

RESEARCH PAPER

## Environmentally Friendly Bleaching Process of Polyester Materials via Direct Process of Water without Heating System

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### ABSTRACT

A great quantity of chemicals, water, and energy are used during the bleaching of raw polyester (Polyethylene terephthalate) fabrics. For this aim, it is highly significant both economically and ecologically to develop eco-friendlier bleaching technology that removes the necessity for heat energy in the bleaching of these substrates. Here, we report on the green method for laboratory-scale bleaching with  $\text{H}_2\text{O}_2$  in the presence of sodium pyrophosphate ( $\text{Na}_4\text{P}_2\text{O}_7$ ), which is used as both wetting agent and pH regulator and contributes to the high whiteness of fabrics. In bleaching, the process of water was used in ambient temperature as forte without any heating process, thus reducing the energy requirement significantly. In the measurements, it was observed that the whiteness value (W.I. = 74.1) was obtained after 10.0 hours of bleaching at 25°C. The SEM image of the polyester (PET) fabrics bleached with  $\text{H}_2\text{O}_2$  for 10.0 hours indicated that few nano-sized remained (dusts, waxes and impurities) on the fabric bleached with  $\text{H}_2\text{O}_2$  for 10.0 hours. In addition, it has been confirmed that PET fabrics bleached with this method have lowest damage on the fabrics (only 0.716% weight loss, insignificant mechanical reduction). This investigation demonstrated that PET fabrics can be effectively bleached in the presence of  $\text{Na}_4\text{P}_2\text{O}_7/\text{H}_2\text{O}_2$  without the necessity for any heating energy.

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### INTRODUCTION

The growing of the world's population will lead to a noteworthy growth in textile manufacture and consumption, which will meaningfully increase the textile industry's energy and water application and release significant amounts of carbon dioxide ( $\text{CO}_2$ ) and other damaging emissions to the environment [1, 2]. Because of the increasing request for textile crops, reducing energy consumption and  $\text{CO}_2$  emissions needs novelty as well as present technologies [3-5]. The procedure of bleaching textile substrates is a challenging process [5-7].

Polyester (PET) ( $(\text{C}_{10}\text{H}_8\text{O}_4)_n$ ) is one of the most significant and extensively used fibers in textiles

owing to its unique properties (Scheme 1) [8,9].

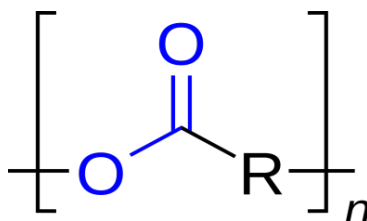
Owing to the variability and assortment of PET bases, the quality and physical properties of fibers such as the appearance of PET like color and luster are powerfully affected by various elements [6, 10, 11]. The color of apparently white PET fibers and especially the variety of its tint attributes will affect the final color appearance of PET fabrics [12-14]. The procedures to eliminate contaminations in PET fabrics are essential in order not to damage the PET structure and to certify its whiteness [15-17]. The hydrogen peroxide bleaching procedure, which is the most applied procedure to eliminate the natural pigments from PET and cotton blends, needs high water consumption and energy input,

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Scheme 1: Chemical structure of PET fibers.

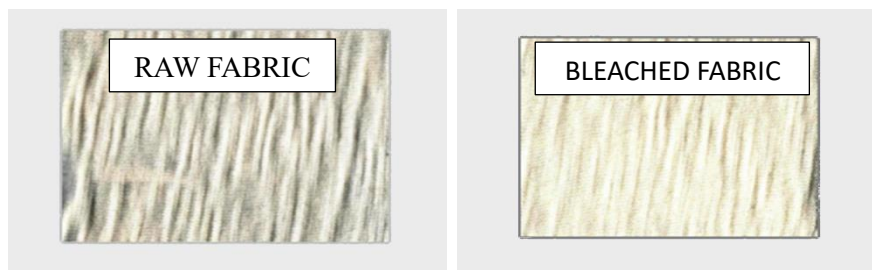


Fig. 1: visual images of raw and bleached PET fabrics.

as well as an alkaline location, stabilizer, and long incubation time at high temperatures [7, 18].

Investigations on the eco-friendly principles of textile finishing processes (such as bleaching and scouring) have been carried out to improve the quality of textile materials and save water and energy [19] (see Table 1).

Considering the benefits of an environmentally friendly process for the treatment of textile materials, this investigation aimed to explore the potential of using Direct Process of Water without heating system on the PET fabrics for the bleaching procedure (Fig. 1).

## EXPERIMENTAL

### Equipment and materials

This investigation employed one set of identically constructed 'piqué' type of knitted fabrics provided by Nature Works LLC, USA and derived from 150/144 dtex/filament Polyester yarns. This kind of manufactured goods is usually used for outerwear garments such as punches, casual clothes, active wear, women's uniform, style wear, and children's clothes [30].

The whiteness of the fabrics was measured as reflectance by the Color Eye-7000 Spectrophotometer, Macbet Gretage. Mettler Toledo S220-K table top pH meter was used for pH measurements. A Libror brand electronic balance (AEU210 Electronic Balance, with an accuracy of 0.0001 grams) was employed to calculate the weight loss of fabrics as a result of bleaching.

### Chemicals

Acetic acid ( $\text{C}_2\text{H}_4\text{O}_2$ , 99% pure) was supplied as a 1% solution in water used for neutralization of samples, hydrogen peroxide solution ( $\text{H}_2\text{O}_2$ , 30%) used for bleaching of samples and sodium pyrophosphate ( $\text{Na}_4\text{P}_2\text{O}_7$ , MW 265.9 g/mol) utilized for pH adjustment obtained from Merck Chemical Company (Germany).

DIADAVIN® EWNOL (D-E) used for scouring agent were supplied by the Iranian Resin Saveh Company.

### Bleaching

#### Process conditions

An average of 5 g was cut from a roll of raw PET fabric for each bleaching experiment. The cut fabric was soaked in a liquor solution. Liquids consisted of hydrogen peroxide, sodium pyrophosphate, a wetting agent (nonionic), and dilute acetic acid. The fabric/liquor ratio specified in the recipes was 1/20. 4 mL  $\text{H}_2\text{O}_2$ , 1 g/L  $\text{Na}_4\text{P}_2\text{O}_7$  and 3 g/L wetting agent were used in the experimentations. The pH of the liquids was adjusted by adding sodium pyrophosphate (TSPP). The resulting solution and PET fabric were transferred into a 100 mL beaker. Then, the fabric bleaching was carried out at different times.

In the last part of the period, the fabrics taken from the beaker were neutralized with dilute acetic acid for 5 minutes and then rinsed with tap water (Fig. 2). Subsequently, the fabric was left to dry at

Table 1. Environmentally friendly approaches in treatment processes of textile materials.

Environmentally friendly Approach	Textile Material	Treatment Process	Ref.
Environmentally friendly Bleaching	Poly(Lactic Acid) Wave Fabrics	UV/O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> System	[20]
Environmentally friendly Scouring	Jute Fibers	Ultrasound-assisted Enzymatic Treatment	[21]
Environmentally friendly Anti-static	Polyester Wave Fabrics	Ultraviolet + O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> /Na <sub>2</sub> SiO <sub>3</sub> System	[22]
Environmentally friendly Scouring	Cotton Knit Fabrics	Enzyme and Soap-nut	[23]
Environmentally friendly Wetting Finishing	Poly lactide Knitted Materials	Ultraviolet/O <sub>3</sub> Irradiation	[4]
Environmentally friendly Anti-pilling	Wool Fabrics	Thiourea Dioxide-Mediated Surface Micro-dissolution	[24]
Environmentally friendly Surface Treatment	Poly(Ethylene Terephthalate) Knitted Materials	UV/Ozone Radiation	[25]
Environmentally friendly Pretreatment	Cotton Fabrics	Ultra-sonication	[1]
Environmentally friendly Surface Oxidation	Poly lactide Knits	UV/O <sub>3</sub> /H <sub>2</sub> O System	[26]
Environmentally friendly Antibacterial, UV-protection, Self-cleaning and Anti-wrinkle Finishing	Viscose/Wool Fabric	TiO <sub>2</sub> , and/or Ag-NPs, as Active Ingredients	[27]
Environmentally friendly Nanoetching of Surface	Ingeo™ Fibers	Ultraviolet/O <sub>3</sub> -Assisted Method	[28]
Environmentally friendly Antimicrobial Finishing	Cotton Fabrics	Bioactive agents from Novel Melia-Azedarachayan Berries Extract	[29]

ambient temperature. Further investigation were carried out with bleached fabrics.

#### Measurements and analysis

##### Labeling of the PET fabrics

Labeling of PET fabrics which were bleached in different situations is briefed in Table 2.

##### Weight loss

The weight loss of the fabric after the bleaching process was calculated on a dry weight basis using

the formula (1):

$$\text{Weight Loss(\%)} = \frac{W_1 - W_2}{W_1} \times 100 \quad (1)$$

where  $W_1$  and  $W_2$  are the dry weights of the PET fabric before and after bleaching treatment, respectively.

##### Degree of whiteness

The color coordinates in terms of CIE Lab and CIE Lch ( $L^*$ ,  $a^*$ ,  $b^*$ ,  $c^*$  and  $h^\circ$ ) and whiteness indexes

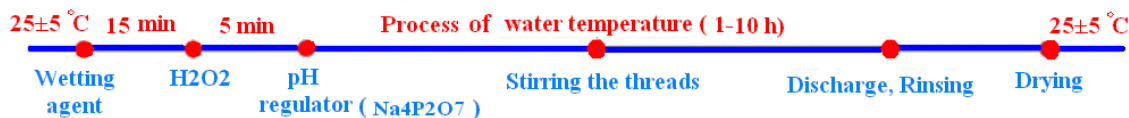


Fig. 2: Diagram of environmentally friendly bleaching process.

Table 2. Identification code and bleaching condition of the PET fabrics.

Code	Bleaching Recipe	Amount	Process of water time (hour)
1 (Virgin)	----		0
2	Bleaching Agent (Hydrogen peroxide)	4 ml	1.0
3		1 g/l	5.0
4	pH Regulator (Sodium pyrophosphate)	3 g/L	10.0
	Wetting Agent		
	Acetic Acid		

(W.I.) of PET fabrics were measured using a Color Eye 7000 spectrophotometer (Macbeth Gretage) under illuminant D65 light at 10° observation angle using 0/d measurement. Whiteness indexes were calculated in terms of CIE Y (green) and Z (blue) reflectance components using the ASTM Method E31373 equation [31, 32].

$$W.I. = \frac{4Z}{1.18} - 3Y \quad (2)$$

where Y and Z are the readings of the device.

#### Tensile strength (RT)

Tensile strength of PET samples was determined by the Instron according to ASTM D2256/D2256M.

#### Scanning Electron Microscopy

Scanning Electron Microscopy examination of the fibers was performed in order to assess whether the various treatments had caused any visible degradation to the fiber surfaces [28]. A XL30 MODEL/PHYLIPS Company/Netherlands instrument was employed for this purpose. In order to avoid problems due to charge build-up, the PET fabrics were previously sputter-coated with gold palladium for two minutes in a SCDOOS MODEL/Bal-Tech Company/Switzerland sputter-coating unit. The images were captured with magnifications of 15000.

#### Software

The use of statistics for investigating the significance of properties and making easy decisions will be the best method to analyze the results [30, 33]. In this research, the SPSS software was used for the statistical analysis of the results. The confidence level of 95% was chosen for cases that required variance analysis.

## RESULTS AND DISCUSSION

### Morphology analysis by scanning electron micrographs

In Fig. 3, SEM images of unbleached raw PET fabric and PET fabrics bleached with H<sub>2</sub>O<sub>2</sub> and Na<sub>4</sub>P<sub>2</sub>O<sub>7</sub> are shown. The SEM image in Fig. 3a belongs to the raw PET fabric, and it is seen from the image that the fibers have homogeneous diameters. When looking at the image, it is observed that there are remains that can consist of dusts, waxes, pigments, and other organic and inorganic impurities, which are also indicated by red arrows among the PET fibers.

Fig. 3b illustrates the SEM image of PET fabric bleached with H<sub>2</sub>O<sub>2</sub> for 1.0-hour. Looking at the image, it is observed that there are remains in the PET fibers, which are also indicated by the red arrows. It is observed that the remains cannot be removed to a large extent after bleaching with H<sub>2</sub>O<sub>2</sub> for 1.0-hour.

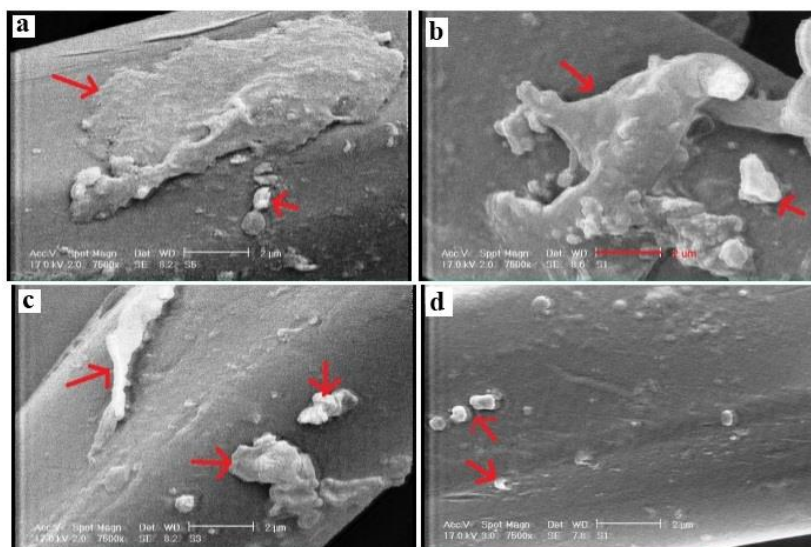


Fig. 3: SEM images of samples of (a) raw fabric, (b) Fabric bleached with  $H_2O_2$  for 1.0 hour, (c) Fabric bleached with  $H_2O_2$  in 5.0-hours(7500x), (d) Fabric bleached with  $H_2O_2$  in 10.0-hours.

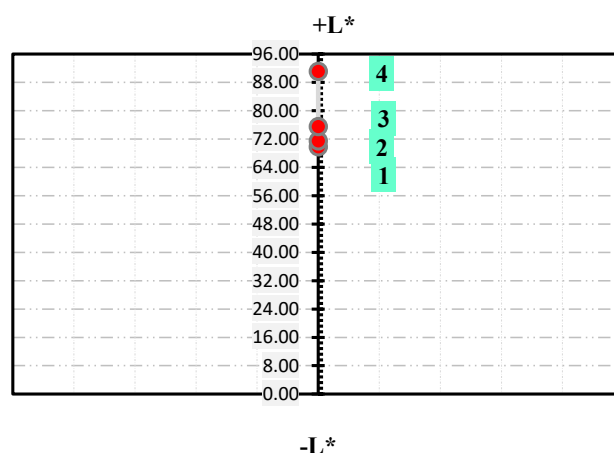


Fig. 4: The  $L^*$ (lightness/darkness) plot of PET bleached fabrics over the CIELAB color space.

Fig. 3c illustrates the SEM image of the experimental PET fabric bleached with  $H_2O_2$  for 5.0 hours. In this figure, it is seen that the surface of the fibers is smoother than the raw fiber. In addition, it was observed that there was much less remain in the fabric bleached with  $H_2O_2$  for 5.0 hours compared to the SEM image of raw and 1.0 hour bleached PET fabrics.

In Fig. 3d, the SEM image of the investigational PET fabric bleached with  $H_2O_2$  for 10.0 hours is specified. In this figure, it is shown that the external surface of the fibers is considerably smoother than the raw fiber. Moreover, it was observed that there was notably fewer nano-sized remains in the fabric

bleached with  $H_2O_2$  for 10.0 hours.

#### Color parameters of PET materials

CIE  $L^*a^*b^*$  color space was used to record the color coordinates [34, 35]. In CIE  $L^*a^*b^*$  space,  $L^*$  indicates the lightness (+ = lighter, - = darker) [36, 37]; the perfect white sample has  $L^* = +100$  [38, 39]. White is recognized by distributions around opposite poles of lightness coordinate in different color order system [40, 41]. As seen from the Fig. 4, the 10.0 hours bleached fabric, has the highest  $L^*$  value (+91.128 %).

Table 2 presents the results of different bleaching procedures on the colorimetric attributes of the

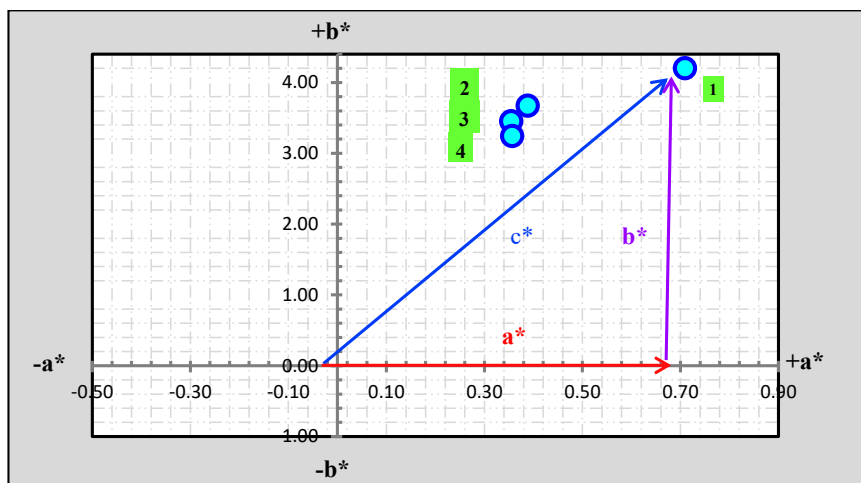


Fig. 5: The  $a^*b^*$  scatter plot of PET bleached fabrics over the CIELAB color space (the  $b^*$  value describes the yellowness/blueness coordinate in certain color space, for example Hunter L, a, b and CIELAB).

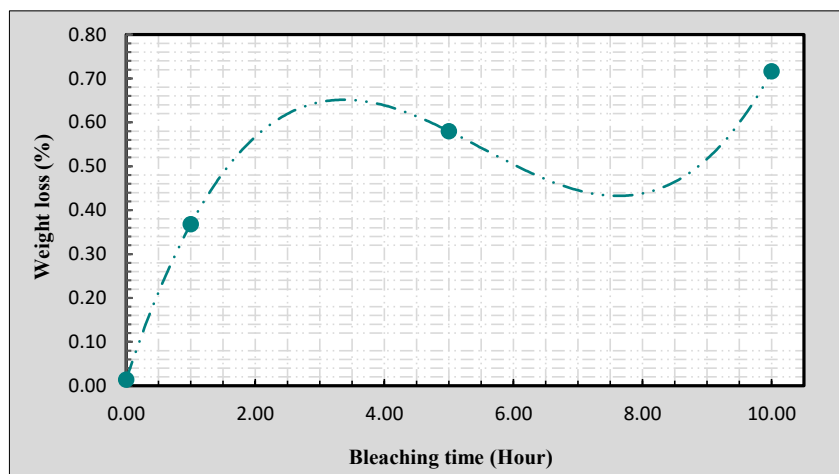


Fig. 6. Relationship between Weight loss of the PET fabric (%) and bleaching time (hour).

PET fabrics. White is identified by low Chroma values in various color order system [20, 42]. As seen in Table 3, the 10.0-hours bleached fabric has the lowest  $C^*$  value (3.2616).

The hue angle formula is depending on which quadrant the color is located [43-45]. In Fig. 5, it appears that the tint attribute of PET bleached fabrics does not shift from the first quarter of hue area. It means that the tint effect of bleached fabrics does not change from reddish-yellowish white shade. Additionally, according to Fig. 5, the minimum value of  $b^*$  belongs to 10.0-hr bleached fabric. The  $b^*$  value is applied as the variance between a specimen and a standard reference color. If  $b^*$  value is positive, there is more yellowness than blueness, whereas when  $b^*$

or  $b^*$  value is negative, more blueness is observe (the samples with  $b^* > 0$  means yellowness and  $b^* < 0$  blueness[46, 47]). Therefore, sample 4 has the lowest yellow shade due to the lower  $b^*$  value (3.45).

#### Whiteness of PET material

Table 4 and 5 exhibit the specifications of bleached PET fabrics used as substrates in this investigation. As stated by Table 3 and 4, the PET fabrics have different whiteness, yellowness, and tint attributes. Based on the CIE whiteness formula, samples are described as whites if their whiteness index (WI) and tint factor ( $T_w$ ) follow from limits defined by the following relations [48, 49].

$$40 < WI < (5Y-280), -4 < T_w < +2 \quad [50, 51]$$



Table 3. The colorimetric parameters of PET bleached fabrics.

PET fabric	Material 1	Material 2	Material 3	Material 4
$c^* = (a^{*2} + b^{*2})^{1/2}$	4.2595	3.6905	3.4682	3.2616
$h^o = \tan^{-1}(b^*/a^*)$	80.39°	83.9°	84.17°	84.11°

Table 4: Whiteness index and yellowness index of PET fabrics.

PET fabric	Material 1	Material 2	Material 3	Material 4
WI (%)	35.5	67.9	68.5	74.1
YI (%)	9.5	7	5.5	2.5
Y	69.811	81.486	85.504	91.128
Shade	Yellow	White	White	White

Table 5: Influence of bleaching routine on the tint factor and shade of PET bleached fabrics.

PET fabric	Material 1	Material 2	Material 3	Material 4
Tw	-4.2	-0.5	-0.4	-0.2
Shade	Yellow	Reddish white		

Based on the limits defined above, bleached PET fabrics are described as white. However, the whiteness index (WI) and tint factor (Tw) of virgin PET fabrics are out of limitations defined by CIE whiteness formulation.

Based on Table 4, all the bleached PET fabrics have negative value of tint factor. Samples with Tw=0 are described as bluish whites and those with positive (Tw>0) and negative (Tw<0) tint factors represent the greenish and reddish whites, correspondingly [52].

It can be indicated that 10.0-hours bleached PET fabrics with the highest whiteness index (WI=74.1) and lowest tint factor (Tw= -0.2) could be described as the whitest substrate among the three bleached fabrics. Obviously, the different whitening treatment of PET fabrics leads to its greater whiteness attribute in comparison with other white substrates. The 10.0-hours bleached PET fabric displayed the top colorimetric properties between these four substrates (WI=74.1, T=-0.2, L\*=+91.128, a\*=+0.106, b\*=0.355, c\*=3.2616, h°=84.11).

#### Weight loss studies

One of the topics to be studied in the bleaching procedure is the weight loss of the fabrics after bleaching. For this aim, the weight losses that happen as an effect of the bleaching procedures carried out at different times are estimated and specified in Fig. 6. As perceived from the diagram, it is realized that the weight loss of the fabric rises as the bleaching time increases. This can be explained by the elimination of undesirable fragments such as waxes, polymeric contaminants, pigments, lipid chemicals, microbiological contaminations and other organic and inorganic impurities from the fabric as the bleaching process continues [17, 32, 53-57]. Fig. 6 exhibits the association between the weight loss percentage of PET fabrics and bleaching time.

#### Tensile strength

The mechanical properties of fabrics are essential for their purposes and parts of usage [58-61]. For this aim, it is necessary that the bleached fabrics display appropriate Tensile strength. Considering this situation, the Tensile strength of pure and

Table 6. Descriptive\* statistics for Tensile strength.

PET	Unit	Std.	Std.	95% Confidence Interval		Minimum	Maximum		
Sample	N	Mean	Deviation	Error	for Mean				
1 (Virgin)	10	5.7140	N/tex	0.05320	0.02379	5.6479	5.7801	5.64	5.77
2	10	5.4680	N/tex	0.01095	0.00490	5.4544	5.4816	5.46	5.48
3	10	5.4600	N/tex	0.03317	0.01483	5.4188	5.5012	5.41	5.49
4	10	5.4860	N/tex	0.01517	0.00678	5.4672	5.5048	5.47	5.50
Total	40	5.4550	N/tex	0.11927	0.01886	5.4169	5.4931	5.27	5.77

\* The descriptive statistics feature of SPSS can also give summary statistics such as the mean, median and standard deviation.

Table 7. Variance analysis results for Tensile strength.

		Sum of Squares	DF	Mean Square	F Value	P Value	
Between Groups	(Combined)	0.484	4	0.069	31.434	0.066 > 0.05	
	Linear Term	Contrast	0.346	1	0.346	157.033	0.065 > 0.05
		Deviation	0.139	3	0.023	5.501	0.057 > 0.05
	Within Groups		0.070	32	0.002		
	Total		0.555	39			

Table 8. Multiple Comparisons (Post Hoc Tests).

(I)	(J)	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
1 (Virgin)	2	0.24600(*)	0.02967	0.066	0.1856	0.3064
	3	0.25400(*)	0.02967	0.065	0.1936	0.3144
	4	0.22800(*)	0.02967	0.057	0.1676	0.2884

\* The mean difference is insignificant at the 0.05 level.

bleached fabrics was measured. These data were analyzed using the SPSS software. A confidence level of 95% (95% Confidence Interval for Mean) was selected for cases that required variance analysis. The results of this study are presented in Tables 6-8. It can be seen that in terms of statistical analysis, all the values have insignificant differences compared to the unbleached raw PET fabrics.

In the first cell in Table 8, which is marked with pink color, it can be seen that the tensile strength value of the raw unbleached PET fabric (code

1) do not have a significant difference with the other bleached fabrics (code 2 to 4) (because it is marked with an asterisk \* have been explained at the end of the table that the asterisk values do not differ significantly from the raw sample at the 95% confidence level).

## CONCLUDING

Nowadays, the textile industry has emphasized on the use of innovative pollution-free and ecologically friendly knowledge called



“green technology”. In this effort an innovative bleaching procedure is applied for eliminating the contaminations of PET fabrics in the mild situations. The following conclusion can be drawn from the present study: 10.0-hours bleaching procedure is described for the 10-hours bleaching of PET fabrics at 25°C in a H<sub>2</sub>O<sub>2</sub>/ Na<sub>4</sub>P<sub>2</sub>O<sub>7</sub> liquor solution. The 10.0-hours bleached PET fabrics showed the maximum L\* value (+91.128) and the minimum C\* value (3.2616). The SEM image of the PET fabrics bleached with H<sub>2</sub>O<sub>2</sub> for 10.0 hours demonstrated that there was few nano-sized remains (dusts, waxes and impurities) on the fabric bleached with H<sub>2</sub>O<sub>2</sub>. The tint attribute of PET bleached fabrics does not shift from the first quarter of the hue area. It means that the tint effect of bleached fabrics does not change from reddish-yellowish white shade. It was observed that the 10.0-hours bleached PET fabrics showed weight loss of approximately 0.07%. This can be explained through the removal of disagreeable wastes such as waxes, pectin, suits, proteins, pigments, hemicelluloses, and other impurities from the fabrics in the bleaching process. It can be realized that in terms of statistical analysis, the Tensile strength of PET fabrics have insignificant differences compared to the unbleached raw PET fabrics.

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#### CONFLICT OF INTEREST

The author declares that there are no conflicts of interest regarding the publication of this manuscript.

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