# Morphology Structure Study of Polypropylene Hollow Fiber Membrane Made by the Blend-Spinning and Cold-Stretching Method

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**ABSTRACT:** This paper proposes one of simplest ways to prepare polypropylene/easy hydrolytic degradation polyester (PP/EHDPET) microporous hollow fibers by blend spinning and cold stretch process. And it details and investigates the structure properties of as-spun fibers, micropore formation mechanism, and pore structure of the hollow fiber membranes by the scanning electron microscope. © 2002 Wiley Periodicals, Inc. J Appl Polym Sci 84: 1390–1394, 2002; DOI 10.1002/app.10280

**Key words:** polypropylene; easy hydrolytic degradation polyester; hollow fiber membranes

## INTRODUCTION

The blend fibers with the sea-island (or matrixfibril) morphological structure are usually obtained by the method of blend spinning of two immiscible polymers. Adjusting the parameters, such as composition, viscosity ratio, and elasticity ratio as well as process conditions during mixing, the two components can form the sea phase, or island phase respectively.<sup>1–3</sup>

Recent studies<sup>8,9</sup> synthesized a new type of copolyesters, easy hydrolytic degradation polyester (EHDPET), which can rapidly dissolve in the hot alkaline solution. Its glass transition temperature is relatively high (its  $T_g$  is about 70°C) compared to that of polypropylene (PP) (its  $T_g$  is about -30°C). So under a certain condition, the blend of PP/EHDPE. The can be easily form two-

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phase structure, in which EHDPET component is used as island phase.

Under the cold-stretching temperature of a bit lower than  $T_g$  of EHPET, such as 50°C, PP component will be oriented along the fiber axis and the EHPET component cannot be deformed. which makes the phase separations appear at the interface between PP phases and EHPET phases. So the surfaces and interface of the hollow fibers will appear as some small pores or narrow slots. Meanwhile, the hydrophilicity of PP/EHDPET membrane will be improved because it contains some of hydrophilic EHPET component. Melting blend and cold-stretching process is a new kind of modified method, which may endue the PP/EHDPET membrane with not only the bigger pores, but also the favorable hydrophilicity.4,5

This study has investigated the morphological structure of the PP/EHDPET blend, the phase separation phenomenon during the stretching

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Figure 1 Micropore formation mechanism of blend spinning and cold stretching.

process, and the pore structure by the scanning electron microscope.

## **EXPERIMENTAL**

#### Materials

PP chips is a commercial product with melt flow index (MFI), 35 g/min (2.16 kg, 230°C), obtained from Yanshan Petrochemical Corporation of China; and EHDPET chips was supported by Tianjin Petrochemical Corporation of China, the intrinsic viscosity number  $\eta$  is 0.37.

#### **Preparative Procedure**

Accorded a certain composition ratio, PP chips and EHDPET chips were well mixed and then extruded at 250–300°C through a single-hole tube-in-orifice type spinneret to manufacture hollow fibers. The outer diameter and inner diameter of the spinneret were 10 and 8 mm, respectively. The as-spun hollow fibers were cold stretched at  $45^{\circ}$ C, and the stretching ratio was 6. The outer diameter, inner diameter, and wall thickness of the resulting PP hollow fibers were  $450 \sim 400$ ,  $350 \sim 200$ , and  $50 \sim 80 \ \mu$ m, respectively.

#### Scanning Electron Microscopy (SEM)

The morphology of PP/EHDPET blend fibers was investigated by using a scanning electron microscopy, KYKY AMRAY1000, from Chinese Academy of Science. The accelerated voltage and probe current were 5 kV and 5 mA, respectively. Specimens were coated with a thin golden layer before observation.

## **Differential Scanning Calorimeter (DSC)**

DSC measurement was performed with a Perkin-Elmer model DSC-7500. During the thermal treatment and DSC scanning, the specimens were fully protected by dry nitrogen purge gas to prevent them from oxidation. Samples were rapidly heated from ambient temperature to 280°C at a heating rate of 100°C/min, holding for 3 min to



Figure 2 DSC curves of PP/EHDPET blend fibers.



Figure 3 Sea-island structure of as spun PP/EHDPET.

destroy all crystalline nuclei. Then the samples were cooled to 100°C at a cooling rate 20°C/min, and naturally cooled from this temperature to 100°C, the crystallization curves were recorded at the same time. After this, the samples were heated to 280°C at a heating rate of 20°C/min, and the melting curves were recorded.

# **RESULTS AND DISCUSSION**

# **Micropore Formation Mechanism**

PP/EHDPET microporous fibers are prepared by cold stretching of the as-spun PP/EHDPET blend described as above. It can be seen from Figure 1 that the stretching causes the splitting of the PP phases at the periphery of the EHPET particles and results in a microporous PP texture, which is also the reason for whitening of the resultant fibers.

# **Thermal Behavior**

Figure 2 shows the heating and cooling behavior of PP, EHDPET, and PP/EHDPET blends. It

is clearly illustrated that the PP/EHDPET blend was thermally immiscible system. In fact, PP and EHDPET crystallize independently.

Figure 3 was SEM images of the cross-section of as-spun PP/EHDPET hollow fibers (70/30 wt %). The blend fiber has typically two-phase structure, in which one component formed the "island" phase as anomalous large spherical particles distributed in the "sea" shown in Figure 3(a). And obviously there is an interface layer between the PP phase and EHDPET phase. EHDPET is dyeable by cation dyes, but PP is not dyed. So it could clarify which is sea and which are islands in the blend. Figure 3(b) indicated that EHDPET were islands and PP was sea since EHDPET particles were dyed in dark.

# Pore Structure of Hollow Fibers

Figure 4 is SEM image of cross-section of as-spun fibers before cold stretching process. It is clearly indicated that the as-spun fiber had sea-island



Figure 4 SEM images of cross-section of as-spun hollow fiber.



Figure 5 SEM images of microporous membrane.

structure but there were no interconnected micropores.

Figure 5 shown SEM images of PP/EHDPET hollow fiber membranes: the whole view, the outer surface, and the cross-section perpendicular and parallel to the fiber axis direction. The results showed that the microporous PP hollow fiber had an interconnected fibrous structure parallel to the fiber axis, and the pores in the surfaces were long elliptical, and paralleled to the fiber axis. Generally each pore was containing one or two EHDPET particles.

# **CONCLUSIONS**

PP/EHPET is an incompatible blend system that can form a two-phase structure, in which polypropylene component as continuous phase (sea) and EHPET component as dispersed phase (island). During the cold stretching process, the sea and island phases of the blend fiber separated along the fiber surfaces and interfaces to a form microporous porous structure. This paper detailed investigated the structure properties of as-spun fibers and micropore formation mechanism and pore structure of the hollow fiber membranes by the scanning electron microscope. And it is noteworthy that the blend spinning and cold stretch process as described above is one of the simplest ways to prepare PP microporous hollow fiber.

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