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Reflectance Properties of Brown Mass Dyed Poly(ethylene terephthalate) Filament Yarns in the Visible-near Infrared Region

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ABSTRACT

Near infrared and visible reflectance properties of brown mass dyed poly(ethylene terephthalate) melt spun filament yarns were studied compared to natural brown hue of desert areas. Pigment Red 177, Pigment Green 7 and carbon black particles were used in this investigation. Near-infrared reflectance, mechanical and thermal properties as well as crystallinity of the camouflage poly(ethylene terephthalate) mass dyed filament yarns were measured. The obtained result indicated that by using an optimal content of the mentioned colored pigments and carbon black particles during the melt spinning operation could be achieved to desirable near infrared reflectance properties in the poly(ethylene terephthalate) drawn filament yarns. Also, mechanical properties of the samples were acceptable evaluated. The samples with the highest concentration of pigments had the highest percentage of crystallinity. XRD results indicated that 2 sample with the highest concentration of pigments and additives among other samples had the greatest distance between the crystalline plates. Maximum space between crystal layers is about 4.01-4.08 Å. Also, microstructural uniformity of the sample was confirmed by a low variation of samples. Prog. Color Colorants Coat. 13 (2020), 93-104© Institute for Color Science and Technology.

1. Introduction

Camouflage means hid or decept to reduce detection and is based on principles and agents of passive defense [1-3]. Concealment is one of the most important war crimes that must be used to deceive the enemy [4]. The idea of camouflage is taken from nature so that all animals have different ways to conceal and consistency with their living environment to survive and protect themselves [5-9].

In recent years, alongside the advancement of the camouflage industry, the equipment of camouflage exploration has been developing and progressing. An example of these rigs could be warfare surveillance devices that operate in various areas with different magnetic waves and are sensitive to UV rays, near infrared (NIR), far infrared (FIR) and radar waves [10]. Therefore, it is necessary to ensure the security of soldiers and their supplies to increase the military strength by protecting them not only in visible areas but also against identification equipment in different spectra of electromagnetic waves [8, 11-13].

To receive an infrared concealment property, textiles are dyed, painted, printed, coated or have received dyes and pigments during the melt spinning process so that the reflectance tendency in the nonvisible area must be assorted with the reflectance tendency of basis [5, 13]. Materials used for camouflage textiles in visible and NIR area consist conventional fibers and fabrics such as

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nylon, polyester, polypropylene, cotton and conventional dyes (e.g., acid, disperse, and sulfur dyes and pigments) [14, 15].

Commonly, the hues made in the most concealment samples are khaki, green, olive, brown and black [1]. Some researchers have reported that adding a percentage of carbon black (CB) nanoparticles to concealment hues in printing pastes could reduce the NIR reflectance of the samples. CB nanoparticles could also perceive a top air permeability and moderate great light fastness, also they didn't show any negative effect on overall fastness of the samples [3, 16-18]. While there are few studies on the production of camouflage fibers during the melt spinning process, Frankel produced modified polyamide-6-filament yarns by using carbon black particles that it is suitable for to create camouflage in desert, urban and mountainous areas [19]. In our previous research works, a little content of CB was used during the melt spinning process that was reported significant reflectance reduction on the Vis-NIR band. By applying a small amount of CB particles not only does not have a damaging effect on color of it also makes more reflectance matching in NIR range. In one of them camouflage polypropylene filament yarns, and in another PET filament yarns were produced, and acceptable mechanical properties were obtained. Camouflage properties in forest areas at visible–NIR band were successfully has been achieved, in a study that PET filament yarns produced. In order to produce olive green camouflage hue. Also the crystallinity percent of the PET camouflage filament yarn samples was decreased, and washing and light fastness values of camouflage PET filament yarn were excellent [20-22].

Lack of published works on mass dyeing of synthetic fibers with brown hue in the NIR region, obliged us to study the reflectance properties of mass dyed poly(ethylene terephthalate) (PET) filament yarns. Two commercial Red (C.I. Pigment Red 177) and Green (C.I. Pigment Green 7) pigments were used to obtain camouflage property in desert zones. The reflectance properties of the modified PET filament yarns in visible-NIR wave band were studied. Mechanical and thermal properties as well as microstructure of the samples were also characterized.

2. Experimental

2.1. Materials

Semi-dull granules (TG 645) prepared by Tondgooyan Petrochemical Co. (Iran) with the intrinsic viscosity of 0.64 ± 0.01 dL/g, melting temperature of 255 ± 3°C, and TiO_2 content of 0.34 ± 0.06 %wt were used as starting material. The applied pigments are listed in Table 1. CB (series N-330) was supplied from Dodeh pars Co. (Iran) with the maximum sulfur content of 2.5%, pour density of 355 - 405 kg/m^3, the maximum ash content of 0.75%, and pH=7. The applied pigments have high infrared absorbance (similar to natural brown hue) and high thermal stability during the melt spinning process.

2.2. Methods

2.2.1. Pre-drying of PET granules and pigments

PET granules were crystallized for two hours at 120 °C and then were dried for four hours at 160 °C before feeding into the melt spinning extruder. Selected green and red pigments and CB particles were dried for 4 h at 120 °C before mixing with PET granules. A hot air oven was used for drying the raw materials.

2.2.2. Melt spinning and cold drawing process

The melt spinning operation was carried out by a laboratory mixing extruder produced by Dynisco, MA (USA). Different amounts of PET granules, pigments, and CB were mixed and then fed into the barrel of the extruder. Orifice header of the extruder was set with a diameter of 1 mm that it is standard for orifice of an extruder. Temperature of the barrel and die zones was set to 270 °C. The extruder screw rotation was set to 12 rpm.

Table 1: Specification of the pigments used in the samples.

Commercial name	C.I. name	Molecular formula	Molecular structure	Molecular weight	Manufacturer company	Manufacturer country
Cromophtal RED A3B	C.I. Pigment Red 177	$C_{28}H_{16}N_2O_4$	Anthraquinone	444.446 g/mol	Ciba Geigy	Switzerland
Pthalo GREEN GLNP	C.I. Pigment Green 7	$C_{32}H_3Cl_{15}CuN_8$	Phthalocyanine	1127.15 g/mol	Ciba Geigy	Switzerland

As-spun filaments were rolled around the cylindrical paper packages at a speed of approximately 70 m/min. Some fiber samples were cold drawn utilizing EMT 3050 tensile tester made by Elima Co. (Iran) at a constant elongation speed of 50 cm/min at room temperature then they were rolled on a card board frame to form an order. Table 2 shows the concentration of selected pigments and carbon black particles applied during the melt spinning process and their codes.

2.2.3. Spectral reflectance evaluation

The reflectance curves of the filament yarns were evaluated with a reflectance spectrophotometer, made by Analytik Jena Specord 250 (Germany) in Visible/NIR band width of 400-1100 nm. Observation geometry spectrophotometer used in this paper was set to 8°/d according to ASTM E1331 [23]. The scan speed of the test was set to 0.5 s. The reflectance behavior of fibers was evaluated against reflection behavior of the reference sample. The spectrum of the reference sample was selected from the official database of the Hochst Company [24] used by many researchers [9, 16-18, 25].

2.2.4. Mechanical properties

Tensile samples were tested using EMT 3050 tensile tester at a constant elongation based on ASTM D3822-95a [26]. Gage distance and gage speed were 10 cm and 50 cm/min, respectively. Ten tensile measurements were carried out for each sample. Their average and coefficient of variants (C.V. %) were reported.

2.2.5. Thermal and crystallinity properties

Differential scanning calorimeters (DSC 214 Polyma made by Germany) was applied for studying the thermal behavior and calculating the crystallinity of the samples. The fibers were heated at the heating rate of 10 °C /min from room temperature to 280 °C and, subsequently, cooled down to room temperature. The glass transition temperature (T_g), melting temperature (T_m), melting and crystallization enthalpies were estimated during the DSC test, and the crystallinity percentage was calculated by equation (1):

$$X_c\,(\%) = \frac{[\Delta H_m - \Delta H_c]}{\Delta H^{\circ}_m} \times 100 \qquad (1)$$

Where X_c is the crystallinity percentage, ΔH_m is the melting enthalpy, ΔH_c is the crystallization enthalpy, and ΔH°_m advert to 100% crystallinity polymer, which in the case of PET equals to 140 j/g [27, 28].

2.2.6. X-Ray diffraction analysis

Crystallinity evaluation was carried out by X-Ray diffraction (XRD) device (E Quinox 3000 made by Inel (French)). The scanning angular of 5-118° was applied in obtaining X-ray diffraction patterns. X-ray scanning of samples was performed employing voltage and current of the test at 40 kV and 30 mA, respectively. The size of the crystal plates was calculated by Scherrer equation (Eq. 2):

$$L_c = \frac{0.9\lambda}{\beta \cos\theta} \qquad (2)$$

Table 2: Concentration of additives applied during the melt spinning process and their codes.

Code samples	CB (%)	C.I. Pigment Red 177 (%)	C.I. Pigment Green 7 (%)
1	0.1	0.9	0.6
2	0.2	0.9	0.6
3	0	0.9	0.6
4	0.02	0.8	0.5
5	0.07	0.8	0.5
6	0.1	0.8	0.5
7	0.12	0.8	0.5
P (Pure)	0	0	0

Where L_c is the size of the crystal plates, λ is the wavelength of X-ray (1.54187 Å), β is the peak width in half-height, and θ is the angle between the incident rays and the crystal surface. According to Bragg's equation (Eq. 3) were calculated the spacing between crystal layers.

$$d=\frac{\lambda}{2sin\theta} \quad (3)$$

Where d is the crystal layer spacing, λ is the wavelength of X-ray (1.54187 Å), and θ is the angle between the incident rays and the crystal surface [29].

3. Results and Discussion

3.1. Vis – IR reflection of brown mass dyed filament yarns

Pigment Green 7 and Pigment Red 177 were used to produce brown mass dyed PET filament yarns. These pigments have a great thermal stability during the melt spinning process and also have infrared absorption property with proper reflectance in this region [30]. CB was also used to adjust the reflectance properties in near infra-red region. CB powders are highly thermal durable during the melt spinning process and great IR rays absorbance [31]. The following figures show the effect of carbon black particles and color pigments on the reflection properties of the samples.

Figure 1 indicates the reflection behavior of PET pure filament yarn and samples containing two different concentrations of CB powder compared to the reference sample in the Vis – NIR region between 400 to 1100 nm. It can be seen that the total Vis – NIR reflectance of the pure PET fiber is about 68-85%, although the NIR reflectance is around 75-85%. The CB in two concentrations of 0.1% and 0.2 % was added to the PET granules with the constant concentration of Pigment Green 7 and Pigment Red 177 (G=0.6 %, R=0.9 %) in two samples for production of brown fiber to investigate the effect of CB powders. By adding 0.1 % of CB to sample 2 that is more than sample 1, we could reduce the NIR reflectance of the sample. The maximum absorbance wavelength of Green Phthalocyanine pigment is about 670 nm because it has a central copper atom. Therefore, it can be appropriate for color matching of the filament yarn. By increasing the CB powder concentrations from 0.1 % to 0.2 %, the reflection is reduced about 5% in the NIR range. Also, there is significant difference between the pure sample and the colored sample in 400 – 1100 nm. These two samples are very similar to the reference sample in the NIR region.

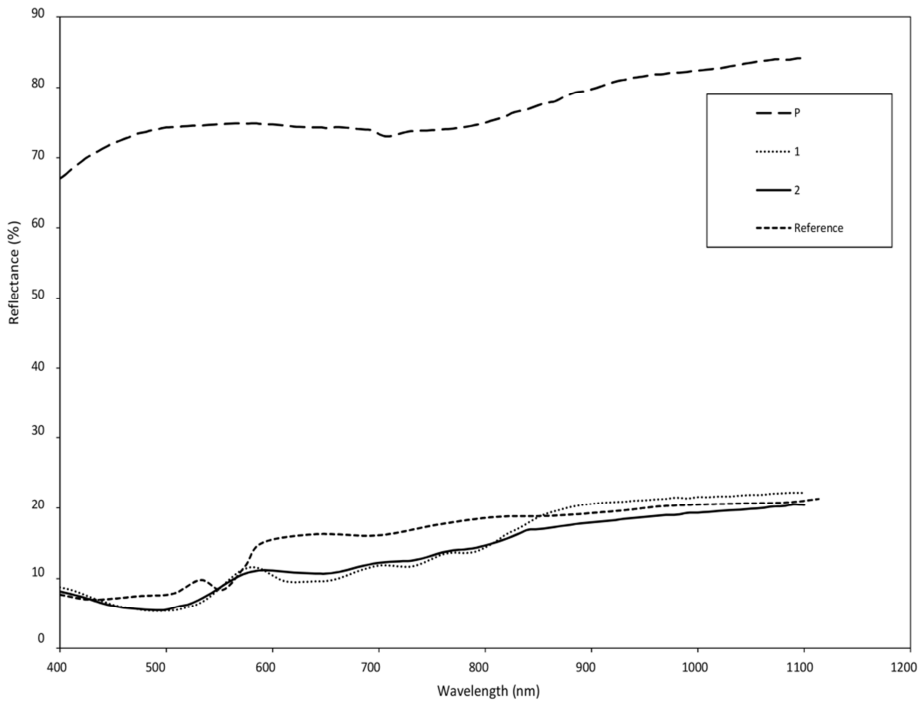

Figure 1: Reflectance spectra of the pure PET, reference and filament yarn samples 1 and 2.

It can be seen in Figure 2 that by the elimination of CB in melt spinning process without changing the dye content according to the two previous samples, we observe about 50% reflection increasing in NIR range. Also, it caused 5% increase in reflection in 400 – 800 nm. It shows that the presence of CB in the reflection reduction is very effective. So, carbon black particles have significant effect on the reflection and infrared absorption in the infrared region.

Then, we reduced 0.1% from colored pigment as compared with first two specimens and reduced CB five times compared to the sample 1, so in Figure 3 have seen about 30% reflection reduction in NIR range due to the sample 3, and this is due to the presence of CB. But there is about 5% increasing in reflection in the range of 400-800 nm compared to the CB-free sample.

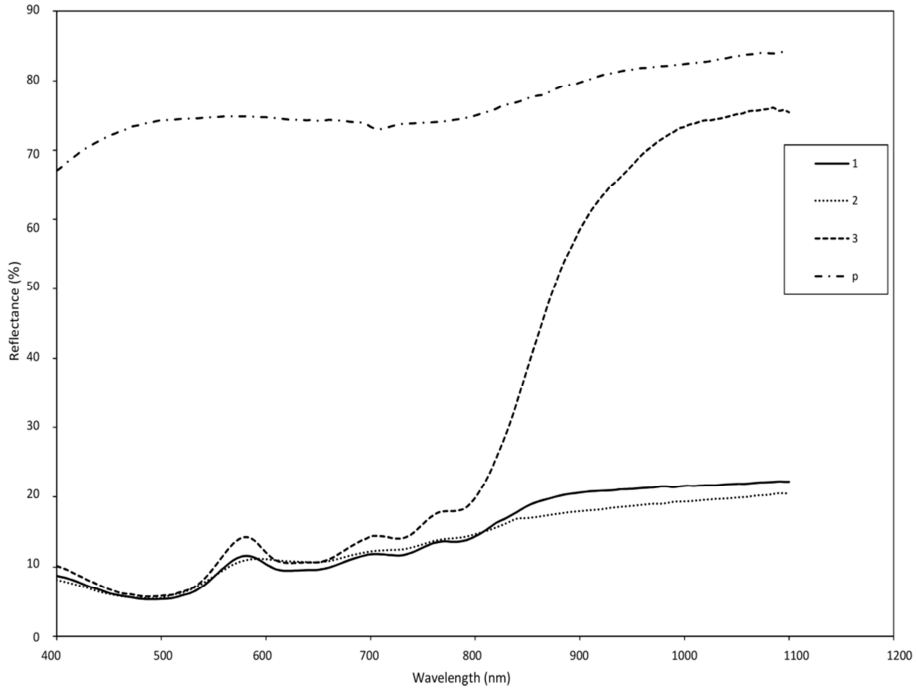

Figure 2: Reflectance spectra of the pure PET and filament yarn samples 1, 2 and 3.

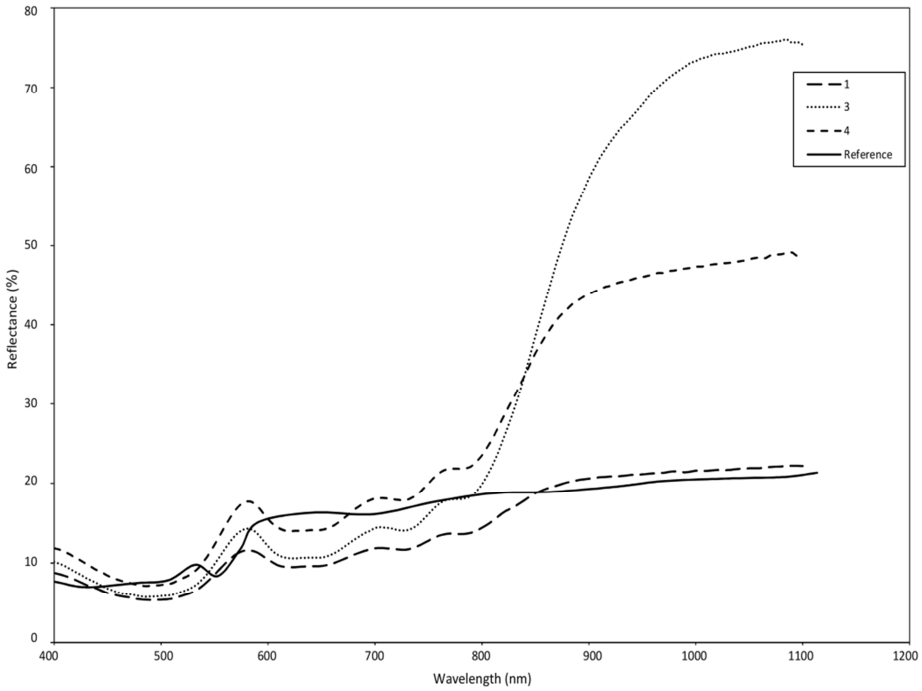

Figure 3: Reflectance spectra of the reference and filament yarn samples 1, 3 and 4.

Then, receiving to the reference reflection, the CB was raised 3.5 times compared to sample 4 without changing the content of the colored pigments. It can be seen in Figure 4 that by 3.5 times increasing of CB in the presence of constant values of Pigment Green 7 and Pigment Red 177 (sample 4), we observed about 15% reflection reduction in NIR range. Although the reflection in NIR range has decreased, the reflection of sample is about 35%. For brown hue, the reflection value is about 10% higher than that of the reference sample. Therefore, we need higher amounts of CB powders in the colored formulation.

As shown in Figure 5, by supplying the concentration of dyes constantly such as the previous sample and increasing 1.4 times CB particles compared to the sample 5, we noticed about 4 – 9% reflection reduction in NIR range and about 2 – 4% reflection decline in visible range compared to the previous sample. This specimen is similar to the first sample by holding 0.1 % colored pigments fewer than sample 1.

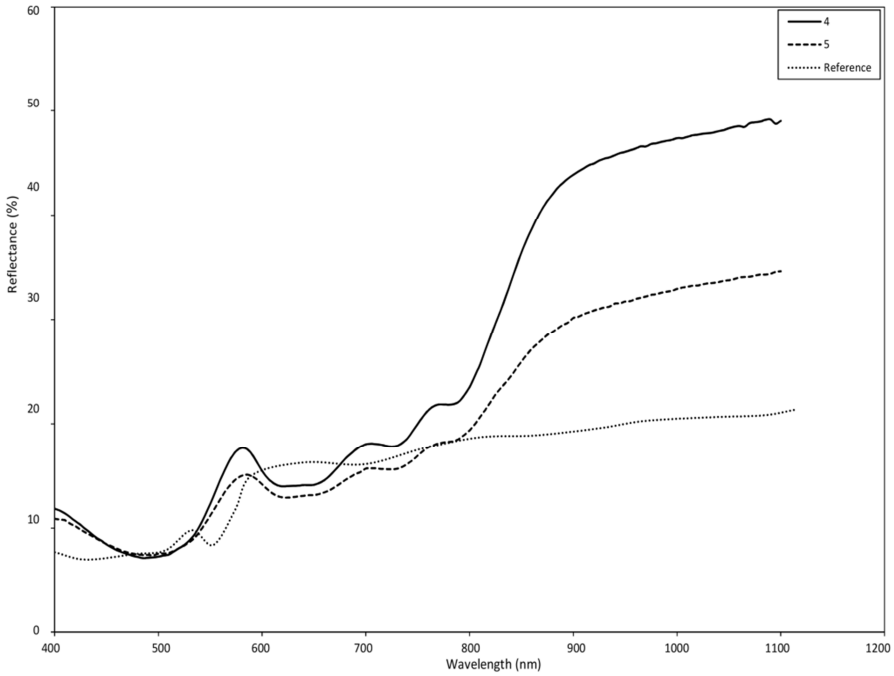

Figure 4: Reflectance spectra of the reference and filament yarn samples 4 and 5.

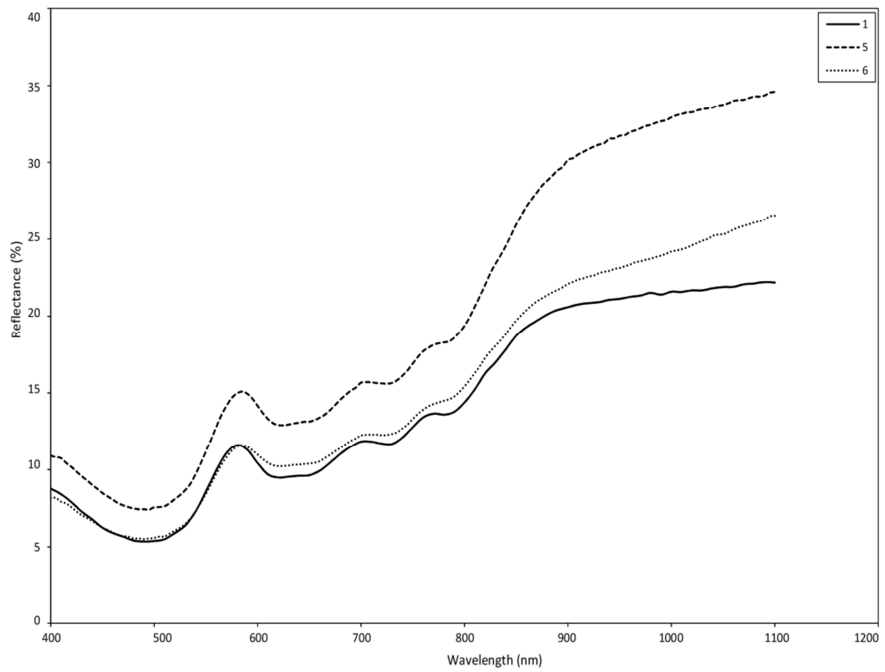

Figure 5: Reflectance spectra of filament yarn samples 1, 5 and 6.

Afterward, we produced one another sample with a constant content of value of colored pigments, and by increasing 1.2 times CB of 6 sample, we found 2 – 5% reflection reduction of sample 6 in NIR range. The effect of CB particles on reduction of the reflection in NIR range is greater than that of the colored pigments. As sample 7 compared with sample 1, it has observed that due to the higher concentration of colored pigments in specimen 1, specimen 7 has about 1% reflection reduction in the NIR region. This reduction is due to the presence of 0. 02 % more CB in sample 7.

Figure 6 shows the reflectance spectra of sample 7 and brown reference. Accordingly, as shown in Figure 6, we can express that the optimum sample for creating brown shade is sample 7 which contains 0.5 % green pigment, 0.8 % red pigment, and 0.12 % CB.

Figure 7 shows four reflectance spectra that are similar to the reference brown hue. They have 25 ± 5% reflectance in NIR region. Among these 4 samples, sample 7 has shown a good matching behavior in the wavelength range of 865-1100 nm.

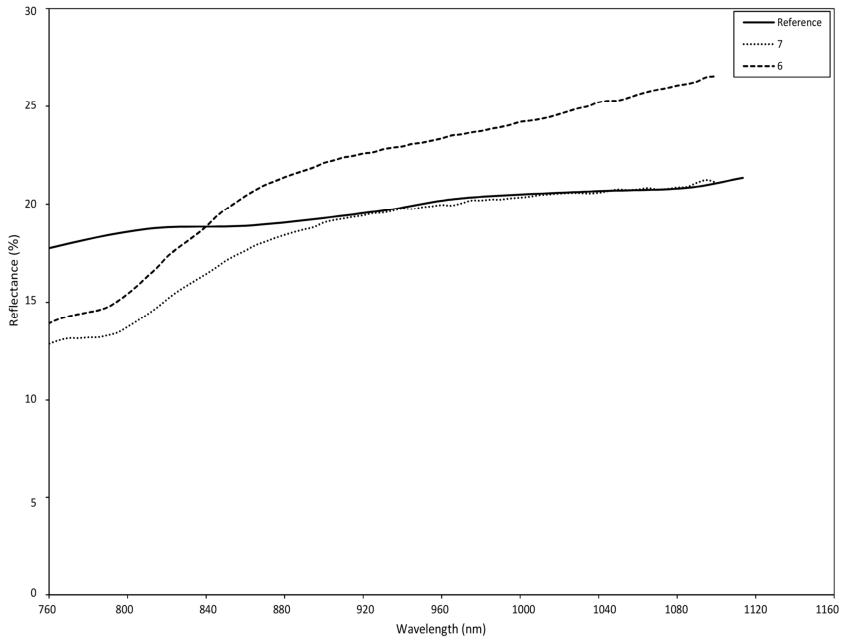

Figure 6: Reflectance spectra of the reference and filament yarn samples 6 and 7.

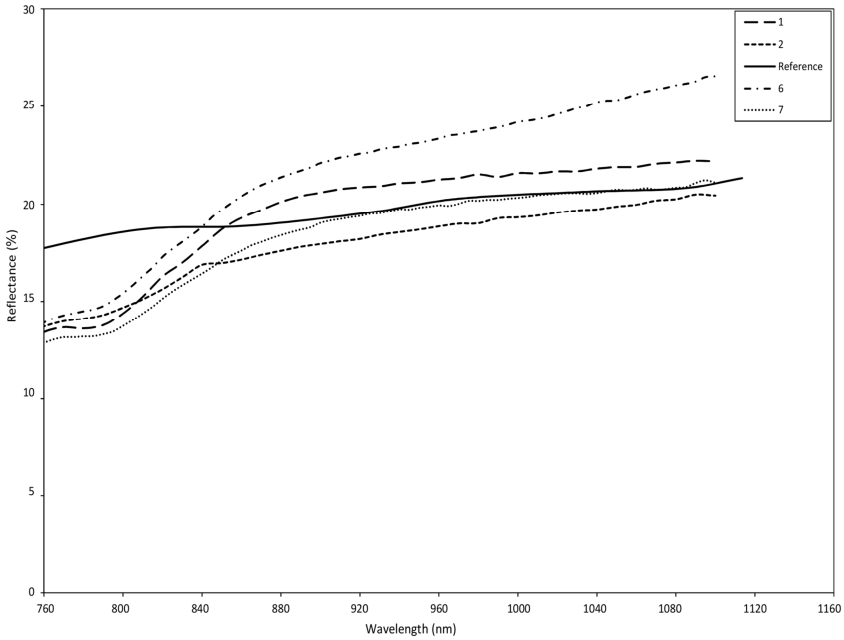

Figure 7: Reflectance spectra of the reference and filament yarn samples 1, 2, 6 and 7.

3.2. Mechanical properties

Mechanical and thermal properties of samples 1, 2, 6 and 7 with similar reflection to the reference sample were studied. The mechanical properties of as-spun and drawn filament yarn samples are shown in Tables 3 and 4, respectively. The obtained results of selected filament samples are also presented to show the effect of pigments and CB on the mechanical properties of the PET filament yarn samples. Hence, elongation at break of as-spun sample 2 is less than other samples because this sample has the largest content of additives. But, the elongation at break of sample 2 was improved. The modulus and work of rupture of as-spun PET filament yarns of specimens are very poor. With increasing the CB concentration, mechanical properties of melt-spun filament yarn samples were reduced. The mechanical properties of PET filament yarn samples enhanced by cold drawing process with the orientation increase of polymer chains along the PET filament yarn axis. With the lowest concentrations of additives, the highest strength is obtained (sample 6). With the highest concentration of additives, the lowest initial modulus has been observed (sample 2).

Withdrawing process and arrangement of molecular chains along the longitudinal axis of the yarn, the initial modulus often increases [32]. By cold drawing process, the crystallization rate increases while the size of the crystals decreases in the longitudinal axis of the fiber. The number of crystal cells and the initial modulus increase with increasing the crystallization [33]. The cause of the decline of elongation at break in drawn samples due to increasing of arrangement or perhaps the adherence of amorphous and crystalline areas to each other. During drawing process, the diameter of the fiber is reduced, hence the linear density of the drawn samples are decreased. Due to the presence of pigments and additives, the strength of the samples before and after drawing has decreased rather than the pure sample. The sample with the lowest amount of additives showed the best mechanical properties, similar to the mechanical properties of the pure PET fiber sample.

Table 3: Mechanical properties of as-spun fibers (pure sample and samples 1, 2, 6, and 7).

Sample	Tenacity (CN/Tex)	Elongation at break (%)	Linear density (Denier)	Modulus (CN/Tex)	Work of rupture (CN/Tex)
Pure PET	4.13 (8.22)[a]	460.2 (4.15)	42	11.23 (7.28)	8.96 (9.61)
1	3.71 (10.22)	447.2 (2.1)	42.3	10.33 (4.93)	7.25 (9.67)
2	1.99 (11.33)	201.7 (7)	39.6	11.71 (7.66)	3.74 (20.83)
6	3 (6.42)	381.2 (6.74)	40.03	10.47 (3.63)	4.88 (11.73)
7	2.67 (6.4)	381.2 (6.74)	45	9.32 (3.62)	4.34 (11.75)

[a] value inside brackets indicate coefficient of variants (C.V.%).

Table 4: Mechanical properties of drawn fibers (pure sample and samples 1, 2, 6, and 7).

Sample	Tenacity (CN/Tex)	Elongation at break (%)	Linear density (Denier)	Modulus (CN/Tex)	Work of rupture (CN/Tex)
Pure PET	19.7 (9.98)[a]	37.4 (7.77)	8.73	96.36 (10.92)	5.37 (11.86)
1	18.5 (11.32)	24.87 (9.58)	9.028	83.41 (10.4)	3.36 (15.79)
2	17.76 (9.77)	42.84 (12.04)	8.75	57.24 (7.2)	4.87 (9.34)
6	19.23 (9.33)	29.24 (6.45)	9.24	102.4 (11.45)	3.99 (14.27)
7	17.09 (9.32)	29.24 (6.45)	10.392	91.08 (11.46)	3.55 (14.31)

[a] value inside brackets indicate coefficient of variants (C.V.%).

The uniformity of mechanical properties was acceptable, and some mass dyed samples (6 and 7) had higher uniformity comparing to pure PET filament yarn.

3.3. Thermal properties

The thermal properties of the pure and selected PET filament yarn samples (1, 2, 6 and 7) are evaluated. The DSC graphs in the heating and cooling operations are shown in Figure 8. Also, the values of thermal properties are reported in Table 5.

According to Figure 8 (a), the presence of CB and colored pigment did not show an impressive effect on final melting, but it reduced the glass transition temperature (T_g) of the PET filament yarn samples compared to pure PET sample. Also, the crystallinity percent of the brown mass dyed PET filament yarn samples was decreased except for samples 1 and 2 because they had more colored pigments in filament sample which play as nucleation centers.

Furthermore, the crystallization temperature (T_c) was increased from 207.4 to 216 °C by increasing the amount of the additives. Accordingly, all values of the samples crystallinity were normal. Crystallization occurs during the cooling process. It can be seen in Figure 8 (b) that the nucleation of crystals was started earlier because of the pigments particles nucleation role and the enhancement and reduction of height and width of the crystallization peaks, respectively. The colored pigments prepare nucleation properties at higher temperatures previous the solidification [34]. While, there are numbers of the nucleating agents, which increases the final percentage crystallinity, the number of spherulites, and crystallization speed that they cause a synergism effect for the nucleation [35, 36]. According to the results shown in Table 4, the thermal properties of the samples do not change dramatically with a little variation, but the crystallinity and mechanical properties by little variation are changed.

3.4. X-ray diffraction analysis

The XRD patterns of pure sample and PET filament yarn samples 1, 2, 6 and 7 are shown in (Figure 9). Thermoplastic polymers have a wide peak and they support crystallization to specific crystalline forms, but mineral material such as colored pigments have a sharper peak due to more crystallinity. Because of the low concentration of colored pigments in the samples, cannot be seen sharp peaks regarding colored pigments in Figure 9. It was considered that the presence of 10% mineral material in the fibers is recognizable using XRD technique [37]. The results of XRD test are given in Table 6. The maximum spacing between crystal layers is about 4.01 – 4.08 Å. Accordingly, size of the spacing between the crystalline plates of the colored samples is somewhat higher than the pure PET filament yarn sample. Sample 2 with the highest concentration of pigments and additives among other samples has the greatest distance between the crystals plates. According to Table 6, it can be concluded that the most important parameter for changing the crystals plates distance is the amount of carbon black. The higher initial modulus of sample 6 is related to its higher crystal plates size among the other samples.

Table 5: DSC test results of the pure and drawn PET filament yarn samples 1, 2, 6 and 7.

Sample	Melting temperature (°C)	Glass transition temperature (°C)	Temperature of crystallinity (°C)	Melting enthalpy (j/g)	Crystallization enthalpy (j/g) (heating)	Crystallization enthalpy (j/g) (cooling	Crystalline percentage (%)
Pure PET	254.1	76.2	207.4	42.81	1.029	44.27	29.84
1	255.1	66.9	216	46.74	1.051	38.52	32.64
2	255.2	69.2	214.2	45.79	1.509	40.91	31.63
6	259.1	67.5	215.6	43.51	4.619	36.12	27.78
7	254.1	63.7	215.8	45.6	11.36	44.62	24.45

Table 6: XRD results of pure sample and as-spun PET filament yarn samples 1, 2, 6 and 7.

Sample	Diffraction angle [2θ]	Peak width in half height (FWHM) [2θ]	Size of the crystal plates (Å)	d-spacing (Å)
Pure PET	22.2	9.25	0.153	4.0119
1	22.18	10.57	0.135	4.0147
2	21.8	9.16	0.154	4.08417
6	22.1	8.87	0.159	4.029
7	22.04	9.27	0.1527	4.04

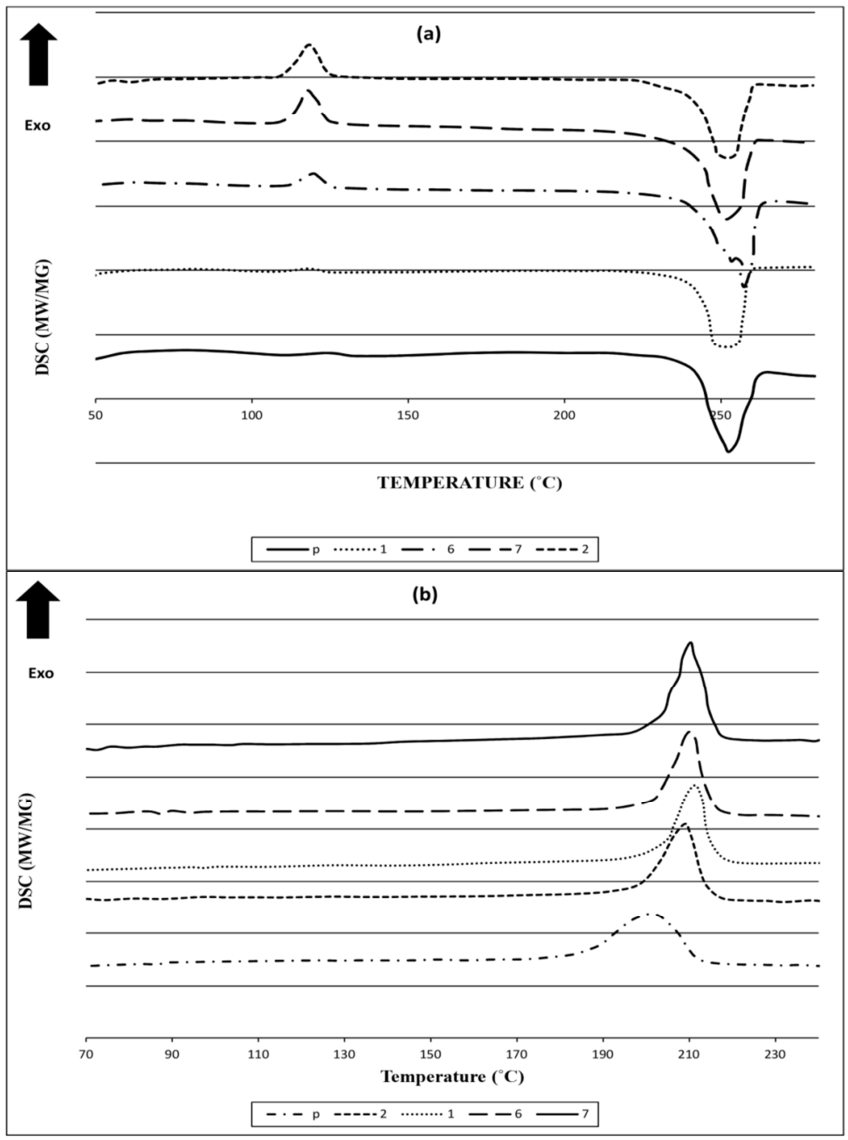

Figure 8: DSC graph for different mass dyed filaments with carbon black and red and green pigments (pure sample and samples 1, 2, 6 and 7): (a) heating graph and (b) cooling graph.

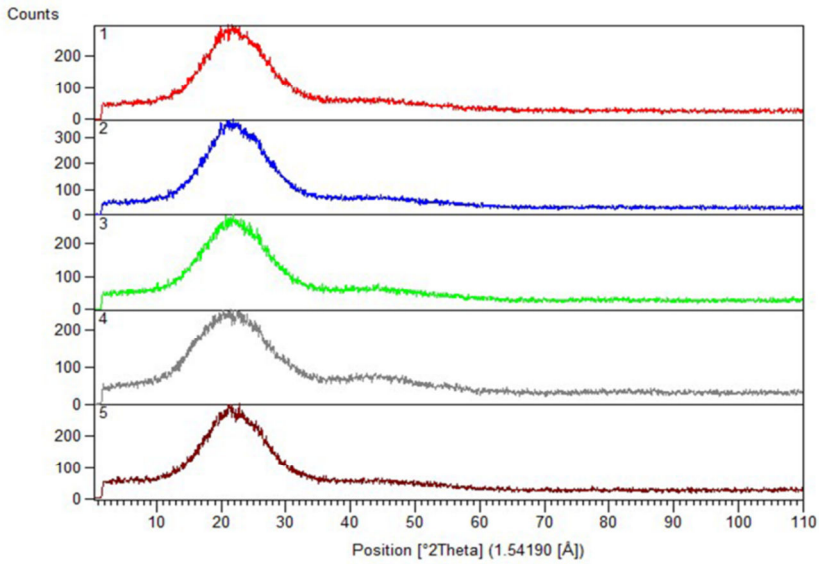

Figure 9: XRD patterns of (1) 1, (2) pure, (3) 7, (4) 2, and (5) 6 PET filament yarn samples.

4. Conclusions

In this research, PET filament yarns with NIR reflectance properties were prepared. Pigment Red 177, Pigment Green 7 and CB particles were used to supply brown mass dyed poly(ethylene terephthalate) filament yarn. The results showed that by decreasing about 0.1% of colored pigments without changing the amount of carbon black, the reflection of samples is increased about 4% in the NIR region. Carbon black not only had no negative effect on the properties of the samples, but also creates a good reflection of brown color in NIR range. Lower values of tenacity and elongation at break of obtained PET filament yarns after adding CB powder and colored pigments are due to the accumulation of additives in the polymer. Reduction of mechanical properties is improved after the drawing process, and they are in acceptable range. CB and pigments caused about 1-5 °C increase in final melting point temperature and about 7–13 °C reduction in glass transition temperature of samples. Furthermore, the crystalline percentage of the brown mass dyed poly(ethylene terephthalate) filament yarns was decreased except for the two samples that had the most colored pigments. By increasing the amount of the additives, the crystallinity temperature of the samples was raised. Additionally, the highest concentration of additives had the greatest distance between the crystals plates.

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