

# Fine Disperse Dyeable Polypropylene Fiber from Polypropylene/Polystyrene Nano-Ceria Blends

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**ABSTRACT:** The blend of polypropylene (PP) and atactic polystyrene (PS) hybrid with nano-ceria (CeO<sub>2</sub>) was prepared for fine dyeable fiber. Nano-CeO<sub>2</sub> modified with stearic acid was in situ added to PS by radical suspension polymerization. The dispersion of hy-PS with nano-CeO<sub>2</sub> in the PP crystals was investigated. The rheological behavior of the blend was similar to that of PP under the testing conditions, and the blend had stable spinning and drawing processability. The fine modified PP filaments were processed from the blend and had practical mechanical

properties. The dyeability of the knitting fabrics from these filaments was studied. The increased amorphous content and the interface between PP and hy-PS allowed the dye to penetrate the fiber, and the modified PP fiber had strong affinity to dye because of the complexation between cerium and dye. These effects resulted in improving *K/S* value and color fastness to soaping. © 2009 Wiley Periodicals, Inc. *J Appl Polym Sci* 113: 1953–1958, 2009

**Key words:** polypropylene; polystyrene; ceria; dye

## INTRODUCTION

Polypropylene (PP) is widely applied in the textile industries because of its good chemical resistance, low cost, low density, good processability, and comfort to the skin. Fine PP fiber is an ideal material for clothing because of its good handling quality, moisture permeability, and other advantages. Recent studies on modified PP fiber have focused on functional modifications, such as dyeability, antistatic properties, and flame resistance, etc.

It is necessary to modify PP to overcome its disadvantages for further developing its uses. Blending is an efficient process for the dyeable modification of PP in all of these modified methods. A wide range of additives has been tried. Dayioglu<sup>1</sup> selected 2-vinyl pyridine/styrene copolymer as an additive to improve the dyeability of PP fiber and found that industrially acceptable shades could be attained by selecting the amount and type of copolymer and the disperse dyes. Son et al.<sup>2</sup> prepared PP fiber dyeable with disperse dye by melt blending with poly(ethylene-covinyl acetate) (EVA) prior to extrusion. A variety of polymers with polar groups has been used to modify the dyeability of PP fiber.<sup>3–7</sup> Inorganic nanoparticles have

been an excellent additive, with which PP were blended to improve its dyeability. It was reported by Fan and coworkers that PP was dyeable by the influence of nano-clay.<sup>8,9</sup> However, there was a disadvantage that the dyeings were uneven, indicating that the nano-clay was not properly dispersed in the PP matrix because of high agglomeration and poor dispersion, and the particle size used was not in the nanometer range. It was also demonstrated by many researchers<sup>10–13</sup> that the *K/S* value and fastness to soaping could be improved by adding rare earth in dye bath.

The resin for fine filament requires excellent spinning and drawing processability. Our colleagues pioneered many studies on dyeable fine PP resins.<sup>14–17</sup> We added up to 8% atactic polystyrene (PS) with a controlled molecular weight and molecular weight distribution to PP fiber. These blends had good spinnability and improved dyeability. Fine fiber with improved dyeability was produced and successfully industrialized. Unfortunately, because of weak affinity between dispersed dye and low polar polyolefin derivative, their fastness to soaping was not acceptable in practical use, especially for medium to dark shades. The goal of this study was to increase the dyeability of PP fiber through the blend of PP and hybrid PS containing nano-ceria (CeO<sub>2</sub>) modified with stearic acid, which maintained acceptable blend processability and good mechanical performance. The key factors of obtaining satisfactory blend filaments, including the processing conditions and blend morphology, were investigated. The

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dyeings of the knitting fabrics made by the blends were used to evaluate the dyeability of these fibers.

## EXPERIMENTAL

### Materials

Nano-CeO<sub>2</sub> as an additive was of 100 nm size from Jiangsu Zhuoqun Nano Rare Earth (Jiangsu, China). Stearic acid was of analytical grade. Styrene as a monomer and benzyl peroxide (BPO) as an initiator were of chemical grade. Sodium thiosulfate as a chain transfer agent was of analytical grade. Polyvinyl alcohol (PVA) as a protective agent was of commercial grade, with molecular weight between 75,000 and 79,000. PP as a matrix polymer specially designed for fine filaments, with a melt-flow rate of 25–35 g/10 min, was supplied by Dong-Hua University Resin Factory (Shanghai, China). Silvatol as a de-oil agent was of commercial grade from Ciba (Shanghai, China). The dyes used were C. I. Disperse Red 167 and C. I. Disperse orange 30, supplied by Dystar (Shanghai, China), which were purified by the recrystallization method.<sup>18</sup> The stock solution was prepared by pasting 1 g of disperse dye with a small amount of dispersing agent and then adding distilled water to make 1% solution.

### Preparation of hy-PS (CeO<sub>2</sub>) and PP blends

Nano-CeO<sub>2</sub> particles were placed in a sealed glass tube with stearic acid and absolute alcohol at 25°C, oscillated with ultrasonic wave for 30 min, then agitated and heated to 75°C and refluxed for 5 h. At the end of modification, the solution was placed until demixing and sedimentary powder was obtained. Finally, the product was dried at 50°C under vacuum for 24 h.

The samples of hybrid-PS (CeO<sub>2</sub>) containing modified nano-ceria (hy-PS (CeO<sub>2</sub>)) were prepared by suspension polymerization.<sup>19</sup> The monomer styrene and modified nano-CeO<sub>2</sub> particles were placed in a sealed glass tube with PVA and oscillated with ultrasonic wave for 30 min; the initiator BPO and sodium thiosulfate were added and agitated in a temperature-controlled water bath from 70 to 95°C progressively, polymerized for 10 h. At the end of preparation, the product was washed with distilled water (40°C). Finally, the product was dried at 60°C under vacuum for 3 h. The number-average molecular weight ( $M_n$ ) and molecular weight distribution ( $W_d$ ) were determined with the Shimadzu LC-2a gel permeation chromatography system (Kyoto, Japan).  $M_n$  of PS was  $1.34 \times 10^5$ , and  $W_d$  was 1.65.

PP pellets were dried in a vacuum dryer at 80°C for 12 h before they were blended with hy-PS (CeO<sub>2</sub>) with an SHL-35 twin-screw extruder ( $D = 35$

mm) at 220°C; the blend was then palletized and kept at 60°C under vacuum for 6 h. To evaluate the agglomeration of nano-CeO<sub>2</sub>, the morphology of the CeO<sub>2</sub> in PS after melt extruded in the SHL-35 twin-screw extruder was observed with JSM-5600LV scanning electronic microscopy (JEOL, Japan). The samples were broken off under liquid nitrogen before gold was sprayed onto these cross-sections.

Based on our former research on dyeable fine PP, the result showed that as the PS component was increased from 0 to 8%, especially from 4 to 8%, there was a further increase in dye uptake. Practically acceptable mechanical properties were attained from the fiber blended with 2 to 8%, and spinnability on a small scale was satisfactory up to 8% of additive.<sup>14–17</sup> The blends of PP/PS (8.0 wt %) and PP/hy-PS (8.0 wt %) were chosen for the research.

### Determination of the morphology and rheology property of the blends

PP, PP/PS, and PP/hy-PS (CeO<sub>2</sub>) blended chips were completely melted at 220°C and crystallized at 138°C. The crystallization of the blends and the dispersion of PS and hy-PS (CeO<sub>2</sub>) in PP were observed. Photographs of the crystal morphology were taken with a polarized microscope (Nikon SE/YS Alphaphot-2 YS2). The morphology of PS and hy-PS (CeO<sub>2</sub>) in PP was taken with a JSM-5600LV scanning electronic microscopy (JEOL, Japan).

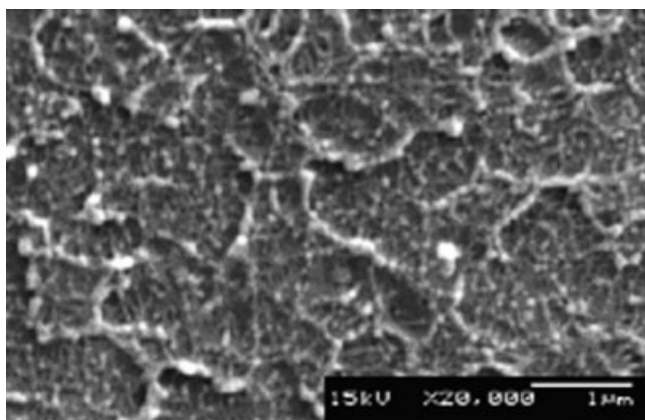
PP, PP/PS, and PP/hy-PS (CeO<sub>2</sub>) blends were dried at 120°C in a vacuum oven for 12 h before the rheology test was performed with an Instron 3211 capillary rheology meter (Instron, England). Test temperatures ranging from 230 to 250°C and shear rates ranging from 10 to  $10^3$  s<sup>-1</sup> were studied to cover the processing conditions.

### Preparation of the fiber

The filaments made of PP blend were processed with Fujifilter MST C400 spinning tester (Tokyo, Japan) with 28 spinnerets ( $L/D = 2$ ) at the take-up speed of 400 m/min. The as-spun filaments were then drawn with a Barmag Sprinnzwirn 3013 drawing-twisting machine (Chemnitz, Germany) at 850 m/min. Drawing ratio of 4–5 was tried to identify the drawability of the as-spun filaments. The yarns with a single end of less than 1.0 dtex/f were collected for evaluation.

### Evaluation of the fiber mechanical and structural properties

The drawn fibers were tested on XL-1 strain–stress tester (Shanghai, China), whose gauge was set to



**Figure 1** The dispersibility of CeO<sub>2</sub> nanoparticles in PS/CeO<sub>2</sub> nanocomposites.

10 mm, and elongation speed was adjusted between 35–45 mm/min to ensure a break time of ~ 20 s.

The degree of crystallinity was calculated from density measurement at 25°C by the density gradient column method with an MD-01 density tester (Zhejiang, China) filled with a mixture of 1-propanol and water.

The orientation of the fibers was measured with an SOM-2 sonic speed orientation tester (Shanghai, China) at a frequency of 10 kHz.

### Dyeing

Three knitting fabrics with the drawn filaments of PP, PP/PS (92/8 w/w), and PP/hy-PS (CeO<sub>2</sub>) (92/8 w/w) were produced to evaluate the dyeability of the blend.

Knitting fabrics were prepared and scoured for 30 min with 1% Silvatol (wt % of fiber) and 1% caustic soda (wt % of fiber) at a material to liquor ratio of 1 : 100 at 80°C, followed by washing with distilled water and drying.

The fabric was dyed in a finite bath (pH = 4.5) at a material to liquor ratio of 1 : 20 at 100°C in a

beaker-dyeing machine for 10 h to obtain equilibrium absorption of dye. After the completion of the dyeing cycle, the samples were soaped for 30 min with 1% soaping agent (wt % of fiber) at a material to liquor ratio of 1 : 50 at 80°C and then rinsed twice with distilled water before color estimation.

The color darkness on the fabric surface ( $K/S$  value) was measured with the use of a Hitachi 307 color photometer.  $K/S$  value was calculated by the Kubelka-Munk Formula.

$$K/S = \frac{(1 - R_\infty)^2}{R_\infty} = kc,$$

where  $K$  represents absorption coefficient,  $S$  represents scattering coefficient, and  $R_\infty$  represents reflecting ratio at certain wave length.

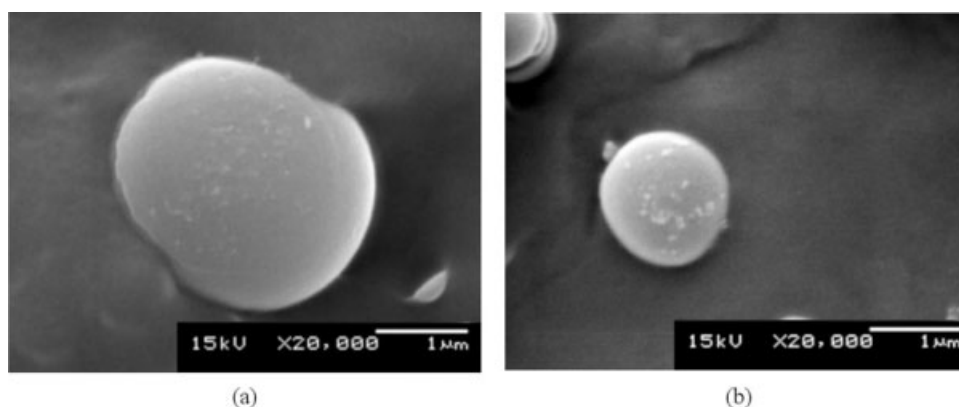
Fastness to soaping of the knitting fabrics was evaluated as per the method described by Huang.<sup>13</sup>

## RESULTS AND DISCUSSION

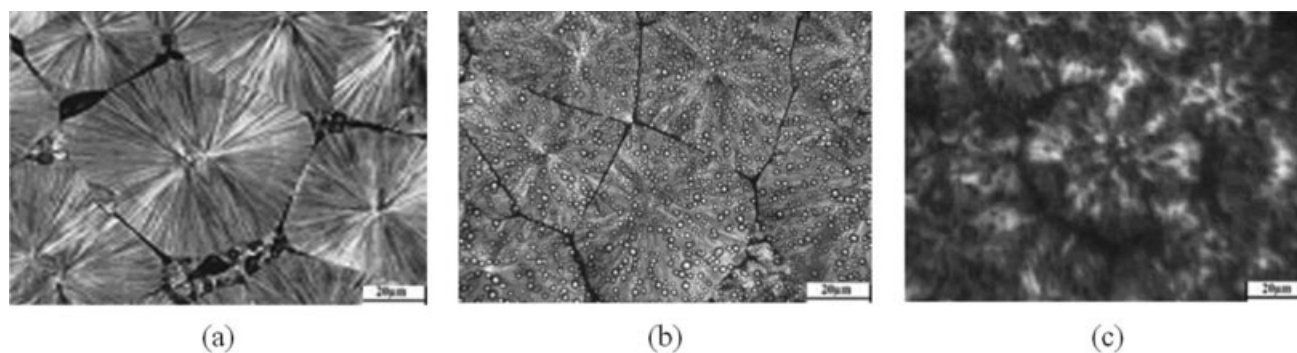
### Rheology and morphology of the PP/hy-PS (CeO<sub>2</sub>) blend

Inorganic nanoparticles are used to blend with a polymer for obtaining material with some improved performance. However, inorganic nanoparticles are not usually properly dispersed in the polymer matrix because of their high agglomeration, poor dispersion, and the particle size used not being in the nanometer range. Nano-CeO<sub>2</sub> modified with stearic acid was added to PS by radical suspension polymerization and melt-extruded with a twin-screw extruder at 210°C. The morphology of the CeO<sub>2</sub> in PS did not agglomerate and was all in ~ 100 nm (Fig. 1), almost the same size as raw nano-CeO<sub>2</sub> material.

Hy-PS (CeO<sub>2</sub>) was applied as an additive in PP to obtain fine PP fiber with maintained mechanical properties and improved dyeability. The viscosity, morphology, and interfacial structure of the PP/hy-PS (CeO<sub>2</sub>) blend were significant factors of the



**Figure 2** The dispersion of modified PS in the PS blends: (a) PP/PS and (b) PP/hy-PS (CeO<sub>2</sub>).



**Figure 3** PP crystal in the PP/modified PS blends under polarized light: (a) PP without additive, (b) PP/PS, and (c) PP/hy-PS (CeO<sub>2</sub>).

spinnability and dyeability. The effects of the hy-PS (CeO<sub>2</sub>) additive on the blend rheology and morphology are discussed in the following.

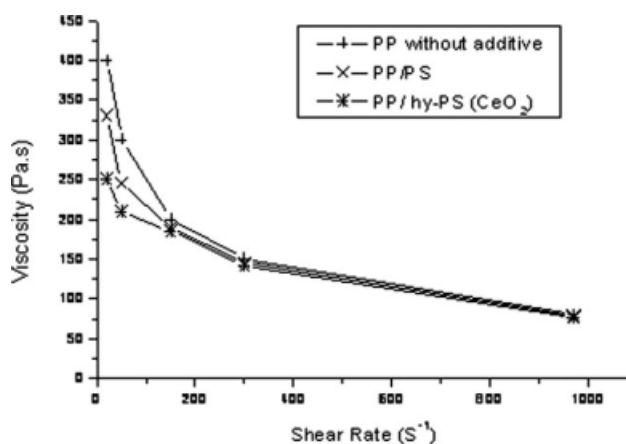
PP and PS formed an immiscible polymer blend. The dispersion of PS and hy-PS (CeO<sub>2</sub>) in the PP matrix was observed by scanning electronic microscopy, as shown in Figure 2. The dispersed PS and hy-PS (CeO<sub>2</sub>) phases were spread uniformly in the continuous PP phases as round particles. The diameters of the dispersed phase ranged from 1 to 5 µm. In the blend system, hy-PS (CeO<sub>2</sub>) was not only used as an additive but also as a dispersing agent because the polarity of nano-CeO<sub>2</sub> modified with stearic acid would be decreased. Thus, the diameter of the hy-PS (CeO<sub>2</sub>) phase in PP [Fig. 2(b)] was smaller than that of PS additive.

With a polarized light source, pictures of the spherulites were taken, as shown in Figure 3. The spherulite shape of the PP crystallites in the PS and hy-PS (CeO<sub>2</sub>) systems, as shown in Figure 3(b,c), was very similar to that of PP [Fig. 3(a)]. There were visible particles within the spherulite areas, not conchoring massively among PS granules and agglomerating among nano-CeO<sub>2</sub> particles, but these additives did not change the morphology of the spherulites much. In the blend of PP and PS, the bound areas between the spherulites were clear, with some dark areas between the spherulites. PP/hy-PS (CeO<sub>2</sub>) systems had better compatibility than PP/PS, which could be confirmed by the phenomenon of blurrier interface of PP/hy-PS (CeO<sub>2</sub>) than that of PP/PS. We suppose this was because PS was immiscible with PP. When PP crystallized, PS tended to be expelled from the PP crystal area. With the growth of the spherulite, the additive aggregated outside the spherulite, hindering it from moving closer and crystallizing into the spherulite structure. Therefore, the dark area formed was closely related with the amorphous part of PP and additive. The amorphous part between the spherulites and the interfaces between PP and the additives within the spherulite areas both helped the penetration of the dye into the modified PP fiber.

The rheology curves of the PP/hy-PS (CeO<sub>2</sub>) blend were tested at 240°C at a shear rate range of 10–10<sup>3</sup> s<sup>-1</sup> (Fig. 4), along with PP and PP/PS for a comparison. All these melt-flowing curves coincided with the curve of pseudoplastic fluid, which their viscosity decreased as the shear rate increased. PP/hy-PS (CeO<sub>2</sub>) showed rheological behavior very close to those of the PP and PP/PS systems, but with slightly lower viscosities, especially at low shear rate. The decreased viscosity was caused by the easier interfacial slide between hy-PS (CeO<sub>2</sub>) and PP matrix. The rheology information helped us to identify the spinning/drawing process settings for PP/hy-PS (CeO<sub>2</sub>) blend.

### Spinnability of PP/hy-PS (CeO<sub>2</sub>) blend

Spinning tests for PP, PP/PS, and PP/hy-PS (CeO<sub>2</sub>) blends were carried out with a spinning tester. Yarn with 28 filaments was taken up at 400 m/min winding speed. These filaments were not as transparent as pure PP fiber when they came from these spinnerets. Those color effects occurred in all the blends. It might have come from the interfaces between PP and



**Figure 4** Rheology curves of the PP/modified PS blends at 245°C.

**TABLE I**  
The Processability and Mechanical Properties of the Modified PP Drawn Filaments

Samples	Denier of the drawn filament (dtex/f)	Strength to break (Cn/dtex)	Elongation to break (%)	Spinning and drawing processability
PP	0.91	3.82–3.85	32.2–32.5	Stable
PP/PS	0.98	3.53–3.57	38.6–39.1	Stable
PP/hy-PS	0.95	3.61–3.63	39.6–40.1	Stable

modified PS when the phases separated. The drawing test with a drawing-twisting machine was carried out at draw ratios ranging from 4 to 5. The spinnability of the blends was acceptable for fine fiber process. PP/hy-PS (CeO<sub>2</sub>) showed good spinnability as PP and PP/PS systems. This blend produced a stable spinning line in the tested output range from 240 to 250°C. These filaments also showed good tensile strength at break and drawability. Table I listed the processability, denier, and mechanical data. All the drawn filaments reached less than 1 dtex/f. Tensile strength and elongation at break of a modified PP filament were very close to that of PP fiber.

#### Dyeability of the fine PP/hy-PS (CeO<sub>2</sub>) fiber

Degrees of orientation and crystallinity of fine dyeable PP have significant effects on its dye uptake.<sup>20</sup> Dye diffusion depends on two facts: the volume of the accessible region (amorphous region) and the tortuosity of the dye diffusion path. The larger the degree of crystallinity of fiber, the smaller is the volume of the accessible region for dye diffusion. Because accessible region and the tortuosity are associated with degrees of crystallinity and orientation greatly, it is easy to dye when degrees of crystallinity and orientation decrease.

In the polymer blend system, PP and PS were immiscible. The dispersed PS and hy-PS (CeO<sub>2</sub>) phases had effects on degree of crystallinity and orientation of the blend fibers. All blend fibers showed largely lower crystallinity than pure PP fiber did, but slightly higher orientation with respect to that of pure PP (Table II).

The knitting fabrics of PP/PS and PP/hy-PS (CeO<sub>2</sub>) were parallel to be dyed and tested with a color photometer and a soaping tester. The results

are listed in Table III. The *K/S* value of the fabric made by PP/hy-PS (CeO<sub>2</sub>) was slightly higher than that of PP/PS, and fastness to soaping was higher than that of PP/PS. Normally, the PP/PS fabrics could be dyed to medium shades but without acceptable fastness to soaping, and this resulted in little chance of practical applications. With the addition of hy-PS (CeO<sub>2</sub>) (8.0 wt %), the average fastness of all the tests reached level 4 or higher.

Because PP is alkyl chain with no polar groups and disperse dyes have strong polarity, the interaction between a dye and PP can only be van der Waals forces, which are relatively weak. Although PS can form the  $\pi$  bond with the groups supplying protons, it is also a weak force. This explains the acceptable color depth of PP/PS fabric but extremely low fastness to soaping.

To improve their dyeability, it is necessary to blend with other compositions with strong affinity to dye. Rare earth is an effective to improve dye uptake and fastness to soaping.<sup>21</sup> When rare earth is added in dye bath, the dye with larger molecular weight and strong forces is formed by a complex reaction between the rare earth ion and lone-pair-electron-supplying groups, such as  $-\text{OCO}-$ ,  $-\text{N}=\text{N}-$ ,  $-\text{NR}_1\text{R}_2$ , and  $-\text{NHCO}-$ , which commonly appear in the disperse dye structure. The complexation helps to increase both the *K/S* value and fastness to soaping. We suggest that some CeO<sub>2</sub> reacted with acid, and cerate was formed first when the PP/hy-PS (CeO<sub>2</sub>) fiber was dyed in the acid dye bath, then the complex reaction occurred between cerate and disperse dye. This explains why the fastness to soaping of PP/hy-PS (CeO<sub>2</sub>) was improved, especially for medium to dark shades. Thus, PP/hy-PS (CeO<sub>2</sub>) (92/8 w/w) is a successful modification for the fine dyeable PP filaments.

**TABLE II**  
The Crystallinity and Orientation of the Modified PP Drawn Filaments

Samples	Denier of the drawn filament (dtex/f)	Crystallinity (%)	Orientation (%)
Pure PP	0.91	58.81–58.87	86.49–86.53
PP/PS (8.0 wt %)	0.98	38.08–38.13	88.52–88.56
PP/hy-PS (8.0 wt %)	0.95	35.08–35.14	89.09–89.13

#### CONCLUSIONS

Hy-PS (CeO<sub>2</sub>) dispersed uniformly in the PP matrix as spherical particles ranging from 1 to 5  $\mu\text{m}$ . The PP/hy-PS (CeO<sub>2</sub>) blend showed a rheological behavior similar to that of PP. The PP/hy-PS (CeO<sub>2</sub>) blend showed stable spinning processability. These fibers had good drawability, and drawn filaments reached less than 1.0 dtex/f. The fine fibers made of the

**TABLE III**  
**Dyeing Properties of the Modified PP Fabrics**

Sample	Dye (C.I. disperse no.)	Concentration of dye (wt % of fiber)	K/S value	Fastness to soaping (off-color of orient sample)	Fastness to soaping (staining of white fabric)
PP/PS (8.0 wt %)	Red 167	0.1	0.7045	3-4	4-5
PP/PS (8.0 wt %)	Red 167	2.0	2.0039	4	3-4
PP/hy-PS (8.0 wt %)	Red 167	0.1	0.8614	4-5	4-5
PP/hy-PS (8.0 wt %)	Red 167	2.0	2.6123	4-5	4
PP/PS (8.0 wt %)	Orange 30	0.1	0.4157	3	4-5
PP/PS (8.0 wt %)	Orange 30	2.0	1.1284	4	3-4
PP/hy-PS (8.0 wt %)	Orange 30	0.1	0.5841	4	4-5
PP/hy-PS (8.0 wt %)	Orange 30	2.0	1.8241	4-5	4

blend showed improved dyeability, and mechanical performance similar to that of PP filaments.

The PP/hy-PS (CeO<sub>2</sub>) blend had good dyeing effect because of the complexation between disperse dye and certium in the blend system. The changes of degrees of crystallinity and orientation of modified PP fiber resulted in an increase of its amorphous region. It was proven to be an efficient additive for improving the dyeability of fine PP filaments with practical processability and mechanical properties.

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