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Effect of maleic anhydride grafted polypropylene compatiblizer in enhancing the properties of Polypropylene/ Poly lactic acid polyblend fibres.

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***Abstract*— In the present work, the effect of maleic anhydride grafted polypropylene (MAPP) as compatibilizer on dyeability properties of polypropylene (PP) and polylactic acid (PLA) polyblend fibres is studied. The PLA content in the polyblend fibres were kept constant at 5% and 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% PLA content in the blend fibre, addition of MAPP further enhanced the dyeability in the range of 16-23%, along with good wash and light fastness properties. The compatibility between the polymer blend 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.**

***Keywords*— Polypropylene, polylactic acid, maleic anhydride grafted polypropylene, dyeability.**

I. INTRODUCTION

Polypropylene (PP) fibres have been made disperse dyeable by using meltblending technique to form polyblend fibres [1-3]. Polymer blending is an effective and economical techniques used to obtain fibres with distinct properties in comparison to those of each blend component. This technique is less complex in comparison to synthesis of new monomers and/or new polymerization processes needed to produce new fibrous polymeric materials. PP based poly-blend fibres have been made disperse dyeable by blending PP with Polyester [4, 5], Nylons [6], Polystyrene [7], etc.

Among polyesters, polylactic acid (PLA) is a biopolymer having the same processing temperature range as that of polypropylene. PP:PLA polyblend fibres formed using meltblending technique [8-11] and bicomponent fibre spinning [12] have been reported. However, PP:PLA polyblend fibres formed are incompatible and efforts to improve compatibility using polymeric compatiblizer needs to be studied. An ideal compatibilizer for PP:PLA blend will react with the carboxylic acid and/or hydroxyl terminal groups of PLA 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 [13]. 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, 14]. A dedicated study to assess the effect of MAPP in improving the compatibility of PP:PLA polyblend fibres has not yet been carried out. Thus, in the present work, systematic study on effect of varying concentration of MAPP such as 1, 3 & 5% having 0.4% maleic anhydride content as shown in Table 1 for polyblend fibres has been reported.

Table 1. PP:PLA:MAPP polyblend fibres denotation and their compositions

Sr. No.	Denotations (PP:PLA:MAPP)	Composition (% by weight)		
		PP	PLA	MAPP
1	95:5:0	95	5	0
2	95:5:1	95	5	1
3	95:5:3	95	5	3
4	95:5:5	95	5	5

II. MATERIALS AND METHODS

A. Materials

PP polymer chips (Repol H350FG), having MFI 35 were supplied by Reliance Industries Ltd (India). PLA polymer chips (Ingeo biopolymer 6201D), having MFI 30 were supplied by NatureWorks LLC, USA 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.

B. Fibre spinning

PP, PLA and MAPP were melt compounded in a co-rotating twin-screw extruder (APV Baker, UK) keeping barrel temperatures 15 to 20^{0}C above melting temperature of polyblend and the screw speed 60 rpm. The extruded pellets were pre-dried in an oven at 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 220^{0}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 process.

C. Dyeing of fibres

The fibres were mild-scoured with 2 g/l of a nonionic detergent at 70^{0}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^{0}C, which was followed by neutralization with 1 g/l acetic acid solution, washing and drying.

D. Characterization

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) [15]. 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^{0}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 [16].

X-ray diffraction studies were carried out using powder technique on XRD-6100 (Shimadzu, Japan) using CuKα radiation. Information on crystalline form and percentage crystallinity was obtained from the I-2θ plots. The crystalline orientation factor (f_c) was calculated by Wilchinsky's method [17] using the azimuthal intensity distributions of the (110) and (040) reflections. The amorphous orientation factor (f_a) was calculated using the Stein-Norris [18] method.

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^0_c is the heat of fusion for a 100% crystalline polymer. Values of ΔH^0_c taken from the literature are 93 J/g for PLA and 207 J/g for PP, respectively.

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^{0}C at a heating rate of 20^{0}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.

III. RESULTS AND DISCUSSION

A. Dyeability

The effect of MAPP on disperse dyeability of polyblend fibres for 3% shade is given in Table 2.

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Table 2. K/S values of disperse dyed polyblend fibres (shade 3%).

Fibre (PP:PLA: MAPP)	Disperse dyeing		
	Dianix Yellow S-6E ER	Dianix Rubine S-2G 150%	Dianix Blue ER
95:5:0	7.4675 (--)	10.5124 (--)	11.907 (--)
95:5:1	8.128 (8.85)	11.1512 (6.08)	12.8452 (7.9)
95:5:3	8.9324 (19.62)	11.9873 (14.03)	13.5392 (13.705)
95:5:5	9.1872 (23.03)	12.3728 (17.7)	13.8925 (16.67)

[a] Values in bracket represent percentage increase in K/S values.

With the addition of MAPP there is a slight increase in the disperse dyeability of the fibres in the range of 16-23% for all the three disperse dyes studied. The presence of maleic anhydride has brought about changes in the internal fibre structure thus increasing the dye-uptake resulting in higher K/S values. However, due to addition of compatiblizer, ease of processing has been greatly improved, which is discussed further.

The fastness properties of the disperse dyed polyblend fibres given in Table 3, indicate that the disperse dyed polyblend fibres gave very good (grade 4) colour fastness to washing. In case of the light fastness too, a maximum rating of 5 i.e. good was achieved for the polyblend fibres.

Table 3. Wash and light fastness properties of disperse dyed polyblend fibres.

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

B. 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. PLA and MAPP have led to formation of polyblend fibres with overall reduced crystallinity thus increasing the surface area for dye diffusion. The presence of maleic anhydride has changed the overall crystallinity, crystalline orientation and amorphous orientation as observed from Table 4. The I-2θ plots of the polyblend fibres show that increasing amounts of MAPP results in gradual decrease in the intensity of X-ray diffraction peaks, thus suggesting an increase in the amorphous content making polyblend fibres more accessible to dyes.

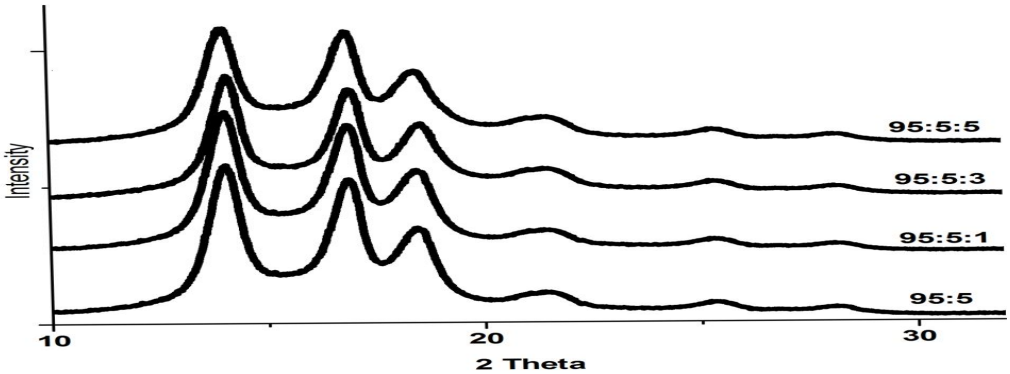

Fig.1. X-ray diffractogram of PP:PLA:MAPP polyblend fibres.

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The overall orientation of the polyblend fibre was affected by the presence of MAPP. The crystalline orientation slightly changed from 0.90 to 0.89 for MAPP addition. The amorphous orientation of the fibres increased from 0.59 to 0.61 for 3% addition of MAPP. The increase in amorphous orientation shows that the amorphous region have become more ordered and slight uniformity introduced in the dispersed phase of PLA. This improvement in amorphous orientation supports the compatiblization effect of MAPP [19]. Thus the addition of MAPP has improved the compatibility in the polyblend fibres formed.

Table 4. X-ray crystallinity and orientation of polyblend fibres.

Fibres (PP:PLA:MAPP)	Crystallinity (%)	Orientation	
		Crystalline	Amorphous
95:5:0	54.15	0.90	0.59
95:5:1	53.49	0.89	0.60
95:5:3	52.30	0.89	0.61
95:5:5	50.45	0.89	0.60

C. Thermal behavior of polyblend fibres

Results of DSC analysis given in Table 5 indicate that only one distinct melting point for polyblend fibre is observed, thus indicating that PLA and MAPP melting peaks have submerged with PP melting peak. With the addition of compatibilizer MAPP, the overall crystallinity of the fibres reduced, thus corroborating the findings of XRD analysis. The overall reduced crystallinity for polyblend fibres could be attributed to the higher interphase region formation with MAPP reacting with the amorphous phase of PLA via anhydride/PLA terminal hydroxyl reaction.

Table 5. The melting behaviour data of polyblend fibres obtained from DSC analysis.

Fibre (PP:PLA:MAPP)	Onset temp (^{0}C)	Endset temp (^{0}C)	Melting peak (^{0}C)	Heat of melting (J/g)	Crystallinity (%)
95:5:0	159.16	170.38	165.36	116.28	55.64
95:5:1	160.02	171.48	166.42	113.13	54.13
95:5:3	160.84	172.3	166.96	112.72	53.93
95:5:5	161.79	172.94	168.19	109.01	52.16

D. Thermal stability

The effect of the MAPP on the thermal stability of the polyblend fibres is shown in Fig. 2 and the temperatures corresponding to 10, 50 and 100% of weight loss obtained by TGA experimental curves are given in Table 6. During thermal degradation, the TGA curves displayed a single step degradation process for all polyblend fibres. The presence of maleic anhydride has shifted the initial weight loss towards lower temperature, a trend also reported by Bertini et. al. [20]. The progressive decrease can be attributed to reduced crystallinity which is evident from the XRD and DSC study of the polyblend fibres. However, the blend fibres are found to be quite stable upto 350^{0}C and their rapid degradation started beyond 380^{0}C only, which is much higher than the regular processing temperature of the fibres.

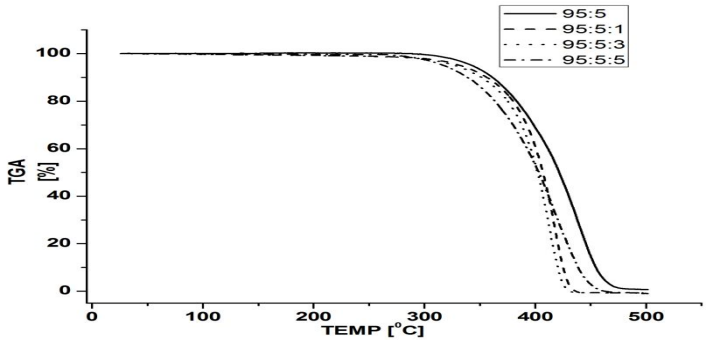

Fig. 2. Thermogravimetric analysis of PP:PLA polyblend fibres.

Table 6. TGA derived decomposition temperatures and weight loss

Fibres (PP:PLA:MAPP)	10% wt loss (^{0}C)	50% wt loss (^{0}C)	100% wt loss (^{0}C)
95:5:0	381	420	461
95:5:1	375	408	438
95:5:3	364	395	432
95:5:5	358	383	444

E. Tensile properties

Results in Table 7 showcase the effect of MAPP on the tensile properties of polyblend fibres. The presence of compatibilizer MAPP has a positive impact on tensile properties, as the tenacity of the fibres increased with MAPP addition though to a limited extent. The enhanced tensile properties is due to the increased compatibility between the PP:PLA polyblend phases. The increase in compatibility is due to formation of chemical bonds and improvement in the uniform internal structural distribution. The maximum increase in tenacity was observed for 3% MAPP and further addition i.e. 5% MAPP led to decrease which could be due to unreacted MAPP acting as a plasticizer.

Table 7. Tensile properties of polyblend fibres

Fibres (PP:PLA: MAPP)	Tenacity (gf/d)	Peak Elongation (%)	Strength Gain (%)
95:5:0	4.08	16.92	-
95:5:1	4.15	17.14	1.72
95:5:3	4.28	17.26	4.9
95:5:5	4.25	17.43	4.16

IV. CONCLUSION

A significant changes in the performance properties of PP:PLA polyblend fibres could be achieved with the addition of MAPP. Addition of MAPP, improved the processing of the fibres with further increase in dye-uptake in the range of 16-23%, with very good fastness ratings. XRD structural analysis showed that for 3% MAPP addition, better amorphous orientation of the fibres is achieved, thus resulting in better tensile properties of the fibres. Thus, the addition of MAPP has led to formation of PP:PLA:MAPP polyblend fibres with slightly reduced crystallinity and increased interphase region with more open structure conducive for its disperse dyeability.

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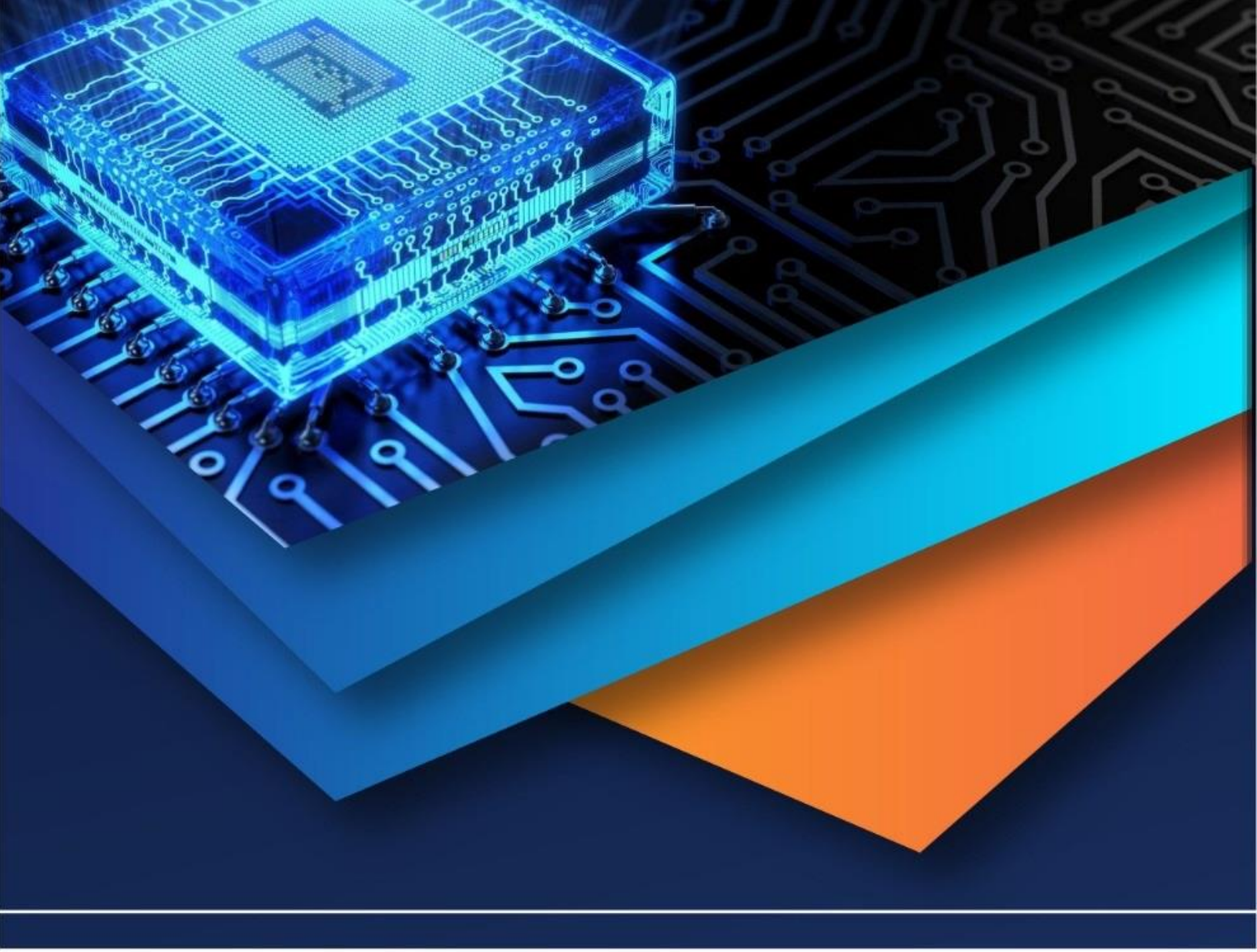

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