# Bio-Bleaching of Cotton/Polyamide Fabric with Different Enzyme System at Low Temperature

Ayşe Usluoğlu Department of Chemistry, Faculty of Arts and Sciences, Sakarya University, Sakarya 54187, Turkey ayseusluoglu@hotmail.com

Abstract— The enzymes in the textile industry used in many chemical often are and technological processes to reduce pollution in the production as they are non-toxic and ecological. Furthermore, the use of enzymes results in reduced process times, energy and water savings, the improved product quality and the potential process integration. Previous studies show that tetraacetyl ethylenediamine (TAED) with peroxide system has potential in the bleaching of cottonbased fabrics with improved bleaching effectiveness under mild conditions. The present work aims to optimize the bleaching process parameters including TAED and concentrations, time, pH, temperature and to compare the bleaching process using TAED and H<sub>2</sub>O<sub>2</sub> with TAED, H<sub>2</sub>O<sub>2</sub> and enzyme combination in the same bath. Quality of bleached cotton/PA fabric was measured in terms of whiteness index, tensile strength, fabric absorbency. The optimum bleaching recipe consisted of 15 g/L, TAED; 5 g/L, hydrogen peroxide; 3 g/L, enzymes and 0.5 g/L non-ionic wetting agent, the treatment was carried out at 60°C for 60 min. The optimized bleaching recipe and processing were compared with conventional process. Results obtained that, Cotton/Polyamide fabric bleached with TAED/ H<sub>2</sub>O<sub>2</sub> and either protease or pectinase enzyme shows excellent wettability and acceptable whiteness index (WI). This bio-process achieved equal quality CO/PA fabric whiteness to the conventional system at much shorter batch times, and with significantly reduced fabric strange lost and alkali consumption that would be beneficial to the textile industry.

Keywords— Cotton, Polyamide, Enzyme, Bio-Bleaching, Tetraacetylethylenediamine

I. INTRODUCTION

Textile and the chemical processing industries always have major share in the environmental global pollution. Enzymes play key role in solving the problem. The use of enzymes in the textile industry is rapidly gaining worldwide interest because of their non-toxic and environmentally friendly properties to reduce pollution in the textile production. [1-2] All natural and regenerated cellulose fibers have undesirable yellow impurities. Therefore, bleaching is commonly essential for the preparation of fibers to remove the colored impurities prior to staining and Gulnur Arabaci Department of Chemistry, Faculty of Arts and Sciences, Sakarya University, Sakarya 54187, Turkey garabaci@sakarya.edu.tr

finishing. Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) is commonly used to bleach the natural and regenerated cellulose fibers and their blends with other synthetic fibers. In textile process, cotton bleaching requires both extremely high temperatures (normally 98 °C) and high alkalinity to reach satisfactory results for cotton stuffs. These harsh conditions cause to extraordinary energy consumptions, great fabric weight loss, high chemical oxygen demand (COD) discharge, and large amount of wastewater formed by beaching and rinsing processes. Therefore, it becomes a serious duty for the textile industry to develop new cotton bleaching processes for reduction of the temperature and consumption [3-5].

In latest years, reasonably investigates have been made to lower temperature and (or) shorten time period for textile bleaching by including bleach activators and catalysts into aqueous hydrogen peroxide solutions.[6-10] These low-temperature bleaching systems have been shown to have advantages over conventional  $H_2O_2$  bleaching methods in reducing energy consumption and protecting cotton fabric from chemical damage.[11-16]

One of the most promising method to decrease energy consumption and fiber damage is to include bleach activators into the peroxide bleaching solution to form peracid which can lower temperature and pH in bleaching process. Thus water consumption and energy both during bleaching and rinsing of the fabric can be decreased. The mostly used activator in this process is tetraacetylethylenediamine (TAED) [17-25]

The application of enzymatic systems in the preparation of cotton fabric is advantageous in terms of decreasing water, chemicals, and energy consumption, reuse of desizing waste baths, process conditions, and environmentally friendly processes.[26] The most common bleaching agent nowadays is hydrogen peroxide, which is dosed in excess to the fibres. Alternatively, the peroxide could be produced in situ by enzymatic system/peracids. The aim of the present study was to develop an enzymatic process for bleaching of cotton/polyamide (PA) fabrics, based on peracidic the use acid (PAA) or tetraacetylethylenediamine (TAED) with H<sub>2</sub>O<sub>2</sub> and enzymes such as lipase, protease, cellulase or pectinase. In the bleaching system, each enzyme with PAA is separately carried out at the same temperatures, pH value and time to determine whiteness index (WI), tensile strength and water

absorbency of cotton/polyamide fabrics. In this way, consumption of water, time and auxiliary agents would be lowered.

## II. MATERIALS AND METHODS

A. Materials

Greige 65% knitted cotton – 35 % polyamide fabric was kindly supplied by Aydın Örme Company, Turkey. Scourzym\_ L (Alkaline Pectinase with an activity 375 apsu/g), Lipolase 100 T, Novacell BL Conc, were kindly supplied by Novo-Nordisk A/S, Copenhagen, Cellulase (BioPrep\_, Novoenzymes A/S, Denmark). Kemastone LH (protease, Kemcolor) Tetraacetylethylenediamine (TAED) were purchased SDC limited company. Solvipol ECL non-ionic wetting agent was kindly supplied Erca Company. H<sub>2</sub>O<sub>2</sub> (%30 w/w), NaOH were analytical grade.

## B. Conventional Bleaching Process

Bleaching of cotton/PA fabrics were carried out using exhaustion technique, was adapted as follows: 5 g of cotton fabric were treated with an aqueous solution containing NaOH (3 g/L), Solvipol ECL (1 g/L),  $H_2O_2$  30% ( 5 g/L) and Stabilizer (0,5 g/L) using material to liquor ratio (LR) 1:20 at 98 °C for 30 min. The samples were washed several times with boiling water then washed with cold water, catalase enzyme processing and finally dried at ambient conditions.

## C. Low temperature bio - bleaching process

Exhaustion technique was employed to carry out bioscouring and PAA bleaching for greige cotton fabric. The experimental technique was adopted as follows: a swatch from greige cotton fabric was immersed in aqueous solution containing 3 g/L, enzymes (pectinase and/or cellulase/lipase and/or protease at pH 7.5) along with 1 g/L Solvipol ECL (non-ionic wetting agent). Different concentrations from TAED (10-20 g/L) and  $H_2O_2$  (3–8 g/L) were subsequently added. Material to liquor ratio (LR) was adjusted at 20. The bath temperature was 40°C - 50°C - 60°C for 45 min. The samples were then washed with boiling water to deactivate the enzyme, followed by tumble drying and conditioning.

#### D. Testing and analysis

Wettability was assessed in terms of drop disappearance time, measured by allowing a drop of water to fall on the sample and recording the time required for drop disappearance (AATCC Test Method 39-1980). The fabric shows wetting time less than 5 s is judged as good wetting property.

Tensile strength and elongation at break were determined according to ASTM standard test method.

The degree of whiteness was measured on the Macbeth Color Eye 7000A spectrophotometer by using the CIE method according to EN ISO 105-J02:1997(E) standard.

III. RESULTS AND DISCUSSIONS

# A. Whiteness Measurement

Cotton material contains yellowish impurities which come from cotton cellulose. Bleaching is often required to remove the impurities for the preparation of cottonbased textiles unless they are dyed in deep or dark shades.[15, 18] Figure 1 shows the effect of different enzymes (lipase, protease, cellulase and pectinase at pH 7.5) on the whiteness of cotton/PA fabric with TAED/H<sub>2</sub>O<sub>2</sub> system at 60°C. TAED/H<sub>2</sub>O<sub>2</sub> and TAED/ H<sub>2</sub>O<sub>2</sub>/enzyme systems were performed at three different temperatures (40, 50 and 60°C). 60°C was the best temperature for the bleaching as seen in Table 1. The lowest WI degrees were observed at 40°C and bleaching process for cotton/PA fabric was not enough. 50°C had the better WI degree than 40°C but it was still not enough for the best peroxide bleaching WI degree for cotton/PA. It represents the optimal bleaching temperature of cotton/PA fabric where the maximum WI (Table 1). The TAED/ $H_2O_2$ recipe showed lower WI against commercial peroxide bleaching system. Addition of enzyme (protease, lipase, cellulase, pectinase) to the low temperature bleaching system was improved WI. As followed the WI degrees at optimum temperature (60°C) were as followed lipase < protease < pectinase < cellulose in Figure 1.



 $\operatorname{Fig.}\xspace{1.5}$  . The effect of enzymes on the bleaching of cotton/PA.

#### B. Water Absorbancy

Table 1 shows biobleaching of Cotton/Polyamide fabric with commercial bleaching, TAED/H<sub>2</sub>O<sub>2</sub>/Enzymes bleaching. Our results showed that the addition of enzyme (protease, lipase, cellulase, pectinase) to the TAED/H<sub>2</sub>O<sub>2</sub> system was improved fabric absorbancy. However, temperature changes in the same system had no effect on the fabric absorbancy (Table 1).

### C. Tensile Strength

In this part of the work, tensile strength of cotton/PA fabric was investigated with different enzymes (protease, lipase, cellulase, pectinase) at different temperatures . The results showed that the TAED/ $H_2O_2$  recipes were comparable to the commercial peroxide bleaching method for improving tensile strength at three different temperatures (40, 50 and 60°C) (Table 1). The best temperature for the highest tensile strength was 40°C which was the optimal temperature for the tensile strength of cotton/PA. Tensile strength was as followed TAED/ $H_2O_2$ /protease < Commercial peroxide bleaching method < TAED/ $H_2O_2$ /protease < TAED/ $H_2O_2$ /protease

TAED/H<sub>2</sub>O<sub>2</sub>/cellulase in Figure 2. Protease and commercial bleaching system had the lowest tensile strength for cotton/ PA fabric (Figure 2). The lowest tensile strength was observed for the peroxide bleaching control because the high alkaline and peroxide system could damaged cotton/PA fabric. However, cellulase enzyme system showed the best tensile strength in Figure 2. The retained tensile strength was gradually decreased as the temperature increased as seen Table 1.



Fig. 2. Tensile strength of CO/PA with different enzymes.

Table 1 shows that, raising the bleaching temperature from 40°C to 60°C is accompanied by increasing the TAED/H<sub>2</sub>O<sub>2</sub> bleaching efficiency. This could be associated with the favorable effect of the temperature on swelling of cotton fabric, diffusion of PAA, enhancement of reactant mobility and their collision with cotton impurities. A close examination of results in Tables 1 would reveals that cotton/PA fabric can be successfully enzymatic TAED/H<sub>2</sub>O<sub>2</sub> bleached. The optimum bleaching recipe consists of 15 g/L, TAED; 5 g/L, H<sub>2</sub>O<sub>2</sub>; 3 g/L, enzymes and 0.5 g/L Solvipol ECL (non-ionic wetting agent) the treatment was carried out at 60°C for 60 min.

The results show that the optimized TAED/ H<sub>2</sub>O<sub>2</sub>/enzymes activated bleaching system could achieve high quality cotton/PA bleaching with comparable fabric whiteness to the conventional system at much shorter batch times, and with significantly reduced fabric strength loss and decreased alkali consumption, which would be beneficial to sustained development of the textile wet processing industry. D.  $TAED / H_2O_2$  Concentration

Hence, the effective concentration of liberated PAA required to achieve bleaching of cotton fabric will depend on the concentration of both TAED and  $H_2O_2$ . As seen Table 2, the optimum recipe for cotton/PA fabric is 15 g/L TAED and 5 g/L  $H_2O_2$  with maximum WI degree and Tensile Strength

TABLE I. TO COMPARE, COMMERCIAL BLEACHING, TAED/H\_2O\_2 BLEACHING, TAED/H\_2O\_2/ENZYMES BIOBLEACHING FOR COTTON/POLYAMIDE FABRIC

	Temperature [°C]	WI	Wett. [sec.]	Max. Force [kgf.]
Blank		- 16.93	> 200	23.22
Bleaching	98°C	65.51	1	13.87
TAED/H <sub>2</sub> O <sub>2</sub>	40°C 50°C 60°C	49,72 54,66 58.83	3 3 2	14.22 14.18 13.93
TAED/H <sub>2</sub> O <sub>2</sub> + pectinase	40°C 50°C 60°C	51.63 57.24 61.91	1 1 1	14.52 14.36 14.19
TAED/H <sub>2</sub> O <sub>2</sub> + lipase	40°C 50°C 60°C	51.78 56.34 60.27	1 1 1	15.47 15.45 15.06
TAED/H <sub>2</sub> O <sub>2</sub> + protease	40°C 50°C 60°C	52.67 58.14 61.35	1 1 1	13.96 13.78 13.54
TAED/H <sub>2</sub> O <sub>2</sub> + cellulase	40°C 50°C 60°C	54.36 58.65 62.23	1 1 1	15.62 15.48 15.24

# E. Effect of time period

Table 3 shows the effect of time of the bleaching process on the aforementioned technical properties of cotton/PA fabric. The results disclose that, prolonging the time of bleaching significantly favors the bleaching process as evidenced by enhancement in fabric whiteness index. At the same time the adverse effect of bleaching time on the tensile strength is not striking. In a more specific sense, prolonging the duration of bleaching from 30 to 60 min is accompanied by increments in whiteness index (WI); meanwhile the fabrics under investigation undergo certain degradation as clarified by the results of tensile strength. WI and tensile strength values reach the best values in 60 min as seen table 3.

FABRIC BLEACHING.					
TAED/ H <sub>2</sub> O <sub>2</sub> Amount [g/L.]		WI	Max. Force [kgf.]		
TAED H <sub>2</sub> O <sub>2</sub>	10 3	52.43	14.62		
TAED H <sub>2</sub> O <sub>2</sub>	15 5	58.83	13.93		
TAED H <sub>2</sub> O <sub>2</sub>	20 8	59.01	13.38		

TABLE II. EFFECT OF TAED/H2O2 CONCENTRATION ON CO/PA

TABLE III. EFFECT OF BLEACHING TIME ON CO/PA FABRIC

Bleaching Time [min]	WI	Max. Force [kgf.]
30	53.27	14.17
45	57.18	14.03
60	58.83	13.93

#### IV. CONCLUSIONS

In the present work, the TAED/H2O2/enzymes system was compared to a commercial bleaching method by measuring WI, tensile strength and water absorbency of cotton/polyamide fabric. All of the data demonstrated that in the presence of the TAED/ H<sub>2</sub>O<sub>2</sub>/Enzyme bleaching of the cotton knitted fabric could be performed at 60 °C with the fabric whiteness and strength comparable to that treated with the traditional procedure performed at 98 °C. The results show that the optimized TAED/ H<sub>2</sub>O<sub>2</sub>/enzyme activated bleaching system could achieve high quality cotton/PA bleaching with comparable fabric whiteness to the conventional system at much shorter batch times, and with significantly reduced fabric strength loss and decreased alkali consumption. Thus, the optimized and enzymatically activated bio-bleaching system can reduce both alkali consumption and batch time remarkably, showing the advantages of high efficiency, pollution reduction and consumption decrease which would be useful to sustained development of the textile industry. In this aspect, the bleaching processes with TAED/H<sub>2</sub>O<sub>2</sub>/Enzyme are shorter, consume less energy and water and are therefore environmentally friendlier than the conventional high-temperature and highly alkaline bleaching processes with  $H_2O_2$ .

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#### REFERENCES

[1] A.R. Ramadan, "Characterization of biobleaching of cotton/linen fabrics", J. Text. App., Technol. Manag., Vol. 6, pp.1-12, 2008.

[2] S.H. Zeronian, M.K. Inglesby, "Bleaching of cellulose by hydrogen peroxide", Cellulose, Vol. 2, pp. 265–272,1995.

[3] E.S. Abdel-Halim, S.S. Al-Deyab, "One-step bleaching process for cotton fabrics using activated hydrogen peroxide", Carbohydrate Polymers., Vol. 92, pp. 1844–1849, 2013.

[4] R.E. Ball, J.O. Edwards, M.L. Hagget, P. Jones," A kinetic and isotopic study of the decomposition of monoperoxyphthalic acid", J. Am. Chem. Soc., Vol. 89, pp. 2331–2333,1967.

[5] J.Y. Cai, D.J. Evans, "Guanidine derivatives used as peroxide activators for bleaching cellulosic Textiles", Color. Technol., Vol. 123, pp. 115–118, 2007.

[6] D.M. Davies, M.E. Deary, "Kinetics of the Hydrolysis of Tetra acetylethylenediamine", J. Chem. Soc., Perkin Transactions, Vol. 2, pp. 1549–1552, 1991.

[7] N.A. Ibrahim, S.S. Sharaf, M.M. Hashem, "A novel approach for low temperature bleaching and carbamoylethylation of cotton cellulose", Carbohydrate Polymers, Vol. 82, pp. 1248–1255, 2010.

[8] J.F. Goodman, P. Robson, E.R. Wilson, "Decomposition of aromatic peroxyacids in aqueous alkali", Transactions of the Faraday Society, Vol. 58, pp. 1846–1851, 1962.

[9] M. Hashem, M. El-Bisi, S. Sharaf, R. Refaie, "Pre-cationization of cotton fabrics: an effective alternative tool for activation of hydrogen peroxide bleaching process", Carbohydrate Polymers, Vol. 79(3), pp. 533–540, 2010.

[10] A. Hebeish, M. Shaker, N. Ramadan, B. El-Sadek, M.A. Hady, "New development for combined bioscouring and bleaching of cotton-based fabrics", Carbohydrate Polymers, Vol. 78, pp. 961–972, 2009.

[11] R.A. Heler, R. Weiler, "Kinetics of the reaction of p-dinitrobenzene with basic hydrogen peroxide", Can. J. Chem., Vol. 65, pp. 251–255, 1987.

[12] J. Hoffmann, G. Just, W. Pritzkow, H. Schmidt, "Bleaching activators and the mechanism of bleaching Activation", J. Prakt. Chem, Vol. 334, pp. 293–297, 1992.

[13] N.C. Gursoy, S.H. Lim, D. Hinks, P. Hauser, "Evaluating hydrogen peroxide bleaching with cationicbleach activators in a cold pad-batch process", Text. Res. J., Vol. 74, pp. 970–976, 2004.

[14] S.J. Scarborough, A.J. Mathews, "Using TAED in bleaching fiber blends to improve fiber quality", Textile Chemist and Colorist & American Dyestuff Reporter, Vol. 32, pp. 33–37, 2000.

[15] X. Long, C. Xu, J. Du, S. Fu, "The TAED/ $H_2O_2$ /NaHCO<sub>3</sub> system as an approach to low-temperature and near-neutral pH bleaching of cotton", Carbohydrate Polymers, Vol. 95, pp. 107–113, 2013.

[16] T. Topalovic, V. Nierstrasz, L. Bautista, D. Jocic, A. Navarro, M. Warmoeskerken, "Analysis of the effects of catalytic bleaching on cotton", Cellulose, Vol. 14, pp. 385–400, 2007.

[17] O.E. Ismal, A.T. Ozguney and A. Arabaci, "Effects of various biotreatment systems on dyeing properties of cotton with reactive dyes", AATCC Rev., Vol. 7, 34, 2007.

[18] C. Xu, X. Long, J. Du, S. Fu, "A critical reinvestigation of the TAED-activated peroxide system for low-temperature bleaching of cotton", Carbohydrate Polymers, Vol. 92, pp. 249–253, 2013.

[19] A. El-Shafie, M.M. Fouda, M. Hashem, "Onestep process for bio-scouring and peracetic acid bleaching of cotton fabric", Carbohydrate Polymers, Vol. 78, pp. 302–308, 2009.

[20] J. Shao, Y. Huang, Z. Wanga, J. Liub, "Cold pad–batch bleaching of cotton fabrics with a TAED/ $H_2O_2$  activating system", Color. Technol., Vol. 126, pp. 103–108, 2010.

[21] N.Ç. Gursoy, H. Dayıoğlu, "Evaluating Peracetic Acid Bleaching of Cotton as an Environmentally Safe Alternative to Hypochlorite Bleaching", Textile Research Journal, Vol. 70, (6), pp. 475-480, 2000.

[22] M. Prabaharan, L. Almeida, "Process optimization in tetraacetyl ethylenediamine activated sodium perborate bleaching of cotton", Indian Journal of Fibre & Textile Research, Vol. 29, pp. 343-349, 2004.

[23] H. Zeng, R.-C. Tang, "Application of a novel bleach activator to low temperature bleaching of raw cotton fabrics", The Journal of The Textile Institute, Vol. 106, No.8, pp. 807–813, 2015.

[24] S. Wang, S. Li, Q. Zhu, C. Q. Yang, "A Novel Low Temperature Approach for Simultaneous Scouring and Bleaching of Knitted Cotton Fabric at 60 °C", Ind. Eng. Chem. Res., Vol. 53, pp. 9985–9991, 2014.

[25] C. Xua, D. Hinksb, C. Suna, Q. Wei, "Establishment of an activated peroxide system for low-temperature cotton bleaching using N-[4-(triethylammoniomethyl)benzoyl]butyrolactam chloride", Carbohydrate Polymers, Vol. 119, pp. 71– 77, 2015.

[26] Nina Spicka and Petra Forte Tavcer, "Complete enzymatic pre-treatment of cotton fabric with incorporated bleach activator", Textile Research Journal, pp. 1–9, 2012.