\lambda=2$ (Figure 4a) the permeability decreases to 171 cm³/m².24h.bar. Compared to that of the non-stretched PP, there was more than 200% improvement in oxygen barrier properties. This is not a significant improvement in the barrier property as compared with the results already reported for EVOH [1-3,10]. However, the advantages of PET compared to EVOH are its lower cost and humidity resistance [3].

The draw ratio in a post-extrusion process is also

an important processing parameter in controlling the morphology of dispersed-phase. The permeability data show that the process of uniaxial film stretching would result in 10-20% decrease in the oxygen permeability. This is due to the enlarged area of PET domains due to the higher degree of deformation of the added dispersed phase, which provides long permeation paths (tortuosity τ) through the blends [1]. Comparing micrographs of the blend samples with draw ratios 1 and 2 shows the influence of draw ratio on the morphology of the blends. It is also observed that a higher draw ratio produces a well-developed laminar structure of the PET with a very large area.

CONCLUSION

Uniaxial orientation methodology enhances barrier properties by generating a well-developed laminar structure in blend films. Regarding the effects of compatibilizer content and draw ratio on barrier properties and morphology of uniaxially stretched blend films, obtained from the studies on rheological [22] and mechanical properties, and morphology of the blends it may be concluded that compatibility of the system is improved by use of a compatibilizer such as PP-g-MA. A large increase in barrier properties of PP/PET blends could be achieved by producing PET platelets of larger area within the blends through post-extrusion process. It is revealed that there exists an optimum level of compatibilizer content (5% for the studied blends) for obtaining enhanced barrier properties with a well-developed laminar structure. The barrier properties of the PP, PET, and their blend films have improved as draw ratio is increased.

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