

Effect of compatibilizer on the dyeability of polypropylene/ polytrimethylene terephthalate polyblend fibres

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Abstract - Polyblend fibres of polypropylene (PP) and polytrimethylene terephthalate (PTT) with maleic anhydride grafted polypropylene (MAPP) as compatibilizer were studied for their dyeability. MAPP content was varied from 1-5% and its role in influencing the dyeability of fibres was studied by investigating the changes in the internal structure of the fibres. For 5% PTT content in the blend fibre, addition of MAPP further enhanced the dyeability in the range of 500-1200%, along with excellent wash and light fastness properties. The compatibility of the polymers in the blends also increased with improvement in tenacity. The increased dyeability is attributed to the reduced crystallinity and increased polarity of fibres due to the presence of maleic anhydride. Although reduced crystallinity led to reduced thermal stability, it was well within the acceptable limits.

Key Words : Polypropylene, dyeability, polytrimethylene terephthalate, maleic anhydride grafted polypropylene

1. INTRODUCTION

The dyeability of polypropylene (PP) fibres is a challenge due to its highly crystalline structure with no polar groups. Different techniques making use of additives, chemical modification of fibre surface like grafting and meltblending of polymers have been employed to impart dyeability [1-3]. However, polymer blending is one of the most effective and economical techniques used to obtain fibres with distinct properties in comparison to those of each blend component. This technique is less complex and economical than the synthesis of new monomers and/or exploring new polymerization processes needed to produce new fibrous polymeric materials. Polypropylene based poly-blend fibres have been made disperse dyeable by blending PP with Polyester [4, 5], Nylons [6], Polystyrene [7], etc.

Among polyester, polytrimethylene terephthalate (PTT) shows excellent dyeability properties with lower melting temperature leading to easier processability of fibres [8]. PP/PTT polyblend fibres have shown good disperse

dyeability with reduced tensile properties [9].

An ideal compatibilizer for PP/PTT blend will react with the carboxylic acid and/or hydroxyl terminal groups of PTT while being miscible with PP. Maleic anhydride grafted polypropylene (MAPP) has been used for improving the compatibility between PP and other polymers such as nylon, polyethylene terephthalate which possess some functional groups [10]. However, the literature indicates that the role of MAPP in improving the compatibility of the polymers in the blend with PP as major polymer component has been studied to a limited extent [7, 11].

Hence, in the present work, systematic study of effect of varying concentration of MAPP in PP/PTT blend on compatibility has been reported. The behaviour of the blend fibres with respect to thermal properties, crystallization behaviour, mechanical properties and dyeability, etc is also investigated.

2. EXPERIMENTAL

2.1 Materials

PP polymer chips Repol H350FG, having MFI 35 were supplied by Reliance Industries Ltd (India). PTT polymer chips (0.93 IV) were supplied by Futura Polymers (India) and MAPP with 0.4% MAH content was procured from Pluss Polymer (India). Disperse dyes Dianix Blue ER (Low Energy), Dianix Yellow S-6E ER (Medium Energy) and Dianix Rubine S-2G 150% (High Energy) were obtained from Dystar India Pvt. Ltd.

2.2 Fibre Spinning

PP, PTT and MAPP were melt blended using a co-rotating twin-screw extruder (APV Baker, UK) keeping barrel temperatures ranging from 210 to 255°C, and the screw speed 60 rpm. The extruded pellets were dried in an oven at 120°C for at least 12 hr before melt-spinning. The melt blended polymer chips were melt spun into fibres using laboratory melt spinning machine (Fair Deal Associates, India). The temperatures of the extruder zones ranged from 180 to 240°C. The filaments extruded from the spinneret were cooled down by blowing cool air in a 1.5 m

quench duct and subsequently guided over godet roller via a metered spin finish passage. These filaments were further passed on to draw rollers where in drawing was carried out by two-stage drawing method at an optimized draw ratio.

2.3 Dyeing of Fibres

The fibres were mild-scoured with 2 g/l of a nonionic detergent at 70°C for 30 min and subsequently rinsed with hot and cold water and then dried in air. The disperse dyeing of the scoured fibres was carried out in a high temperature / high pressure beaker dyeing machine (Rota dyer, India) using a standard method of the dyeing of synthetic fibres. The dyed samples were then subjected to reduction clearing treatment with 2 g/l caustic soda and 2 g/l sodium hydrosulphite for 20 min at 70°C, which was followed by neutralization with 1 g/l acetic acid solution, washing and drying.

2.4 Characterization

The thermal properties and crystallization behaviour were studied using differential scanning calorimetry (Shimadzu, Japan). The measurements were carried under nitrogen atmosphere (flow rate 20ml/min). Crystallization exotherms and melting endotherms were recorded and the degree of crystallinity (X) was calculated from the DSC data using the following relationship:

$$X = \frac{\Delta H_e}{\Delta H_c^0} \times 100$$

where, ΔH_e is experimental heat of fusion, ΔH_c^0 is the heat of fusion for a 100% crystalline polymer. Values of ΔH_c^0 taken from the literature are 145.48 J/g for PTT [12] and 207 J/g for PP [13], respectively.

X-ray diffraction studies were carried out using powder technique on XRD-6100 (Shimadzu, Japan) using $\text{CuK}\alpha$ radiation. Information on crystalline form and percentage crystallinity was obtained from the I-2 θ plots. The crystalline orientation factor (f_c) was calculated by Wilchinsky's method [14] using the azimuthal intensity distributions of the (110) and (040) reflections. The amorphous orientation factor (f_a) was calculated using the Stein-Norris [15] method. The birefringence of fibre were obtained using polarizing microscope fitted with Berek compensator by retardation method [16].

The thermal stability of the samples was studied by carrying out thermogravimetric analysis (TGA) using DTG-60H thermogravimetric analyzer (Shimadzu, Japan). Samples of about 7.5 mg were heated from 50 to 500°C at a heating rate of 20°C/min in a nitrogen atmosphere, and the corresponding degradation levels at various temperatures were noted.

Tensile testing of filaments was measured on Tinius Olsen Tensile testing machine. The gauge length was 100 mm and strain rate was kept at 50 mm/min. Ten samples was

tested and their average values are reported.

The dyed samples were evaluated for colour depth in terms of Kubelka Munk function (K/S), using Spectra Flash SF 300 computer colour-matching system (Datacolour International, USA) [17]. Kubelka Munk function (K/S) is given by the following equation:

$$\frac{K}{S} = \frac{(1 - R)^2}{2R}$$

where, R is reflectance at complete opacity, K is the absorption coefficient and S is scattering coefficient.

Colour fastness to washing was evaluated as per ISO 105-C10:2006 B washing fastness test conditions in a Launder-O-meter for 30 min at 60°C, using 5 g/l non-ionic soap and 2 g/l soda ash at a liquor to material ratio of 50:1.

Colour fastness to light was evaluated as per BS 1006 test method. Dyed samples were placed on cardboard paper, and its half portion was covered by a black sheet of paper and remaining portion was exposed to mercury lamp continuously for 17 hours. The fading of exposed samples was graded with reference to blue wool standards subjected to same testing conditions [18].

3. RESULTS AND DISCUSSION

3.1 Thermal Behaviour of Polyblend Fibres

Results of DSC analysis given in Table 1 indicate that there are two distinct melting points for polyblend fibres, thus exhibiting the presence of PP and PTT while MAPP melting peak submerged with PP melting peak. In case of pure polypropylene, the melting peak was observed at 169.56°C. At 5% PTT additive, the melting peak of PP shifted slightly to lower temperature of 166.66°C. However, melting peak of PTT was not apparent indicating existence of PTT in the amorphous form and/or being uniformly dispersed in the PP matrix. With the addition of compatibilizer MAPP however, PTT peak was observed at 225.59°C for 1%, 226.06°C for 3% and 226.13°C for 5% MAPP. The maleic anhydride is always present in the amorphous zone as it does not take part in chain folding [19,20] and PP-g-MA being miscible in PP, may act as a nucleating agent due to its easier dispersion, favouring the crystallization of PTT within its own domain. Similar behaviour is reported by Wang et al. [21] that the presence of MAPP increased the crystallization rate of PTT, whereas, that of PP component was retarded.

PTT melting peak became increasingly dominant with the addition of MAPP, which was evident from increase in heat of melting. Simultaneously the melting peaks of PP were hindered which was quite evident from decrease in heat of melting. The depression of PP melting point with increasing MAPP content suggests the occurrence of co-crystallization and/or defect insertion or isomorphous crystallization of MAPP and PP in the blends. Duvall et al.[22] also observed the absence of a distinct MAPP

Table 1. The melting behaviour data of polyblend fibres obtained from DSC analysis.

Fiber (PP/PTT/MAPP)	Onset temp (°C)		Endset temp (°C)		Melting peak (°C)		Heat of melting (J/g)		Crystallinity (%)
	PP	PTT	PP	PTT	PP	PTT	PP	PTT	
100/0/0	161.92	-	172.56	-	169.56	-	141.9	-	68.55
95/5/0	156.43	-	169.78	-	166.66	-	124.96	-	60.37
95/5/1	156.59	217.79	170.53	231.52	166.89	225.59	110.88	2.87	55.53
95/5/3	155.42	219.7	170.92	230.18	166.97	226.06	107.96	3.05	54.25
95/5/5	157.64	219.53	171.18	230.31	167.03	226.13	96.88	3.71	49.35

melting point in the blends and that pure MAPP crystalline structures were not detected, due to their small proportion in the blend or due to the fact that they co-crystallize with PP. Thus overall reduced crystallinity or enthalpy loss for polyblend fibres could be attributed to the higher interphase formation by the reaction of MAPP with the amorphous phase of PTT via anhydride/PTT terminal hydroxyl reaction and reduced PP crystallinity.

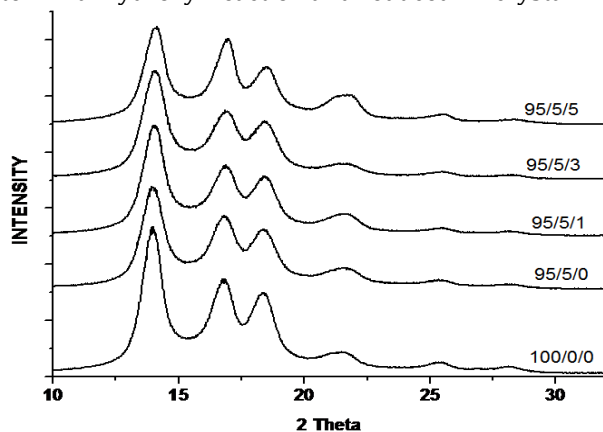


Fig. 1. X-ray diffractogram of polyblend fibres.

3.2 X-Ray Diffraction Analysis

The x-ray diffraction plots of polyblend fibres shown in Fig. 1 exhibit the influence of MAPP on fibre structure formation. PTT and MAPP have led to formation of polyblend fibres with reduced crystallinity thus corroborating the findings of DSC analysis. The presence of maleic anhydride has brought about a slight change in the overall crystallinity, crystalline orientation (f_c) and amorphous orientation (f_a) as observed from Table 2.

Table 2. X-ray crystallinity and orientation of polyblend fibres.

Fibres (PP/PTT/MAPP)	Crystallinity (%)	Orientation	
		Crystalline	Amorphous
100/0/0	62.2	0.94	0.67
95/5/0	55.6	0.93	0.60
95/5/1	55.1	0.93	0.61
95/5/3	54.7	0.92	0.62
95/5/5	54.0	0.92	0.61

Improvement in f_a leads to enhanced tensile properties of fibres [23], thus showcasing the positive influence of MAPP on polyblend fibre properties.

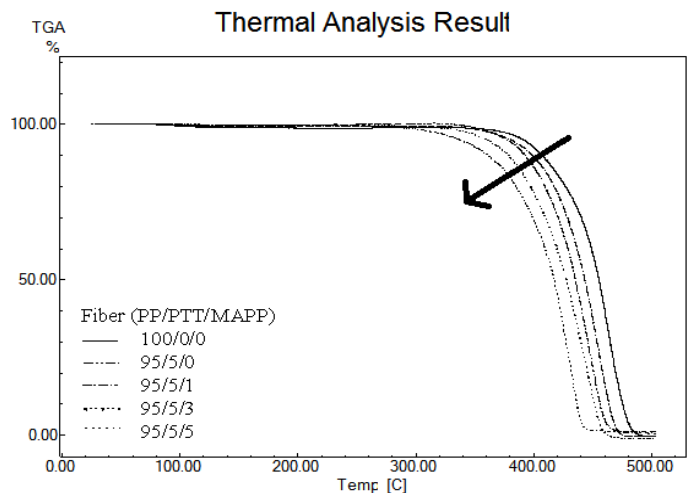


Fig. 2. Thermogravimetric analysis of PP/PTT/MAPP blend fibres.

3.3 Thermal Stability

The effect of the MAPP on the thermal stability of the polyblend fibres was studied by means of thermogravimetric experiments. The temperatures corresponding to 10, 50 and 100% of weight loss obtained by TGA experimental curves are given in Table 3. During thermal degradation, the TGA curves displayed a single step degradation process for all samples as seen in Fig. 2. For the polyblend fibres it is observed that the initial weight loss began at lower temperatures than for corresponding pure PP fibre.

Table 3. TGA derived decomposition temperatures and weight loss

Fibre (PP/PTT/MAPP)	10% wt loss (°C)	50% wt loss (°C)	100% wt loss (°C)
100/0/0	426	465	498
95/5/0	403	446	482
95/5/1	396	444	479
95/5/3	388	432	465
95/5/5	362	416	438

The presence of maleic anhydride further shifted the initial weight loss towards lower temperature, a trend also reported by Bertini et al. [24]. The progressive decrease can be attributed to reduced crystallinity which is evident from the DSC study of the blend fibres. However, the blend fibres are found to be stable upto 360°C and their rapid degradation started beyond 400°C only, which is much higher than the regular processing temperature of the fibres.

Table 4. The effect of PTT content on tensile properties of PP/PTT blend fibres

Fibre (PP/PTT/MAPP)	Tenacity (gf/d)	Peak elongation (%)	Tenacity loss (%)
100/0/0	5.5	14.2	--
95/5/0	4.3	15.5	22.6
95/5/1	4.4	15.1	20.2
95/5/3	4.8	14.8	12.5
95/5/5	4.6	15.3	16.9

3.4 Tensile Properties

Results in Table 4 indicate that tenacity for polyblend fibres was lower than that of pure PP fibres, which was obvious due to its highest crystallinity. For 5% PTT in the polymer blend, the tenacity was found to be decreasing whereas elongation slightly increased which is obviously due to immiscible blends formation with distinct decrease in the crystallinity. The addition of compatibilizer MAPP had a positive impact since tenacity of the fibres increased with MAPP addition, a behaviour exhibited probably due to the increased compatibility between the two polymer phases by formation of chemical bonds and improvement in the internal (structural) unevenness of the PP/PTT blends with MAPP favouring uniform dispersion and size of PTT phase as observed by different researchers [25-27] for polyester/PP blends too.

Table 5. K/S values of disperse dyed polyblend fibres (shade 3%).

Fibre (PP/PTT/MAPP)	Dianix Blue ER	Dianix Yellow S-6E ER	Dianix Rubine S-2G 150%
100/0/0	1.6768 (100)	0.7392 (100)	2.4353 (100)
95/5/0	21.8629 (1203.8)	13.5902 (1738.5)	16.4481 (575.4)
95/5/1	26.9228 (1505.6)	15.9754 (2061.2)	23.1416 (850.3)
95/5/3	28.5852 (1604.7)	18.7288 (2433.7)	27.4034 (1025.3)
95/5/5	25.7246 (1434.2)	17.7119 (2296.1)	25.6476 (953.2)

^a Values in bracket represent percentage increase in K/S values over that of pure PP.

The maximum tenacity was observed for 3% MAPP and further addition i.e. 5% MAPP led to decrease in tenacity and increase in elongation at break. This behaviour can be attributed to unreacted MAPP acting as a plasticizer due to its amorphous nature.

3.5 Dyeability

The results relating to disperse dyeability of the blend fibres for 3% shade given in Table 5, indicate that initial very low K/S values showing mere tinting of 100% PP fibre, obviously due to its highly crystalline structure, were found to increase by more than 500 to 1200% when PP was substituted by 5% PTT.

The increase in dyeability can be attributed to reduced fibre crystallinity due to introduction of aromatic fibrous polymers PTT and formation of interfaces between the phases which lead to increased surfaces for dye adsorption. On subsequent addition of MAPP from 1 to 3% there was significant further increase in K/S values, to the order of 1200 to 2500%. This may be attributed to the presence of maleic anhydride in the amorphous zone which induces polarity and increases the interactions between the fibre and dye resulting in higher K/S values and fastness property [7]. Both of these changes in internal structure of the PP/PTT/MAPP blend thus resulted in enormous increase in dyeability in general from 575 to 2400% for all the three disperse dyes studied.

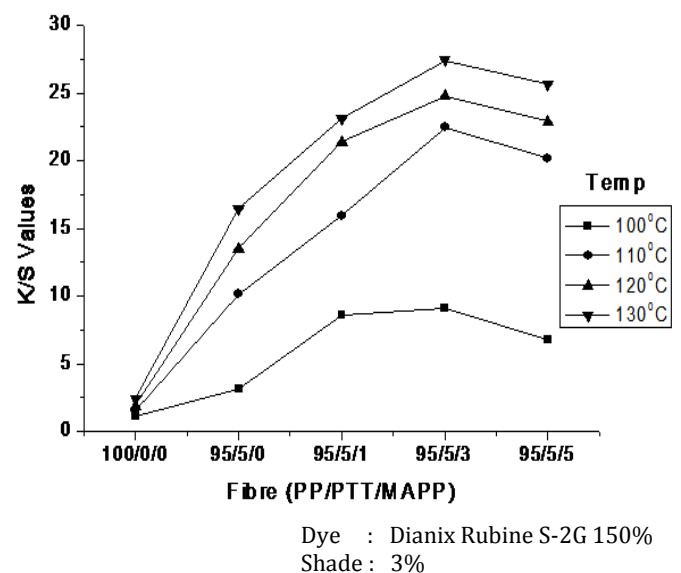


Fig. 3. Effect of dyeing temperature on K/S values of polyblend fibres.

The K/S values increased with the addition of MAPP and 3% MAPP addition in PP/PTT blend found to give optimum K/S values for all the three dyes studied. Any further addition i.e. 5% MAPP led to slightly reduced colour values although this blend fibre exhibited lowest crystallinity values. The possible reason for this behaviour

Table 6. Light and washing fastness properties of disperse dyed PP/PTT blend fibres (shade 3%).

Fibre (PP/PTT/MAPP)	Light Fastness			Washing Fastness		
	Dianix Blue ER	Dianix Yellow S-6E ER	Dianix Rubine S-2G 150%	Dianix Blue ER	Dianix Yellow S-6E ER	Dianix Rubine S-2G 150%
100/0/0	2	2	2	1	1	1
95/5/0	4	4	4	4	4	4
95/5/1	5	5	5	4	4	4
95/5/3	5	6	6	4	4	4
95/5/5	4	5	5	3	3	3

may be attributed to the physical structure formed of the blend fibres which does not permit higher dye build up further possibly due to dye saturation or its inability to retain dye molecules, and there could be desorption of the dye.

In order to further study the dyeing behaviour of melt blend fibres with MAPP addition, the dyeability of polyblend fibres was studied with respect to dyeing temperature and shade (%) dyed. The results in Fig. 3 show that the K/S values increased with increase in dyeing temperature, from 100 to 130°C for 3% shade. This may be attributed to corresponding increase in accessible volume available for dye diffusion in thermoplastic blend fibres as the temperature of dyeings increases [28]. However, in this case too, the K/S values were lower for 5% MAPP addition as compared to 3% MAPP. This once again confirms the possibility of desorption occurring in case of 5% MAPP addition in blend, reducing the capacity of internal fibre structure in retaining the dye molecules.

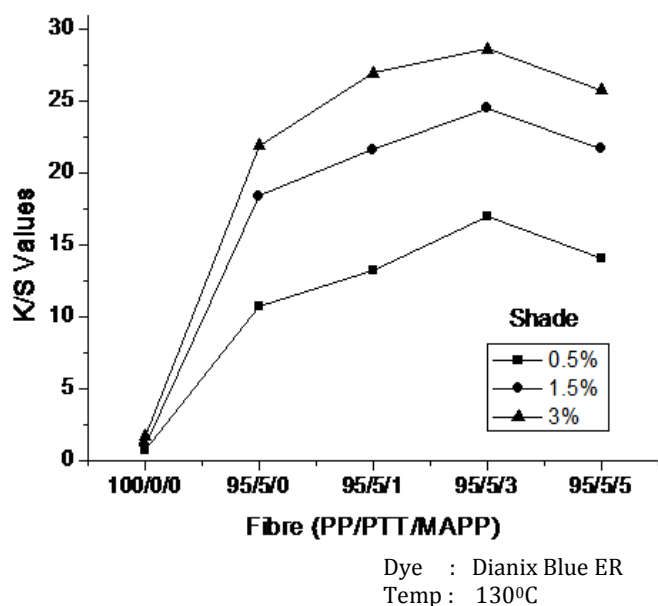


Fig. 4. Effect of dyeing shade % on K/S values of polyblend fibres.

Dye : Dianix Blue ER
Temp : 130°C

The results in Fig. 4 show that the trend of increase in the K/S values remained same even when shade (%) dyed varied from 0.5 to 3%. It is obvious that higher was the shade (%) dyed, higher would be the K/S values. This may be attributed to the fact that increased number of dye molecules are present in the solution to diffuse into the fibre surface, at higher shade. It is to be noted that even at lower shade (%), 3% MAPP addition gave optimum K/S values, higher than that for 5% addition. Hence, the reduced K/S values were not due to dye saturation since plateau region was not observed, but may be due to desorption or due to the inability of the physical structure of the fibre to retain the adsorbed dye molecules completely.

3.6 Colour Fastness

The results with respect to the fastness properties of the dyed fibres given in Table 5, indicate that the enormous increase in dyeability of modified fibres was also accompanied with very good (grade 4) colour fastness to washing. In case of the light fastness too, a maximum rating of 6 i.e. good was achieved for the polyblend fibres. However, it is to be noted that 5% MAPP containing polyblend fibre showed slightly inferior wash fastness properties as compared to 3% MAPP polyblend which can be attributed to more open fibre structure of the former as compared to 95/5/3 polyblend making it difficult to retain the dye molecules within fibre structure when subjected to wash fastness tests. The slight inferior light fastness may be due to the higher accessibility of the blend fibre to light radiation and lower amount of dye on the fibre due to lower K/S values.

4. CONCLUSION

A significant increase in dyeability of hitherto almost undyeable PP could be achieved by substituting it with 5% PTT. This is attributed to the reduced crystallinity and increased interphase, evident from fibre structure analysis. Addition of MAPP, not only improved processability but also further accelerated the enhancement in dye uptake and at 3% MAPP addition, the increase in dyeability reached to as high as 1000 to 2400%, with very good wash and light fastness properties. Thermal analysis of blend fibres showed that the addition of MAPP further reduced the crystallinity of PP component

and favoured PTT crystallization thus forming fibres with reduced crystallinity and open structure. Hence, the thermal stability of the fibres slightly decreased with MAPP addition, although it was well within the acceptable limits.

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