

# EFFECTS OF PRE- AND INTERMEDIATE CAUSTICISATION ON PATTERN FORMATION AND FASTNESS PROPERTIES OF THREE- AND TWO-BATH DYEINGS OF WOVEN POLYESTER/CATIONIC DYEABLE POLYESTER/RAYON FABRICS

## POLİESTER/KATYONİK BOYANABİLİR POLİESTER/RAYON KUMAŞLARIN ÜÇ VE İKİ BANYOLU BOYANMASINDA ÖN VE ARA KOSTİKLEMENİN DESEN OLUŞUMU VE HASLIKLAR ÜZERİNE ETKİSİ

İlhan ÖZEN

*Erciyes University, Department of Textile Engineering, Kayseri, Turkey*

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### ABSTRACT

In this study, poly(ethylene terephthalate) (PET)/cationic dyeable PET (CD-PET)/viscose rayon (CV) woven fabrics were pre-treated and intermediate-treated with different amounts of sodium hydroxide (NaOH) (15, 20, 25, 30, 35, 40, and 45 g/l) at different temperatures (80°C and 100°C). Afterwards, PET, CD-PET and CV parts were dyed using three-bath (separate dyeing) and two-bath (PET and CD-PET parts together and CV part separately) methods. The effects of causticisation order and processing conditions on fabric weight, pattern, and fastness properties were investigated. The best results were obtained with pre- or intermediate-causticisation realized at 80°C or 100°C using 20 g/l or 25 g/l NaOH concentrations both in three-bath and two-bath dyeings.

**Key Words:** Polyester, Cationic dyeable polyester, Rayon, Dyeing, Causticisation, Pattern.

### ÖZET

Bu çalışmada poli(etilen tereftalat) (PET)/katyonik boyanabilir PET (CD-PET)/viskon (CV) karışımı dokuma kumaşlar farklı konsantrasyonlarda (15, 20, 25, 30, 35, 40 ve 45 g/l) sodyum hidroksit (NaOH) kullanılarak farklı sıcaklıklarda (80°C ve 100°C) ön ve ara işlemlere tabi tutulmuştur. Ardından PET, CD-PET ve CV üç banyolu (ayrı ayrı) ve iki banyolu yöntemle (PET ve CD-PET kısımlar beraber, CV kısım ayrı) boyanmıştır. Kostikleme işlem sırası ve şartlarının kumaş ağırlığına, desene ve haslık özelliklerine etkisi incelenmiştir. Hem üç banyoda hem de iki banyoda en iyi sonuçlar 80°C veya 100°C'ta, 20 g/l veya 25 g/l NaOH kullanılarak yapılan ön veya ara kostikleme ile elde edilmiştir.

**Anahtar Kelimeler:** Poliester, Katyonik boyanabilir poliester, Rayon, Boyama, Kostikleme, Desen.

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**Corresponding Author:** İlhan Özen, iozen@erciyes.edu.tr, Tel: +90 352 207 66 66, Fax: +90 352 437 57 84

### 1. INTRODUCTION

Polyester (PES) dominates the world market for synthetic textile fibers (1-3). There are various polyester fiber types such as poly(ethylene terephthalate) (PET), poly(butylene terephthalate) (PBT), poly(trimethylene terephthalate) (PTT), and modified polyester types. One of the most common modified

polyesters which finds usage as textile fiber is a PET copolymer containing a sodium salt of a dicarboxylic acid, e.g. of 5-sulfoisophthalic acid. The acidic sulfo group allows the attachment of cationic dye molecules. This copolymer is called cationic dyeable PET (CD-PET) which provides PET ionic dyeability (1).

Polyester fibres are used either as pure material or mixed with cellulose-based fibres. The selection of these fibres ensures reduced pilling, ability to absorb water, and comfort in wear resulting mainly from the use of cellulose-based fibres, as well as suitable mechanical properties such as tensile strength, abrasion resistance

and dimensional stability characteristics of synthetic fibres. The mostly used cellulose-based semisynthetic fiber is viscose rayon (CV) since it possesses versatility in processing and applications, combining the best characteristics of both natural and man-made fibres (4). Polyester fibres show a hydrophobic character, and swell to a very small extent in water, whereas hydrophilic cellulose fibres swells in water. This requires that the single fibers have to be dyed one after the other in blends of polyester and viscose (5-7). Addition of a third component makes the dyeing process more complex which requires a very high input of labor, when dark shades are to be dyed at high quality.

In literature, only PES/CV blends have been studied and considering their dyeing difficulty related studies have focused on the shortening of dyeing procedure of these blends (1,8,9). Different from other studies, we used a woven blend fabric consisting of polyester/cationic dyeable polyester/viscose rayon. In our previous study (10), we showed that causticisation and reductive cleaning processes could be combined for polyester/cationic dyeable polyester/viscose rayon fabrics and established the optimum processing conditions without sacrificing the desired properties. But in that study, causticisation were performed only at 80°C and 120°C using just 30 g/l and 45 g/l NaOH concentrations. Moreover, only three-bath dyeing process was applied. In the present study however, we extended the causticisation conditions (80°C, 100°C, and 120°C, NaOH concentrations changing between 15 g/l and 45 g/l with 5 g/l increments) and thus enabling to compare the causticisation processes realized at 80°C and 100°C and to determine the most suitable causticisation conditions. Furthermore, we investigated the effects of two-bath dyeing process in addition to the three-bath dyeing process used in our previous study.

## 2. MATERIALS AND METHODS

### 2.1. Materials

Woven polyethylene terephthalate (PET)/cationic dyeable polyethylene terephthalate (CD-PET)/viscose rayon (CV) fabrics (155 g/m<sup>2</sup>) were kindly

supplied by ASY Textile, Bursa, Turkey and used throughout this study. Yarn number used in weft (PET/CD-PET) is 150 dtex and in warp (CV) 50/2 Ne. The warp and weft densities of the pattern used are 36 cm<sup>-1</sup> and 32 cm<sup>-1</sup>, respectively. The commercial dyes used in this study were Setapers Yellow P66 (dispers dye, C.I. Dispers yellow 114), Setacryl Black FDL (cationic dye, a mixture of C.I. Basic Blue 159, C.I. Basic Red 46, and C.I. Basic Yellow 28), and Setazol Red 3BS (reactive dye, C.I. Reactive Red 195). Imerol PCJ (Clariant, Turkey) used for dyeing of PET and CD-PET is a nonionic dispersing/levelling agent. Lyocol RDN (anionic dispersing/levelling agent, Clariant, Turkey) was used for CV dyeing. Tanaterge PURA® (Tanatex, Holland) was used as anionic soaping agent. All other chemical reagents were of laboratory grade. The chemicals and dyestuffs were used as received.

## 2.2. Method

### 2.2.1. Causticisation

Sodium hydroxide (NaOH, 38°Bé) was used for the causticisation. The viscose part of the PET/CD-PET/CV blend fabrics was intended to remove partially by treating with NaOH. This treatment has been carried out at 80°C and 100°C for 30 minutes by using 15, 20, 25, 30, 35, 40, and 45 g/l NaOH (38°Bé). After this treatment the fabric was rinsed three times at 95 °C for 10 min and neutralized at 60 °C for 20 min with acetic acid. Pre-causticisation process was carried out before PET (three-bath) or PET/CD-PET (two-bath) dyeing under conditions mentioned above. Intermediate-causticisation process to be made after PET (three-bath) dyeing, was combined with reductive cleaning since both processes are performed at basic conditions thus allowing to reduce the costs related to dyeing. It is known that cationic dyestuffs are sensitive to reductive cleaning. Therefore, intermediate-causticisation process was not applied for two-bath dyeings. In order to be able to perform reductive cleaning at the same time, 6 g/l sodium dithionite (Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub>) was added to the causticisation recipe. Pre-causticized and dyed samples were subjected to a reductive cleaning additionally using the following recipe:

2 g/l NaOH and 6 g/l Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> at 80°C for 20 minutes. Afterwards, the samples were treated with acetic acid (pH:5) at 55-60°C.

### 2.2.2. Dyeing Procedures

All dyeings were carried out in IR sample dyeing machine (Termal Laboratuvar Aletleri San. ve Tic. Koll. Şti., Turkey) using 1% of dye concentration in distilled water with a liquor ratio of 10:1. The samples were either pre-causticized or intermediate-causticized.

#### 2.2.2.1. Three-Bath Dyeing

Samples were pre-washed at 70°C for 10 minutes without using a soaping agent before dyeing process. All fiber types were dyed separately in this dyeing process. Dyeing profiles for PET, CD-PET and CV are given in Figure 1, 2, and 3, respectively. Amounts of chemicals used for PET dyeing are as follows: 1 g/l acetic acid (CH<sub>3</sub>COOH), 1 g/l sodium acetate (CH<sub>3</sub>COONa), 6 g/l sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>), 1.5% Imerol PCJ®, 1.5% Setapers Yellow P66 (pH: 4-4.5). After completing the dyeing process, a reductive cleaning (pre-causticisation) or causticisation (intermediate-causticisation) was performed. Cationic dyeing recipe was as follows: 1 g/l Na<sub>2</sub>SO<sub>4</sub>, 2% Imerol PCJ®, 0.5% Setacryl Black FDL (pH: 4-4.5). Before starting the viscose dyeing, the fabric was rinsed at 70 °C. 1 g/l Lyocol RDN, 20 g/l Na<sub>2</sub>SO<sub>4</sub>, 10 g/l sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>), and 0.5% Setazol Red 3BS were used in viscose dyeing. After completion of the whole dyeing process, the samples were subjected to neutralisation with CH<sub>3</sub>COOH (pH: 6) for 10 min, to washing with soaping agent (1.5 g/l) at 85°C for 10 min, and to rinsing at 60°C for 10 min.

#### 2.2.2.2. Two-Bath Dyeing

PET and CD-PET were dyed together using the CD-PET dyeing profile and afterwards CV part was dyed. 1 g/l CH<sub>3</sub>COOH, 1 g/l CH<sub>3</sub>COONa, 6 g/l Na<sub>2</sub>SO<sub>4</sub>, 2% Imerol PCJ®, 1.5% Setapers Yellow P66, and 0.5% Setacryl Black FDL were added to the dyebath (pH: 4-4.5). Chemical amounts for dyeing of CV moiety were the same as the three-bath dyeing.

## 2.2.3. Characterization

### 2.2.3.1. Weight Reduction

The weight loss of the fibres was determined. The total weight loss of the samples was measured as the difference in the weight of the fabric sample before and after treatment. Details related to the test procedure can be found elsewhere (10).

### 2.2.3.2. Rubbing Fastness Tests

Color fastness to rubbing (dry/wet) was determined according to ISO 105 X12:2001. The color fastnesses were assessed according to ISO 105 A03 by

using a grey scale in light box which was illuminated with D65-daylight.

### 2.2.3.3. Optical Microscopy

In order to determine the optimized amount of NaOH and to make the patterns formed distinctive, images of the causticized and dyed samples were taken by using an Olympus SZ61-TR optical microscope.

## 3. RESULTS AND DISCUSSION

The pre- or intermediate-causticized and dyed samples using three-bath and two-bath were characterized in terms of weight reduction, appearance, and rubbing fastness properties. Weight loss

values of the treated samples are given in Table 1. Considering the three-bath dyeing and pre-causticisation, mass loss of the pre-treated fabrics was determined to increase with increasing amount of sodium hydroxide. There was no appreciable differences observed between treatment temperatures of 80°C and 100°C. The same trend was detectable in the intermediate-causticized samples and almost no difference was observed between pre- and intermediate-causticisation. The samples dyed using the two-bath dyeing process represent the same situation but showing less weight loss meaning less fiber damage.

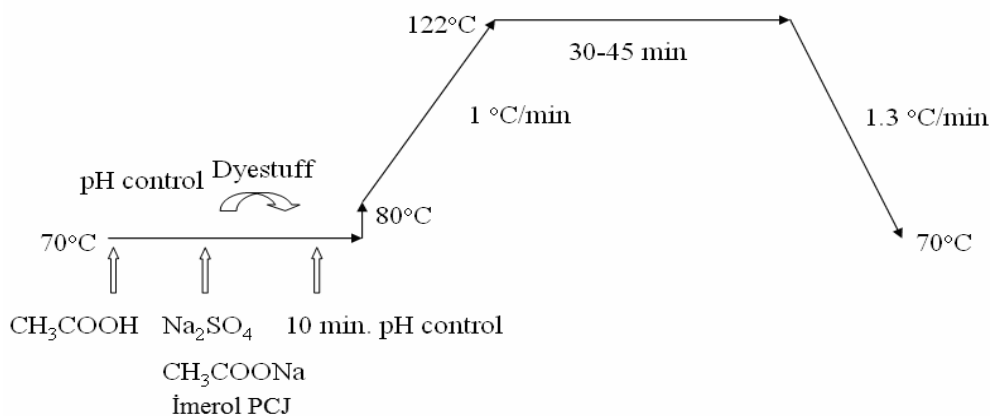


Figure 1. Dyeing profile for PET

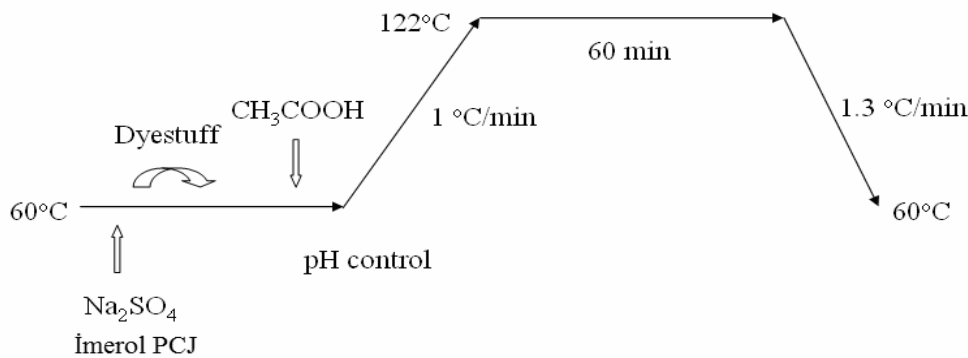


Figure 2. Dyeing profile for CD-PET

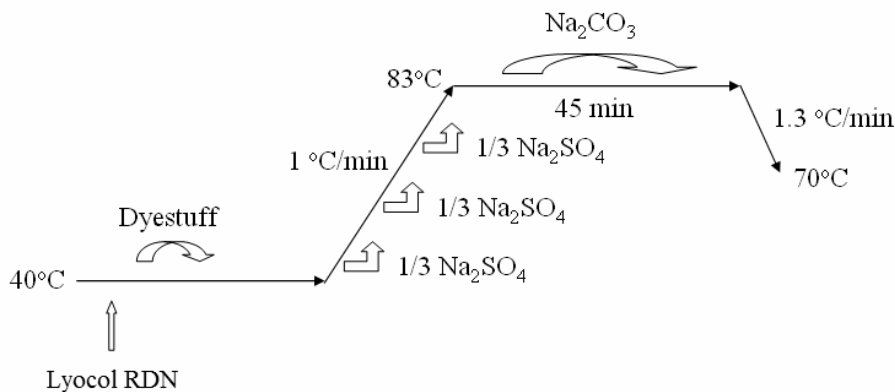


Figure 3. Dyeing profile for CV

**Table 1.** Weight loss values of the causticized (pre- or intermediate-) and dyed (three- or two-bath) PET/CD-PET/CV blend fabric samples

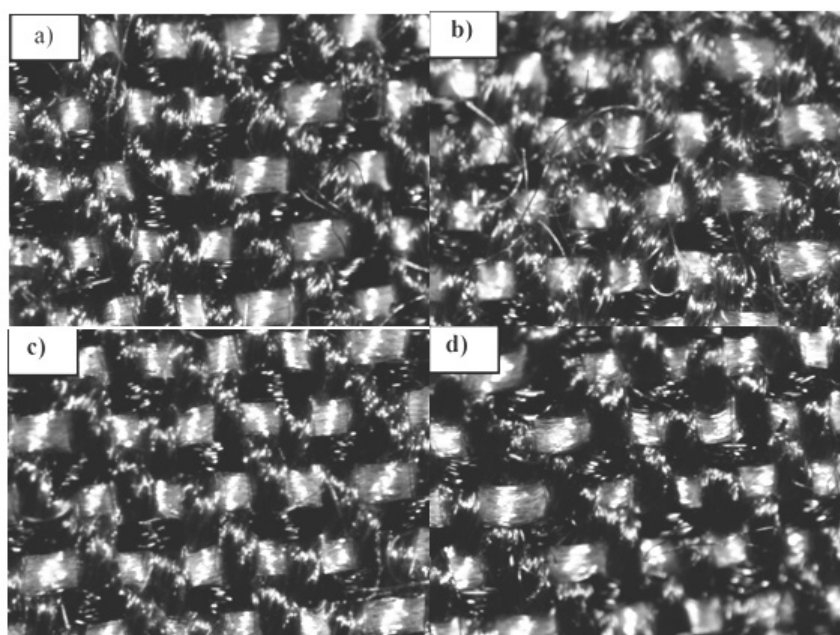
Dyeing Conditions	NaOH (g/l)	Weight loss (%)			
		Pre-causticisation		Intermediate-causticisation	
		80°C	100°C	80°C	100°C
3-bath	15	1.8	2.1	2.2	4.1
	20	2.0	2.2	2.4	4.4
	25	1.8	3.3	2.3	4.3
	30	3.6	4.1	2.9	4.8
	35	5.4	6.2	4.8	6.6
	40	6.8	6.6	5.7	8.4
	45	9.5	9.4	7.9	11.5
2-bath	15	2.0	2.1	-*	-*
	20	2.1	2.7	-*	-*
	25	1.9	3.6	-*	-*
	30	4.5	4.3	-*	-*
	35	6.2	6.5	-*	-*
	40	7.3	6.9	-*	-*
	45	9.7	10.4	-*	-*

\*: not available

In order to determine the pattern formation of the dyed fabrics, optical microscopy images were taken. The most striking images are given in Figure 4. The pattern formation in pre-causticized at 80°C and two-bath dyed samples was improved in comparison to the untreated (Figure 4,a) and the treated fabric samples with 15 g/l NaOH (Figure 4,b) when employed a NaOH concentration of 20 g/l (Figure 4,c) or 25 g/l (Figure 4,d) by showing a more distinctive PET part (yellow color). On the other hand, fabric patterns were worsened as of 30 g/l NaOH

concentration. Pre- or intermediate-treated samples at 100°C delivered the same results. PET/CD-PET/CV fabrics pre- or intermediate-causticized and dyed using three-bath presented the same results in terms of pattern formation. According to the light microscopy images, the most suitable conditions seem to be the one in which the fabric was pre- or intermediate-treated at 80 °C by using a sodium hydroxide concentration of 20 g/l or 25 g/l and dyed using three- or two-bath dyeing process.

The causticized (pre- or intermediate) and dyed (three- or two-bath) fabric samples were subjected to dry/wet rubbing fastness measurements and their results are depicted in Table 2. Wet rubbing fastness values are given in parantheses. Independent upon the dyeing process, causticisation order, and causticisation conditions, all samples gave a dry rubbing fastness value of 5. It can be said that all samples delivered nearly the same wet rubbing fastness values being either 4/5 or 5.



**Figure 4.** Micrographs of the pre-causticized and two-bath dyed PET/CD-PET/CV blend fabric samples. Abbreviations: a) Sample without causticisation, b) Pre-causticisation at 80°C with 15 g/l NaOH (38°Bè), c) Pre-causticisation at 80°C with 20 g/l NaOH (38°Bè), d) Pre-causticisation at 80°C with 25 g/l NaOH (38°Bè), (Magnification: 10x3)

**Table 2.** Rubbing fastness (dry/wet) values of the causticized (pre- or intermediate-) and dyed (3- or 2-bath) PET/CD-PET/CV blend fabric samples

Dyeing Conditions	NaOH (g/l)	Rubbing fastness (dry/wet)			
		Pre-causticisation		Intermediate-causticisation	
		80°C	100°C	80°C	100°C
3-bath	0	5 (5)	5 (5)	5 (5)	5 (5)
	15	5 (5)	5 (4/5)	5 (5)	5 (5)
	20	5 (4/5)	5 (4/5)	5 (5)	5 (5)
	25	5 (5)	5 (4/5)	5 (4/5)	5 (4/5)
	30	5 (4/5)	5 (4/5)	5 (4/5)	5 (4/5)
	35	5 (4)	5 (4)	5 (5)	5 (4/5)
	40	5 (4)	5 (4/5)	5 (4/5)	5 (5)
	45	5 (3/4)	5 (4)	5 (5)	5 (4/5)
2-bath	0	5(4/5)	5(4/5)	-*	-*
	15	5 (4/5)	5 (5)	-*	-*
	20	5 (5)	5 (5)	-*	-*
	25	5 (5)	5 (3/4)	-*	-*
	30	5 (4/5)	5 (5)	-*	-*
	35	5 (4/5)	5 (4)	-*	-*
	40	5 (4)	5 (3)	-*	-*
	45	5 (4/5)	5 (3/4)	-*	-*

\*: not available

#### 4. CONCLUSION

We have established the optimum temperature (80°C) and optimum amount of sodium hydroxide concentration (20 g/l) for causticisation process in woven poly(ethylene

terephthalate) (PET) / cationic dyeable poly(ethylene terephthalate) (CD-PET) / viscose rayon (CV) fabrics. And it was revealed that both the causticisation treatment/reductive cleaning and dyeing of PET/CD-PET parts in the same bath could be

combined for less fiber damage and cost savings without sacrificing pattern formation and fastness properties. These quasi-yarn dyed fabrics can be used mainly in production of trousers.

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