Dyeing of Polypropylene Blends by Using Microwave Energy

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ABSTRACT

In this work, C.I. Disperse Blue 79 was used to dye 100 % isotactic polypropylene (iPP) fibers, the most widely used fiber in the industry, 100 % linear low density polyethylene (LLDPE) polymer fibers and ternary polymer blends obtained from four different proportions of these polymer fibers with an elastomer of ethylene-vinyl acetate (EVA) in the absence and presence of microwave. The samples were tested for color measurement, color fastness, mechanical and morphological properties. The results of the investigation show that addition of up to 15% EVA in ternary polymer blends results in good color and mechanical properties for both dyeing methods in both the absence and presence of microwave energy. The SEM images indicate there is no porosity on the surfaces of the iPP/LLDPE/EVA ternary polymer blends dyed in the presence of microwave energy. Using microwave energy for dyeing provided a saving of almost 90% of the time normally required and thus a significant amount of energy.

Keywords: polypropylene; ternary polymer blends; ethylene vinyl acetate elastomer; microwave energy

INTRODUCTION

Microwave energy is an alternative heating method that is used widely for pre-treatment, dyeing, finishing, drying, fixation, sanitization, surface modification, and grafting of textile materials. Compared to conventional heating methods, it provides fast, effective, uniform, and energy efficient heating because of microwave energy absorption in textile materials.

The dyeing of synthetic fibers such as polyester, acrylic, polyamide, polypropylene and poly (butylene terephthalate) has been studied in the presence of microwave energy; and results were compared with conventional methods. The direct dyeing of cotton and reactive dyeing of flax with microwave energy have also been investigated. The use of microwave heating enhanced the hydrophilicity, dyeability, and color fastness properties of the fibers, and caused acceptable color differences after repeated dyeing ($\Delta E^* < 1$ CIELab unit) [1-4].

Polypropylene fibers are increasingly being used in industry because they are inexpensive, light, and have good thermal stability [5]. Polypropylene, with its aliphatic and stereoregular polymer structure, is a hydrophobic and highly crystalline fiber [6]. Limited investigations have been performed on dyeing of polypropylene fibers. A masterbatch method is normally employed for coloration of these fibers. The cost of masterbatch method is quite high and it is difficult to obtain medium and dark colors [7]. To improve the dyeability and to reduce the problems associated with dyeing polypropylene fiber, polypropylene can be blended with other polymers or additives [8-19], or graft copolymerization can be used to attach dye accepting functional groups [20]. Alternatively, polypropylene fibers can be treated with gamma radiation [21] and plasma technology [22] to improve coloration. In industry, a 2-vinylpyridine-co-styrene additive is used to improve the dyeability of polypropylene fibers and produce adequate color values [23]. Polymer blends increase the function and variation of the fibers. Fiber blends can be prepared to improve the dyeability of fibers. These include polypropylene / polyethyleneterephthalate, polyamide/polyamide 6, polypropylene / polyamide 6, and polypropylene / polyethylene [24-36]. Polypropylene (PP) / linear low-density polyethylene (LLDPE) blends can be used for technical, home and medical textiles. The melting point of LLDPE is low and it is compatible with many of the higher temperature polymers used for textiles [37].

In this work, 100% isotactic polypropylene (iPP) fibers, 100% linear low-density polyethylene (LLDPE) fibers, and ternary polymer blends (obtained from four different proportions of these polymer fibers with an elastomer of ethylene-vinyl acetate (EVA)) were dyed with C.I. Disperse Blue 79 by using conventional methods and microwave

energy. Mechanical, morphological, light fastness and color properties of the fibers are characterized.

MATERIALS AND METHODS

Production of Ternary Polymer Blends

This work investigated the dyeability of fibers produced from 100% isotactic polypropylene (iPP), 100% linear low density polyethylene (LLDPE) and ternary polymer blends in four different proportions of these polymers with an elastomer of ethylene-vinyl acetate (EVA). The ternary polymer blends had iPP/LLDPE/EVA ratios of 80/19/1, 70/25/5, 60/30/10, and 50/35/15. Granule samples of polymer blends were prepared using a twin-screw extruder at a screw speed of 170 rpm, temperature range of 170-245˚C, and head pressure of 21 bars. The granular materials were then used to melt spin fibers using the conditions summarized in *Table II.* Melt flow index (MFI), density, tensile strength, and elongation values of the samples are given in *Table I.* MFI values of iPP/LLDPE/EVA ternary polymer blends were obtained from melt indexer at 230°C and 2160g load. Tensile test specimens were prepared from the fibers.

TABLE I. Material properties.

	MFI (g/10min)	Density (g/cm^3)	Tensile Strength (MPa)	Elongation (%)
iPP	35	0.90	32	10
LLDPE	50	0.92	12.4	150
EVA	35-45	0.95	11	700-1000

TABLE II. Production condition of ternary polymer blends.

Parameter	Value
Temperature	180-200 °C
Extruder pressure	50 bar
Pump rotation	80 rev/min
Spinning ratio	1:3
Velocity of first spinning mill	300 rev/min
Velocity of second spinning mill	900 rev/min
Winging speed	183.7 m/min
Linear density of single fiber	20 dtex
Number of spinneret	40x2
Diameter of spinneret	0.4 mm

Sarapol 349 U *(CHT)* was used as a carrier and C.I. Disperse Blue 79 (monoazo) was used for the sample dyeing. Perlavin OSV *(Dr. Petry)*, a non-ionic surfactant and sodium carbonate *(Merck)* were used to wash the dyed samples.

Dyeing Methods

All dyeing processes were carried out under atmospheric conditions using conventional and microwave heating with Disperse Blue 79. Fiber samples weighing 1.5g were used for each dyeing. Dye was applied at 1% owf (on the weight of fiber) to obtain the desired depth of shade. Carrier was added to the dye bath at a concentration of 1 g/L with a liquor ratio of 50:1. Dyeing processes, which were carried out with conventional heating were initiated at 30°C. The dye bath temperature was than increased to 98°C at a heating rate of 2°C/min and this temperature was maintained for 45 minutes. Finally, the bath was cooled and the dyed fiber samples were removed. The dyed samples were washed and then rinsed with cold water. During the washing process, Perlavin OSV at 2 g/Land 0.5 g/L sodium carbonate were added to the washing bath; and washing was carried out at 75°C for 15 minutes with a liquor ratio of 25:1. The total dyeing process took 79 minutes in the absence of microwave, including the time for heating the dyebath.

The dyeing processes in the presence of microwave were carried out in a microwave oven (White Westinghouse, model KM06VF2W), with a maximum output power of 700 W and operating at 2450 MHz. The time-temperature diagram of the dyeing process in the presence of microwave is given at Figure 1. The dyebath (in a glass beaker) was placed into the microwave oven and processed at 30°C. The energy level of the oven was adjusted to medium level (460 W). The temperature was raised and kept at 98°C for 3 minutes. Then, the energy level of the oven was shifted to a lower level (120 W) for 5 minutes. The dyebath removed from oven and cooled to 60°C using ambient conditions. The cooled sample was removed from the dyebath, washed as described previously and then rinsed with cold water. The total processing time, including heating the dyebath, was 8 minutes for dyeing in the presence of microwave.

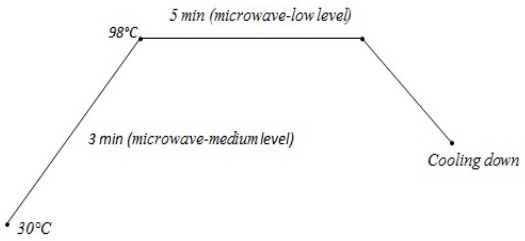


FIGURE 1. The diagram of dyeing method in presence of microwave.

Equipment and Standards Used for the Tests

The reflectance values of dyed samples were measured using Datacolor SF600+ spectrophotometer with specular included mode and LAV (6.6 mm) viewing aperture. The color (CIELab) values of these samples were calculated using D65 illuminant and 10° standard observer values. At the maximum absorption wavelength (λ_{max}) for dye, the K/S (color

strength) values of dyed samples were calculated by using the Kubelka-Munk equation. The CIELab 1976 Color Differences Formula was used to find and express the color differences. For fastness tests, the Light Fastness Tester *(James H.Heal)*, the Wash Fastness Tester [Gyrowash] *(James H.Heal)* and the Rubbing Fastness Tester [Crockmeter] *(James H.Heal)* were used.

The light, washing and rubbing fastness tests were carried out in accordance with the methods described in ISO 105-B02, ISO 105-C06 (A1S test conditions; 40 °C temperature, 30 min, and 10 steel balls), and ISO 105 X12, respectively. The dyed samples were exposed to the light for 100 hours.

The tensile strength tests of the samples were carried out using an Instron 4411 testing machine according to the ASTM D 2256 standard (10 mm/min speed).

The morphological properties of the samples were analyzed with a scanning electron microscope (JEOL JSM-T330), which was operated at 5 and 10 kV, according to TS EN ISO 9220 standard. The samples were coated with gold in order to increase their conductivity.

RESULTS AND DISCUSSION

Color Measurement Results of Dyed Samples

The color measurement results of dyed samples in absence and presence of microwave are given in *Table III* and *Figure 2*. The data in *Table III* and *IV* show that dyed samples in the presence of microwave are darker than the dyed samples in the absence of microwave. The dyed 100% iPP fiber in the presence of microwave is slightly less green (Δa^*=-0.01), more blue (Δb^*=-2.84) and saturated (ΔC^*=2.81) than the dyed samples in the absence of microwave. The K/S values are 0.45 and 0.71 for dyed samples in absence and presence of microwave respectively.

The 100% LLDPE fiber is greener (Δa^*=-1.87), more blue (Δb^*=-3.86) and saturated (ΔC^*=3.78) than the dyed samples in the absence of microwave. The K/S values are 0.52 and 0.69 for dyed samples in absence and presence of microwave respectively.

80/19/1% iPP/LLDPE/EVA ternary polymer blends are greener (Δa^*=-1.75), more blue (Δb^*=-8.67) and saturated (ΔC^*=8.83) than the dyed samples in the absence of microwave. The K/S values are 1.23 and 2.10 for dyed samples in absence and presence of microwave respectively.

70/25/5% iPP/LLDPE/EVA ternary polymer blends are less green (Δa^*=0.47), slightly less blue (Δb^*=0.68) and slightly less saturated (ΔC^*=-0.71) than the dyed samples in the absence of microwave. The K/S values are 5.21 and 6.28 for dyed samples in absence and presence of microwave respectively.

60/30/10% iPP/LLDPE/EVA ternary polymer blends are slightly greener (Δa^*=-0.03), slightly less blue (Δb^*=0.34) and slightly less saturated (ΔC^*=-0.34) than the dyed samples in the absence of microwave. The K/S values are 9.97 and 11.42 for dyed samples in absence and presence of microwave respectively.

50/35/15% iPP/LLDPE/EVA ternary polymer blends are slightly redder (Δa^*=0.01), less blue (Δb^*=3.28) and less saturated (ΔC^*=-3.28) than the dyed samples in the absence of microwave. The K/S values are 10.31 and 12.11 for dyed samples in absence and presence of microwave respectively.

TABLE III. CIELab values of the dyed samples.

Dyeing	Sample	L^*	a^*	b^*	C^*	h°
Absence of Microwave	iPP**	72.29	-1.36	-7.83	7.95	260.15
	LLDPE**	71.65	-1.24	-8.02	8.12	261.21
	80/19/1***	62.13	-1.37	-10.67	10.76	262.68
	70/25/5***	43.53	-2.03	-25.66	25.74	265.48
	60/30/10***	34.86	-0.54	-26.37	26.38	268.83
	50/35/15***	33.59	0.29	-27.19	27.19	270.61
Presence of Microwave	iPP**	67.13	-1.37	-10.67	10.76	262.68
	LLDPE**	68.01	-3.11	-11.48	11.89	254.84
	80/19/1***	54.88	-3.12	-19.34	19.59	260.84
	70/25/5***	40.41	-1.56	-24.98	25.03	266.43
	60/30/10***	31.73	-0.57	-26.03	26.04	268.75
	50/35/15***	33.05	0.3	-23.91	23.91	270.72

** 100%
*** iPP/LLDPE/EVA

TABLE IV. CIELab color differences of the dyed samples[a].

Samples	ΔL^*	Δa^*	Δb^*	ΔC^*	ΔH^*	ΔE^*
iPP**	-5.16	-0.01	-2.84	2.81	0.41	5.89
LLDPE**	-3.64	-1.87	-3.46	3.78	1.09	5.36
80/19/1***	-7.25	-1.75	-8.67	8.83	0.47	11.44
70/25/5***	-3.12	0.47	0.68	-0.71	0.42	3.23
60/30/10***	-3.13	-0.03	0.34	-0.34	0.04	3.15
50/35/15***	-0.54	0.01	3.28	-3.28	0.05	3.32

** 100%
*** iPP/LLDPE/EVA;
[a] Dyed samples in the absence of microwave were taken as "standard".

For each dyeing method, the K/S values were increased with increasing blend rate of EVA with iPP and LLDPE ternary polymer blends; but somehow for higher proportions of EVA, more than 15%, the structure of the polymer blends deteriorated and fiber spinning became impossible.

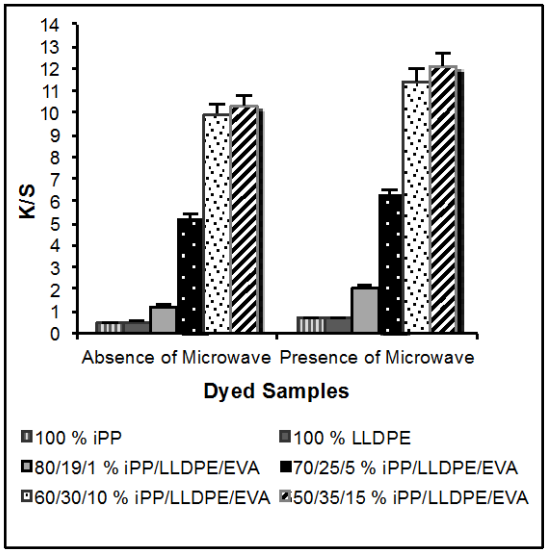


FIGURE 2. K/S values of dyed samples at the maximum absorption wavelengths (600 nm).

Color Fastness Test Results of Dyed Samples

The light, washing, and rubbing fastness test results are summarized in *Table V*. The light fastness of the samples dyed in the presence of microwave (5) was quite good when compared with those samples dyed in the absence of microwave (3). It was observed that light fastness values were not affected by fiber composition. The color change (CC) values of all samples after the washing fastness tests were 5 and the staining test results of adjusted multiphasers were 3-5 greyscale ratings. The color fastness to rubbing of all fiber samples dyed in the absence and presence of microwave were quite good with levels of 4 and 4-5 respectively, for dry and wet rubbing. In a previous study, poly(butyleneterephthalate) fabric dyed in the presence of microwave was compared with conventionally dyed fabric based on color and washing fastness, and similar results have been reported. [1].

TABLE V. Color fastness results of dyed samples.

Dyeing	Sample	Washing fastness [b]							Light fastness	Rubbing fastness	
		CC	CA	Co	PA	PES	PAN	Wo		Dry	Wet
Absence of Microwave	iPP**	5	4	4-5	4	3-4	5	4-5	3	4	4-5
	LLDPE**	5	4	4-5	4	3-4	5	4-5	3	4	4-5
	80/19/1***	5	4	4-5	4	3-4	5	4-5	3	4	4-5
	70/25/5***	5	4	4-5	4	3-4	5	4-5	3	4	4-5
	60/30/10***	5	4	4-5	4	3-4	5	4-5	3	4	4-5
	50/35/15***	5	4	4-5	4	3-4	5	4-5	3	4	4-5
Presence of Microwave	iPP**	5	4	4-5	4	3-4	5	4-5	5	4	4-5
	LLDPE**	5	4	4-5	4	3-4	5	4-5	5	4	4-5
	80/19/1***	5	4	4-5	4	3-4	5	4-5	5	4	4-5
	70/25/5***	5	4	4-5	4	3-4	5	4-5	5	4	4-5
	60/30/10***	5	4	4-5	4	3-4	5	4-5	5	4	4-5
	50/35/15***	5	4	4-5	4	3-4	5	4-5	5	4	4-5

** 100%; *** iPP/LLDPE/EVA; [b] CC, color change; CA, cellulose acetate; Co, cotton; PA, polyamide (nylon 6.6); PES, polyester (Terylene); PA, polyamide (Nylon 6.6); PAN, acrylic (Courtelle); Wo, wool.

Mechanical Properties of Samples

The tensile strength, elongation, and Young's modulus values of the fibers, obtained from the samples before and after dyeing, are given in *Figures 3-5* respectively. The dyeing processes generally reduced these properties. The tensile strength, elongation, and Young's modulus values of the fibers dyed in the presence of microwave were higher than the values of the samples dyed in the absence of microwave. For most fiber compositions, the difference in tensile strength and Young's modulus between the undyed fibers and dyed fibers in the presence of microwave radiation were not significant. Based on these results, it is reasonable to conclude that there have been minimal changes in the molecular chain structure of the polymers processed in the presence of microwave due to the shorter process time.

The results show that increasing the EVA content increased the tensile strength, elongation, and Young's modulus values of the ternary polymer blends. This is due to increased orientation of the molecular chains during the fiber spinning and resulting increases in crystalline structure of the fibers. Other studies have shown that increased proportion of EVA elastomer in similar ternary polymer blends and also acetate groups in the structure of EVA have been effective in increasing fiber tensile strength properties [38].

The results demonstrate that iPP and LLDPE polymer blends with addition of EVA at levels as high as 15% can be used as industrial polymers.

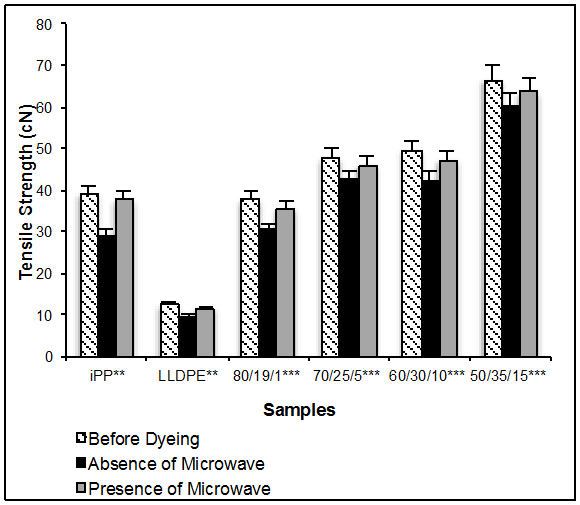


FIGURE 3. Tensile strength (cN) values of samples before and after dyeing (** 100%; *** iPP/LLDPE/EVA).

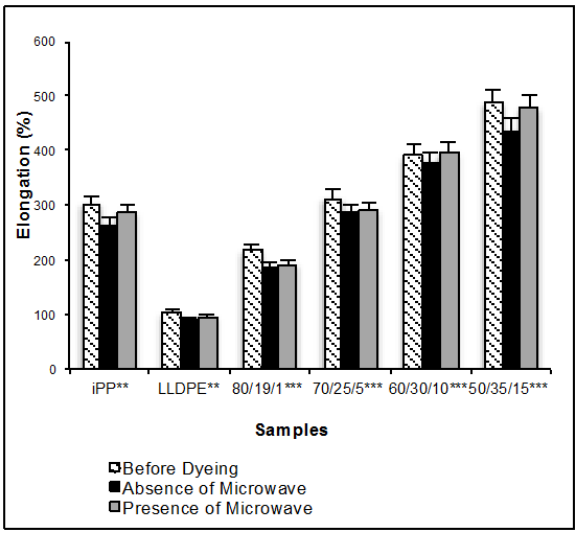


FIGURE 4. The change on elongation (%) of samples before and after dyeing (** 100%; *** iPP/LLDPE/EVA).

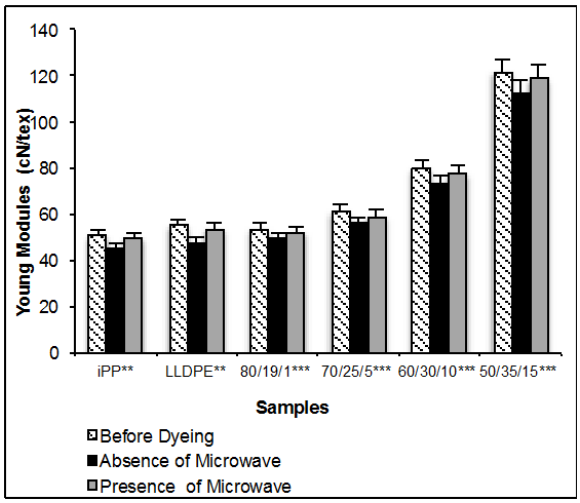
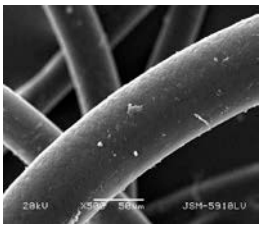


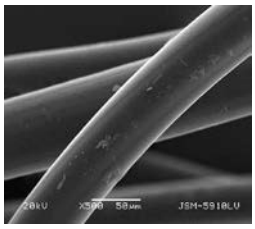
FIGURE 5. The changes on young modulus (cN/tex) of samples before and after dyeing (**100%; *** iPP/LLDPE/EVA).

Morphological Properties of Fibers

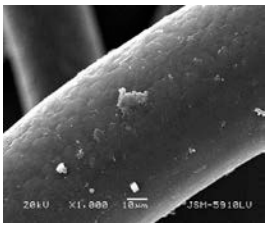
SEM images of iPP/LLDPE/ EVA ternary polymer blends dyed in the absence and presence of microwave energy are shown at *Figure 6*. A lot of porosity was observed on the surface of iPP/LLDPE/EVA ternary polymer blends dyed in the absence of microwave. The preasence porosity shows clearly that the dye had a non-dispersive effect on the surfaces of the blends. No porosity or deformation was observed on the surface of iPP/LLDPE/EVA ternary polymer blends dyed in the presence of microwave, indicating higher fiber structure preservation. Color measurement results showed that dye exhibited better adhesion to the fibers dyed in the presence of microwave energy as a result of the smoother, pore free surfaces.



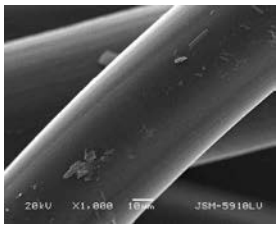
Dyed 50/35/15 % iPP/LLDPE/EVA ternary polymer blends in the absence of microwave (x500)



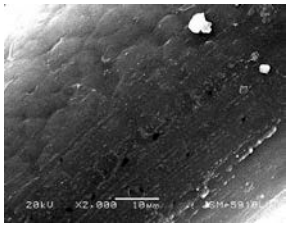
Dyed 50/35/15 % iPP/LLDPE/EVA ternary polymer blends in the presence of microwave (x500)



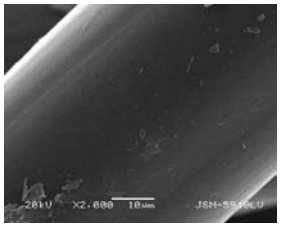
Dyed 50/35/15 % iPP/LLDPE/EVA ternary polymer blends in the absence of microwave (x1000)



Dyed 50/35/15 % iPP/LLDPE/EVA ternary polymer blends in the presence of microwave (x1000)



Dyed 50/35/15 % iPP/LLDPE/EVA ternary polymer blends in the absence of microwave (x2000)



Dyed 50/35/15 % iPP/LLDPE/EVA ternary polymer blends in the absence of microwave (x2000)

FIGURE 6. SEM images of ternary polymer blends after dyeing.

CONCLUSION

Based on the results presented, the following conclusions are possible:

- EVA incorporated at levels up to 15 % improved the coloristic and mechanical properties of ternary polymer blends.
- The color fastness values to washing and rubbing were adequate. In the presence of microwave energy, the light fastness values of the fibers improved from 3 to 5.
- SEM images indicated there was no porosity or deformation on the surface of dyed ternary polymer blends dyed in the presence of microwave energy.
- The total dyeing time, including heating up the dye baths, was 79 minutes in the absence of microwave and 8 minutes in the presence of microwave. This time reduction of about 90% indicates potential for significant energy savings by using microwave energy during the dyeing process.

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