

Influence of Ethylene Vinyl Acetate on the Spinnability and Mechanical Properties of Poly(propylene)/Zeolite-Ag Blend Fibers

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ABSTRACT: The spinnability and mechanical properties of poly(propylene) (PP)/zeolite-supported Ag⁺ (zeolite-Ag)/ethylene vinyl acetate (EVA) ternary blend fibers were studied. It was found that the spinning temperature of the ternary blend fibers was decreased in the presence of EVA. The addition of 2 wt % EVA substantially improved the spinnability of the blend system by enhancing its flowability. It was also found that the ternary fiber with EVA₂₈ (28 wt % vinyl acetate content) showed balanced improvement of mechanical properties by a concomitant increase in modulus and tensile strength. The improvements of spinnability

and mechanical properties suggested that a core-shell structure of zeolite-Ag/EVA₂₈ particles, with zeolite-Ag as the core and EVA₂₈ as the shell, was formed and remained during the melt-mixing process of the blended chips and during the course of fiber processing. EVA probably enhanced the binding between the zeolite-Ag and the PP matrix, as made evident in SEM microphotographs. © 2005 Wiley Periodicals, Inc. *J Appl Polym Sci* 96: 1460–1466, 2005

Key words: poly(propylene) (PP); blends; phase structure; mechanical properties; fibers

INTRODUCTION

Poly(propylene) (PP) has significantly progressed from the early stages of development to its present status as one of the common synthetic fibers. Much attention has been devoted to the development of functional PP fibers for many specific applications. Through the course of fiber processing, functional PP fibers can be produced by chemical and physical modifications. Blend-spinning is a simple methodology to modify many properties of PP fibers. Inorganic additives, such as titanium oxide, zinc oxide, magnesium hydrate, zirconium anhydride, and cuprous iodide, for example, are usually used in the blend-spinning of PP fibers, through which many characteristics, such as UV protection, fire resistance, far infrared light harvesting, and antistatic properties, can be imparted.^{1–4} An antibacterial property is an important function of PP fibers for PP fiber-based fabrics and nonwovens. Three types of additives are commonly used for the antibacterial modification of many synthetic fibers. The first type of additives includes nanosized inorganic powders, such as titanium oxide, zinc oxide,

magnesium oxide, and calcium oxide. The second type of additives constitute synthetic or natural zeolite-supported metal ions (Ag⁺, Cu²⁺, Zn²⁺, NH₄⁺). The third type of additives includes compounds of the two types of additives mentioned above.^{5–10} Nanosized inorganic powders show a remarkable antibacterial effect because of their photocatalytic actions, although this function is effective only under UV illumination.^{11,12} Zeolite-supported Ag⁺ (zeolite-Ag) is widely used because Ag⁺ exhibits an excellent antibacterial effect against *Escherichia coli* and *Streptococcus faecalis*.¹³ However, the spinnability and the mechanical properties of the fibers blended with the inorganic antibacterial additives normally deteriorate, although the dispersion of the inorganic additives could be modified by surface coating with coupling agents. It is still difficult to spin-blend PP fibers with zeolite-Ag, particularly because of the high loading ratio of the inorganic additive.

Ethylene vinyl acetate (EVA), an elastomer, is a known candidate to modify the processing of organic-inorganic composites and to balance improvements of impact strength and stiffness that have been achieved in several plastic products. Rita and Bluma¹⁴ found that blended PP fibers with a small amount of EVA showed higher elastic modulus than that of pure PP fiber. A similar improvement of mechanical properties with the addition of EVA was also found in colored PP fibers.¹⁵ Gupta et al.¹⁶ reported the melt-rheological

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TABLE I
Ethylene Vinyl Acetates Used and Their Melt Indices

	EVA ₁₄	EVA ₂₁	EVA ₂₈
Vinyl acetate content, %	14	21	28
Melt index, g/10 min	4.0	3.0	2.2
Producer	Mitsui Petrochemical Co., Japan	Formosa Petrochemical Co., Taiwan	Formosa Petrochemical Co., Taiwan

properties of PP/EVA blends by varying of the blend ratios and by using EVAs with altered vinyl acetate content. Good dispersion of EVA spherical droplets in the PP matrix was found. The mean diameter of the dispersed droplets is affected not only by the blend ratio but also by the shear stress. We previously reported the rheological properties of ternary blends of PP, zeolite, and EVA.¹⁷ It was found that the apparent viscosities of the ternary blends could be lowered, compared to that of PP/zeolite binary systems, when the content of vinyl acetate in EVA was judiciously controlled. Furthermore, surface modification of zeolite with coupling agents substantially influenced the viscosity of ternary systems, and use of polar coupling agents resulted in lower apparent viscosity because of the better interaction between the coupling agent and EVAs.

Based on the rheological properties of the ternary blends of PP, EVA, and zeolite, it is possible that EVA could modify the spinnability of PP fiber blended with zeolite. In this article, we report on the use of EVA as an additive to spin antibacterial PP fibers with zeolite-Ag. The spinnability, mechanical properties, and morphology of the blended fibers were investigated.

EXPERIMENTAL

Materials

Commercially isotactic poly(propylene) Prolen[®] [melt index (MI) of 30 g/10 min; Himont, São Paulo, Brazil] was used as the matrix material for the antibacterial fibers. Three ethylene vinyl acetates, provided by different companies, are listed in Table I, with vinyl acetate contents ranging from 14 to 28 wt %. The melt indices of the EVAs are also listed in Table I. All the melt indices of polymers were tested according to ASTM D 1238. The zeolite, used as a carrier in antibacterial agents, was kindly provided by Nanhai Mineral Co. (China) in the form of a needlelike solid; the content of SiO₂ was 58.44 wt %, Al₂O₃ was 14.98 wt %, and the specific area was 175 m²/g. The antibacterial zeolite-Ag, with a medium diameter in the range of 10 to 30 μm, was prepared in our laboratory by an ion-exchange method.⁹ The surface of zeolite-Ag was modified with the coupling agent YDH-201 (Nanjing Coupling Agent Factory, China).¹⁸

Preparation of blended masterbatches

A two-step process were adopted to prepare the ternary PP/zeolite-Ag/EVA masterbatches. First, the treated zeolite-Ag and EVA were melt mixed, and then the ternary composites of PP/zeolite-Ag/EVA were prepared, with proportions as listed in Table II. All were conducted on a Brabender rheometer (Brabender Technologie KG, Duisburg, Germany) at a temperature of 185°C and a torque of 60 rpm for 10 min. The blends were then diced with a dicing cutter (Nanjing Keya Machine Co., Ltd., China). The blended chips were dried in a vacuum oven at 65°C for 12 h before spinning.

Preparation of as-spun fiber

The as-spun fibers were prepared by adding different ternary masterbatches by 20 wt % into 80 wt % PP, and fiber samples were all the same as those in the masterbatches (e.g., F1 was prepared by adding 20 wt % M2 into 80 wt % PP). All as-spun fibers were spun at a pick-up speed of 18 m/min by a melt-spinning machine with a screw diameter of 25 mm and length to diameter ratio of 24, under a rotation speed of 13 rpm, at temperatures ranging from 200 to 230°C. A spinneret having 12 holes (0.5 mm diameter) was used. The melt line was cooled by conditioned air (28°C, 65% relative humidity).

Drawing and heating

A one-stage drawing was carried out for these as-spun fibers. The fibers were preheated by a heating plate, at

TABLE II
Proportions of Poly(propylene)/Zeolite-Ag/Ethylene Vinyl Acetate Masterbatches

	Poly(propylene) (wt %)	Zeolite-Ag (wt %)	EVA ₁₄ (wt %)	EVA ₂₁ (wt %)	EVA ₂₈ (wt %)
M1	95	5	0	0	0
M2	90	10			
M3	85	5	10	0	0
M4	85	5	0	10	0
M5	85	5	0	0	10
M6	80	10	10	0	0
M7	80	10	0	10	0
M8	80	10	0	0	10

a temperature ranging from 60 to 70°C, and drawn at a drawing ratio of 5.5. The line speed of the first roll was set at a constant rate of 12 m/min. The drawn fibers were dried and heat-treated at a temperature of 100 to 110°C for about 10 min.

Mechanical properties

Mechanical properties of the blended fibers, including the tensile strength and the elongation at break, were measured with a YG001 single-fiber tensile tester (China), at a downward rate of 24 mm/min. The results were obtained by averaging the outcomes of 24 runs.

Microstructure

The morphology of the blended fibers was examined by use of a scanning electron microscope (Hitachi S-500, Osaka, Japan). Specimens of ultrathin sheets (10–30 μm) were prepared by cutting tested sample bundles with a fiber-slicing knife. To examine the distribution of EVA in the PP matrix, some specimens were immersed in hot butylene oxide for 5 min to remove EVA from the blends. Some fiber samples were prepared by curing of the fibers with epoxy and then breaking them in liquid nitrogen. Before testing, all specimens were surface sprayed with gold.

RESULTS AND DISCUSSION

Influence of EVA on the spinnability of the PP/zeolite-Ag/EVA blend systems

At present, three different routines are commonly applied to determine the spinnability of fibers: (1) the method used to determine the tenuous flow's longest tensile length, (2) the method used to determine the tenuous flow's extension at break, and (3) the method used to determine the largest drawing ratio.¹⁹ Here we used the number of times of breakage during a 15-min period under continuous spinning conditions based on the first method, finding that the fewer the times of breakage, the better the spinnability of blends.

The influence of EVA on the melt spinnability of the PP/zeolite-Ag/EVA blend system is listed in Table III. For the binary blends with low zeolite-Ag content of 1 wt %, the spinnability is good—with one breakage in 15 min (Table III, F1). However, for the binary blends with 2 wt % zeolite-Ag, the spinnability deteriorated: two or more times of breakage occurred in 15 min (Table III, F2). The spinnability was substantially improved for the ternary blended chips with EVAs. No breakage, or fewer occurrences of breakage, occurred in 15 min for the two ternary blends with different contents of zeolite-Ag (Table III, F3–F8), indicating that the EVAs play an important role in the improve-

TABLE III
Spinning Conditions of the Blended Fibers^a

Sample	Zeolite-Ag (wt %)	EVA ₂₈ (wt %)			Spinnability ^b
		EVA ₁₄	EVA ₂₁	EVA ₂₈	
F1	1	0	0	0	+
F2	2	0	0	0	–
F3	1	2	0	0	++
F4	1	0	2	0	++
F5	1	0	0	2	++
F6	2	2	0	0	+
F7	2	0	2	0	+
F8	2	0	0	2	+

^a Spinning temperature: 220°C.

^b The symbols ++, +, and – denote excellent (no breakage in 15 min), good (one time of breakage in 15 min), and bad (two or more times of breakages in 15 min) spinnability, respectively.

ment of the spinning processing of PP/zeolite-Ag blends.

According to rheology theory, the shear flow of a fluid passing through the spinning head and the drawing flow of the spinning thread are the most related to the form of fiber. Two kinds of factors affect the flow behaviors: (1) the composition and structure of the melt; and (2) an external cause: spinning temperature, shear speed, winding speed, and the structure of the spinneret.¹⁹

The spinning temperature influenced the spinnability of ternary blends under the same shear speed and winding speed. For ternary blends with EVAs with different vinyl acetate contents, the spinnability was excellent at 2 wt % content of zeolite-Ag during a particular temperature range, and no breakage took place in 30 min (Table IV). The spinning was carried out smoothly at a relative low temperature of 205°C with EVA₂₈ (28% vinyl acetate content), compared with that of ternary blends with EVA₁₄ and EVA₂₁. The mass flow data were used as an indication of the flow condition of the ternary melts and, only in the range from 10.5 to 11.5 g/min, the spinnability was excellent under particular rolling speeds, such as 150 m/min used here. The results are consistent with the rheological behaviors of ternary blends of PP/zeolite-Ag/EVA, for which the lowest melt viscosity was found for PP/zeolite-Ag/EVA₂₈. It was also found that the spinning temperatures needed to be increased if the ternary blended chips were prepared by first mixing PP with the treated zeolite-Ag and then adding the EVAs.

Influence of EVA on mechanical properties of the PP/zeolite-Ag/EVA blend fibers

A one-stage drawing was carried out for the as-spun fibers, at a drawing ratio of 5.5. The mechanical properties of the drawn fibers were tested and the results

TABLE IV
Influence of Ethylene Vinyl Acetate with Various Content on the Spinning of Blended Fibers

Sample	Spinning temperature (°C)	Rolling speed (m/min)	Mass flow rate (g/min)	Spinnability ^a
F2	220	150	10.9	–
F6	208	150	10.5	++
F7	215	150	11.0	++
F8	205	150	11.2	++

^a The symbol ++ denotes excellent spinnability (no breakage in 30 min), and – denotes two or more times of breakages in 15 min.

are listed in Table V. In the absence of EVA, the elongation at break of PP/zeolite-Ag fibers was 31%. The elongation at break of PP/zeolite-Ag/EVA₁₄ fibers was virtually identical to that of the binary blend fibers, although the elongation at break of PP/zeolite-Ag/EVA₂₁ fibers decreased. The elongation at break for PP/zeolite-Ag/EVA₂₈ fibers was 35.5%, with an approximately 15% increase compared to that of the binary blend fibers. The tensile strength and the modulus of all the PP/zeolite-Ag/EVAs fibers were higher than those of the binary blend fibers, indicating that the addition of EVA, an elastomer, had a reinforcing effect. We also tested Hook joint elongation at break and Hook joint strength in the fibers under investigation, with the three EVAs, and identified tendencies that were similar to those of elongation at break and tensile strength. The reinforcement as well as the increase of tenacity with EVA₂₈ demonstrated that EVA₂₈ was the most suitable additive to achieve balanced modifications of the mechanical properties of the blending fibers. Under the same addition level and processing conditions, the difference of EVAs on the mechanical properties of ternary blend fibers was related to the vinyl acetate content in EVAs and the average molecular weight of ethylene in EVAs.

The incorporation of vinyl acetate, compared to low-density polyethylene, in the ethylene chain increases flexibility, toughness, and clarity. These factors are primarily attributed to a reduction of the crystallinity of the polyethylene. EVA properties are influenced by vinyl acetate content and melt index. One of

the outstanding characteristics of EVA is its toughness at low temperature, and it exhibits excellent stress crack resistance, similar to that of polyethylene.¹⁹ EVA copolymers that have a vinyl acetate content < 30 wt % obey the Flory–Burfield theories of copolymer crystallization. When the vinyl acetate content is increased, the relative quantity of polyethylene amorphous phase increases and the degree of crystallinity decreases.²¹ Based on the rheology analysis, the PP/zeolite-Ag/EVA₂₁ system showed the highest apparent viscosity compared to that of other systems¹⁷ (e.g., the poorest movability of the molecular chain under the same stress, attributed to entanglement of molecular chain, or other reasons that also influence the mechanical properties of the ternary fibers). Thus in the mechanical test, the PP/zeolite-Ag/EVA₂₁ system showed the poorest mechanical properties with respect to both elongation at break and tensile strength.

The breaking mechanism of a fiber is normally regarded as a combination of slip and breakage of microfibrils. Poly(propylene) usually forms spherulites during the course of fiber processing. An abundance of microvoids should form as a result of the deformation of spherulites during the drawing process of as-spun fibers. Because most inorganic fillers and PP are incompatible, microvoids form in the interface between the inorganic fillers and the PP matrix, thus normally inducing a decrease of mechanical properties of the blending fibers. In the present study, the EVA layer on the surfaces of zeolite-Ag particles served as a stress-transition layer, conducting the plas-

TABLE V
Influence of EVAs on the Mechanical Properties of PP/Zeolite-Ag/EVA Blended Fibers^{a,b}

Property	Control	EVA ₁₄	EVA ₂₁	EVA ₂₈
Elongation at break, %	31.0 (±2.0)	31.2 (±2.7)	25.8 (±2.5)	35.5 (±1.9)
Tensile strength, cN/dtex	2.91 (±0.10)	3.10 (±0.08)	3.00 (±0.11)	3.19 (±0.09)
Modulus, cN/dtex	19.4 (±0.5)	22.8 (±0.4)	20.5 (±0.6)	24.0 (±0.3)
Hook joint elongation at break, %	24.9 (±1.2)	26.9 (±1.1)	24.8 (±1.2)	32.4 (±1.3)
Hook joint strength, cN/dtex	2.62 (±0.12)	2.84 (±0.10)	2.87 (±0.11)	3.00 (±0.13)

^a All the data for mechanical properties were the averages of 24 run times. The largest deviation is given in parentheses.

^b The contents of zeolite-Ag and EVA in blend fibers were all 2 wt %; all the as-spun fibers were drawn at a drawing ratio of 5.5.

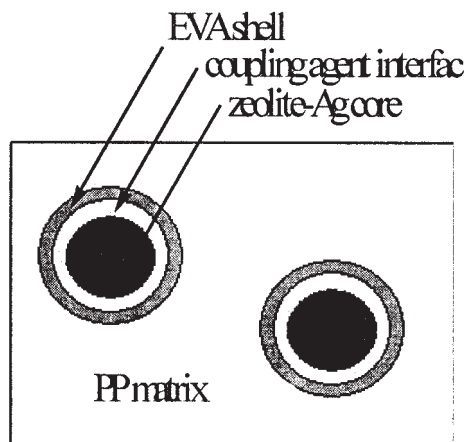


Figure 1 Schematic representation of the PP/zeolite-Ag/EVA ternary blend system.

tic deformation under the stress and maintaining proximate combinations of zeolite-Ag particles and the PP matrix.²² With increasing vinyl acetate content in the EVAs used in this study, the amorphous portion increases in EVA, which should improve the stress-induced deformation of the EVA layer and improve the mechanical properties of the blended fibers.

Gupta et al.¹⁶ reported that PP/EVA blends were only partially compatible, although Ramírez-Vargas et al.²³ reported that PP and EVA were compatible when the content of EVA was low. Wang and Zhao¹⁹ reported that EVA and PP were thermodynamically incompatible and had typical sea-island structures. In some PP/EVA/filler ternary blends,^{24–26} an encapsulation of filler with EVA or other elastomer might be formed. The phase-structure formation of these composites revealed that the main factor determining the composite phase structure constituted the surface characteristics of the components, such as wettability, surface energy, and interfacial adhesion between phases. In our study, the lower spinning temperature and the balanced improvements of the mechanical properties for PP/zeolite-Ag/EVA₂₈ blend fibers suggested that the treated zeolite-Ag particles were encapsulated by EVA₂₈ and the encapsulated zeolite-Ag particles were uniformly dispersed in the PP matrix. A schematic representation of the morphological structure of the PP/zeolite-Ag/EVA ternary blend system is shown in Figure 1. A core-shell structure of the system is presumed for zeolite-Ag particles after the modifications with the coupling agent and EVAs. Zeolite-Ag particles serve as a core and the coupling agent forms an interface on the core after the surface treatment by the coupling agent. Furthermore, an EVA forms a shell on surface-treated zeolite-Ag particles after the melt-mixing process. If no core-shell structure is formed in the ternary system, the zeolite-Ag particles may break away from the PP matrix in

the drawing stage, and microvoids should form, after which the mechanical properties of blend fiber should become weak. The EVA shell on zeolite-Ag can be regarded as a stress-transition layer that can conduct plastic deformation under the drawing stress and maintain a proximate combination between zeolite-Ag particles and the PP matrix.²² Judging from the mechanical properties of the ternary fibers, as listed in Table V, EVA₂₈ is the best of the studied EVAs to form an ideal core-shell structure because of the highest content of vinyl acetate to zeolite.

As shown in Figure 2, the structure of zeolite-Ag, which is a needlelike solid [Fig. 2(a)], changed only slightly during the mixing and spinning processes [Fig. 2(b)–(d)]. After the melt-spinning process, it was found that the color of zeolite-Ag particles changed from being initially white to slightly yellow. Similar results were also previously reported for polymer/zeolite-Ag blends. The color change of zeolite-Ag had only a limited influence on the antibacterial property.^{27,28} The SEM microphotographs of PP/Zeolite-Ag/EVA₂₈ masterbatches are shown in Figure 2(d) and (e). Without any EVA, zeolite-Ag particles in masterbatch M2 break off easily from the PP matrix under the shear stress of cutting during the sample preparation [Fig. 2(b) and (c); the arrow indicates the index of the shear stress]. Some zeolite-Ag was completely debonded from the matrix [Fig. 2(b)] and the gap between the zeolite-Ag and the polymer matrix was as large as 10 μm [Fig. 2(c)]. However, this phenomenon was not found in the PP/zeolite-Ag/EVA₂₈ masterbatch (M8) [Fig. 2(d)]. With the addition of EVA, the gap between the inorganic particles and the polymer matrix either decreased or disappeared under the shear stress. When EVA₂₈ was removed from the matrix by immersing the specimen in hot butylene oxide for 5 min, the SEM microphotograph of the etched specimen of M8 shows many dark holes on the surface [Fig. 2(e)]. The dark holes represent the EVA droplets that were dissolved during the selective etching, and the brighter, irregularly shaped particles were of the zeolite-Ag filler, which remained in the matrix. After etching, voids were observed around the filler particles, which should be encapsulated by EVA before the etching. The results suggest that EVA partially covered the surfaces of zeolite-Ag particles before etching, which supported our supposition of a core-shell structure. Apart from formation of an interlayer around the filler particles, EVA was also dispersed as small droplets in the PP matrix. The PP/zeolite-Ag/EVA₂₈ fibers showed a regular profile after the drawing process [Fig. 2(f)].

The morphology of PP/Zeolite-Ag/EVA₂₈ fibers is also shown in Figure 3. The PP/zeolite-Ag/EVA₂₈ fibers showed a regular profile after the drawing process [Fig. 3(a)]. The breakage surface of the fibers showed that white granules might be the zeolite-Ag/

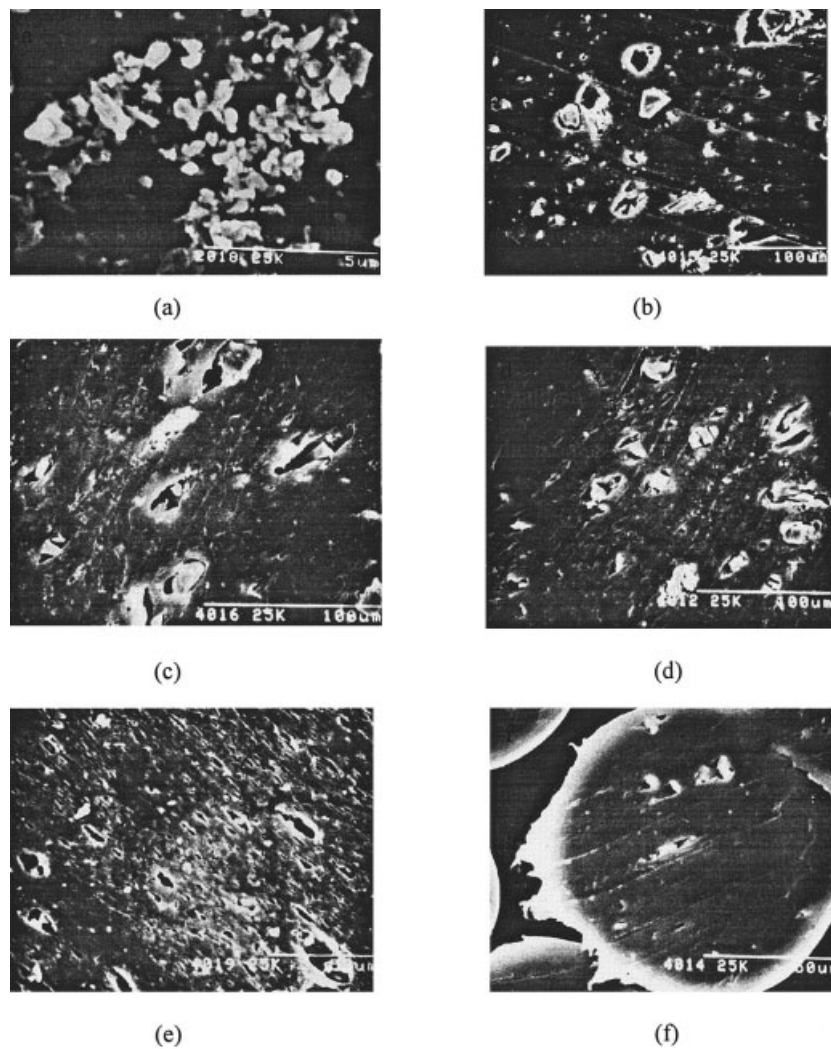


Figure 2 SEM microphotographs of (a) zeolite-Ag, (b) zeolite-Ag in PP/zeolite-Ag blend fiber, (c) the cross section of PP/zeolite-Ag masterbatch, (d) the cross section of PP/zeolite-Ag/EVA₂₈ masterbatch, (e) the cross section of PP/zeolite-Ag/EVA₂₈ masterbatch after selective etching by hot butylene oxide, and (f) the cross section of PP/zeolite-Ag/EVA₂₈ fiber. All blended samples were prepared by cutting with a fiber-slicing knife.

EVA₂₈ particles, whose size was as small as $<1.5 \mu\text{m}$. Thus the zeolite-Ag/EVA₂₈ particles, characterized by a core-shell structure, were well dispersed in the PP matrix.

CONCLUSIONS

Excellent spinnability of PP/zeolite-Ag/EVA ternary blend fibers, at a relatively lower spinning tempera-

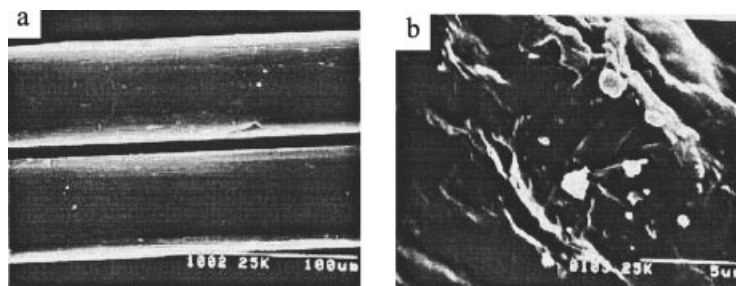


Figure 3 SEM microphotographs of PP/zeolite-Ag/EVA₂₈ fibers: (a) the fiber profile; (b) the break surface of the fiber. The sample was broken in liquid nitrogen.

ture, was achieved; this is in sharp contrast to the poor spinnability of PP/zeolite-Ag binary blend fibers. The exceptional spinnability can be attributed to the judiciously controlled melt-mixing sequences of the blended chips: melt mixing of EVAs and surface-treated zeolite-Ag particles first, followed by the addition of PP. It was found that the ternary fiber with an EVA₂₈ (28% vinyl acetate content) showed a balanced improvement of mechanical properties with a concomitant increase in modulus and tensile strength. The improvements of the spinnability and the mechanical properties suggested that a core-shell structure of zeolite-Ag/EVA₂₈ particles was formed and remained during the melt-mixing process of the blended chips and during the course of fiber processing, respectively.

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