

# Reducing Steaming Times in the Printing of Nylon/Elastane Blend Fabrics via Chemical Modification

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## Abstract

Nylon fibres are commonly used in blends with elastane. One of the problems encountered in the printing of nylon/elastane blend fabrics is the necessity of long steaming times for fixation, which causes an increase in energy consumption. In this study, the possibility of printing polyamide fabrics with 1:2 metal complex dyes for short steaming times was investigated. For this aim, laboratory and sample scale trials were performed to develop a method of chemically modifying fibres so that polyamide fabrics can be printed in short steaming times. Based on the results obtained in both trials, it was noticed that the steaming time for fixation could be reduced from 30 minutes to 15 minutes by printing with 1:2 metal complex dyes without causing a loss of printing yield and without affecting the fastness adversely in case of applying cationisation pretreatment with a modified quaternary polyalkylamine compound based product.

**Key words:** nylon/elastane blend fabric, printing, cationisation pretreatment, colour, fastness.

## Introduction

The first synthetic fibre produced by synthesis in the world is polyamide (PA). Nylon (PA 6,6) and Perlon (PA 6) fibres are the most common types of globally available PA fibres. Nylon was experimentally discovered in 1938 in the United States by Wallace H. Carothers, and in the year 1939 DuPont began a pilot production of this fibre. At the same time, Perlon fibre was also discovered by the German scientist Prof. Dr. Paul Schlack [1]. PA 6,6 is obtained by the polycondensation of hexamethylene diamine [ $\text{H}_2\text{N}-(\text{CH}_2)_6-\text{NH}_2$ ] with adipic acid [ $\text{HOOC}-(\text{CH}_2)_4-\text{COOH}$ ], while PA 6 fibres are obtained from caprolactam [ $\text{C}_6\text{H}_{11}\text{NO}$ ] [2]. The importance of synthetic fibres is increasing day by day due to the wide range of their application. PA fibres are an important synthetic fibre group used in many fields as carpet, clothing and tyres as reinforcement materials [3].

PA fibres can be dyed with acid, chromium and metal complex dyes, as well as disperse dyes due to their hydrophobic structure [4]. Acid and 1:2 metal complex dyes used in the dyeing of polyamide fibres can also be applied for printing. Printed and dried fabrics are rinsed and washed after 20-30 minutes of steaming for fixation. It is beneficial to use an anionactive reserving agent with affinity to the fibre in order to prevent the white background from getting stained by blocking the amino groups in the places that are not being printed during the washing process [5].

As is known, nylon fibres are commonly used in blends with elastane. One of the problems encountered in the printing of nylon/elastane blend fabrics is the necessity of long steaming times for fixation, which cause an increase in the energy consumption of the process. In this study, the possibility of printing polyamide fabrics with 1:2 metal complex dyes for short steaming times was investigated. Laboratory and sample scale trials were performed in order to develop a chemical modification method so that polyamide fibres can be printed for short steaming times. The amount of anionic dyes that polyamide fibres can bind to their structure is limited, since free amino groups are present only at the ends of the macromolecules. If the number of cationic groups in their structure can be increased by chemical modification, an increase in their affinity to anionic dyes could be obtained compared to untreated fibres. Although there are many studies in the literature on the cationisation of cellulosic fibres [6-16], studies on the cationisation of polyamide fibres are limited, and to the best of the authors' knowledge, till today there has been no research on the effect of cationisation on the printability of polyamide fabrics.

In the study carried out by Khalfaoui *et al.*, experimental adsorption isotherms of four dyes (Acid Blue 25, Acid Yellow 99, Reactive Yellow 23, and Acid Blue 74) from aqueous solution on cationised PA 6.6 were analysed using a double layer adsorption model. The parameters involved in the analytical expression of this model, such as the number or fraction

of adsorbed dye molecules per site, the number of receptor sites per gram of adsorbent, and the concentration at half-saturation were determined from adsorption isotherms at four temperatures between 293 and 353 K. The evolution of these parameters with temperature is discussed in relation to the adsorption process and the behaviours of the different dyes, taking into account their particular structure. The results are compared with those already published dealing with the adsorption of these same dyes onto cationised cotton. It was found that a double-layer adsorption model was best at describing the experimental adsorption isotherms for all of the dyes. The n plots showed that all the dyes are anchored parallel to the surface even if the temperature varies [17].

Bahtiyari investigated the dyeability of polyamide fabrics treated with a cationisation agent using the box Behnken statistical design. It was found that colour efficiency values were increased when dyeing treated samples with reactive dyes, and the fastness values were not adversely affected [18].

El-Molla *et al.* investigated the usage possibility of the cationisation process with agents having different cationic groups (Solfix E, Tinofix ECO, Acramine Berfix K and cetyl trimethyl ammonium bromide) in the dyeing of cotton and PA 6 fabrics with commercially available anionic dyes. Both the impregnation and exhaust methods were used in the study. Fabrics cationised with a commercial product of quaternary ammonium structure showed better colour yield in all dye-

**Table 1.** Paste recipes.

Stock thickening paste		Printing paste	
Thickening agent	120 g/kg	Nyloset Navy Blue M-BR	18 g/kg
Anti-foaming agent	20 g/kg	Levelling agent	30 g/kg
Ammonium sulphate	120 g/kg	Urea	30 g/kg
Water	740 g/kg	Stock thickening paste	461 g/kg
		Water	461 g/kg

ing conditions than other products. It was found that the cationic process improves the colour fastness of cotton and PA 6 fabrics as well as their colour depth [19].

## Materials and method

All experiments were carried out using an 84/16 nylon/elastane blend plain knitted fabric (260 g/m<sup>2</sup>). In laboratory and sample scale production experiments, pure and soft mill water were used, respectively.

Fabric samples subjected to cationisation treatment and untreated ones were printed using a 1:2 metal complex dye (Nyloset M, Setaş Inc.). In these trials, the cationisation process was carried out at the conditions of a previously published study [20] with a modified quaternary polyalkylamine compound based product. Treatment conditions were as follows: 5% cationisation agent, liquor ratio 1:15, pH 7, 30 min., 60 °C. This process was carried out on an HT jet dyeing machine with a capacity of 20 kg. ATR/FTIR measurements were carried out to determine the changes in the functional groups of the treated fibres. In addition, SEM photographs were also taken to observe if there was a change in the surface structures or not.

The fabrics were printed with paste prepared in a specified manner (Table 1) and then subjected to fixation by saturated steam for 15 and 30 minutes.

The colour yield (K/S) and CIE L\*a\*b\* values of the printed fabrics were measured using a spectrophotometer, and the average of 3 measurements were taken. The colour fastness properties of the printed fabrics with respect to washing, rubbing and light were all tested.

Optimum conditions determined in laboratory scale experiments were also tested in sample scale industrial production. For this purpose, fabric samples subjected to cationisation treatment and untreated ones were printed using a 1:2 metal complex dye according to the recipe given be-

low by the Serhas Tekstil Company located in Ergene-Tekirdağ, Turkey (Table 2).

In sample scale production experiments, the fixation of printed and dried fabrics with saturated steam was done for 30 minutes for untreated fabric and 15 minutes for fabric which was subjected to cationisation. After steaming, fabric samples were subjected to 5 cycles of cold washings with 0.5 g/l NaOH and 0.5 g/l Setawash QW (cationic washing soap). The duration of each washing cycle was 10 minutes, after which neutralisation was performed. Afterwards, color yield (K/S) and CIE L\*a\*b\* values of the printed samples were measured by spectrophotometer and the average of 7 measurements were taken. In addition, washing, rubbing and light fastness tests were also performed according to ISO standards.

**Color measurements:** The reflectance (R) and CIE L\*a\*b\* values of the dyed samples were measured with a Datacolor SF600 Plus C-T reflectance spectrophotometer under illumination/observer conditions set at D65/10°. Measurements were carried out at 630 nm, which was the wavelength of maximum absorption. Then the colour yield, expressed as K/S, was calculated from the Kubelka-Munk Equation (1), given below:

$$K/S = (1 - R)^2/2 * R \quad (1)$$

R = reflectance of dyed sample measured at 630 nm ( $\lambda_{max}$ )  
 K = absorption coefficient  
 S = scattering coefficient  
 The total colour difference ( $\Delta E$ ) values were calculated according to Equation (2) below:

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \quad (2)$$

L\* = lightness-darkness value of the colour  
 a\* = value + a\* represents redness and value -a\* - greenness  
 b\* = value + b\* represents yellowness and value -b\* - blueness.

**Determination of colour fastness to washing:** Colour fastness determination

**Table 2.** Chemicals and auxiliaries used for printing with 1:2 metal complex dye.

Nyloset Black MRX	50 g/kg
Thickening agent (Guar)	60 g/kg
Citric acid	5 g/kg
Urea	50 g/kg
Ammonium sulphate	20 g/kg
Levelling agent	50 g/kg
Water	765 g/kg

of the dyed samples with respect to washing was carried out according to the ISO-105 C06 [21] standard test method. For this aim, the sample was adjusted for testing by sewing a multifibre (4 cm x 10 cm) on one side of it, and then it was treated with a detergent solution of 4 g/l for 30 minutes at 40 °C. Afterwards, results were evaluated with the grey scale.

**Determination of colour fastness to rubbing:** Colour fastness determination of the dyed samples with respect to rubbing was carried out in a dry and wet state with a crockmeter (Prowhite) according to the ISO 105-X12 [22] standard test method and evaluated with the grey scale.

**Determination of colour fastness to light:** Determination of the colour fastness of the dyed samples with respect to light was made according to the ISO 105 B02 [23] standard test method on an SDL Atlas brand 150S + PLUS model light fastness test device and evaluated with the standard blue scale.

**Fourier-Transform Infrared (FTIR) analysis:** FTIR analyses of samples were carried out using a Bruker brand Vertex 70 ATR model Fourier transform infrared spectrophotometer in order to detect changes in the functional groups of the fibres as a result of the cationisation process.

**Scanning Electron Microscopy (SEM) analysis:** SEM photographs of samples were taken at 5000x magnification using FEI brand Quanta FEG 250 model scanning electron microscope to determine whether there was a change in the surface structure of the fibres after cationisation.

## Results and discussion

Colour yield (K/S) values of the fabric samples subjected to cationisation treatment and untreated ones printed using a 1:2 metal complex dye, on a laboratory scale, are given in Figure 1.

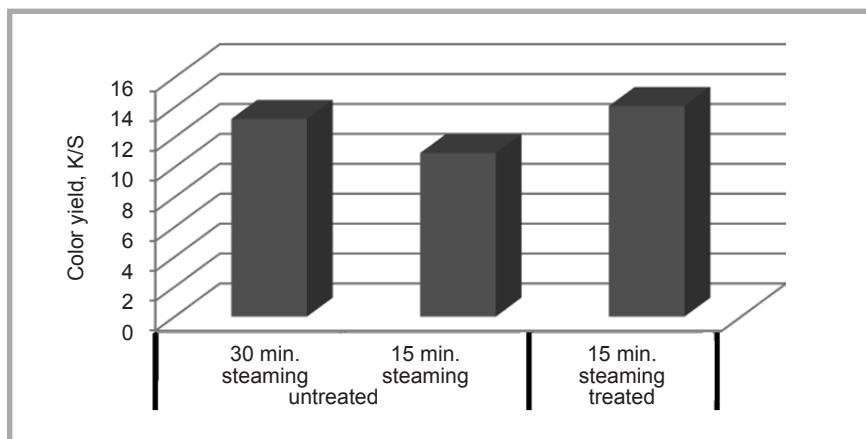
From **Figure 1** it can be seen that if the steaming time is reduced from 30 to 15 minutes, the colour yield decreases by approximately 15%. However, for the sample which was subjected to cationisation prior to printing, nearly the same colour yield can be achieved even after 15 minutes of steaming as compared to the untreated sample steamed for 30 minutes. CIEL\*a\*b\* values of the printed fabrics are given in **Table 3**.

From **Table 3** it can be clearly seen that the L\* value of the untreated fabric sample increases if the steaming time is reduced from 30 to 15 minutes showing, that the colour is getting lighter. Furthermore, it is visible that for the sample which was subjected to cationisation prior to printing, after 15 minutes of steaming the L\* value is lower, which means that its colour is darker compared to the untreated sample steamed for 30 minutes. Based on the results obtained, it can be concluded that the changes in the nuance of the colour are quite low. Results of the colour fastness of the printed fabric samples on a laboratory scale are given in **Table 4**.

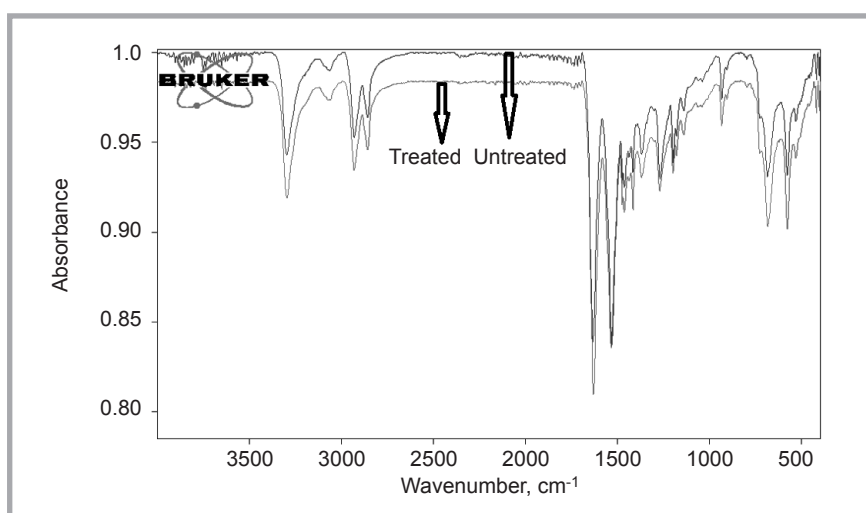
From **Table 4** it can be seen that very good light, washing and rubbing fastness values were obtained in all trials. The cationisation process did not have any negative effect on colour fastness values.

FTIR analyses were carried out in order to detect the changes in the functional groups of fibres as a result of the cationization process, results of which are summarised in **Figure 2**.

It is observed that there is an increase in the band frequencies of the carbon-hydrogen single bond (2910-2930 cm<sup>-1</sup>), amid I (C = O stretching) (1620-1640 cm<sup>-1</sup>), amid II (NH twist) (1510-1530 cm<sup>-1</sup>) and amid III (NH bending/CN stretching) (1230-1240 cm<sup>-1</sup>) in the samples treated with a cationizing agent [24, 25]. In addition, the peaks around 3300 cm<sup>-1</sup> show free and hydrogen bonded N-H stretching. This corresponds to the NH groups in the polyamide structure [18], and it can be said that an increase occurred in these groups after cationisation. Considering the chemical structure of the cationisation agent used in the experiments (**Figure 3**), it can be understood that an increase will occur in the aforementioned groups as a result of their addition to the fibre structure. The possible binding mechanism of the modified quaternary



**Figure 1.** Colour yield (K/S) values of the fabric samples subjected to cationisation treatment and untreated ones printed using a 1:2 metal complex dye (Nyloset Navy Blue M-BR).



**Figure 2.** FTIR analysis results of untreated and treated fabric samples.

**Table 3.** CIEL\*a\*b\* values of the fabric samples subjected to cationisation treatment and untreated ones printed using 1:2 metal complex dye (SD: Standard deviation).

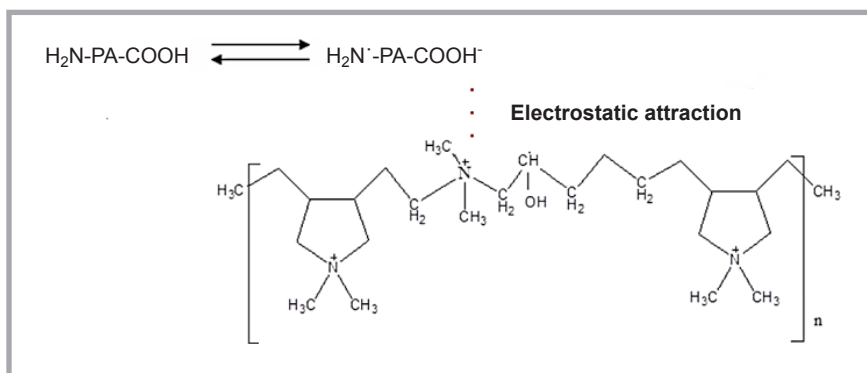
Fabric sample	Steaming time, min	L*	SD	a*	SD	b*	SD
Untreated	30	26.56	0.29	0.67	0.02	-15.07	0.11
Untreated	15	29.10	0.39	0.39	0.06	-15.51	0.09
Treated	15	25.85	0.10	0.57	0.05	-15.10	0.04

**Table 4.** Light, washing and rubbing fastness values of fabric samples subjected to cationisation treatment and untreated ones printed using a 1:2 metal complex dye.

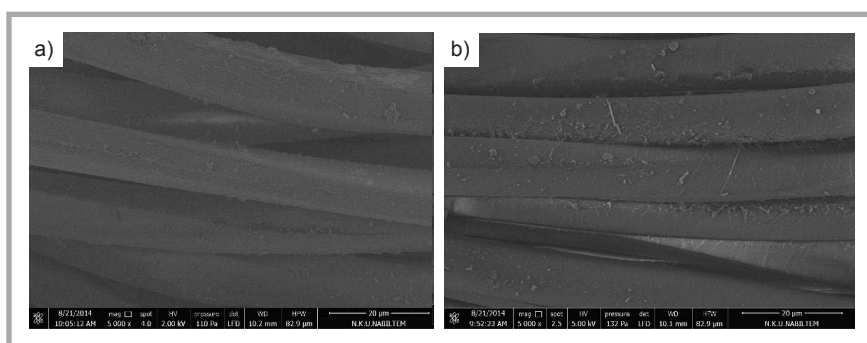
Fabric sample	Steaming time, min	Light fastness	Washing fastness						Rubbing fastness	
			CA	CO	PA	PES	PAN	WO	Dry	Wet
Untreated	30	6	5	5	4-5	5	5	5	5	4-5
	15	5-6	5	5	4-5	5	5	5	5	4-5
Treated	15	5-6	5	5	4-5	5	5	5	5	4-5

**Table 5.** K/S and CIE L\*a\*b\* values of printings carried out in sample scale industrial production with Nyloset Black MRX dye (SD: Standard deviation).

Fabric sample	Steaming time, min	K/S	SD	L*	SD	a*	SD	b*	SD
Untreated	30	1.63	0.02	24.32	0.19	-0.49	0.01	-2.87	0.03
Treated	15	1.64	0.03	23.,70	0.40	-0.53	0.01	-3.07	0.04



**Figure 3.** Possible binding mechanism of modified quaternary polyalkylamine-based cationizing agent to PA fibres.



**Figure 4.** SEM (5000x) analysis results of untreated fabric samples (a) and those treated with a cationisation agent (b).

**Table 6.** Light, washing and rubbing fastness values of printings carried out in sample scale production with Nyloset Black MRX dye.

Fabric sample	Steaming time, min	Light fastness	Washing fastness					Rubbing fastness		
			CA	CO	PA	PES	PAN	WO	Dry	Wet
Untreated	30	5	4-5	5	3-4	5	5	5	5	4-5
Treated	15	5	4-5	5	4	5	5	5	5	4-5

polyalkylamine compound based cationizing agent to the polyamide fibres is given in **Figure 3**.

As can be seen from **Figure 3**, the product based on the modified quaternary polyalkylamine compound is bonded to the carboxylate anions (–) in the polyamide fibres through electrostatic attraction forces. In this respect, new cationic groups are added to the structure of the polyamide fibres, and the amount of anionic dyes which the fibre is able to bind is increased.

After determining the changes in the chemical structure of the fibres with FTIR analyses, scanning electron microscopy (SEM) analyses were carried out to determine whether there was a change in the surface structure of the PA fibres or not. The results obtained are given in **Figure 4**.

As can be seen from the SEM photographs presented in **Figure 4**, the chemical modification did not change the surface structure of the PA fibres. Cationisation is a chemical modification in which new functional groups are attached to the fibres, and it is not expected to cause a significant change in the crystallinity or surface structure of the fibres.

It is determined in laboratory scale studies that steaming times can be reduced from 30 minutes to 15 minutes if cationization is performed before printing. However, the results should also be checked in industrial conditions. For that purpose, fabric samples treated by the cationisation process and untreated fabric samples were printed with a 1:2 metal complex dye (Nyloset Black MRX) under industrial conditions as well. The fixation of printed and dried fabrics with saturated steam was performed for 30 minutes for

untreated fabric and 15 minutes for fabric subjected to cationisation. K/S and CIE L\*a\*b\* values of the fabric samples produced under industrial conditions are given in **Table 5**.

From **Table 5** it can be seen that the fabric sample which was subjected to cationisation prior to printing gives nearly the same colour yield (K/S) and lightness-darkness (L\*) values after 15 minutes of steaming as compared to the untreated sample steamed for 30 minutes. Furthermore, it can be concluded that the changes in the nuance of the colour are quite low. Colour fastness results of the printed samples are given in **Table 4**. If the nuance of the colours of both samples is compared, it can be said that the difference between them is quite small. When the total colour difference ( $\Delta E$ ) value is calculated from CIE L\*a\*b\* values, it is found to be 0.2, which indicates that the colours of both samples are very similar. Colour fastness test results related to the printings carried out in sample scale production are summarised in **Table 6**.

From **Table 6** it is visible that very good light, washing and rubbing fastness values were obtained in both trials. All these results reveal that after cationisation, the steaming times of polyamide fabrics after printing can be shortened from 30 minutes to 15 minutes without any loss in colour yield and fastness values.

## Conclusions

In this study, analysis was conducted with the aim of developing a method of chemically modifying polyamide fibres in order that nylon/elastane blend fabrics can be printed with short steaming times. In both laboratory and sample scale trials, it was determined that it would be possible to reduce the steaming times from 30 minutes to 15 minutes without causing a loss of yield in printing and without affecting the fastness adversely in the case of applying cationisation pretreatment with a 5% modified quaternary polyalkylamine compound based product at pH 7, 60 °C for 30 min to nylon/elastane blend fabrics before printing with a 1:2 metal complex dye. The total colour difference between the treated fabric sample with a short-term steam fixation (15 min) and the untreated sample with a long-term steam fixation (30 min) after printing with a 1:2 metal complex dye remained within the acceptance limits.

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