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The Effects of Hydrogen Peroxide and Sodium Hypochlorite Oxidizing Treatments on the Color Properties of Naturally Colored Green Cotton

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Abstract

The cultivation of naturally colored cotton has captured the attention lately due to the increasing environmental concerns and problems. The green color in cotton fibers is owing to a lipid biopolymer (suberin) sandwiched between the lamellae of cellulose microfibrils in the secondary wall. Naturally colored green cotton fiber does not need dyeing or coloration process due to their inherent color characteristics leading to more ecological and sustainable textile production. Naturally colored cotton growers and producers have less requirement for the pesticides, insecticides since these varieties have already insect and disease-resistant qualities as well as they exhibit property for drought and salt tolerant leading to more environmental friendly cotton fiber production. The colorimetric (CIE L*, a*, b*, C*, h°, K/S etc.) properties of studied naturally green colored Turkish cotton fiber were explored before and after scouring (with NaOH), and oxidizing processes with hydrogen peroxide and sodium hypochlorite (with different sodium hypochlorite concentrations and different oxidizing treatment periods and at different pH levels, with or without scouring process as a precursor treatment etc.) in comparison with their greige (un-treated) counterpart. The application of scouring process with NaOH to naturally green colored cotton fiber increases the color yield levels leading to darker appearance. Similarly, hydrogen peroxide and sodium hypochlorite oxidizing processes resulted in an increase in the color yield levels of naturally green colored cotton fibers leading to darker appearance. Even at high sodium hypochlorite concentrations such as 200 ml/l at pH 12 and room temperature for 48 hours, the naturally green colored cotton fibers maintain their color.

Key words

naturally colored cotton, oxidizing, sodium hypochlorite, hydrogen peroxide, color

1. INTRODUCTION

It is known that naturally colored cotton originated around 5,000 years ago in the Andes region. Therefore, naturally colored cotton is not a novel concept and its cultivation history dates back 5000 years [1-4]. Naturally colored cottons, containing natural cotton fibers with natural pigments, have been planted for thousand years but have captured limited interest till the recent years owing to their short fiber length and poor fiber strength features [5, 6]. With the sophisticated genetic breeding techniques, nevertheless, naturally colored cotton fibers in various

shades of green and brown have been effectively manufactured with existing conventional textile machineries [7]. Unlike conventional cotton fibers dyed with synthetic dyes, the colors of which tend to fade, the natural color of the naturally colored cotton fiber actually deepens after repeated washings [8]. Apart from their inherent color characteristics, these fibers also display natural flame resistance properties leading to increased attention for different end-use applications [9,10]. Naturally colored cotton fiber is naturally pigmented fiber which grows in different shades of green and brown. Historical records also state the existence of browns with the shades of pink and lavender tints [11]. Naturally colored cottons have a minor niche market and naturally colored cotton fibers available today are usually shorter, weaker, and finer than regular Upland cottons. However, these fibers can be spun effectively into ring and rotor yarns for many different applications [12]. These fibers also can be blended with conventional off-white cotton fibers or mixed amongst themselves. Naturally colored cotton fibers are currently grown in China, Peru, and Israel. Also, Turkey produces naturally colored cotton fibers in different shades of brown and green. Different shades of brown and green naturally colored fibers are the main colors which are available in the market. Other different colors such as mauve, mocha, red are also available in Peru in a very limited supply and some other new shades are under investigation. The color for brown and red-brown naturally colored cotton fibers appears to be in vacuolar tannin material bodies in the lumen of the fiber. The different shades of brown and red-brown are mostly because of catechin–tannins and protein–tannin polymers [13].

On the other hand, green color in naturally colored cotton fibers is because of a lipid biopolymer (suberin) sandwiched between the lamellae of cellulose microfibrils in the secondary wall [13-14]. It is right point to state that brown naturally colored cotton fibers (and white lint) do not comprise suberin. Green naturally colored cotton fibers are categorized by high wax content (14 –17% of the dry weight) while white and brown fibers comprise about 0.4 –1.0% wax [13]. Latest research studies regarding naturally colored cottons reported that naturally colored brown cotton fiber is very similar in morphology to off-white (white) cotton fiber while naturally colored green cotton fiber is different as it comprises suberin. Suberin comprising primarily bifunctional fatty acids can theoretically generate a three dimensional network in the existence of glycerol, which is also found in green cotton fiber nevertheless not in white cotton fiber. How this 3D network affects the structure of the individual crystallites of cotton fiber cellulose was examined in that study. According to their outcomes, the researchers stated that the presence of suberin does not affect the structure of the individual crystallites nonetheless obstructs the development of the crystallites in the naturally colored green cotton fibers [15]. In the literature, it is stated that green is the second important commonly occurring lint color in cotton fibers. Therefore, green color in naturally colored cotton fibers is less common than brown shade and ensues primarily in two shades such as green and lighter green shade. Green color is more prone to fading and naturally green colored cotton fiber fades quicker than the naturally brown colored cotton fiber. Extended exposing to sunlight during boll opening resulted in quick fading of green color and the color turns to white, off-white or brownish shades. It is also reported that part of lint that is not directly subjected to sunlight preserves its original lint color [16]. It is known that the loom state cotton fiber fabric contains approximately 8-12% natural impurities of total weight of the cotton fiber [17]. Alkali treatment, scouring or hydropilizing treatment are generally applied to cotton fibers in order to remove these impurities in the cotton fibers. Although bleaching and/or dyeing processes may generally not be applied to naturally colored cotton fibers, scouring process (alkali treatment or hydropilizing treatment) is generally applied to naturally colored cotton fibers in order to increase the hydrophilicity levels of cotton fibers. In comparison to off-white cotton fibers, naturally brown colored cotton fibers have similar percentage of wax in their content however naturally green colored cotton fibers have much higher wax content in the fiber. This higher wax content of naturally green colored cotton fiber with pectin and lignin could decrease the moisture absorbency of treated naturally green colored cotton fibers with warm water and NaOH solution to decrease the hydrophobic content and enhance moisture regain of green cotton fibers. After alkali treatment process, naturally green colored cotton fibers displayed higher moisture absorbency and decrease on the crystalline region of the fiber. [18].

Hypochlorites as bleaching substances are still popular even though the anti-chlorine lobby and ecological pressures against the usage of hypochlorites in textile processes. Sodium hypochlorite, a sodium salt of hypochlorous acid (HOCl), solution is strongly alkali (pH ~ 11.55) and the free caustic presence in the solution acts as a stabilizer. After bleaching with hypochlorite, the fiber is generally treated with dilute hydrochloric acid to neutralize any alkaline present in the fabric. An antichlor treatment with sodium thiosulphate or bisulphite can be also advised to eliminate any residual chlorine from the fabric. Finally, the fabric is then washed with water to rinse out acid from the fabric [17]. Hydrogen peroxide is an oxidizing agent and also very common for cotton bleaching process. As mentioned earlier, even though bleaching process may generally not be applied to naturally colored cotton fibers, the behavior of naturally colored cotton fibers and their colorimetric property changes (such as color permanency performance and shade shifting possibility etc.) after the application of bleaching treatments such as hydrogen peroxide and sodium hypochlorite oxidizing treatments can arouse curiosity and interest. Therefore, in this study, colorimetric (CIE L^*, a^*, b^*, C^*, h^0, K/S etc.) properties of studied naturally green colored cotton fiber were investigated before and after scouring (with NaOH), and oxidizing processes with hydrogen peroxide and sodium hypochlorite (with different sodium hypochlorite concentrations and different oxidizing

treatment periods, with or without scouring process as a precursor treatment etc.) in comparison with their greige (un-treated) counterpart.

2. MATERIALS AND METHODS

Naturally green colored cotton unique fiber type was selected and cultivated for this study under the control of the Turkey Nazilli Cotton Research Institute in the Aegean region of Turkey. This utilized naturally green colored unique Turkish cotton fiber is *Gossypium hirsutum L.* Visual appearance and properties of the used naturally green colored cotton fiber type are given in the Table 1.

Table 1 Detailed information of used Turkish naturally green colored cotton fiber [19]

Registered Name of the Cotton Fiber	**Akdemir**
Visual appearance	
Color of the fiber	Green
Scientific Species Name	*Gossypium hirsutum* L.
Registered fiber length (UHM) (mm)	25.8
Registered fiber fineness (micronaire)	3.3
Registered average fiber strength (1000 lb/inch^2)	72.9
Registered Ginning efficiency (%)	21.6
Average Days of maturity (days)	116
Plant type	In conical form

Scouring and Oxidizing Processes

Scouring process (with NaOH), conventional bleaching process [with hydrogen peroxide (H_2O_2), and sodium hypochlorite (NaOCl)] and the combination application of scouring and bleaching processes together (scouring + bleaching) were applied to studied naturally colored green Turkish cotton fiber type in order to examine their colorimetric (CIE L^*, a^*, b^*, C^*, h^0, K/S) properties before and after different wet pre-treatments. The application procedures of implemented scouring and bleaching processes for studied naturally colored green cotton fibers are shown in Table 2. In this study, there are three different types of pre-treatment processes. First one is only scouring process with sodium hydroxide. The second one is a direct bleaching process with hydrogen peroxide or sodium hypochlorite without any scouring process involvement (directly from greige to bleached). The third process type is the combination sequential usage of scouring and bleaching processes (scouring then bleaching = scouring + bleaching with hydrogen peroxide or sodium hypochlorite). Moreover, different sodium hypochlorite oxidizing treatments (in varying concentrations: 40-100-200 ml/l sodium hypochlorite at various pHs: pH 11, pH 11.7, and pH 12) were applied to naturally green colored cotton fibers. Scouring and hydrogen peroxide bleaching processes of naturally green colored cotton fibers were carried out at 90ºC for 30 minutes and 60 minutes, respectively, using Ataç Lab Dye HT model IR sample dyeing machine via the exhaustion process. Different sodium hypochlorite oxidizing treatments were applied to green cotton fibers at ambient room temperature (~20ºC) for 6, 24 and 48 hours. In this study, the effects of bleaching with sodium hypochlorite at their inherent natural pH levels but without the involvement NaOH on the color properties of naturally green colored cotton fiber was also examined at ambient room temperature (The rightmost hand side column of Table 2). Afterwards, all treated naturally green colored cotton fibers were washed firstly with warm water for 10 minutes then rinsed under tap water for 10 minutes. After washing cycles, fibers were flat-air-dried at room temperature. Then, the changes in the colorimetric properties of green naturally colored cotton fibers were determined after these pre-treatment processes in detail in comparison with their greige cotton fiber counterpart.

Table 2 Application conditions of implemented scouring and oxidizing processes to the naturally green colored cotton fiber

Application Conditions	Scouring	Bleaching with hydrogen peroxide	Bleaching with sodium hypochlorite	Bleaching with sodium hypochlorite
Concentrations	1 ml/l sequestering agent 1 ml/l non-ionic wetting agent % 2 caustic soda (NaOH)	2.5 g/l caustic soda 2.5 ml/l hydrogen peroxide (50%) (H_2O_2) 2 g/l non-ionic wetting agent 1 g/l stabiliser	3 g/l caustic soda 1 ml/l non-ionic wetting agent 40 ml/l sodium hypochlorite (at pH 12)	1 ml/l non-ionic wetting agent 40-100-200 ml/l sodium hypochlorite (at pH 11, 11.70, 12)
Temperature (°C) **Time** **Liquor ratio**	90 30 minutes 1/25	90 60 minutes 1/25	20 6, 24, 48 hours 1/25	20 6, 24, 48 hours 1/25
After treatment (Washings)	Rinsing at 50 °C for 2 minutes then cold washing for 2 minutes	Rinsing at 50 °C for 2 minutes then cold washing for 2 minutes then neutralization with 1ml/l acetic acid for 2 minutes afterwards cold washing for 1 minute	Rinsing at 20 °C for 30 minutes with 10 g/l sodium sulfite	Rinsing at 20 °C for 30 minutes with 10 g/l sodium sulfite

Colorimetric Measurements

The CIE L^*, a^*, b^*, C^*, and h^0coordinates were measured and the K/S (Color strength) values calculated from the reflectance values at the appropriate wavelength of maximum absorbance (λmax) for un-treated and treated naturally colored green cotton fiber samples using a DataColor SpectraFlash 600 (DataColor International, Lawrenceville, NJ, USA), spectrophotometer (D65 day light, 10° standard observer). Each naturally colored green cotton fiber sample was read in four different areas, twice on each side of the fibers for consistency, and the average value was calculated and reported. The definitions and color measurement equations are shown below [20];

$L^* = Lightness, darkness\ (100 = white, 0 = black)$

$a^* = red(+a), green\ (-a)$

$b^* = yellow(+b), blue\ (-b)$

$$\text{Kubelka} - \text{Munk equation}\ (K/S) = (1-R)^2/2R \quad (1)$$

$$C^*(Chroma) = [(a^*)^2 + (b^*)^2]^{1/2} \quad (2)$$

$$h^0 = \arctan(b^*/a^*) \quad (3)$$

3. RESULTS AND DISCUSSION

Color Properties of naturally green colored cotton fibers before and after bleaching with hydrogen peroxide

When Table 3 and Figure 1 were examined, the color yield of the greige (un-treated) naturally green colored cotton was 3.7 (K/S). After scouring treatment, the color yield value increased to 6.77 (K/S) leading to darker appearance. This determination is in line with the results of the study of Kang et. al. [21]. In their study, the influence of scouring process on the colorimetric properties of naturally colored cotton fibers was investigated. Three naturally colored cotton fibers (buffalo brown, coyote brown, and green cotton) were treated with two alkali solutions (sodium carbonate and sodium hydroxide) and one enzyme solution (mixture of pectinase and cellulase). In their research, similar darkening effect was obtained for colored cotton fibers after alkali treatment. After alkali treatment, the shade of cotton fiber became deeper and darker, and the alkali treatment solutions were also deeply colorized. Their outcomes displayed that the naturally colored cotton fibers became swollen and pigment in the colored cotton fibers moved toward the outer layer of the cotton fibers following alkali treatment. The colorized solutions could be expressed by the release of pigments from the cotton fiber [21]. Direct bleaching with hydrogen peroxide without the scouring treatment application as a precursor treatment resulted in 5.75 (K/S) leading to higher color yield than greige (un-treated) green cotton fiber (Table 3 and Figure 1). It is important to state that only scoured green cotton fiber exhibited stronger color yield with darker appearance in comparison with only bleached, with hydrogen peroxide, green cotton fiber (6.77 versus 5.75 K/S). Also scoured+ bleached, with hydrogen peroxide, green cotton fiber displayed higher color yield value than greige green cotton fiber (5.44 versus 3.70; Table 3). It can be stated that overall scouring process alone, bleaching process alone with hydrogen peroxide and scouring + bleaching process with hydrogen peroxide led to stronger color yield with darker appearance in comparison with greige (un-treated) naturally green colored cotton fiber. These measured results are actually in line with the visual observation from Table 3.

Table 3 Color properties of naturally green colored cotton fiber before and after hydrogen peroxide bleaching processes

Pre-Treatment type	L^*	a^*	b^*	C^*	h^0	K/S	
Greige (un-treated)	63.4	1.5	22.3	22.3	86.1	3.70	
Scoured	50.4	-0.3	17.4	17.4	91.0	6.77	
Bleached with hydrogen peroxide	50.1	1.1	17.0	17.0	86.2	5.75	
Scoured+ Bleached with hydrogen peroxide	52.5	2.2	19.0	19.2	83.5	5.44	

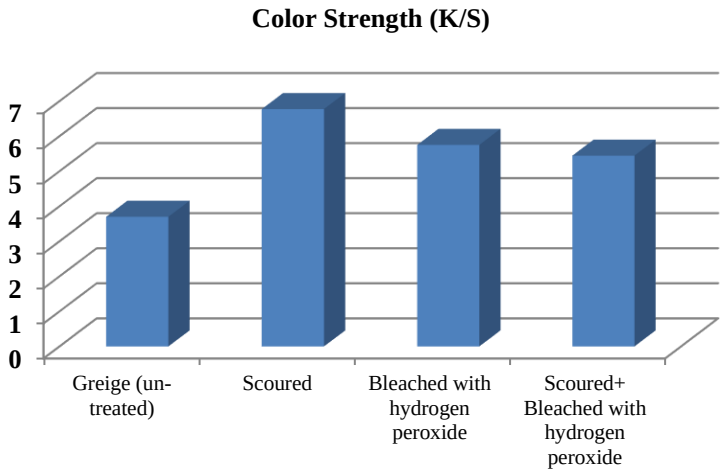

Figure 1 Measured color strength levels of green cotton fiber after scouring and hydrogen peroxide bleaching processes

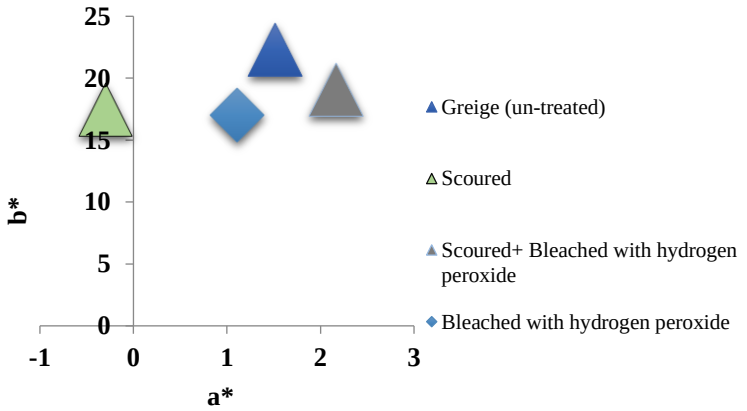

Figure 2 $a^* - b^*$ plot

Lightness (L^*) values of naturally green colored cotton fiber decreased after all three treatment types of scouring process alone, bleaching process alone with hydrogen peroxide and scouring + bleaching process with hydrogen peroxide leading to darker appearance in comparison with the greige green cotton fiber, as expected, which is in parallel with the aforementioned color yield increase after these processes (Table 3). When $a^* - b^*$ plot (Figure

2) is examined, scoured naturally green colored cotton fiber were slightly less yellow and slightly more green. When it comes to hue angle (h^0) results (Table 3), the hue angle of naturally green colored cotton fiber increased after scouring alone treatment from 86.1 to 91.0. Green cotton fiber bleached with hydrogen peroxide alone treatment displayed similar hue angle level with the greige green cotton fiber. On the other hand, scouring + bleaching with hydrogen peroxide combination process resulted in lower hue angle value than greige green cotton fiber (83.5 versus 86.1, Table 3). These measured slight hue shifts were in line with the visual observation from Table 3.

Color properties of naturally green colored cotton fiber before and after bleaching with sodium hypochlorite

When Table 4 and Figure 3 were examined, bleaching treatments with sodium hypochlorite in company with NaOH, without scouring process involvement as a precursor treatment, (direct bleaching without scouring process) displayed higher color yield values with higher K/S values (5.54, 5.49, 5.92; Table 4) than the color yield value of greige green cotton fiber (3.7).

It was found that the period (in hours) of hypochlorite bleaching application at room temperature did not exhibit an important influence on the color yield level of green colored cotton fiber. Scoured and then bleached, with sodium hypochlorite in company with NaOH, green cotton fibers also displayed high color yield values (K/S of 6.34, 6.56, and 6.29 for 6, 24 and 48 hours, respectively; Table 4 and Figure 3) which were slightly lower than the color yield of only scoured green cotton fiber (6.77, K/S).

Table 4 Color properties of naturally green colored cotton fiber before and after sodium hypochlorite bleaching processes

Pre-Treatment type	L^*	a^*	b^*	C^*	h^0	K/S	
Greige (un-treated)	63.4	1.5	22.3	22.3	86.1	3.70	
Scoured	50.4	-0.3	17.4	17.4	91.0	6.77	
Bleached (with 40 ml/l hypochlorite at pH 12) for 6 hours	53.6	0.6	18.3	18.3	88.0	5.54	
Bleached (with 40 ml/l hypochlorite at pH 12) for 24 hours	54.6	1.0	19.3	19.3	87.0	5.49	
Bleached (with 40 ml/l hypochlorite at pH 12) for 48 hours	53.3	0.6	19.0	19.0	88.1	5.92	
Scoured + *Bleached (with 40 ml/l hypochlorite at pH 12) for 6 hours*	52.0	0.6	19.0	19.0	88.1	6.34	
Scoured + *Bleached (with 40 ml/l hypochlorite at pH 12) for 24 hours*	51.0	-0.1	18.8	18.8	90.4	6.56	
Scoured + *Bleached (with 40 ml/l hypochlorite at pH 12) for 48 hours*	49.7	-0.2	17.2	17.2	90.8	6.29	

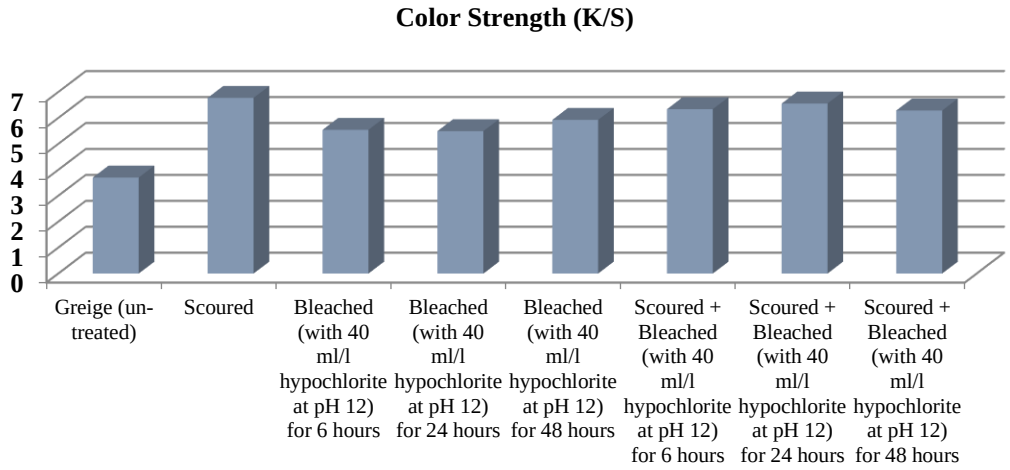

Figure 3 Measured color strength levels of green cotton fiber after scouring and sodium hypochlorite bleaching processes

Lightness (L^*) values of naturally green colored cotton fiber decreased after all three treatment types of scouring process alone, bleaching process alone with sodium hypochlorite and scouring + bleaching process with sodium hypochlorite leading to darker appearance in comparison with the greige green cotton fiber, as expected, which is in parallel with the aforementioned color yield increase after these processes (Table 4 and Figure 3). When $a^* - b^*$ plot (Figure 4) is examined, both bleached (bleached with 40 ml/l sodium hypochlorite at pH 12) green cotton fiber and the scoured and then bleached (scoured + bleached with 40 ml/l sodium hypochlorite at pH 12) green cotton fiber sample were slightly less red and slightly less yellow in comparison with greige green cotton fiber. Only scoured naturally green colored cotton fiber were slightly greener and slightly less yellow when compared with sodium hypochlorite bleached green cotton fiber samples (Figure 4). It is known that sodium hypochlorite solution is strongly alkali (pH ~ 11.55) and the free caustic presence in the solution behaves as a stabilizer.

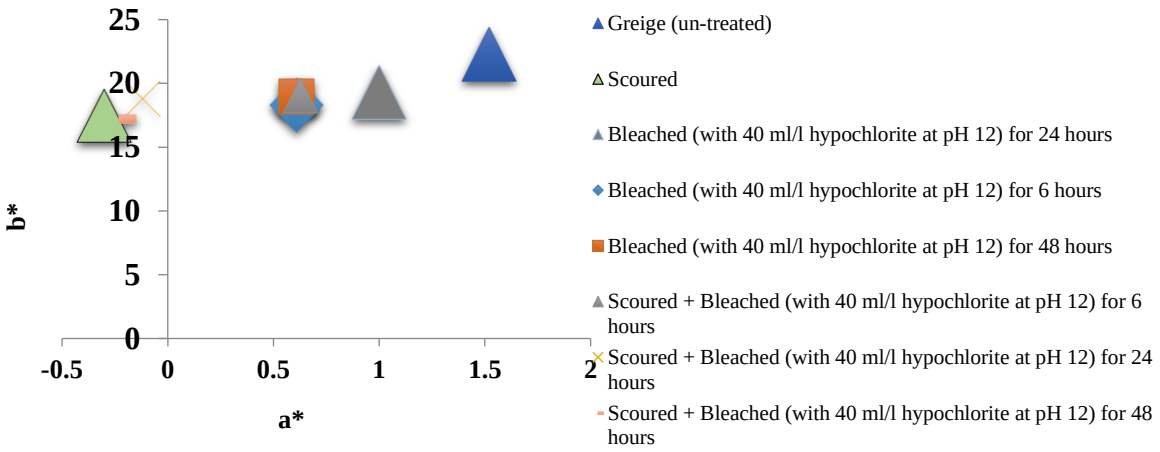

Figure 4 a -b* plot*

The effects of bleaching with sodium hypochlorite at their inherent natural pH levels but without the involvement NaOH on the color properties of naturally green colored cotton fiber was also examined at ambient room temperature. When Table 5 and Figure 5 were investigated, the bleaching process pH value was found to vary depending on the sodium hypochlorite concentration in the bleaching process. As the sodium hypochlorite concentration increases, the pH value of the bleaching bath increases. It is obvious that the color yields of naturally green colored cotton fibers after various hypochlorite bleaching processes are lower than the color yield of scoured green cotton fiber (Table 5 and Figure 5). It seems that the period of sodium hypochlorite bleaching does not cause any significant change in color yield. However, the increase in sodium hypochlorite concentration causes significant changes in color yield. As the sodium hypochlorite concentration increases, the color yield value decreases and the lightness (L^*) value increases leading to lighter appearance with lower color yield levels.

The naturally green colored cotton fiber bleached (with 200 ml/l sodium hypochlorite, such high concentration, at pH 12) for 48 hours displayed significantly lower color yield value than the color yield of scoured green cotton fiber (3.77 versus 6.77, K/S; Table 5 and Figure 5). However, it is right place to point out that this sample (bleached with 200 ml/l sodium hypochlorite at pH 12 for 48 hours) exhibited similar color yield level with the greige (un-treated) naturally green colored cotton fiber (3.77 versus 3.70, K/S; Table 5 and Figure 5). Naturally green colored cotton fibers do not lose their color after sodium hypochlorite bleaching. Sodium hypochlorite bleached naturally green colored cotton fibers are redder and yellower than the scoured naturally green colored cotton fiber (Figure 6).

Table 5 Color properties of naturally green colored cotton fiber before and after sodium hypochlorite bleaching processes varying in sodium hypochlorite concentration and application period

Pre-Treatment type	***L****	***a****	***b****	***C****	h^0	K/S	
Greige (un-treated)	63.4	1.5	22.3	22.3	86.1	3.70	
Scoured	50.4	-0.3	17.4	17.4	90.1	6.77	
Bleached (with 40 ml/l hypochlorite at pH 11) for 6 hours	52.1	0.1	17.9	17.9	89.6	5.80	
Bleached (with 40 ml/l hypochlorite at pH 11) for 24 hours	52.2	0.1	18.9	18.9	89.6	5.74	
Bleached (with 40 ml/l hypochlorite at pH 11) for 48 hours	53.4	0.1	18.3	18.3	89.4	5.50	
Bleached (with 100 ml/l hypochlorite at pH 11.7) for 6 hours	57.7	2.3	21.4	21.5	84.0	4.82	
Bleached (with 100 ml/l hypochlorite at pH 11.7) for 24 hours	58.2	2.8	21.4	21.6	82.5	4.66	
Bleached (with 100 ml/l hypochlorite at pH 11.7) for 48 hours	58.0	1.61	19.89	19.95	85.4	4.35	
Bleached (with 200 ml/l hypochlorite at pH 12) for 6 hours	63.8	3.82	23.5	23.81	80.8	3.65	
Bleached (with 200 ml/l hypochlorite at pH 12) for 24 hours	62.5	3.34	22.29	22.54	81.5	3.65	
Bleached (with 200 ml/l hypochlorite at pH 12) for 48 hours	61.6	3.32	21.77	22.02	81.3	3.77	

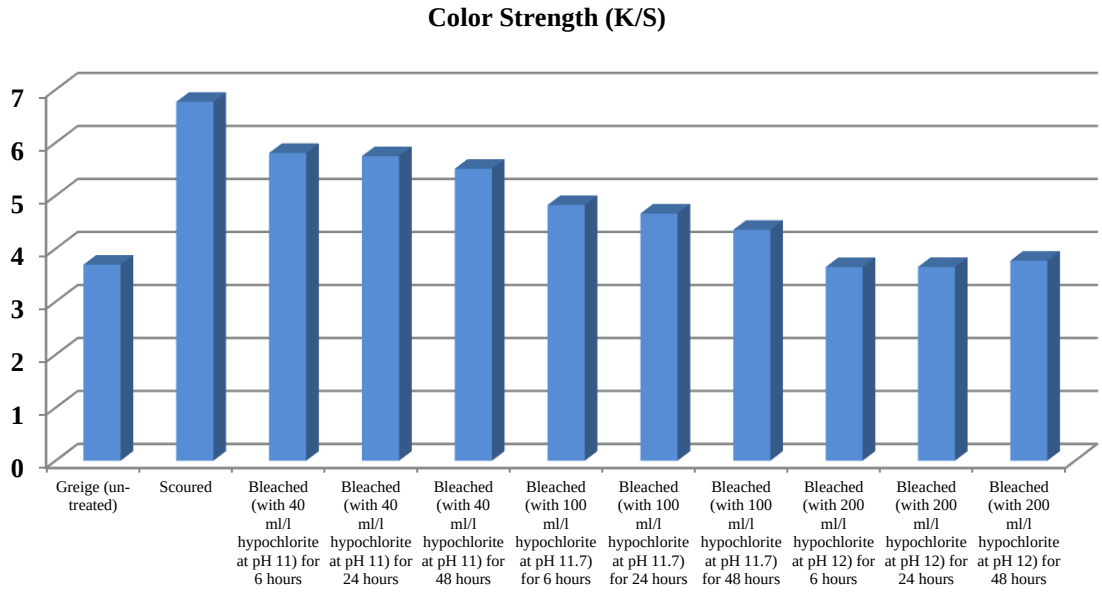

Figure 5 Measured color strength levels of green cotton fiber after scouring and sodium hypochlorite bleaching processes varying in sodium hypochlorite concentrations

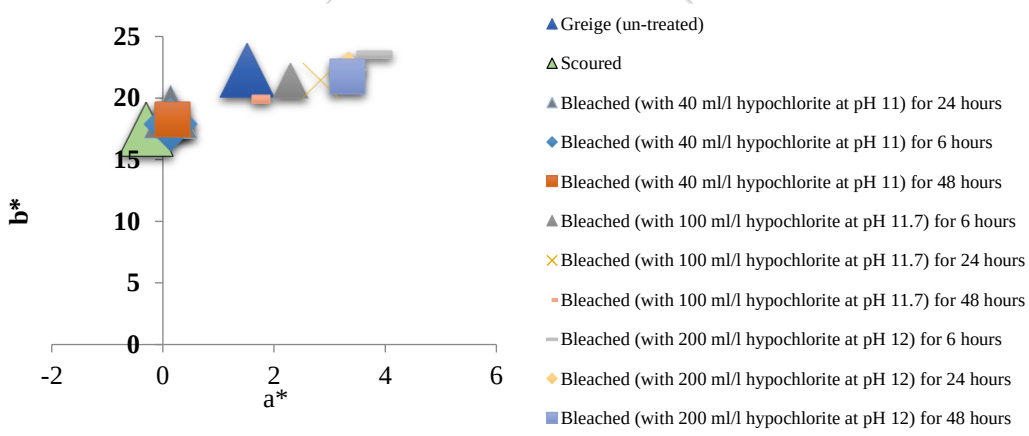

Figure 6 a -b* plot*

4. CONCLUSIONS

The cultivation of naturally colored cotton fiber has captured the attention lately due to the increasing ecological concerns and problems. In this study, colorimetric (CIE L^*, a^*, b^*, C^*, h^0, K/S etc.) properties of studied naturally green colored Turkish cotton fiber were explored before and after scouring (with NaOH), and oxidizing processes with hydrogen peroxide and sodium hypochlorite (with different sodium hypochlorite concentrations and different oxidizing treatment periods, with or without scouring process as a precursor treatment etc.) in comparison with their greige (un-treated) counterpart. Naturally green colored cotton unique fiber type (*Gossypium hirsutum L.*) was selected and cultivated for this study under the control of the Turkey Nazilli Cotton Research Institute in the Aegean region of Turkey. According to the results of this study, the application of scouring process with NaOH to naturally green colored cotton fiber increases the color yield levels and darkens the color leading to darker appearance. It is known that natural inherent pigments inside the naturally colored cotton fibers moves toward the outer layer of the naturally colored cotton fibers after alkali scouring process leading to darkening effect and therefore darker appearance. Hydrogen peroxide and sodium hypochlorite bleaching oxidizing processes resulted in an increase in the color yield levels of naturally green colored cotton fibers leading to darker appearance. The further increase of the sodium hypochlorite concentration leads to the decrease on the color yield levels leading to lighter appearance. However, this drop in color yield is close to the color values of the un-treated greige naturally colored green cotton fiber sample. Even at high sodium hypochlorite concentrations such as 200 ml/l at pH 12, the naturally green colored cotton fibers maintain their natural color.

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