

AMINODDIN HAJI<sup>1</sup>  
REZA M.A. MALEK<sup>2</sup>  
FIROOZMEHR MAZAHERI<sup>2</sup>

<sup>1</sup>Department of Textile, Birjand  
Branch, Islamic Azad University,  
Birjand, Iran

<sup>2</sup>Textile Department, Amirkabir  
University of Technology, Hafez  
St., Tehran, Iran

SCIENTIFIC PAPER

UDC 677.494.674:677

DOI 10.2298/CICEQ101205022H

## COMPARATIVE STUDY OF EXHAUSTION AND PAD-STEAM METHODS FOR IMPROVEMENT OF HANDLE, DYE UPTAKE AND WATER ABSORPTION OF POLYESTER/COTTON FABRIC

*In this study, a pad-steam process for treatment of polyester/cotton fabric with sodium hydroxide is developed and the effects of process parameters on selected properties of the fabric are investigated. The results are compared with the conventional exhaustion process. Both processes improved the handle, dyeability and water absorption of the polyester/cotton fabric, but the pad-steam process has the advantages of less strength loss of the fibers, shorter treatment time, lower consumption of water and chemicals that make the process less hazardous to the environment.*

*Key words: polyester; dyeability; weight reduction; cotton; sodium hydroxide.*

Aside from the outstanding features of polyester fabrics such as tensile strength, dimensional stability, washability and abrasion resistance, certain drawbacks such as low water absorbency, pilling, and poor handle have also been reported. Blending polyester with cellulosic fibers is one way to overcome the drawbacks, so that better water absorbency, handle and much more comfort can be achieved [1]. Polyester/cotton fabrics are widely used in the textile industry as they combine the advantages of both components that they are built of. By treating polyester/cotton fabrics in alkaline conditions (using sodium hydroxide) further improvement in the fabric properties can be obtained [2-5].

Alkali treatment of polyester fabric is a well-known and conventional process in textile industry [3]. Sodium hydroxide can hydrolyze the ester groups in the polyester chains. Several researchers have found different mechanisms for alkaline hydrolysis of polyester fiber [1,2]. The weight reduction of polyester ranging between 15 and 30%, gives a silky handle, luster, soil repellency and anti static properties to the polyester fabric [4], and its stiffness, tenacity and elongation are decreased [4-6,12]. Although alkaline hydrolysis decreases the diameter of the fibers, the

density of the fibers is not affected [7-11]. It has been stated by some researchers that the rate of polyester hydrolysis can be improved by using certain accelerants [13].

Xu and Yang have used the microwave irradiation to increase the rate of hydrolysis of polyester with sodium hydroxide [14]. Raslan and Bendak have concluded that the use of sodium hydroxide in alcoholic media is more effective than sodium hydroxide aqueous solution [15]. Konovalova has shown that magnetic activation of the water media can increase the hydrolysis rate of polyester [16]. Kimizu and co-workers have reported that under high pressure conditions (over 100 MPa) the decomposition of PET fabric will be resulted [17].

A wide range of studies has been carried out to investigate the effect of alkali on polyester/cotton fabric [18]. However, no one has worked on continuous processes such as pad-steam in this subject. In this study, a pad-steam process is proposed and compared with the conventional exhaustion method. Due to its lower costs, shorter required time, fewer chemicals used, much less environmental pollution and higher production [19], continuous processes can be a solution, as stated in this work.

## EXPERIMENTAL

### Materials

Plain weave polyester/cotton (67/33) fabric (123 g/m<sup>2</sup>, yarn count, Nm = 40, 50 warp/cm, 30 weft/cm)

Corresponding author: A. Haji, Islamic Azad University, Ghafari St., Birjand, Iran.

E-mail: ahaji@iaubir.ac.ir

Paper received: 5 December, 2010

Paper revised: 14 June, 2011

Paper accepted: 15 June, 2011

was obtained from Brujerd Textile Co., Iran. Tinegal PAC (quaternary ammonium salt), Ultravon CN (non-ionic surfactant) and Invadine VB (a weakly anionic wetting agent), Terasil blue BGE, Terasil black SRL-01 (disperse dyes), Cibacron turquoise P-GR and Cibacron black P-GR (reactive dyes) was kindly supplied by Ex-Ciba, Iran. All other chemicals used in this work were laboratory grade reagents obtained from Merck.

## Methods

To remove any natural or synthetic impurities, all fabric samples were scoured using a solution containing Ultravon CN (2 g/l) and sodium carbonate (1 g/l) for 20 min at 60 °C then rinsed and dried. Finally, the samples were heat set at 170 °C for 30 s in a laboratory stenter.

The alkali treatment was processed by the following methods:

1) *Exhaustion*. Like the conventional process, the samples were immersed in a sodium hydroxide solution (10 up to 40 g/l) at 50:1 liquor ratio. The initial temperature was 40 °C and the temperature was raised to boil in 30 min and then the boiling time was determined and reported. After the appropriate boiling time (1 h), washing, neutralization with 1% v/v acetic acid solution and final rinse of the samples followed.

2) *Pad-steam*. The samples were padded in a bath containing sodium hydroxide (5-30 g/l), Tinegal PAC, Invadine VB and urea, at 100% pick up. Steaming process at 120 °C was practiced. Then washing, neutralization and final rinse of the samples were followed.

The weight loss ( $WL$ ) is expressed as relative  $WL$  according to the equation:

$$WL = 100(W_1 - W_2)/W_1$$

where  $W_1$  and  $W_2$  are the weights of the samples before and after alkaline treatment, respectively.

Hydrolysis of PET can be controlled to affect fabrics at varying levels, *i.e.*, from surface hydrolysis to extensive removal of the constituent polymer. In this study, to avoid from imposing significant changes in the bulk of PET fiber, and restrict the hydrolysis to the surface of the fiber, a weight loss between 15 to 20% has been considered as optimum weight reduction value.

## Physical properties measurement

The tensile strength of treated sample fabric was determined on an Instron tensile tester model TM-SM 1026 according to ASTM D5034-95 (2001) and wicking test was measured according to BS 3424: part 18, method 21A.

Stiffness, or limpness, of a fabric can be determined by measuring bending length. Flexural rigidity of fabric is proportional to the product of fabric areal density and bending length to the third power [9]. To determine fabric handle, bending length of the fabric was measured by a Shirley tester according to Booth [20], and the bending stiffness of the samples was calculated using the following equation:

$$BS = 1000WC^3 \quad (1)$$

where  $BS$ ,  $W$  and  $C$  are bending stiffness, weight of 100 cm<sup>2</sup> of fabric in g and bending length in cm, respectively.

## Wicking test

The experimental setup described in reference [21] was used for the measurement of vertical wicking of fabrics. The 1.5 cm×7 cm strips of samples were lowered to touch the water reservoir, at which moment the timer was started, and the position of water front line (cm) over time was recorded [21].

## Dyeing and color measurements

Alkali treated and untreated PET/cotton fabrics were dyed using the following recipe.

Padding at 75% pick up with a solution containing: 1 g/l disperse dye, 1 g/l reactive dye, 2 g/l Invadine LUN, 50 g/l urea, 5 g/l Invadine DP, 5 g/l Irgapadol MP and 15 g/l sodium bicarbonate, then drying at 110 °C and fixing at 210 °C for 1 min and finally washing with 2 g/l Ultravon CN and 1 g/l sodium carbonate at 50 °C for 30 min and then rinsing thoroughly. To affirm the effect of alkali treatment on dye ability of each fiber, pure cotton and PET fabrics of the same fibers as blended fabric were alkali treated and dyed with the same method. When dyeing cotton fabric, only the reactive dyes were used and only the disperse dyes were used when dyeing PET fabric.

The reflectance of dyed samples and color coordinates CIE  $L^*$ ,  $a^*$  and  $b^*$  values were measured on a X-Rite CA22 spectrophotometer using illuminant D65 and 10° standard observer.

It should be noticed that conditioning of the samples was performed according to ISO 139:2005 standard prior to all measurements.

## RESULTS AND DISCUSSION

### Effect of pad-steam method conditions on weight loss of PET/cotton fabric

Figures 1 and 2 show the effect of sodium hydroxide concentration on weight loss percentage, with and without Tinegal PAC in two methods. It is obvious that weight reduction (%) is higher for exhaustion than

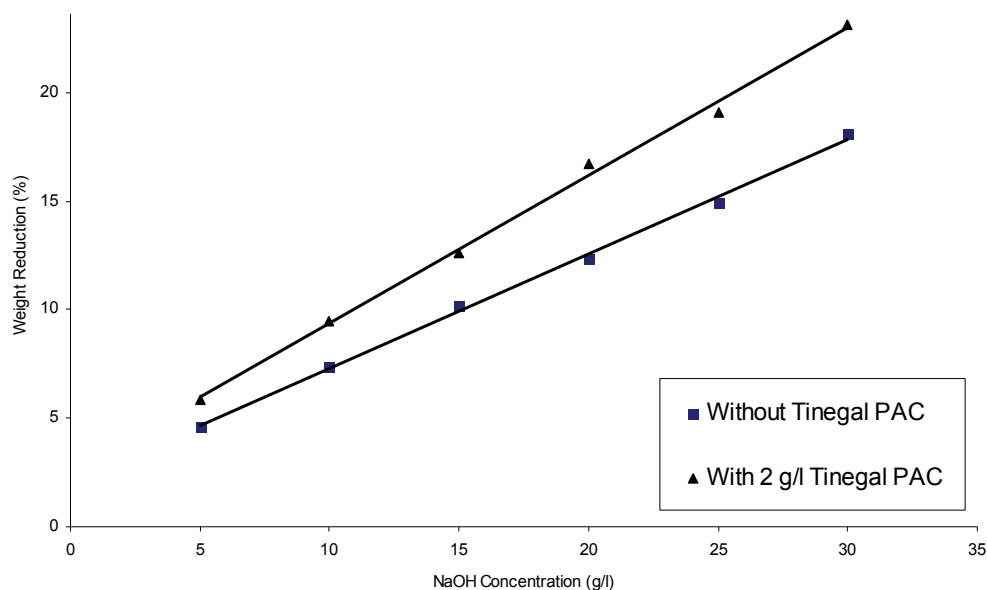


Figure 1. Effect of sodium hydroxide concentration on weight loss percentage in pad-steam method (5 min steaming time).

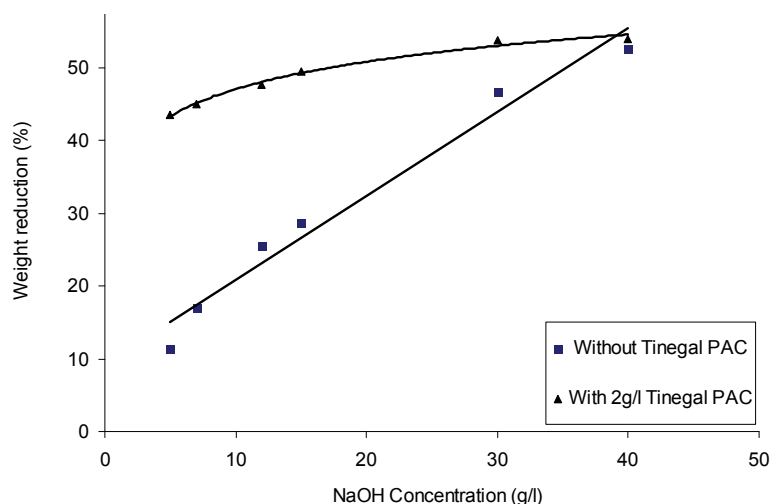


Figure 2. Effect of sodium hydroxide concentration on weight loss percentage in exhaustion method (1 h boiling time).

pad-steam method at a certain alkali concentration, but it should be noticed that the treatment time is 1 h in conventional exhaustion method (compared to 5 min for pad-steam method). According to the figures 1 and 2, the presence of Tinegal PAC has an important role in weight loss of the samples and accelerates the process of alkali treatment. Tinegal PAC is a quaternary ammonium salt with cationic charge, so it seems to be able to cover the negative group indicated in Figure 3, caused by the attack of alkali on the polyester chain, which provide further alkaline attack.

Figure 4 shows that the weight loss of sample increases with an increase in steaming time linearly.

#### Physical properties of alkali treated PET/cotton fabric

Figures 5 and 6 show the effect of steaming time and Tinegal PAC concentration on the tensile strength

of alkali treated fabric with pad-steam method, respectively. It seems that the higher the steaming time the lower would be the tensile strength. Steaming time of five minutes at 120 °C resulted in the lowest loss of tensile strength and was considered as the optimized steaming time.

Increase in Tinegal PAC concentration over 2 g/l in the pad-liquor caused an increase in loss of fabric tensile strength.

It can be seen from Table 1 that the exhaustion method (1 h) has a highly destructive effect on tensile strength of PET/cotton fabric compared with pad-steam method (5 min) because of high degree of hydrolysis of PET fibers.

Figure 7 shows the effect of weight loss percentage on bending stiffness of treated fabric in pad-steam

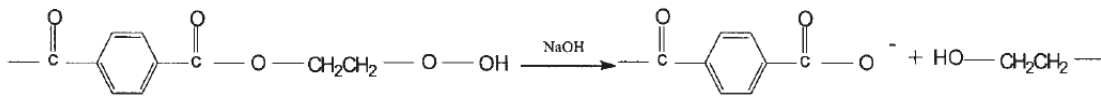


Figure 3. Polyester chain alkaline hydrolysis [18].

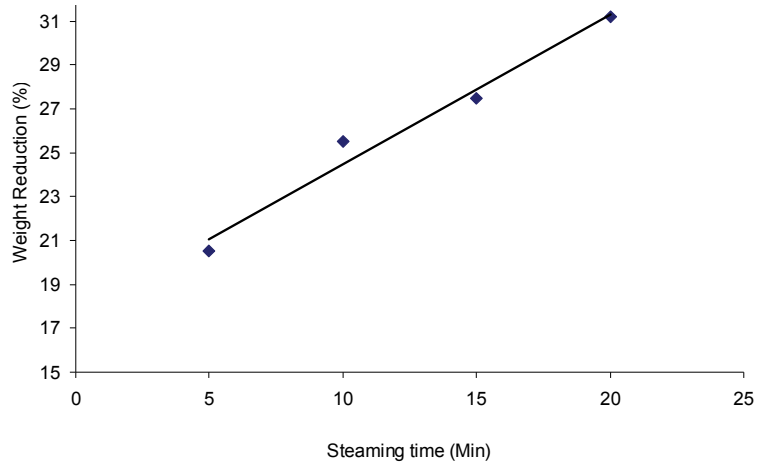


Figure 4. Effect of steaming time on weight loss percentage in different steaming times.

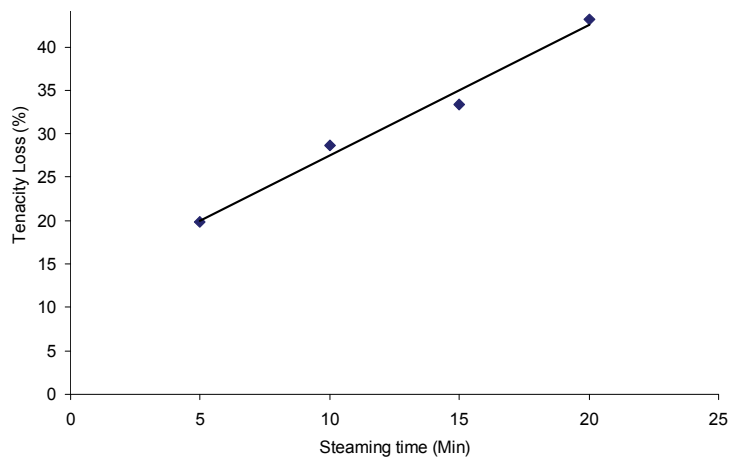


Figure 5. Effect of steaming time on tenacity loss percentage of fabric in pad-steam method at 120 °C with 2 g/l Tinegal PAC.

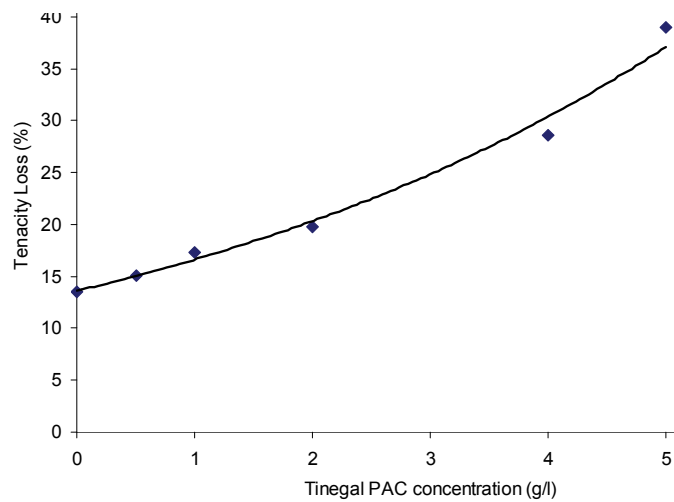


Figure 6. Effect of Tinegal PAC concentration on tenacity loss percentage of fabric in pad-steam method at 120 °C for 5 min.

method. Decreasing in bending stiffness is due to changing in handle properties of treated fabric. Decreasing in bending length of treated fabric might be due to the reduced diameter of fibers and thickness of fabric [22,23] after an increase in concentration of sodium hydroxide or presence Tinegal PAC in the pad-liquor.

Table 1. Effect of two methods on tensile strength of PET/cotton fabrics (2 g/l Tinegal PAC)

Method	Time, min	Reduction of tensile strength, %
Pad-steam	5	20
Exhaustion	60	50.2

As shown in Figure 8, the water front line in the treated sample is pronouncedly higher than the un-

treated one for both methods. This might be due to the increase of hydrophilic groups on the surface of PET fibers. The nucleophilic attack of NaOH on PET chains causes chain scissions at the ester linkages, producing carboxyl and hydroxyl polar groups, which lead to improved polarity and hydrogen bonding capacity with water molecules, and thus better water wettability. Furthermore, the microscopic roughness caused on the surface of the fiber after removal of surface polymers can be responsible for better spreading of water over the fiber surface. There is no significant difference between water absorption of samples treated with two methods.

According to the above-mentioned results, pad-steam method was chosen as the preferred method for alkali treatment of PET/Cotton fabric and the dyeing tests were performed only on pad-steam alkali

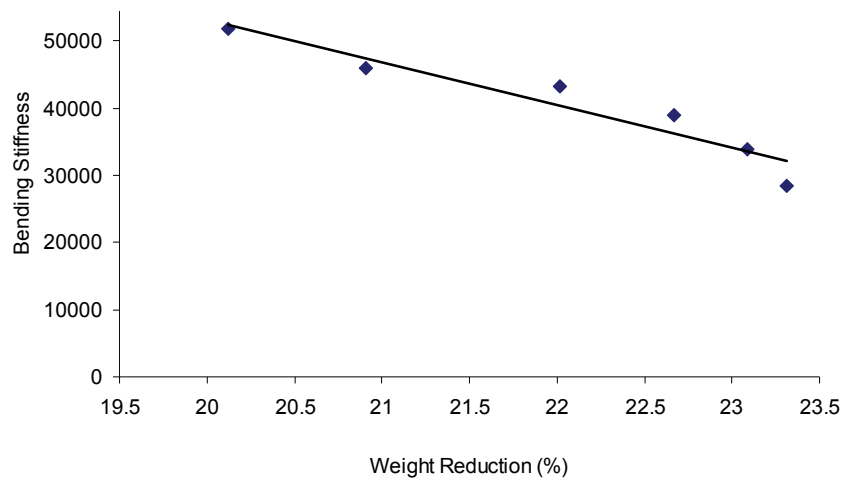


Figure 7. Effect of weight loss percentage on bending stiffness of fabric in pad-steam method at 120 °C with 2 g/l Tinegal PAC for 5 min.

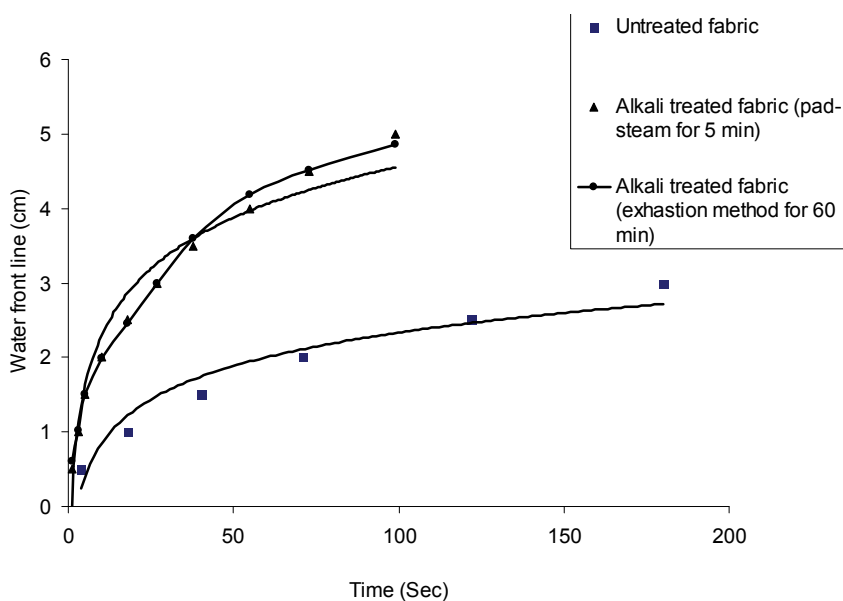


Figure 8. Wicking test results of untreated and alkali treated fabrics with two methods.

treated samples. To specify the effect of the alkali treatment of dye absorption of each fiber, pure cotton and PET fabrics of the same fibers as blended fabric were alkali treated and dyed with the previously mentioned method. As shown in Table 2, the  $L^*$  of dyed alkali treated PET/Cotton samples is less than dyed untreated fabrics indicating an increase in color depth of alkali treated fabric. This increase in dye absorption can be due to the caustification effect of the cotton part as well as the formation of terminal hydroxyl and carboxyl groups on the polyester surface. The increase for cotton portion is more than the PET portion as can be seen from Table 2.

- [3] N. Kallay, *Text. Res J.* (1990) 663-668  
 [4] R.T. Shet, S.H. Zeronian, *Text. Chem. Color.* **14** (1982) 11-16  
 [5] J. Lee, *Proceedings of the 3<sup>rd</sup> Asian textile Conference*, Vol. 1, 1995, pp. 256-261.  
 [6] A. Khosravi, *Dyeing Polyester and Cellulose Acetate*, Jahad Daneshgahi, Tehran, 1994, p. 12  
 [7] H.L. Needles, *Text. Chem. Color.* **17** (1985) 177-185  
 [8] H.L. Needles, S. Holmes, *J. Soc. Dyers Colour.* **106** (1990) 385-388  
 [9] M. Haghghatkish, M. Nouri, *J. Appl. Polym. Sci.* **72** (1999) 631-637  
 [10] K.V. Datye, *J. Soc. Dyers Colour.* **107** (1991) 218-219

Table 2. Color coordinates of untreated and alkali treated (pad-steam method at 120 °C for 5 min) polyester/cotton fabric dyed with blue and black dyes

Fabric	$L^*$	$a^*$	$b^*$	$\Delta E$
Polyester/cotton alkali treated-blue	53.73	-24.3	-27.4	5.013
Polyester/cotton untreated-blue	58.48	-24.4	-25.8	
Polyester/cotton alkali treated-black	18.93	-1.08	-2.82	4.787
Polyester/cotton untreated-black	23.56	-1.97	-3.65	
Cotton alkali treated-blue	48.45	-20.1	-24.9	5.52
Cotton untreated-blue	53.12	-22.5	-23.2	
Polyester alkali treated-blue	63.15	-17.4	-33.4	2.7
Polyester untreated-blue	64.38	-18.6	-31.3	

## CONCLUSION

Alkali treatment of PET/cotton fabric by exhaustion and pad-steam method was studied. Both methods lead to considerable weight reduction and strength loss of the fabric, which is higher in the case of exhaustion method. Steaming or biling time, concentration of alkali and Tinegal PAC have largest effect on the amount of weight reduction in both methods. Alkali treatment by pad-steam method at 120 °C for 5 min reduces the weight of polyester/cotton fabric resulting in minimum loss of tenacity, increase in hydrophilicity and dye absorption and better handle of the finished fabric. Considering the reduced amount of water, alkaline conditions, time, energy consumption and environmental requirements, the proposed process seems to be more appropriate.

## REFERENCES

- [1] E.P.G Golh, L.D. Vilensky, *Textile Science*, 2<sup>nd</sup> ed., Longman Cheshire, Melbourne, Australia, 1983, p. 187  
 [2] R. Betschewa, P. Wangelov, *Melliand Textilber.* **70** (1989) 599-604  
 [11] M. Haghghatkish, M. Yousefi, *Iran. J. Polym. Sci. Technol.* **1** (1992) 56-60  
 [12] M.C. Yang, H.Y. Tsai, *Text. Res. J.* **67** (1997) 760-766  
 [13] S.R. Shukla, M.R. Mathur, *J. Appl. Polym. Sci.* **75** (2000) 1097-1102  
 [14] W. Xu, C. Yang, *Color. Technol.* **118** (2002) 211-214  
 [15] W.M. Raslan, A. Bendak, *J. Appl. Polym. Sci.* **98** (2005) 1829-1837  
 [16] M.V. Konovalova, *Fibre Chem.* **37** (2005) 93-96  
 [17] M. Kimizu, Y. Shimbo, T. Abe, T. Hori, *Seni Gakkashi* **61** (2005) 109-114  
 [18] M. Montazer, A. Sadighi, *J. Appl. Polym. Sci.* **100** (2006) 5049-5055  
 [19] G. Kazakeviciute, V. Valiene, A. Abraitene, *Environ. Res. Eng. Manage.* **28** (2004) 40-47  
 [20] J.E. Booth, *Principle of Textile Testing*, 3rd ed., Butterworth-Heinemann, London, UK, 1968, p. 334  
 [21] A. Patnaik, R.S. Rengasamy, V.K. Kothari, A. Ghosh, *Text. Prog.* **38** (2006) 1-105  
 [22] H. Tavanai, *J. Text. Inst.* **100** (2009) 633-639  
 [23] F. Mousazadegan, S. Saharkhiz, M. Maroufi, *J. Text. Inst.* **101** (2010) 716-728.

AMINODDIN HAJI<sup>1</sup>  
REZA M.A. MALEK<sup>2</sup>  
FIROOZMEHR MAZAHERI<sup>2</sup>

<sup>1</sup>Department of Textile, Birjand Branch,  
Islamic Azad University, Birjand, Iran

<sup>2</sup>Textile Department, Amirkabir University  
of Technology, Hafez St.,  
Tehran, Iran

NAUČNI RAD

## UPOREDNO ISPITIVANJE METODA ISCRPLJIVANJA I PAD-STEAM RADI POBOLJŠANJA NEKIH SVOJSTAVA (OPIPA, PRIJEMA BOJE I APSORPCIJE VODE) TKANINE OD MEŠAVINE POLIESTAR/PAMUK

*U ovom radu, razvijen je pad-steam process za tretiranje tkanine od mešavine poliestar/pamuk natrijum-hidroksidom i istraživana uticaj parametara procesa na odabrana svojstva tkanine. Rezultati su poređeni sa konvencionalnim procesom iscrpljivanja. Oba procesa su poboljšala opip, sposobnost bojenja i apsorpcije vode tkanine od mešavine poliestar/pamuk, ali pad-steam proces ima niz prednosti, kao što su: manji gubitak jačine vlakana, kraće vreme tretiranja, manja potrošnja vode i hemikalija, koje proces čine manje opasnim po životnu sredinu.*

*Ključne reči: poliestar, sposobnost bojenja, smanjenje mase, pamuk, natrijum-hidroksid.*