# Dyeing of Innovative Bicomponent Filament Fabrics (PET/PTT) by Disperse Dyestuffs: Characterization and Optimization Process

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#### Abstract:

PET/PTT bicomponent filaments yarn is produced by two polymers: the polyethylene terephthalate (PET) and the polytrimethylene terephtalate (PTT) extruded side by side. This yarn is known for its high mechanical properties in particular elasticity and elastic recovery. However, differences between physical and chemical properties of the two components make the dyeing step of this yarn complicated. The aim of this work is the development of a dyeing process for bicomponent filaments without altering their physical and chemical properties. Different techniques such as SEM, FTIR, and differential scanning calorimetry (DSC) were used to characterize the studied yarn. For dyeing, three different disperse dyes CI Disperse Red 167.1, CI Disperse Yellow 211, and CI Disperse Red 60 with different energy classes were studied. The influence of dyeing conditions in particular dyeing temperature, pH of dye bath, dyeing time, and carrier concentration in the dye bath was evaluated. Responses analyzed are color strength (K/S), colorimetric coordinates and color fastness of samples dyed with studied dyes. In addition, the stability of elasticity and elastic recovery of bicomponent filament fabrics after the dyeing process has been also verified and proved.

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# Dyeing of Innovative Bicomponent Filament Fabrics (PET/PTT) by Disperse Dyestuffs: Characterization and Optimization Process

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**Abstract:** PET/PTT bicomponent filaments yarn is produced by two polymers: the polyethylene terephthalate (PET) and the polytrimethylene terephtalate (PTT) extruded side by side. This yarn is known for its high mechanical properties in particular elasticity and elastic recovery. However, differences between physical and chemical properties of the two components make the dyeing step of this yarn complicated. The aim of this work is the development of a dyeing process for bicomponent filaments without altering their physical and chemical properties. Different techniques such as SEM, FTIR, and differential scanning calorimetry (DSC) were used to characterize the studied yarn. For dyeing, three different disperse dyes CI Disperse Red 167.1, CI Disperse Yellow 211, and CI Disperse Red 60 with different energy classes were studied. The influence of dyeing conditions in particular dyeing temperature, pH of dye bath, dyeing time, and carrier concentration in the dye bath was evaluated. Responses analyzed are color strength (K/S), colorimetric coordinates and color fastness of samples dyed with studied dyes. In addition, the stability of elasticity and elastic recovery of bicomponent filament fabrics after the dyeing process has been also verified and proved.

**Keywords:** bicomponent filaments; polyethylene terephthalate (PET); polytrimethylene terephthalate (PTT); dyeing; disperse dyes

# 1. Introduction

Nowadays, the textile industry is looking for new alternatives and tends to use more efficient, innovative, and ecological methods while guaranteeing quality and competitiveness. The focus of many recent research has been on improving the performances of textiles in terms of durability [1], resistance [2], permeability [3], elastic recovery [4–6], biodegradability [7], etc. In this context, conventional fibers can no longer meet the demands of textile market which continues to grow and change. To meet these industrial needs, recently, several studies have succeeded to combine several polymers in a single filament in order to obtain the so-called bicomponent, tricomponent, and even more fibers or filaments. These fibers are composed of two or more polymers extruded from the same spinneret [8–10]. They differ from each other in terms of physical and chemical properties such as



molecular weight, strength, melting point, and crystallinity [11]. The characteristics of bicomponent (or even more) fibers depend on the nature and properties of used materials, their arrangement in the fiber, their relative proportions, and the thickness of the obtained fiber [12].

Recently, considerable attention has been paid to innovative bicomponent polyester filaments marketed as Lycra T400<sup>®</sup> by DuPont Company [13–15]. This bicomponent filaments yarn is composed of 60% polyethylene terephthalate (PET) and 40% polytrimethylene terephthalate (PTT). The two polymers making up the yarn are extruded from the same spinneret, adjacent and arranged side by side [8–10]. As shown in Figure 1, each bicomponent filament is melt spun in a specially designed spinneret. Separate extruders and metering pumps are used for both components. The ratio of the polymers can be controlled by varying the speed of the metering pumps. The bicomponent filament thus obtained has a spiral morphological structure that provides it with significant elastic properties [9,16–18]. This new yarn allows to bring a stretch effect to the fabric while ensuring better behavior during various chemical treatments [19,20].



Figure 1. Spinning process of bicomponent filaments using side by side configuration.

The use of PTT into the bicomponent filaments is of great interest as it contributes to the yarn a very soft feel, great elasticity, and better elastic recovery. Indeed, PTT is one of the newest synthetic fibers [21]. It is manufactured by the two major companies, Shell Chemical and DuPont, under the commercial names Corterra and Sorona, respectively [15]. This fiber is synthesized in the same way as PET. It is known for its great extensibility which is attributed to the arrangement and orientation of the polymers in the chain. As reported by [22,23], PTT have more elastic filaments than those of PET, a softer feel and excellent elastic recovery because of the length of the trimethylene unit between the benzene cycles along the polymer chain. In addition, thanks to this increased flexibility of its macromolecular chain, PTT fiber can be dyed at lower temperatures with disperse dyes compared to PET fiber [20].

However, no studies on dyeing bicomponent filaments are available in the literature and several practical questions arise. So, it is essential to develop an adequate process to dye this yarn composed of two polymers having a different affinity and behaviors during dyeing.

This paper presents an original work by giving a newly developed dyeing bath and optimizes new dyeing process of textile fabrics containing bicomponent filaments without altering their physical and chemical properties. The molecular weight and number of polar groups in the molecule directly affect the dyeing performance [24]. So in order to have a complete and thorough study, three different disperse dyes, namely Terasil Rubin 2GFL (Dye #H), Terasil Yellow 4G (Dye #M), and Terasil Red FBN (CI Disperse Red 60) belong respectively to high energy, medium energy, and low energy classes, were used. Many techniques such as scanning electron microscope (SEM), Fourier transform infrared spectroscopy (FTIR), and differential scanning calorimetry (DSC) were performed to characterize the studied yarn. This study is a first part of a more ambitious project to develop a dyeing process that is applicable at the industrial scale.

# 2. Materials and Methods

# 2.1. Textile Support

Textile samples used during this study are jersey fabrics knitted with 100% bicomponent filaments marketed as Lycra T400<sup>®</sup> purchased from Invista Company (Wichita, KS, USA). Table 1 summarizes the characteristics of the used yarn as well as the knitted fabrics thus obtained. Jersey knitted fabrics were made using circular knitting machine type Tricolab gauge 12 (Sodemat, Gent, Belgium).

Structures	Characteristics	Values	ISO Standards
	Composition of filament	60% PET/40% PTT	_
	Number of filaments per yarn	64	_
T400 multifilament yarn	Average count of yarn (Tex)	16.5	ISO 7211/5
	Strength at break of yarn (N)	5	ISO 3377-2
	Elongation at break of yarn (%)	25.41	ISO 3377-2
	Composition	100% T400 multifilament yarn	_
	Type of binding	Jersey knit	_
Fabric knitted with T400	Knit thickness (mm)	0.92	ISO 5084
multifilament yarn	Knit weight (g/m2)	215	EN 12127
	Strength at break (%)	540	ISO 13934-1
	Elongation at break (%)	250	ISO 13934-1

Table 1.	Physical	characteristics	of T400	multifilament	yarn and	knitted	fabrics
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# 2.2. Disperse Dyes

Three disperse dyes with different molecular weights (low, medium, and high) were used in this study. These dyes are namely: Terasil Rubin 2GFL (Dye #H), Terasil Yellow 4G (Dye #M), and Terasil Red FBN (Dye #L). These dyes were purchased from Huntsman Company (Basel, Switzerland) and used without further purification. Their chemical characteristics are presented in Table 2.

# 2.3. Dyeing Procedure

Before dyeing step, knitted fabrics were pretreated in a solution containing water, 1 g/L of Eriopon R detergent (Huntsman Company, Basel, Switzerland) and 2 g/L of sodium carbonate for 10 min at 70  $^{\circ}$ C and using a bath ratio of 20:1.

Table 2. Chemical characteristics and configuration of studied disperse dyes.

Dyes	Chemical Characteristics and Configuration						
	Generic name: CI Disperse Red 167.1 Commercial name: Terasil Rubin 2GFL Chemical formula: C <sub>22</sub> H <sub>24</sub> ClN <sub>5</sub> O <sub>5</sub> Molecular weight: 473.15 g·mol <sup>-1</sup> Minimum steric energy: 6.1873 kcal·mol <sup>-1</sup>						
Disperse Dye N°1 (Codified #H)	$C_2$ $N$ $N$ $C_2$ $H_4$ $COCH_3)_2$ $C_1$ $N$ $C_2$ $H_4$ $COCH_3)_2$ $C_1$ $C_2$ $H_4$ $COCH_3)_2$ $C_1$ $C_2$ $H_4$ $COCH_3)_2$ $C_1$ $C_2$ $C_3$ $C_4$						





Figure 2 shows the dyeing process of T400 knitted fabrics with disperse dyes.





In fact, for each dyeing, 1.2% disperse dye and 1 g/L Albatex DBC (Huntsman Company, Basel, Switzerland) dispersant were added. The liquor-to-fiber ratio is equal to 10:1. Mcilvaine Buffer solution containing disodium phosphate (Na<sub>2</sub>HPO<sub>4</sub>) and citric acid (C<sub>6</sub>H<sub>8</sub>O<sub>7</sub>) is used to ensure that the pH of the dye was stable throughout the dyeing process [25]. The pH is varied from an acidic pH of 4 to a basic pH of 8 and the various dyeing temperatures evaluated are 90 °C, 100 °C, 110 °C, 120 °C, and 130 °C.

In order to have good dyeing performances and energy savings, the effect to add a carrier agent, namely Livster BF and considered less toxic, was tested. It is a derivative of phthalamides purchased from (Huntsman Company, Basel, Switzerland). In order to remove residual dyes deposed at the surface and improve washing fastness, dyed samples were soaped with bath containing 2 g/L sodium hydrosulphite and 2 mL/L sodium hydroxide solution (36°Be) at 50 °C for 10 min, with a liquor-to-fiber

ratio of 20:1. All pre-treatments, dyeings, and post-treatments were performed in a pressure autoclave type Ahiba Nuance (Datacolor, NJ, USA).

#### 2.4. Tensile Testing

Tensile testing was done in order to evaluate the elongation at break as well as the elastic recovery of knitted samples before and after dyeing. Measurements were carried out using tensile testing machine type Lloyd instruments LR5K and according Standard ISO 13934-1. All tests were performed using a gauge of 100 mm and speed of 300 mm/min. All results obtained are the mean values of three different measurements plus the standard deviations.

During these testing, knitted samples before and after dyeing underwent an elongation value between 20% and 50% of the elongation at break for five successive cycles, and the breaking strength after each cycle was recorded. Then, these samples are released, allowing them to return to their original state. The elastic recoveries as well as the permanent deformation are calculated according to the following equations:

Recovery, 
$$\% = \left(\frac{L_{\max} - L_i}{L_{\max}}\right) * 100$$
 (1)

Permanent Deformation, 
$$\% = \left(\frac{L_5}{L_{gl}}\right) * 100$$
 (2)

where:  $L_{max}$  is the imposed extension,  $L_i$  is the extension at zero strength for cycle index i (i = 1 to 5), and  $L_{gl}$  is the gauge length.

#### 2.5. Color Measurement

After each dyeing, the color of knitted samples was measured with a Datacolor's spectrophotometer type Spectraflash 600+ (Lawrenceville, NJ, USA). This allowed us to evaluate the color strength (K/S) and the color coordinates of dyed samples. (K/S) spectra were measured from 400 to 700 nm with 10 nm intervals. For color measurements, samples were presented with four layers and measured three times in different locations in order to minimize errors of color measurements [26,27]. So, all results obtained are the mean values of three different measurements plus the standard deviations.

#### 2.6. Color Fastness Testing

Wash, crock, and light fastnesses of dyed samples were evaluated using ISO standards numbers 105-C06, 105-X12, and 105-B02, respectively. A Suntest CPS+ machine (Atlas Material Technology, Mount Prospect, IL, USA) was used to measure the light fastness. The light intensity of its xenon lamp light is 765 W/m2 and each dyed sample was exposed for 72 h.

#### 2.7. Techniques of Characterization

#### 2.7.1. Scanning Electron Microscopy (SEM)

A scanning electron microscope (SEM) type Hitachi S-2360 (Tokyo, Japan) was used to observe and analyze the morphology of the bicomponent filaments. Characteristics of yarn and filament were evaluated using image analysis captured by a CCD video-camera.

#### 2.7.2. Fourier Transform Infrared Spectroscopy (FTIR)

Bicomponent filaments were examined using the Fourier infrared transform (FTIR) spectra. The FTIR spectra were obtained using Perkin-Elmer spectrophotometer (Waltham, MA, USA). It covers the spectral range from 400 to 10 cm<sup>-1</sup>. The acquisition conditions of this analysis are 4 scans and 4 cm<sup>-1</sup> resolution.

#### 2.7.3. Differential Scanning Calorimetry (DSC)

The thermal analysis of bicomponent filaments was carried out using Mettler machine having Stare SW 9.20 software. A temperature ranging from 0 °C to 300 °C with a heating rate of 10 °C/min, a nitrogen gas flow rate of 50 mL/min and a weight sample of 1 mg are the conditions used in this analysis.

#### 2.8. Experimental Design

Response surface methodology was used to develop and optimize dyeing process of bicomponent filaments with the three studied dyes. For this, a Box-Behnken experimental design having three factors with three levels for each factor was used. This kind of design makes it possible to obtain graphical representations concerning the effect of each variable, the interactions between them, to deduce the optimal conditions and a mathematical model between analyzed response and studied factors [28–30].

The studied parameters are: dyeing temperature, dyeing duration, and concentration of carrier in the dyeing bath. These parameters and their levels are shown in Table 3.

	Unite		Variation Levels	
ractors	Units	Low Level (-1)	Middle Level (0)	High Level (+1)
Dyeing temperature	°C	110	120	130
Dyeing time	min	25	50	75
Concentration of carrier	g/L	0	6	12

 Table 3. Factors and levels used in the Box-Behnken experimental design.

The analyzed response is the color yield (K/S) of the dyed samples. The rest of the dyeing parameters are kept constant as follows: dye concentration of 1.2%, dispersant concentration of 1 g/L, pH of dyeing bath equal to 7, and a liquor ratio equal to 10:1. The Box-Behnken experimental design thus used is shown in Table 4.

Burn Orden		Factors	
Kun Order	Dyeing Temperature (°C)	Dyeing Time (min)	Carrier Concentration (g/L)
1	110	25	6
2	130	25	6
3	110	75	6
4	130	75	6
5	110	50	0
6	130	50	0
7	110	50	12
8	130	50	12
9	120	25	0
10	120	75	0
11	120	25	12
12	120	75	12
13	120	50	6
14	120	50	6
15	120	50	6

Table 4. The three-level Box-Behnken experimental design used in this study.

#### 3. Results and Discussion

#### 3.1. Characterization of Bicomponent Filaments

#### 3.1.1. Morphological Analysis

The scanning electron microscope (SEM) is used to evaluate the surface state and the morphology of bicomponent filaments (pristine filaments). Based on Figure 3, it can be seen that filaments have latent crimps which explain the elasticity of the resulting yarn and its high elastic recovery power.

It is also observed that each filament is composed of two bonded filaments (PET and PTT) arranged side by side. These observations confirm results reported by several authors in the literature [9,11,12]. Image of the filament cross section shows larger and smaller circular surfaces; the larger one corresponds to PET filament (almost 60% of the total area) and the smaller surface corresponds to the PTT filament (almost 40% of the total area) [13].



**Figure 3.** Scanning electron micrographs of bicomponent filaments. (**a**,**b**) Configuration of filaments in the yarn; (**c**) Lengthwise view; (**d**) Cross section view.

#### 3.1.2. FTIR Analysis

Fourier transform infrared spectroscopy provides information concerning the chemical composition and identifies the characteristic functional groups of the material under study. Figure 4 shows the IR spectrum obtained for the bicomponent filaments. The existence can be observed of one strong peak attributed to esters groups at around (1712.20 cm<sup>-1</sup>).

Furthermore, another peak is present at (1408 cm<sup>-1</sup>) which corresponds to the bending vibrations of methylene. Other characteristic bands related to the chemical structure of these filaments are to be noted, namely: the coexistence of the bands which correspond to the C=C vibration in the benzene ring. On the one hand, as reported by Wu et al. [31] and Donelli et al. [32], the bands at 1016.54 cm<sup>-1</sup> and 722.83 cm<sup>-1</sup> correspond to the bending vibration of the phenylic C-H bonds. They indicated also that PET can be distinguished from PTT by the coexistence of one peak at around 1339 cm<sup>-1</sup>, which corresponds to the O-C-H bending vibration due to the arrangement trans-trans of the methylene units. These authors have demonstrated also that PTT spectrum shows one characteristic peak at 1039 cm<sup>-1</sup>, which is absent in the PET spectrum. This peak corresponds to the C-C stretching mode of the PTT's three methylene units, which are arranged in a gauche–gauche conformation. Thus, FTIR analyze can be used to confirm the presence of the two types of polyester in this bicomponent filament.



Figure 4. IR spectra of bicomponent filaments.

#### 3.1.3. DSC Analysis

Figure 5 shows the thermograph of bicomponent filaments obtained by differential scanning calorimetry (DSC). Results show the existence of two principal peaks. The first peak is located at a temperature equal to 247.27 °C. It corresponds to the melting point of the polyethylene terephthalate (PET). This is confirmed by several researchers which found that the melting temperature of pure PET is around 250 °C [13,14]. The second important peak is observed at 223.25 °C, and corresponds to the melting point of (PTT). In fact, as reported by [14] for pure PTT, the melting peak was detected at 226 °C, thus confirming that the second peak (observed in our case at 223.25 °C) corresponds well to the melting point of (PTT). In addition, the existence of a small peak can be seen at 245 °C. This peak may be due to the copolymer created at the interface between PET and PTT filaments [13].



Figure 5. Differential scanning calorimetry (DSC) spectrum of bicomponent filaments.

#### 3.1.4. Recovery Elasticity

Among the main characteristic properties of bicomponent filaments yarn, the object of the present study is its elastic recovery. Knitted samples made from 100% of multifilaments yarn are subjected to an elongation of 20%, 30%, 40%, and 50% of the breaking strength. In each case, the sample is subjected to five consecutive cycles. For each cycle, an elongation is imposed followed by relaxation to evaluate its ability to regain its initial state before excitation.

From obtained results shown in Figure 6, it can be observed that the knitted fabric made with these bicomponent filaments has excellent elastic recovery with values equal to 88.20%, 71.36%, 64.58%, and 59.98% for, respectively, 20%, 30%, 40%, and 50% elongations of that at break after five cycles. So this knit can withstand 50% of its elongation at break after 5 cycles; it is a high performance knit that can be used in the manufacture of technical garments like sportswear thanks to its elasticity and its great elastic recovery [33–35].



Figure 6. Recovery elasticity of knitted samples made of 100% bicomponent filaments.

#### 3.2. Study of Dyeing Process

Several authors have investigated the dyeing mechanism of disperse dyes on hydrophobic fibers [24,25]. They found that the substantivity of disperse dyes can be attributed to various interaction forces. Indeed, the interactions between the dye molecules and the PET and PTT chains involve the formation of hydrogen bonds. In fact, the fiber contains carboxyl groups (as proton acceptors) while the disperse dyes have hydrogen groups (as proton donors). It is true that the hydrogen bond has the predominant quasi-covalent force in the interaction between the studied fibers (PET/PTT) and the disperse dyes, but also the relative contribution of the dispersion forces, and possibly the induced dipolar–dipolar forces by the dipoles also contribute to the interaction between the fiber and the dye [24,25].

More importantly, these interaction forces between studied filaments and disperse dyes are affected by different parameters. In this context, the effect of dyeing conditions of bicomponent filaments was analyzed and the optimum process was then determined. The studied dyeing parameters are namely: dyeing temperature, pH of dye bath, dyeing duration, and carrier concentration added to the dye bath. Each dyeing parameter was varied in order to study its effect while the other parameters were kept constant. As mentioned below, three disperse dyes with different molecular weights (low, medium, and high) were used to dye bicomponent filaments. The effects of dyeing parameters were analyzed for dye concentration of 1.2%. The dyeing process is illustrated in Figure 2.

# 3.2.1. Effect of pH Dyeing Bath

To analyze the effect of pH dyeing bath on dyeing performances, a series of dyeing of bicomponent filaments with the three disperse dyes were carried out with various values of pH (4–8). In fact, the most of dyes are hydrolyzed by acids or alkalis; the hydrolyzed form of the dye may give rise to a different shade and a different affinity [36]. In some cases, some changes induced by the alkaline pH may result in permanent deterioration of the dye. [36] has concluded that the color yield obtained with disperse dyes decreases with increasing the dye bath pH; in some cases, dyeing with a pH value greater than 8 destroys the dye. In order to solve these two problems of hydrolysis and reduction of the disperse dyes encountered in dyeing of polyester fibers, several authors have proved the efficiency of the use of a buffer system [37]. As reported by [38], the dye absorption of PET fiber reaches its peak value at a pH adjusted between 5.5 and 6. Other studies have shown that PTT fiber is ideally dyeing with disperse dyes in a neutral pH [39]. So, referring to the literature, the dye bath pH was varied from an acidic value of 4 to a slightly basic value of 8 (with a step of 1). In addition, Mcilvaine

buffer solutions were used to ensure stability of the dye and avoid any problems that may affect dyeing results. This study therefore gives us the opportunity to see the behavior of both PTT and PET filaments together during dyeing so that the optimal dyeing conditions will be obtained. Resulting values of color strength (K/S) are shown in Figure 7. It can be seen that for the three disperse dyes used for dyeing the bicomponent filaments, the pH value does not have a great effect on the dyeing; (K/S) value remains almost constant according to the dye bath pH. So, in order to have a cleaner process, a pH value of 7 was chosen as the ideal value to dye bicomponent filaments.



**Figure 7.** Effect of dye bath pH on the color yield (K/S) of bicomponent filaments dyed with: (**a**) Dye #H; (**b**) Dye #M; (**c**) Dye #L (Temperature = 130 °C, Concentration of carrier = 0 g/L, Time = 50 min).

The effect of temperature on dyeing performances was studied in order to deduce the optimum value to use for dyeing bicomponent filaments. Tested temperatures were 90, 100, 110, 120, and 130 °C. Figure 8 shows the evolution of K/S of dyed samples according to dyeing temperature.



**Figure 8.** Effect of dyeing temperature on the color yield (K/S) of bicomponent filaments dyed with: (a) Dye #H; (b) Dye #M; (c) Dye #L (pH = 7, Carrier concentration = 0 g/L, Time = 50 min).

Results obtained for the high energy dye, CI dye Disperse Red 167.1, show that increasing the dyeing temperature from 100 °C to 130 °C significantly increases the color strength (K/S). In fact, as the results of thermal analysis show (DSC), the relatively high glass transition temperature of PET/PTT bicomponent filaments (Tg = 39.27 °C) and the two melting temperatures of PTT and PET (Tm = 223.25 °C and 247.27 °C, respectively) obtained with DSC analysis indicate that the dyeing of

PET/PTT bicomponent filaments can only be performed at high temperatures. At low temperatures the fibrous structure is not yet well open for dye penetration and sorption. As a result, while increasing the dyeing temperature, polymer chains in the amorphous zones are more and more mobile which allows them to create sufficiently large voids in which dye molecules can diffuse and thus the dye absorption increases [24].

In the case of disperse dye having medium energy (Dye #M), the temperature required to have the highest color yield is 120 °C. Whereas, for the low energy dye, namely CI Disperse Red 60, the color yield (K/S) reaches its maximum value at a temperature of 110 °C. Indeed, the more the molecules of the dye have a lower molecular weight and a lower polarity, the better the migration inside the fiber and thus the higher the exhaustion rate at a given time and at a lower temperature [24].

### 3.2.3. Effect of Dyeing Time

The effect of dyeing duration was studied. This allows deducing the time needed to reach the dyeing balance and to have dye saturation at the amorphous areas in the fiber. For this, bicomponent filaments were dyed using the three studied disperse dyes by varying the dyeing time from 5 min to 100 min (using a dyeing temperature of 130 °C and a neutral pH). As shown in the Figure 9, the value of the color yield reached its maximum when the dyeing time is 40, 30, and 20 min for Dye #H, Dye #M, and CI Disperse Red 60, respectively. The short dyeing time observed in the case of the Dye #L can be explained by the fact that this dye has the lowest molecular weight and has a low polarity, and therefore has a good migration power inside the fiber [24].



**Figure 9.** Effect of dyeing time on the color yield (K/S) of bicomponent filaments dyed with: (**a**) Dye #H; (**b**) Dye #M; (**c**) Dye #L (pH = 7, Temperature =  $130 \degree$ C, Concentration of carrier = 0 g/L).

#### 3.2.4. Effect of Carrier Concentration

In order to have a more economical dyeing process, the effect of adding a carrier based on phthalamides has been evaluated. The dyeing conditions are neutral pH, dyeing temperature equal to 110 °C and carrier concentration varied from 0 to 15 g/L. The results thus obtained are presented in Figure 10. It is noted that (K/S) value of samples dyed with all studied disperse dyes improves by increasing the carrier concentration to 3–6 g/L. Beyond this concentration, there is a slight decrease in the (K/S) parameter.



**Figure 10.** Effect of carrier concentration on the color yield (K/S) of bicomponent filaments dyed with: (a) Dye #H; (b) Dye #M; (c) Dye #L (pH = 7, Temperature = 110 °C, Time = 50 min).

In fact, the carrier agent plays the role of high temperatures; it breaks the internal links between the macromolecular chains of the fiber and thus facilitating the penetration of dye molecules in the fiber [24]. For concentrations ranging from 0 to 6 g/L, there is an absorption phenomenon. Beyond this value of 6 g/L, there is a desorption phenomenon observed by the decrease of (K/S) values, in particular in the cases of the Dye #L and Dye #H. In addition, when comparing results obtained in Figures 8 and 10, it can be deduced that for temperature equal to 110 °C and carrier concentration equal to 6 g/L, the (K/S) value is very close to that obtained by dyeing samples at 130 °C (without adding carrier agent). So, 20 °C can be saved when adding carrier agent.

#### 3.3. Dyeing Process Optimization

#### 3.3.1. Analysis of the Main Effects and the Interactions Plots

The evolution of the response (the color strength (K/S)) according to each of the three studied factors is shown in Figure 11.



**Figure 11.** Mains effects plots for color yield (K/S) of bicomponent filaments dyed with: (**a**) Dye #H; (**b**) Dye #M; (**c**) Dye #L.

It can be noted that the dyeing temperature has a great influence on the (K/S), especially for Dye #H and Dye #M. Indeed, in the case of Dye #H (disperse dye with high energy), it is observed that the value of (K/S) increases from 110 to 120 °C and slightly from 120 to 130 °C. In the case of Dye #M (medium energy dye), the value of (K/S) reaches its maximum at a temperature equal to 120 °C. Beyond this value, the temperature has no effect on the (K/S) response. For the dye with low energy, CI Disperse Red 60, the curve of (K/S) remains almost constant; in this case the temperature does not affect the value of (K/S).

It can also be seen that time of dyeing is also a significant parameter. The optimum dyeing time is around 50 min for the three studied dyes.

For the third factor, the concentration of the carrier agent, the color yield (K/S) increases slightly with the increase in the carrier concentration from 0 to 6 g/L. Then, it decreases considerably with the increase of the carrier concentration.

The diagram of interactions between the different studied factors is presented in Figure 12. It can be observed that there are significant interactions between dyeing parameters for the three studied dyes; this justifies the fact of using an experimental design which makes it possible to find the optimum dyeing conditions while taking into account the interactions between the various dyes factors and their levels.



**Figure 12.** Interactions plots for color yield (K/S) of bicomponent filaments dyed with: (**a**) Dye #H; (**b**) Dye #M; (**c**) Dye #L.

#### 3.3.2. Analysis of Variance

In order to verify the significance of factors and interactions for the case of the three disperse dyes, values of F (Student) and P (Fisher) coefficients are calculated and presented in Table 5. Factors are considered statistically significant if *P*-value  $\leq 0.05$  [28–30].

**Table 5.** ANOVA table of the color yield (K/S) for the studied parameters ( $R^2(adj) = 92.32\%$ , 99.83% and 94.83% for Dye #H, Dye #M, and Dye #L, respectively).

Dyes	Term	Effect	Coef. (Uncoded Data)	Coef. (Coded Data)	SE Coef.	F-Value	P-Value
	Constant	-	-276.600	22.500	0.670	33.600	0.000
	А	3.175	4.430	1.580	0.410	3.870	0.010
	В	2.050	0.665	1.020	0.410	2.500	0.050
	С	-1.275	2.230	-0.630	0.410	-1.550	0.181
Dve #H	$A \times A$	-3.375	-0.016	-1.680	0.604	-2.800	0.038
Dyc #11	$B \times B$	-3.125	-0.002	-1.560	0.604	-2.590	0.049
	$C \times C$	-5.775	-0.080	-2.880	0.604	-4.780	0.005
	$A \times B$	-1.500	-0.003	-0.750	0.580	-1.290	0.253
	$A \times C$	-1.350	-0.011	-0.675	0.580	-1.160	0.297
	$B \times C$	-0.700	-0.002	-0.350	0.580	-0.600	0.573
	Constant	-	-173.900	25.800	0.165	156.530	0.000
	А	1.500	2.830	0.750	0.101	7.430	0.001
	В	3.470	0.607	1.730	0.101	17.210	0.000
	С	2.370	2.580	1.180	0.101	11.770	0.001
Dve #M	$A \times A$	-2.150	-0.010	-1.075	0.149	-7.240	0.000
Dyc min	$B \times B$	-3.900	-0.003	-1.950	0.149	-13.130	0.000
	$C \times C$	-2.100	-0.029	-1.050	0.149	-7.070	0.001
	$A \times B$	-0.850	-0.001	-0.420	0.143	-2.980	0.031
	$A \times C$	-1.850	-0.001	-0.925	0.143	-6.480	0.001
	$B \times C$	-1.100	-0.003	-0.550	0.143	-3.850	0.012
	Constant	-	-49.300	22.270	0.614	36.260	0.000
	А	0.100	1.000	0.050	0.376	0.130	0.890
	В	1.300	0.236	0.650	0.376	1.730	0.145
	С	-3.100	1.780	-1.550	0.376	-4.120	0.009
Dve #L	$A \times A$	-0.827	-0.004	-0.410	0.554	-0.750	0.489
Dyent	$B \times B$	-1.927	-0.001	-0.963	0.554	-1.740	0.142
	$C \times C$	-8.827	-0.122	-0.410	0.554	-7.970	0.001
	$A \times B$	0.000	0.000	0.000	0.532	0.000	1.000
	$A \times C$	-0.100	-0.000	-0.050	0.532	-0.009	0.929
	$B \times C$	-2.800	-0.009	-1.400	0.532	-2.630	0.046

Where: A is the temperature of dyeing (°C), B is the time of dyeing (min), C is the concentration of carrier (g/L), F-value is the Student coefficient, and P-value is the Fisher coefficient. Factors and interactions with P-value  $\leq 0.05$  are considered significant.

By analyzing obtained values of *P* coefficient, it can be observed that the temperature and the duration of dyeing process are very significant factors for dyeing bi-component filaments with Dye #H and Dye #M and no significant for dyeing with Dye #L. The third factor which is the carrier concentration is significant for dyeing with Dye #M and Dye #L and no significant for dyeing with Dye #H.

In addition, by analyzing the coefficients of Henry's lines (correlation coefficient R<sup>2</sup>(adj)), it can be noted that the obtained values are very close to 1 (0.92, 0.99, and 0.94 for dyeing with Dye #H, Dye #M, and with Dye #L, respectively). It is therefore obvious that the obtained models for the three dyes are highly significant.

#### 3.3.3. Mathematical Models and Contour Plots

The mathematical models allowing deducing (K/S) values according to the studied factors and interactions between them are presented in Table 5 (in columns corresponding to coefficients with coded and uncoded data). Based on these models, surface plots are calculated and presented in Figure 13. Using these plots, it is possible to have an idea which areas tend to maximize the (K/S) response; i.e., areas in which the value of (K/S) are colored in dark green.



**Figure 13.** Surface plots for color yield (K/S) of bicomponent filaments dyed with: (**a**) Dye #H; (**b**) Dye #H; (**c**) Dye #L.

Table 6 shows the optimum dyeing conditions allowing maximizing the color yield (theoretical values of K/S) of PET/PTT bicomponent filament fabrics dyed using studied disperse dyestuffs.

**Table 6.** Optimal conditions obtained by Box-Behnken design and the corresponding theoretical K/S values of samples dyed with used dyes.

Dyes	K/S	Dyeing Temperature (°C)	Dyeing Time (min)	Concentration of Carrier (g/L)
Dye #H	23.03	124.55	55.80	4.96
Dye #M	26.41	120.90	59.34	8.60
Dye #L	22.60	117.53	62.87	4.48

Table 7 shows obtained values of color strength (K/S) and CIELab coordinates of bicomponent filaments dyed using disperse dyes under the optimum dyeing conditions. Each dyeing was repeated three times in three different pots to prove the reproducibility of results.

**Table 7.** Obtained values of color yield (K/S) and colorimetric coordinates <sup>1</sup> (in D65/10°) of samples dyed with used dyes under optimum dyeing conditions.

Dyes	K/S	$L^*\pm\Delta L^*$	$a^* \pm \Delta a^*$	$b^{*}\pm\Delta b^{*}$	$C^* \pm \Delta C^*$	$\Delta E_{\text{CMC}(2:1)}^2$
Dye #H	23.18	$29.37 \pm 0.32$	$46.91 \pm 0.26$	$6.00\pm0.21$	$47.29 \pm 0.32$	0.46
Dye #M	26.32	$73.63 \pm 0.16$	$11.09 \pm 0.30$	$46.51 \pm 0.09$	$47.81 \pm 0.10$	0.35
Dye #L	22.25	$33.50\pm0.47$	$56.68 \pm 0.20$	$6.69 \pm 0.39$	$57.07 \pm 0.24$	0.64

 $\overline{1}$  Mean values of three different samples dyed separately (± mean values of deviations);  $^{2}$  Mean values of color differences CMC (2:1) between three different samples dyed separately.

It can be deduced that the experimental values of (K/S) obtained under the optimum dyeing conditions (equal to 23.18, 26.32, and 22.25 for Dye #H, Dye #M, and Dye #L, respectively) coincide with that found theoretically by the experimental design (Table 6). This proved that our models for the three studied dyes are well predictable. In addition, for each studied disperse dye, very small color differences were observed between the different dyeing all lower than the unit. This proved the good reproducibility of dyeing bicomponent filaments under the optimum conditions which confirm that the obtained models are well validated. On the other hand, samples dyed under optimum conditions present a good level of evenness [40,41] as well as a good union-shade of both components (PET and PTT) of filaments. These results indicate that dyeing was well uniform throughout the textile substrates with a regular distribution of dyes on the whole samples.

According to ISO standards 105-C06, 105-X12, and 105-B02 (used for wash, crock, and light fastnesses, respectively), dyed samples were evaluated for the three studied disperse dyes. Table 8 presents results thus obtained. It is noted that Dye #H has excellent washing, rubbing, and light fastnesses due to its high molecular weight, followed by Dye #M (dye with medium molecular weight) and Dye #L (dye with low molecular weight).

Dyes	Was (ISC	h Fastness ) 105-C06)	Crock I (ISO 1	Fastness 05-X12)	Light Fastness (ISO 105-B02)
	Staining	Color Change	Dry	Wet	_ (100 100 202)
Dye #H	5	4–5	5	4–5	8
Dye #M	4	4	4–5	4–5	7–8
Dye #L	4	4	4	3–4	7–8

Table 8. Colorfastness of bicomponent filaments dyed with used dyes under optimum dyeing conditions.

#### 3.4. Study of Physical Properties of Dyed Bicomponent Filaments

After dyeing under optimum conditions, a series of mechanical tests was carried out to evaluate the stability of the dyed samples and to make sure that they keep the same starting performances, i.e., their extensibility and elastic recovery before dyeing. Table 9 summarizes the results thus obtained. It is clear that the elongation at break has decreased in the case of the three studied dyes but this decrease remains negligible and does not exceed 7% of the initial elongation of the sample before dyeing.

Similarly, it is obvious that knitted fabrics made of 100% bicomponent filaments have excellent elastic recovery since after having undergone as much elongation which is equal to 50% of that at break, we still record values of approximately 80% after the first cycle and 60% after the fifth cycle for each of the three dyes. It is important to note that the major property of articles made using 100% bicomponent filaments, i.e., its elastic recovery, remains almost constant after dyeing.

Samples	Elongation at Break (%)	Strength at Break (N)	Recovery Elasticity after 1 Cycle (%)	Recovery Elasticity after 5 Cycles (%)	Permanent Deformation (%)
Before dyeing	309.40	538.03	79.80	59.98	50.02
After dyeing with Dye #H	278.66	584.06	80.80	60.36	49.55
After dyeing with Dye #M	292.76	569.25	80.72	60.27	49.66
After dyeing with Dye #L	277.16	571.13	80.92	60.41	49.48

**Table 9.** Obtained results of elongation at break, strength at break, recovery elasticity, and permanent deformation of bicomponent filaments knitted samples before and after dyeing under optimum conditions.

# 4. Conclusions

This paper studied the dyeing process of PET/PTT bicomponent filaments using three disperse dyes (with different energy classes). The objective was to develop an appropriate dyeing process allowing optimizing dyeing performances without altering physical and chemical properties of these bicomponent filaments in particular elasticity and elastic recovery. The characterization of filaments from point of view morphology, chemical, thermal, and physical properties was carried out using different techniques such as SEM, FTIR, and DSC. Experimental designs (type Box-Behnken) were carried out to analyze the effect of dyeing conditions and then to optimize the color strength (K/S) of samples dyed with the three studied disperse dyes having different energy classes (low, medium, and high) without altering their elasticity and elastic recovery.

Based on the obtained results, an appropriate process for dyeing fabrics knitted using 100% PET/PTT bicomponent filaments has been successfully developed for each class of disperse dyes (low, medium, and high energies), and without altering the characteristic properties of the material.

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