

Improving Photo-Stability of Linen Fabrics by Gallic Acid Doped Silica Sol Coating

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ABSTRACT: The fading mechanism in colored textile material caused by the light effect occurs by degradation of the dyestuff structure with UV radiation or photo-oxidation in material with the visible light. In this current paper, different solutions containing silica sol with crosslinker, crosslinker-free silica sol and only aqueous antioxidant solutions were prepared by using gallic acid as additive which is also known as oxidation inhibitor. It is aimed to investigate the effect of using antioxidant with silica sol as binder on fabrics. Solutions were transferred to blue, red and yellow colored linen woven fabrics by applying pad-dry-cure method at two different curing temperatures. Untreated and treated fabrics were exposed to artificial light for 72 hours. In every twenty-four hour period, the color changes of fabrics were measured by using spectrophotometer. In this way, total color difference (ΔE), difference of lightness (ΔL^*), difference of redness-greenness (Δa^*), difference of yellowness-blueness (Δb^*) between reference and sample was obtained. The effect of prepared solutions on surface morphology and change in colors of fabrics was also investigated. It was found that gallic acid doped silica sols with cross-linker improve the light fastness of fabrics slightly.

KEY WORDS: Silica Sol, Gallic Acid, Linen Fabric, Light Fastness.

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I. INTRODUCTION

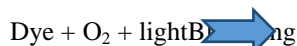
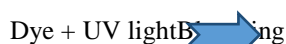
Many textile products expose to light at the stage of production and use. Photo-degradation is the breaking of the polymer chains caused by light. Oxygen and the energetic free radicals trigger the polymer degradation and are often accelerated by factors such as sunlight, heat, mechanical stress and metal ion contaminants [1]. There are two principle types of photo-degradation [2]. The first principle is change in shade and/or depth of shade of the colorant and the second is chemical degradation of the substrate. When a colored polymeric exposed to light, it will absorb radiation with a wavelength of 300-400 nm and cause degradation of any colorant present, changing or removing the color of product [3]. It was declared that UV light-induced unimolecular decomposition and visible light-induced photo-oxidation were the two most important pathway [4].

Oxidation is the major cause of degradation of colorant. Antioxidants are organic compounds that are added to oxidizable organic materials to retard auto-oxidation [5]. Hindered phenols, secondary aromatic amines, certain sulphide esters, trivalent phosphorous compounds, hindered amines, certain sulphide esters, trivalent phosphorous compounds, hindered amines, metal dithiocarbamates and metal ditiophosphates are effective chemical classes of antioxidant to prevent oxidation. In this study, gallic acid and l-ascorbic acid were used to prevent light-induced oxidation in linen fabrics. Gallic acid (GA, 3, 4, 5 trihydroxybenzoic acid) is a natural plant triphenol that constitutes tannin and some alkylesters including ethyl-, propyl-, butyl-, octyl-, and laurylgallates are known as antioxidants [1]. L-ascorbic acid is the primary dietary sources of vitamin C and includes esters of ascorbic acid (vitamin analysis for the health and food sciences). Those two both of antioxidants have high water solubility.

In a previous study, Cristea and Vilarem used vitamin C and gallic acid to improve the light fastness of natural dyed fabrics and they found that these antioxidants were efficient additives for weld [6]. Thiagarajan and Nalankilli concluded that combined application of antioxidant and ultraviolet absorber enhanced the light fastness of reactive dyed cotton fabrics [7].

Sol-gel technology has become one of the most common technique used in manufacturing functional products. Organosilica functional coatings obtained by the versatile sol-gel process which can be basically prepared by hydrolysis of the precursor material and subsequent condensation reactions, the coating process and the next drying and curing [8,9]. Silica sol is used to incorporate silica particles as filler [10].

It has been reported that light fastness of dyes can be enhanced by incorporation into inorganicnanosol coating [11]. Mahltig et al. (2005) has reported for modification of a purely inorganic nanosol by addition of epoxysilane or perfluoroalkylsilane compounds in small amounts to improve the photo-stability [12]. Co-embedding of UV absorbers and the organic dyestuff into sol-gel derived matrix can distinctly improve the bleaching stability. It was concluded even embedding in a pure SiO₂ sol coating with no further additive enhance the photo-stability. In this paper, antioxidants (gallic acid and l-ascorbic acid) with silica sol as binder were transferred to colored linen fabrics on the purpose of improving the photo-stability.



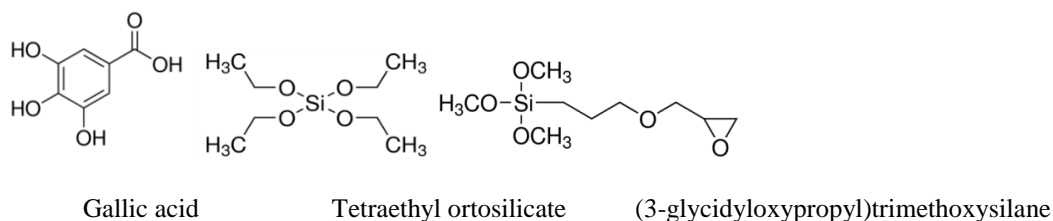
II. MATERIAL AND METHODS

2.1. Materials and Chemicals

In this study, 2/2 panama-weave, 100% linen fabrics (385 gr/m², 17 picks/cm, 20 ends/cm) were used. Warp count and weft count are 10/1 Nm.

The reactive dyestuffs used for dyeing fabrics are Bezaktiv Blue S-Matrix 150, Bezaktiv Red S-Matrix 150, Bezaktiv Yellow S-Matrix 150. All dyestuffs and dyeing chemical agents are obtained from CHT Bezema company.

Chemicals used for solutions are tetraethyl orthosilicate (TEOS) (SigmaAldrich-CAS 78-10-4, 99%); ((3-glycidyloxypropyl)trimethoxysilane (GPTS) (SigmaAldrich- CAS 2530-83-8, 98%); Gallic acid (SigmaAldrich-CAS 149-1-7, 97,5%); sodium hydroxide (NaOH) (Tekkim, 99%) and ethanol (Tekkim, 96%).



2.2. Dyeing

The linen fabrics were dyed (dye conc., 10 g/L; colorcontin, 2 g/L; meropan XPR pearls, 3 g/L; migrasol MV conc./SAP, 7 g/L; urea, 150 g/L; sodium bicarbonate, 15 g/L) by applying pad-dry-cure method at 30°C pad temperature, with pick up of 80%. The fabrics were dried at 110°C. Afterwards, dyed fabrics were cured for 180 sec at 160°C. Later, the fabrics were rinsed at 95°C and dried at 120°C.

2.3. Preparation of Solutions and Samples

Firstly, gallic acid was dissolved in distilled water (10 g/L conc.) at 70°C. Three different solutions were prepared by using gallic acid aqueous solution as additive;

(S1) The first solutions were obtained by adding TEOS (22 ml) and GPTS (8 ml) into ethanol (100 ml) and distilled water (64 ml). 0,1M NaOH (5 ml) was used for hydrolysis and the mixture was stirred (HP 220) until transparent silica sol was obtained. The last step, 100 ml gallic acid solutions was added into sols and stirred together for 30 min at room temperature (pH 3.8).

(S2) The second solutions were prepared using the previous procedure. Only difference is TEOS was used as precursor without any cross-linker addition (pH 3.5).

(S3) The third solutions did not contain any silica sol. In this method, the only gallic acid solution was used on the purpose of examining the contribution of SiO₂ (pH 2.8).

For transferring these solutions to samples, pad-dry-cure method was applied. Before this process, the samples were dried in ATAC – EV 250 oven at 105°C for 4 hours. The transfer process is presented in Figure 1. ATAC-FY-350 model fulard machine was used for the padding process and ATAC – GK ram machine was used for drying and curing process. Two different curing temperature (120°C and 150°C) was applied to examine the effect of curing temperature.

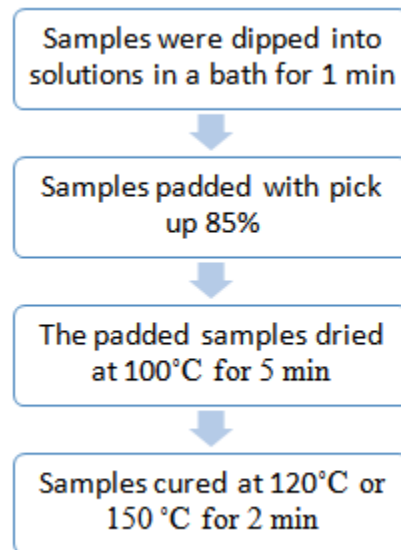


Figure 1: The transfer process

2.4. Characterization of Coated Samples

For characterization of the samples treated with silica sol, scanning electron microscope (SEM) analysis was performed. The surface morphology of untreated and treated with silica sol samples that obtained by using FEI Quanta 650 Field Emission SEM was investigated.

2.5. Color Measurement

The color measurement was performed to investigate the effect of the chemical treatments and light exposure on color change of samples. The rate of color changes of samples were evaluated by using Minolta CM 3600 spectrophotometer. Total color difference values (ΔE), difference of lightness (ΔL^*), difference of redness-greenness (Δa^*), difference of yellowness-blueness (Δb^*) between treated and untreated samples were calculated by CIELAB system. In CIELAB color space; lightness increases with the decrease of L^* value, positive a^* is red, negative a^* is green; positive b^* is yellow, negative b^* is blue [13].

$$\begin{aligned} \Delta L^* &= (L^* \text{ sample} - L^* \text{ standard}) \dots\dots\dots(1) \\ \Delta a^* &= (a^* \text{ sample} - a^* \text{ standard}) \dots\dots\dots(2) \\ \Delta b^* &= (b^* \text{ sample} - b^* \text{ standard}) \dots\dots\dots(3) \\ \Delta C^* &= (C^* \text{ sample} - C^* \text{ standard}) \dots\dots\dots(4) \\ \Delta h^* &= (h^* \text{ sample} - h^* \text{ standard}) \dots\dots\dots(5) \\ \Delta E &= [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \dots\dots\dots(6) \end{aligned}$$

2.6. Light Exposure

The treated and untreated blue, red, yellow samples were exposed to fading under xenon light source in Atlas Xenotest Alpha light fastness tester based on ISO 105 B02:2014 standard. During the fading process, the humidity in the device was 40%; the detected temperature at the black standard temperature (BST) was 48°C, the radiation amount was measured at 81 W/m². The samples exposed to light for 72 hours. All tests were performed in two replicates.

In some previous studies, color changes of fabrics due to light exposure was measured by using CIELAB system and total color difference value (ΔE) was obtained. Hence, even the slightest color change can be determined and it is possible to perform statistical analysis by this method. In our study, the color measurement of the samples before and after fading were performed in every twenty four hour period and ΔE , ΔL^* , Δa^* , Δb^* values were obtained.

2.7. Wash Durability

Durability of applied chemical treatments after washing was evaluated by washing samples for 30 minutes at 40°C with ECE reference detergent according to the ISO 105 C01 standard. After five washing cycle was performed, the samples were exposed to light with unwashed samples at the same time and then color measurements were performed.

III. RESULTS AND DISCUSSIONS

3.1. Characterization

The images of untreated and silica sol coated samples are given in Figure 2. It is seen that the surface morphology of the samples treated with silica sol is clearly different from the untreated sample. The coating materials wrapped around the fiber surface. SiO₂ matrix made the fiber structure thicker and caused roughness. It is seen that bridges between the fibers are formed. In addition, different shapes of particles on the fiber surface is observed.

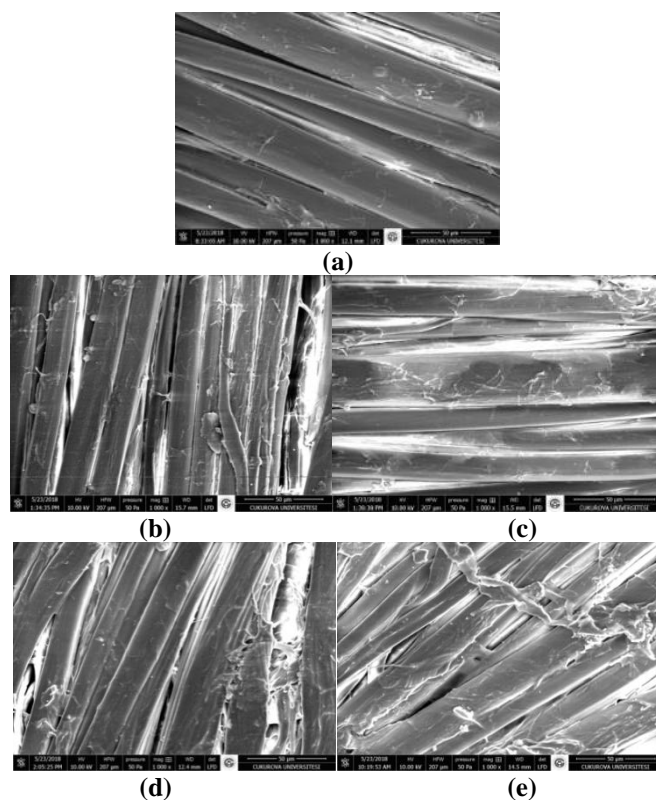


Figure 2: Images of samples; untreated (a), treated with gallic acid doped silica sols containing GPTS and cured at 120°C (b), treated with gallic acid doped silica sols containing GPTS and cured at 150°C (c), treated with gallic acid doped silica sols without GPTS and cured at 120°C (d), treated with gallic acid doped silica sols without GPTS and cured at 150°C (e)

3.2. Color Changes After Treatments

Changes in the color values of the blue, red and yellow linen samples after chemical treatments are shown in Table 1. The difference of lightness values (ΔL^*) of all of the samples treated with silica sol is negative, ie, their color became darker. The roughness of the fiber surface with the silica sol coating caused the surface to reflect less light. This resulted in a darker color of the fabric [14].

All of the chemical treatments increased the greenness value of blue samples and the redness value of the red samples. Silica sol treatments increased the yellowness degree of red and yellow samples and the blueness degree of red and yellow samples was increased by gallic acid aqueous solution. Considering the total color difference, the silica sol coatings caused color difference in all samples remarkably, which varies in 1.78-4.37 interval.

Table I:Color difference values of samples after treatments

Treated Sample	Color Difference Values			
	ΔL^*	Δa^*	Δb^*	ΔE
S1-120-B	-2.94	-0.396	-0.001	2.92
S1-120-R	-1.735	1.589	1.103	2.6
S1-120-Y	-0.987	1.339	2.72	3.19
S1-150-B	-2.234	-0.471	0.039	2.28
S1-150-R	-1.702	1.434	0.803	2.36
S1-150-Y	-1.487	1.91	3.104	3.94
S2-120-B	-3.512	-0.414	-0.133	3.54
S2-120-R	-3.317	2.243	1.745	4.37
S2-120-Y	-1.371	1.506	2.784	3.45
S2-150-B	-3.433	-0.38	0.053	3.45
S2-150-R	-1.462	1.164	1.076	2.16
S2-150-Y	-1.671	0.684	1.605	2.47
S3-120-B	2.573	-1.493	0.855	3.1
S3-120-R	-1.224	0.999	0.027	1.58
S3-120-Y	-0.341	0.091	-0.699	0.78
S3-150-B	1.243	-1.24	0.778	1.92
S3-150-R	-0.23	0.382	-0.392	0.59
S3-150-Y	-0.323	-0.341	-1.675	1.74

3.3. Color Changes After Light Exposure

As a result of periodic fading for seventy-two hours, the difference in color values of treated and untreated samples is given in the Table 2. The difference of lightness values of all treated samples is lower than the untreated samples. Also, the yellow samples treated with gallic acid aqueous solutions(S3) have become darker after light exposure. It is concluded that in the presence of gallic acid in solutions decreased the lightness value of the samples with light absorption. Nevertheless, the total color difference(ΔE) of samples in S3 group are higher than the untreated samples. This result indicates that the color of the samples shifted to different shades with the effect of light due to gallic acid aqueous solutions. After exposure to light, the color shades of the blue samples shifted to yellowness; red and yellow samples showed a trend towards green tone.

When results are examined, it is observed that the total color change of samples decreased with the contribution of silica sol. The best light fastness results in blue, red and yellow samples are treated with gallic acid doped silica sol with GPTS. The differences of ΔE values between these mentioned samples and uncoated samples are 2.83, 1.74, 1.87 for blue, red and yellow samples respectively.

Examination of the mean periodic color difference values after light exposure for the treated and untreated samples are given in Figure 3. The fading rate of all blue samples increased gradually after 48 h exposure. There is no difference in the rate of photo-fading among the blue samples in S3 group and untreated sample. When silica sol and gallic acid existed together in solutions, they increased the photo-stability of blue samples. The red samples of group S3 had been faded rapidly in seventy-two hours. Treatment with gallic acid aqueous solution caused negative effect on light fastness of samples. The red samples in S1 show more stable to light than other red samples.

Fading rates of yellow samples show different behavior by comparison with blue and red samples. Samples in S2 group had been faded with increasing acceleration. Again, yellow samples in S1 group had slower fading rate than untreated yellow one. Addition of GPTS to silica sol slightly suppressed photo-fading in all samples. Especially, when this treatment was cured at 150°C degree, the blue and red samples is much more stable to light and oxidation than untreated samples.

Table 2: Color difference values of treated and untreated samples after periodic light exposure

Sample	24 hoursexposure				48 hoursexposure				72 hoursexposure			
	ΔL^*	Δa^*	Δb^*	ΔE	ΔL^*	Δa^*	Δb^*	ΔE	ΔL^*	Δa^*	Δb^*	ΔE
S1-120-B	1,36	-0,068	2,160	2,44	2,35	0,311	2,455	3,69	5,168	0,261	3,881	6,46
S1-120-R	0,62	-4,258	-0,581	4,34	2,29	-5,918	0,061	6,39	5,222	-8,256	-0,228	9,78
S1-120-Y	0,26	-3,200	-1,538	3,56	0,55	-1,510	-3,210	3,59	0,93	-2,541	-4,581	4,83
S1-150-B	0,75	-0,049	2,246	2,42	2,05	0,042	2,921	3,65	4,2	0,142	4,102	5,87
S1-150-R	0,61	-5,020	0,363	5,07	0,89	-7,142	0,157	7,21	3,44	-9,043	-0,103	9,67
S1-150-Y	0,62	-0,710	-4,103	4,21	0,31	-3,203	-5,276	6,88	0,04	-2,816	-5,829	7,5
S2-120-B	0,79	-0,341	1,975	2,17	1,65	-0,299	3,559	3,94	3,31	-0,380	5,88	6,77
S2-120-R	0,28	-4,620	0,497	4,85	0,64	-7,636	2,074	7,95	2,33	-11,461	3,51	12,24
S2-120-Y	-0,14	-1,826	2,170	2,84	-0,09	-2,849	-4,558	5,32	-1,23	-3,122	-6,828	8,31
S2-150-B	0,98	-0,168	2,444	2,65	3,58	-0,030	3,973	4,33	4,59	0,136	6,773	8,20
S2-150-R	0,82	-4,119	1,453	4,44	1,55	-6,418	1,796	7,24	3,4	-11,014	2,932	11,89
S2-150-Y	-0,18	-1,378	-2,602	2,95	-0,26	-2,649	-4,763	5,5	-0,99	-5,890	-5,946	9,23
S3-120-B	0,56	0,270	2,617	2,69	1,3	0,216	4,442	4,67	2,74	0,464	7,601	8,18
S3-120-R	0,63	-4,580	1,875	5	1,3	-7,832	3,187	8,57	2,69	-12,347	5,384	13,77
S3-120-Y	0,26	-1,928	-2,321	3,77	-0,02	-2,569	-5,918	6,51	-1,29	-2,732	-6,748	10,90
S3-150-B	0,85	0,007	2,350	2,51	1,78	0,712	7,799	8,03	3,01	0,619	7,834	8,52
S3-150-R	1,14	-5,049	1,846	5,5	1,82	-8,050	2,853	8,87	3,99	-13,297	5,168	14,82
S3-150-Y	0,67	-2,370	-2,415	3,45	-0,12	-2,595	-5,919	6,6	-1,47	-3,121	-7,078	8,72
N-B	2,27	0,607	1,478	2,81	4	0,351	2,793	4,9	6,88	0,582	4,205	8,7
N-R	1,77	-3,946	-0,731	4,39	4,24	-6,678	-1,172	7,38	5,94	-9,618	-1,52	11,41
N-Y	0,64	-1,691	-2,820	3,35	0,84	-3,058	-4,796	5,75	1,03	-2,69	-6,05	6,7

*N : Untreated reference samples

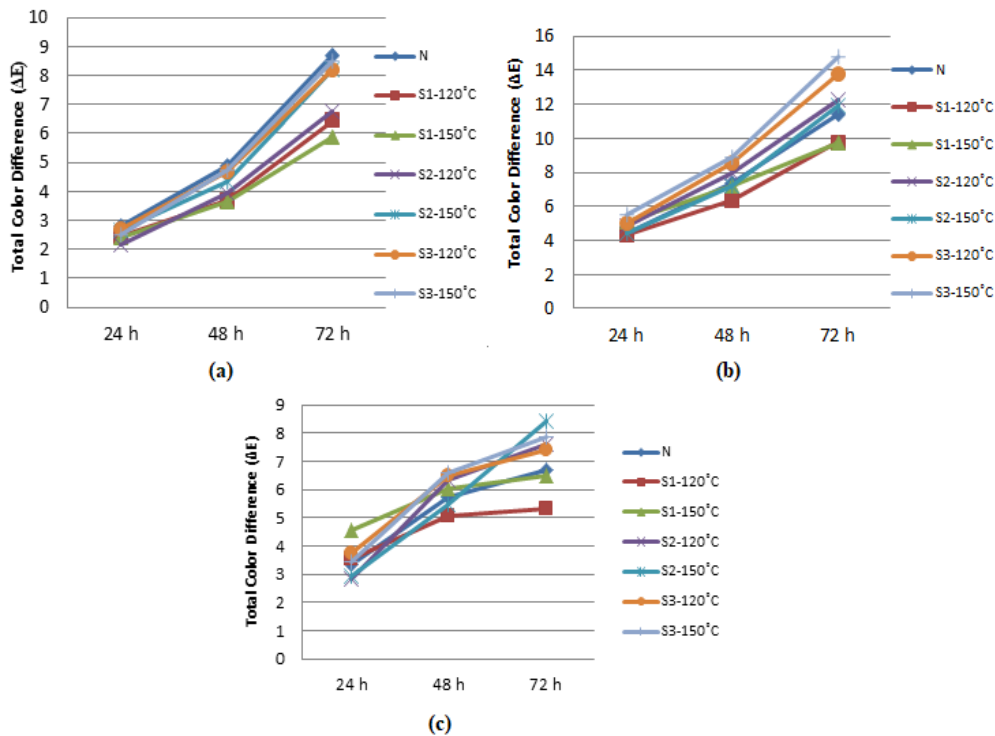


Figure 3: Color difference values for untreated (N) and treated blue (a), red (b), yellow (c) samples after light exposure

3.4. Wash Durability

The durability of treatments to five wash cycles was examined by applying the light fastness tests for all samples. The color difference values of all washed samples due to light exposure were presented in Table 3. The light fastness degree of all samples decreased after washing. The effect of light exposure on shade of all samples are; shifting to yellow in blue samples; shifting to green in red samples and shifting to blue in yellow samples. Color difference values of the blue and red samples in S1 and S2 group are still lower than the untreated samples. The presence of GPTS in the silica sol made the solutions more stable to washing, thus photo-fading in the blue and red samples could be reduced. Applying 150°C curing temperature to blue and red samples after padding process, gave better results than 120°C degree. However, the photo-fading behavior of

treated yellow samples after washing is inconsistent. The chemicals in S1 solutions which remained after washing are inefficient to increase the photo-stability of yellow samples.

The difference of lightness value of blue samples in S1 group is lower than the untreated samples. However, it was found that the lowest ΔL^* values belong to untreated red and yellow samples. In the previous section, it was suggested that gallic acid decreased the ΔL^* value of samples during photo-fading. It is estimated that, this results in the washed samples are due to some amount of gallic acid which could not bind with SiO_2 is removed from the fabric surface by washing thus those remaining on the red and yellow fabrics after washing were not effective in reduce ΔL^* value.

Table 3: Colordifferencevalues of treatedanduntreatedfivecyclewashedsamplesafter 72 h lightexposure

Fivecyclewashedsamples	ColorDifferenceValues			
	ΔL^*	Δa^*	Δb^*	ΔE
S1-120-B	8.364	1.32	4.634	9.65
S1-120-R	7.273	-12.287	-2.353	14.5
S1-120-Y	2.103	-4.803	-8.401	9.9
S1-150-B	6.784	1.278	4.227	8.09
S1-150-R	7.37	-11.229	-2.254	13.62
S1-150-Y	1.438	-4.604	-9.094	10.29
S2-120-B	7.198	0.756	6.152	9.5
S2-120-R	9.053	-14.475	-1.064	17.11
S2-120-Y	2.346	-3.527	-7.471	8.59
S2-150-B	8.289	1.152	6.228	10.43
S2-150-R	7.115	-11.43	-0.944	13.5
S2-150-Y	2.407	-3.079	-7.14	8.14
S3-120-B	8.962	0.401	5.345	10.44
S3-120-R	8.129	-11.77	-2.119	14.46
S3-120-Y	1.911	-3.067	-7.312	8.16
S3-150-B	8.473	0.607	5.746	10.26
S3-150-R	5.705	-10.724	-4.562	12.98
S3-150-Y	2.484	-3.664	-8.458	9.55
N-B	10.064	0.418	5.412	11.43
N-R	6.25	-13.149	-1.743	14.66
N-Y	1.09	-2.866	-7.244	9.79

*N : Untreated reference samples

IV. CONCLUSIONS AND RECOMMENDATIONS

As an oxidation inhibitor the gallic acid, was transferred to linen samples using the SiO_2 as a binder which prepared by sol-gel technique, thereby reducing the oxidation in the samples with the visible light was aimed. It is observed that treatments with gallic acid doped silica sol caused color differences ranging from 1.47-4.37 in all samples.

The combined treatment of gallic acid and GPTS containing SiO_2 is efficient to improve photo-stability of blue, red and yellow samples. Through this treatment, improvements in color difference values (ΔE) after light exposure are 2.83, 1.74, 1.87 for blue, red and yellow samples respectively. Treatments with gallic acid aqueous solution increased the total difference value, decreased the difference of lightness value after light exposure. Thus, it is concluded that changes in color shades of samples caused by light exposure can be reduced with the contribution of silica sol and the difference of lightness value can be decreased with the addition of gallic acid. Besides, it is observed that addition of GPTS in silica sols, provided the durability of treatments to five cycle washing slightly for the blue and red samples.

The recommended studies are as follows;

1. Gallic acid and l-ascorbic acid can be obtained in different concentrations and combined with silica sols and transferred to the samples. Other light stabilizer antioxidants may also be used.
2. The processes containing antioxidant and silica sol can also be applied to fabrics dyed with different types of dyestuff. In this way, the effect of the applied process on light fastness according to the dyestuff property can be examined.
3. UV absorber and antioxidant materials can be combined with sol-gel technique in order to improve the limited light fastness improvement obtained in the study.

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