



Influence of Public Museum Conditions on Polymer Degradation and Color Change of Historical Dyed Wool Textiles

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Abstract

Huge number of historical valuable textiles was created during civilization process. These kinds of textiles preserve and reflect the history and culture of people throughout the ages. However, some damage occurs during the storage and display of historical textiles in museums due to exposure to different environmental conditions. Hence, in this study mechanics of polymer degradation and color change of historical wool textiles by temperature in museums were investigated. To this end, wool fabrics were dyed with natural dyes, namely Madder and Safflower, to resemble the historical textiles in museums. Then, the dyed textiles have been subjected to thermal accelerated aging for variable time exposure. The mechanical, chemical, optical properties in terms of surface morphology and color parameters (CIELab) and air permeability were evaluated. The aim of this study is to present an easier approach for evaluation of the effect of temperature variation on historical textile display in the Egyptian museum.

Keywords: Aging; CIE Lab; Dyes; FTIR; Historical textiles; SEM; Wool.

1. Introduction

Historical textiles are recognized as a genuine source of information and data for history of human and anthropological request [1-6]. Historic textiles have complex structures; the weave structure of materials is irregular, making zones of shortcoming where distinctive colored yarns meet. The degradation of historical textiles happens along numerous decades of uncontrolled museums environmental conditions. This forces the scientists to understand textile's physical properties. On the other hand, subjecting textile to thermal aging procedure create changes that might require several years to occur under the natural aging, under uncontrolled museum conditions [7-9].

Therefore, accelerated-aging procedure is used to achieve important three major purposes. The first one is to create a short time relative ranking of materials with respect to their chemical stability or physical durability. The second one is to anticipate continuous safety of material damage under expected conditions of use.

Finally, clarification of the chemical reactions and the physical consequences thereof in the lab by speeding the deterioration processes [7-12]. Characteristic phenomenon, termed as aged wool fiber Figure 1, is an irreversible re-swelling behavior of natural dyed wool fabric upon thermal aging, in addition to failure in mechanical properties, change in color, and change in chemical structure of wool fibers. Therefore, there are international standards that deal with the recommended conditions for the display of historical items in museum [13, 14]. The chemical changes that occur in historical textiles during deterioration are the underlying cause of the physical changes [15-18]. To this end, the influence of thermal accelerated aging on the mechanical properties, change in color and chemical structure of dyed wool textiles was studied. Additionally, the relationship between the change in chemical structure, its potential reactions and the tendency change degree in physical property were evaluated.



Figure 1: The deterioration aspects of historical wool textiles in different Egyptian museums.

2. Research Methodology

2.1. Materials

Natural coloring Matter

Dry safflowers (Binomial name: *Carthamus tinctorius*) and madder roots were purchased from the Egyptian local market.

Fabrics

Egyptian wool fabric used in this study was supplied by Goldentex Co., Cairo-Egypt. Potassium aluminum sulphate (Alum), ferric chloride FeCl_3 and Copper sulfate $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ were purchased from Fluka. Madder dye and Safflower dye were obtained from Wild Colors, Birmingham, UK.

2.2. Dyeing Process

2.2.1. Dye Extraction

Extraction of natural yellow coloring matter:

The safflowers (*Carthamus tinctorius*) petals were peeled and crushed to the powder form. Then the coloring matter was extracted. The extraction was carried out at (40-100°C) for (10-60 min). Finally, the solution was filtered off and left to cool down.

Madder dye extraction steps:

- 1- Grinding the dry roots, to obtain a fine powder.
- 2- Prepare a 10% solution (w/v)
- 3- Soaking the powder in the distilled water, for 24 hr., to extract the color.

4- Heating the extract, with the continuous stirring, to the boiling temperature for 2hr. It may require adding a water to compensate the evaporated water during the heating process.

5- Allow the extract to be cooled and then filter, to obtain a clear colored solution.

2.2.2. Dyeing procedures

The dyeing process was performed by the exhaustion method using a material to liquor ratio (LR) of 1:20. For 1 g of goods, a dye bath volume of 20 ml is applied. Mordents, alum $\text{Al}_2(\text{NH}_4)_2(\text{SO}_4)_4 \cdot 24\text{H}_2\text{O}$, FeCl_3 , and $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ were added as concentrated solution (50 g L^{-1}) to give a final dye bath concentration of 2.5 g L^{-1} or 5 g L^{-1} mordant. After dyeing, the unfixed dyestuff was removed by rinsing three times with cold water (5 min, room temperature, LR 1:20). Afterwards, the wool fabrics was soaked in water with natural soap for 12 hours and then boiled for 1 hour and rinsed to get rid of finishing materials. Wool fabrics were placed in the dye solution with magnetic stirring at 70°C for 1.5 hours. Finally, soaking the dyed wool fabric in the mordant bath for 30 min at 60°C , then rinsed the dyed fabric with water [16, 19].

2.3. Thermal Aging

Accelerated thermal aging was used as a mean to study the effect of natural aging on the mechanical, chemical and visual properties of historical textiles in museums. In literatures, any scientist have indicated that the accelerated temperature limitation is 100°C degrees for 72 hours which is equivalent to natural aging in the museum for 25 years for historical textiles [20-21]. Therefore, the Wool samples were aged separately at 100°C for 72 hours and 144 hours in a temperature-controlled oven.

2.4. Characterization

The surface morphology of the aged dyed wool fabrics were investigated using Scanning Electron Microscope (SEM- HITACHI-SU-1500) [22]. The CIE Lab values of the thermally aged wool fabrics were measured using a double beam Optimatch spectrophotometer (Datacolor international Spectraflash SF450-UK). It is well known from the literatures that; (L^*) corresponds to the brightness (100 = white, 0 = black); (a^*) refers to the red–green coordinate (positive sign = red, negative sign = green) and (b^*) refers to the yellow–blue coordinate (positive sign = yellow, negative sign = blue) [23]. While, the change in chemical functional groups of an aged wool fabrics were monitored by BRUKER'S VERTEX 70- Fourier Transform Infra-Red Spectroscopy with Attenuation Total Reflection (FTIR-ATR) with resolution of 4 cm^{-1} [24].

The mechanical properties of un-aged and aged wool fabrics such as tensile strength (T.S) and elongation at break percentage (E%) were tested using Shimadzu Universal Tester of type S-500 Japan according to ASTM D 3822-96 [25]. Furthermore, the air permeability of dyed wool fabrics before and after aging was verified by using FX3300 Air Permeability tester SDL at $65 \pm 2 \%$ humidity and $20 \pm 2^\circ\text{C}$ according to EN ISO 9237 standard [26].

3. Results and discussions

3.1. Effect of thermal accelerated aging on wool fiber morphology

The surface morphology examination shows the manifestations of damage to wool fibers after thermal aging. Fig. 2(A) shows the typical morphology of the wool fibers before aging, the wool fibers have severe distinctive shape and smooth homogeneous surface. Fig. 2(B, C) shows the effect of thermal accelerated aging on the surface morphology of wool fibers. Different deterioration aspects of aged wool fiber are obvious with respect to shrinkage and long cracking in the wool fibers. In addition, deformity of fibers is noticed due to severe drought caused by the effect of aging.

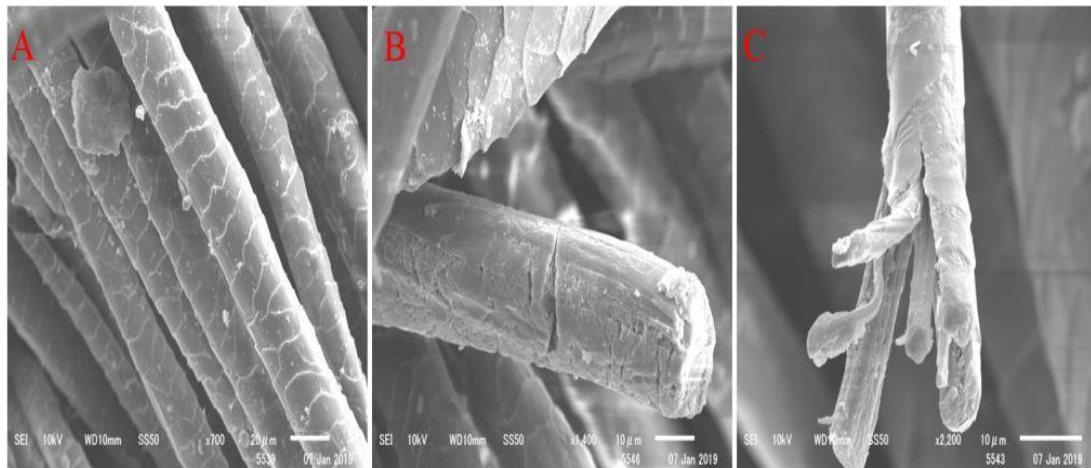
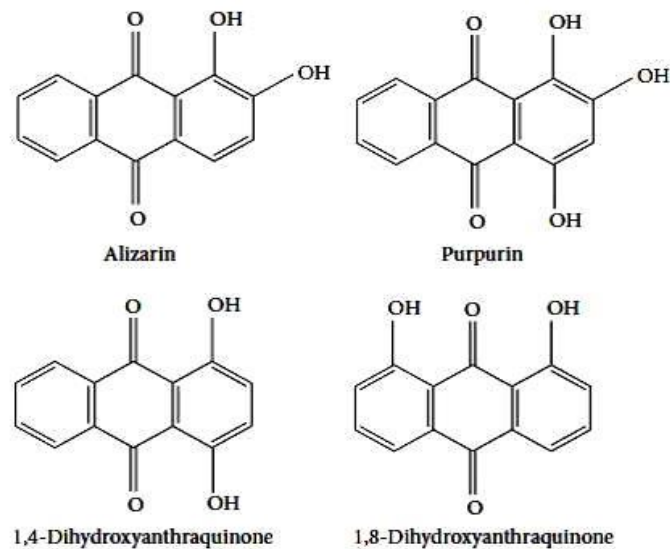


Figure 2. SEM images of wool fabrics before (A) and after aging (B, C).

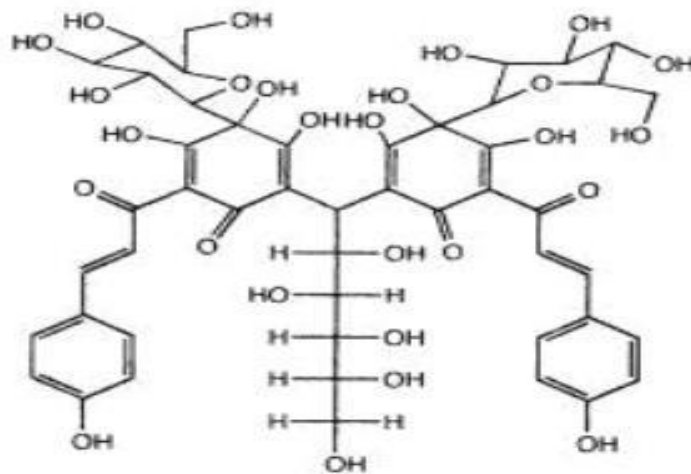
3.2. Effect of thermal aging on the chemical structure of dyed wool fabric

Madder is an old and famous dye for dyeing wool, silk, and cotton fibers [27]. It is a main source of a natural dye producing a variety of anthraquinone dyes in its roots and rhizomes. The main components are di- and tri-hydroxy-anthraquinones, alizarin and purpurin and their derivatives; ruberythric acid (alizarin-primeveroside), pseudopurpurin, and lucidin-primeveroside. Rubiadin, munjisti, quinizarin (1,4 dihydroxyanthraquinone), lucidin, nordamnacanthal, xanthop-urpurin, and 1,8-dihydroxyanthraquinone are also identified from plant tissues [28]. The chemical structure of the most important coloring compounds in madder structure is presented in Scheme 1.



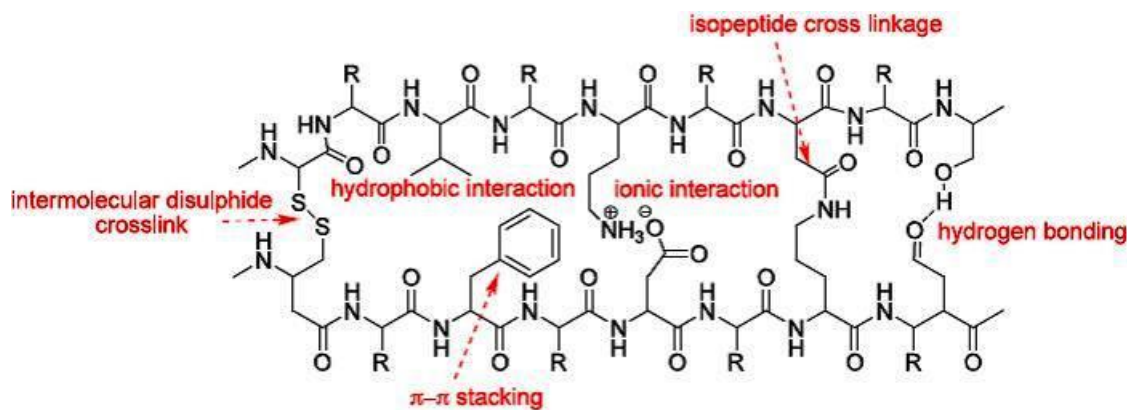
Scheme 1. Chemical structure of the main compounds in madder [29].

Safflower flower petals contain two pigment dyeing's red (Carthamine) and yellow (safflower yellow A&B). The researchers discovered that the structure of safflower red, as well as yellow pigment have unique structures of C-glycosyl - quinochalcone moieties that exist only in *Carthamus Tinctorius* [30]. The structure of safflower yellow dye is illustrated in Scheme 2.



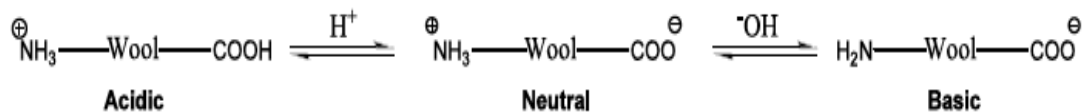
Scheme 2. Chemical structure of safflower yellow dye [30].

Meanwhile, the chemical structure of wool, shown in Scheme 3, is more complex and random. There are several interactions within the chemical structure of the wool; ranging from hydrogen bonding to intermolecular disulfide cross-linkages. These interactions are desirable in the dyeing process, to form hydrogen bonds with dye compounds, π - π stacking with aromatic compounds and ionic interactions with acid dyes [31].



Scheme 3. Chemical bonds within the structure of wool (R = 18 different amino acid groups) [31].

Wool is a zwitterion at neutral pH. The amine group can become protonated ($-\text{NH}_3^+$) and the carboxylic acid group can become deprotonated ($-\text{COO}^-$) group. Under acidic conditions the carboxylic acid groups will become protonated again and exist in their neutral form ($-\text{COOH}$). However, under basic conditions the NH_3^+ group becomes deprotonated and exists as a free amine group ($-\text{NH}_2$) [32]. This ability of wool to change its chemical properties with ease helps in the dyeing processes; as they can form hydrogen bonds with the polar functional groups (hydroxyl and carbonyl) on the dye molecules. A Simplified graphical scheme of wool in different pH conditions can be seen in Scheme 4 [31].



Scheme 4. Simplified graphical scheme of wool in different pH conditions [31]

Figures 3 & 4 show the FTIR of wool fabric dyed with madder and safflower dyes after the thermally accelerated aging. It is appeared that the amide I and amide II appeared at 1620 and 1513 cm^{-1} respectively, referring to the dyed wool fabric present are mainly in β -sheet structure, which are polypeptide chains that are hydrogen bonded to each other. The increase in the OH stretching or bending at 1650 cm^{-1} indicates the hydrolysis of the polypeptide chain of dyed wool fabric. On the other hand, the oxidation of the polypeptide chain of dyed wool fabrics would be absorbed in the $1650\text{--}1750 \text{ cm}^{-1}$ region as a result of the production of carbonyl compounds due to thermal ageing.

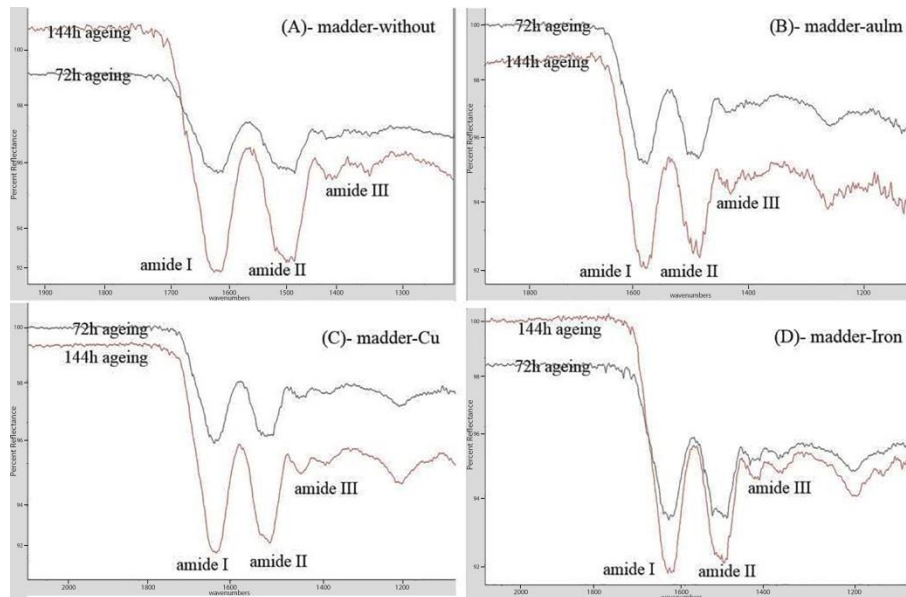


Fig. 3. The FTIR spectrum of dyed wool fabric with madder dye after the thermal accelerated aging.

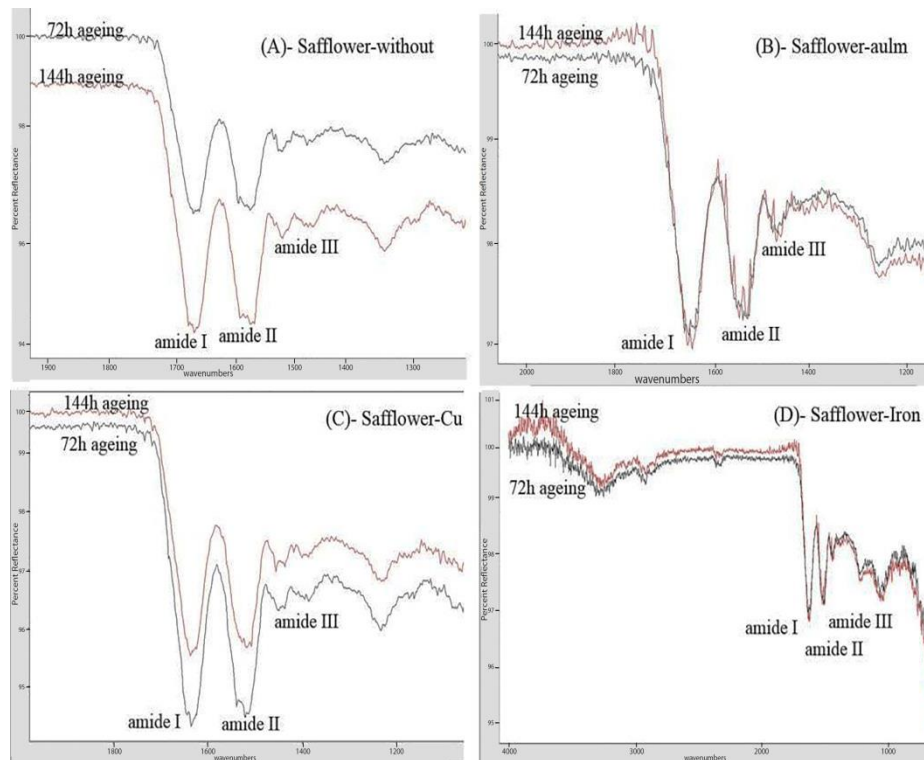


Fig. 4. The FTIR spectrum of dyed wool fabric with safflower dye after the thermal accelerated aging.

3.3. Effect of thermally accelerated aging on wool fabric mechanical properties

It is reported that natural aging in museums has a negative effective on the mechanical properties of historical wool textiles. As, the historical wool textiles loses the durability and tensile strength property upon aging. Therefore, this research will examine the effect of this phenomenon on the dyed wool textile to develop a new policy for the standard exhibition of the museum. In order to study the effect of thermal accelerated aging on the wool fabric mechanical properties. The wool fabric samples were cut with the dimensions $25 \times 5 \text{ cm}^2$, according to the standard of warp

test specimen. The warp strips were produced by raveling away yarns on each side and the test results was an average of five test samples.

Table 1 and Figure 5(A & B) designate the effect of thermal accelerated aging on tensile strength (T.S) and elongation at break percentage (E %) of wool fabric samples. It is clear from the above table and figure that, there is a decrease in the T.S of the wool samples upon aging. The T.S values of wool dyed with safflower dye mordanted with alum was 251.3 N, while, after aging for 72 hours it decreases to 237.9 N. Further decrease in T.S was noticed after prolonged aging for 144 hours to 230.2 N. In addition, there is a decrease in the E% values of the wool samples after aging. For example, the E% values of wool dyed with safflower dye mordanted with alum were 27.57%; then, decreases to become 25.47% after aging for 72 hours.

Table 1. Mechanical properties of dyed wool fabric before and after thermal aging.

No	Samples	Mechanical Properties	
		Tensile Strength (N)	Elongation (%)
Wool Fabrics dyed with natural dyes before ageing			
1	Wool + Saff+ Alum	251.3	27.57
2	Wool + Saff	234.2	31.88
3	Wool + Saff+ Cupper	242.1	36.27
4	Wool + Saff+ Iron	246.5	33.45
5	Wool + Madder+ Alum	245.3	36.45
6	Wool + Mad	235.9	34.57
7	Wool + Madder+ Cupper	244.6	34.25
8	Wool + Madder+ Iron	248.6	35.25
Wool Fabrics dyed with natural dyes after thermal aging for 100°C at 72 h			
9	Wool + Saff+ Alum	237.9	25.47
10	Wool + Saff + Without	227.4	27.14
11	Wool + Saff+ Cupper	230.2	32.38
12	Wool + Saff+ Iron	242.5	30.75
13	Wool + Madder+ Alum	239.9	30.45
14	Wool + Mad+ Without	231.9	26.43
15	Wool + Madder+ Cupper	231.3	29.54
16	Wool + Madder+ Iron	244.3	29.89
Wool Fabrics dyed with natural dyes after thermal aging for 100 °C at 144 h			
17	Wool + Saff+ Alum	230.2	20.65
18	Wool + Saff	219.8	18.18
19	Wool + Saff+ Cupper	220.7	18.19
20	Wool + Saff+ Iron	231.4	21.78
21	Wool + Madder+ Alum	230.1	24.78
22	Wool + Mad	229.3	23.68
23	Wool + Madder+ Cupper	238.9	19.37
24	Wool + Madder+ Iron	238.9	21.17

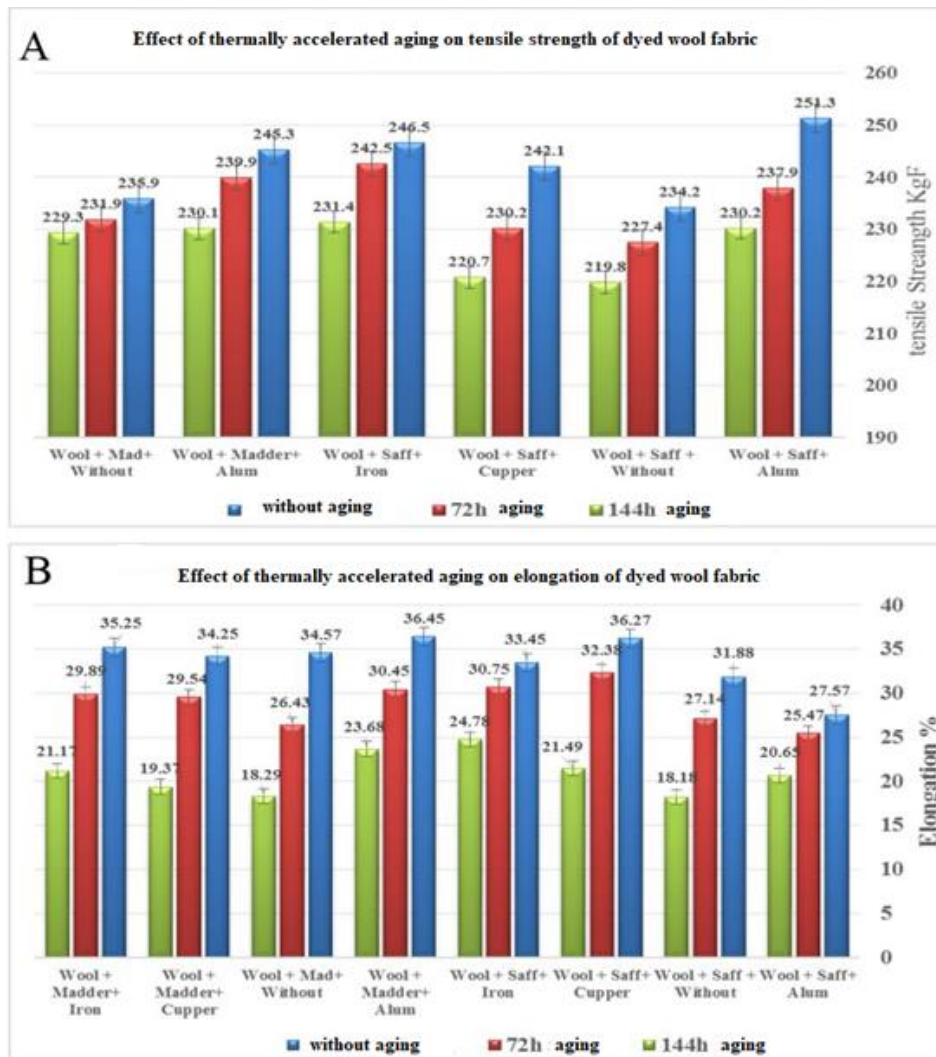


Fig. 5 . (A) Tensile Strength of dyed wool fabric before and after thermal aging; (B) Elongation of dyed wool fabric before and after thermal aging.

3.4. Effect of thermally accelerated aging on color change of dyed wool fabric

Table 2 and Figure 6 represent the effect of thermal accelerated aging on color change of dyed wool fabric. It is a clear that there are severe changes in the optical properties of the dyed wool fabrics upon aging. Figure 6 (A&B) show that the wool fabric dyed with madder or safflower dye mordanted with different type of mordents become more darker after aging. For example, the brightness coordinate (L) of wool dyed with safflower mordanted with alum was 65.41, however, after aging for 72 hours it becomes 64.34. While, on further aging for 144 hours, it recorded 63.24. In other words, ΔL of wool dyed with safflower mordanted with alum after aging for 72 h and 144 h is -1.07 and -2.17 CIELab unit, respectively. Besides, the notable decrease in brightness values for all dyed wool samples are shown in Table 2.

In addition, the red-green coordinate (a) of dyed wool fabric was changed after aging. For example, Δa of wool dyed with safflower mordanted with alum after aging for 72 h and 144 h was -2.24 and -3.13 CIELab unit respectively. While Δa of wool dyed with madder mordanted with alum after aging for 72 h and 144h was 1.26 and 3.76 CIELab unit respectively. Furthermore, following with the same trend, the effect of thermal accelerated aging on the yellow-blue coordinate (b) of dyed wool fabric are shown Table 2 and Figure 6 (E&F). It is clear for the reader that the aged

wool fabrics samples become blueness. For example, the yellow–blue coordinate (b) of wool dyed with safflower mordanted with alum was 34.8 after aging for 72 h became 30.02 and then after further aging for 144 h became 25.64 CIELab unit. In other words, Δa of wool dyed with safflower mordanted with alum after thermally aging for 72 h and 144 h is -4.78 and -9.16 CIELab unit respectively. While the yellow-blue coordinate (b) of wool dyed with madder mordanted with alum was 32.76 after thermally aging for 72h become 17.89 and then after thermally aging for 144 h become 11.51 CIELab unit. In other words, Δa of wool dyed with safflower modanted with alum after thermally aging for 72 h and 144h is -14.87 and -21.25 CIELab unit, respectively. One can see the less in brightness for all dyed wool samples in Table 2. The results show after of thermally accelerated aging, the dyed wool fabric became darker ($-\Delta L$), more red ($+\Delta a$), and more blue ($-\Delta b$) in character.

The effect of the thermal accelerated aging on the red-green coordinate (a) of the dyed wool fabric is shown in Figure 6 (C & D). The effect of the thermally accelerated aging on the yellow–blue coordinate (b) of the dyed wool fabric is shown in Figure 6 (E & F).

Table 2. The color parameters of dyed wool fabric before and after thermal aging.

Wool Fabrics dyed with natural dyes before ageing				
No	Samples	Color parameters		
		L	a	b
1	Wool + Saff+ Alum	65.41	16.73	34.8
2	Wool + Saff	68.45	15.22	24.56
3	Wool + Saff+ Cupper	58.40	12.04	20.56
4	Wool + Saff+ Iron	54.78	16.12	28.91
5	Wool + Madder+ Alum	65.61	16.94	32.76
6	Wool + Mad	66.72	16.02	31.46
7	Wool + Madder+ Cupper	66.06	18.42	28.54
8	Wool + Madder+ Iron	45.81	25.32	19.82
Wool Fabrics dyed with natural dyes after thermal aging for 100°C at 72 h				
9	Wool + Saff+ Alum	64.34	14.49	30.02
10	Wool + Saff	66.91	12.09	22.01
11	Wool + Saff+ Cupper	58.75	9.36	19.23
12	Wool + Saff+ Iron	52.45	13.24	26.18
13	Wool + Madder+ Alum	58.37	18.2	17.89
14	Wool + Mad	57.06	16.95	16.53
15	Wool + Madder+ Cupper	53.88	19.86	14.75
16	Wool + Madder+ Iron	52.14	27.10	12.47
Wool Fabrics dyed with natural dyes after thermal aging for 100°C at 144 h				
17	Wool + Saff+ Alum	63.24	13.60	25.64
18	Wool + Saff	64.60	11.91	21.15
19	Wool + Saff+ Cupper	54.71	8.77	17.73
20	Wool + Saff+ Iron	49.54	9.95	24.15
21	Wool + Madder+ Alum	57.71	19.88	11.51
22	Wool + Mad	57.19	18.55	12.21
23	Wool + Madder+ Cupper	53.81	22.11	8.61
24	Wool + Madder+ Iron	51.66	28.44	8.01

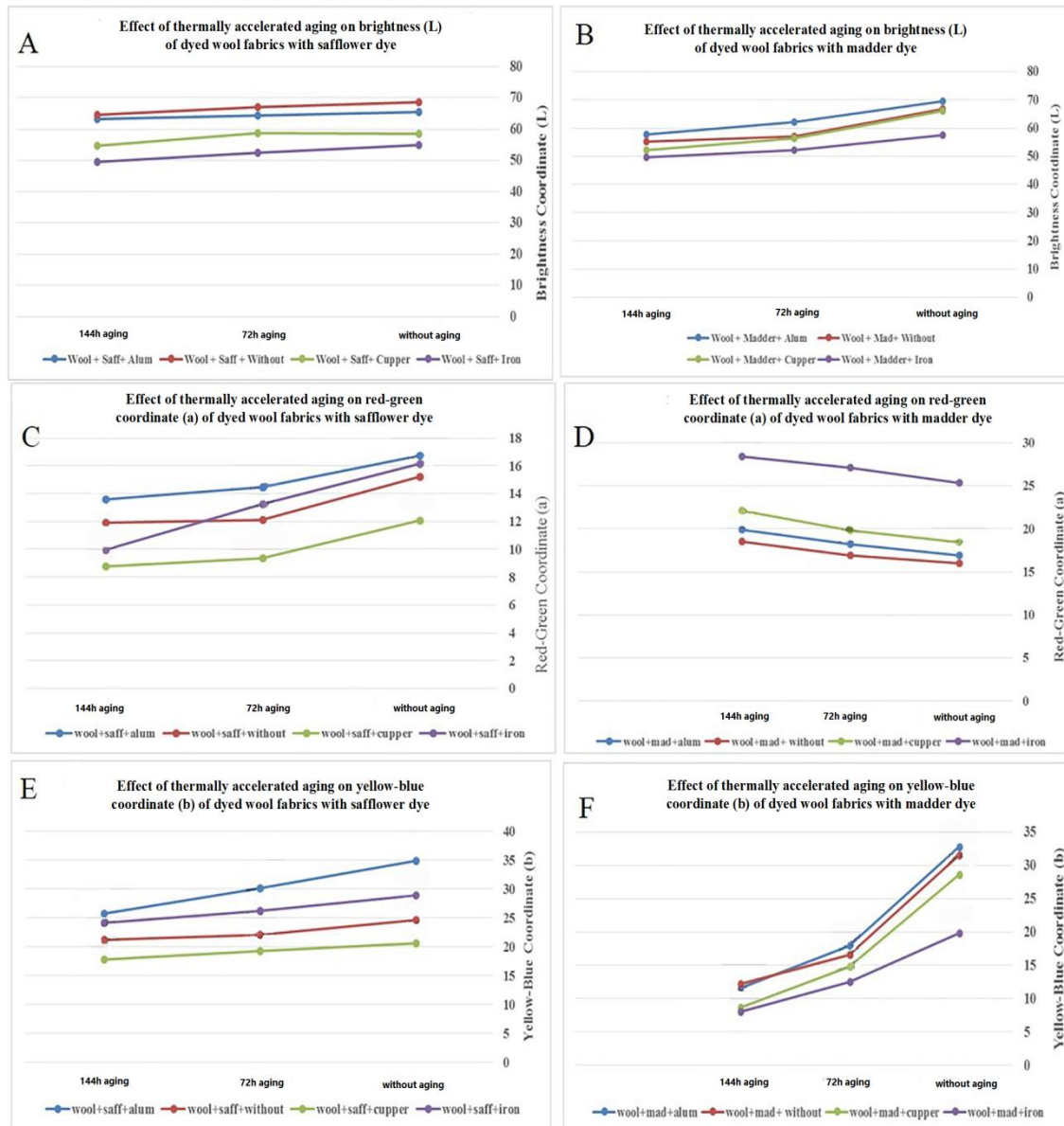


Fig. 6. The effect of the thermal accelerated aging on the brightness coordinate (L,a and b) for the dyed wool fabric.

3.5. Effect of thermal aging on the air permeability of dyed wool fabric

Table 3 shows the effect of thermal aging on the air permeability of the dyed wool fabric. Air permeability affects the wind resistance, water vapor permeability and filtering properties of dyed wool fabrics. This helps to understand the behavior of aged wool fabric for the restoration process using cleaning and reinforcing materials. It is clear that the air permeability of dyed wool fabric was decreased after aging process via reducing the interstellar spacing between the warp and weft yarn.

Table 3. Air permeability property of the dyed wool fabrics before and after aging.

Wool Fabrics dyed with natural dyes after thermal aging for 100°C at 72 h						
No	Samples	Air permeability				
1	Wool + Saff+ Alum	27.0	25.0	25.6	24.4	23.6
2	Wool + Saff	25.8	25.9	25.2	25.0	25.6
3	Wool + Saff+ Cupper	24.3	23.6	22.9	24.2	23.8
4	Wool + Saff+ Iron	26.1	26.4	25.7	26.0	25.1
5	Wool + Madder+ Alum	25.0	24.7	23.7	25.9	24.7
6	Wool + Mad	24.8	24.1	24.5	25.3	24.4
7	Wool + Madder+ Cupper	26.7	26.2	27.1	28.4	28.3
8	Wool + Madder+ Iron	27.0	25.2	25.8	26.7	25.0
Wool Fabrics dyed with natural dyes after thermal aging for 100°C at 144 h						
9	Wool + Saff+ Alum	26.5	24.6	25.1	23.9	23.9
10	Wool + Saff	25.5	25.2	25.3	24.1	25.1
11	Wool + Saff+ Cupper	23.8	23.3	22.3	22.3	23.5
12	Wool + Saff+ Iron	25.5	26.0	25.7	25.8	24.7
13	Wool + Madder+ Alum	24.4	23.2	23.8	25.1	24.5
14	Wool + Mad	24.3	24.4	24.0	24.6	24.2
15	Wool + Madder+ Cupper	25.4	25.7	24.7	25.7	26.0
16	Wool + Madder+ Iron	22.6	24.0	23.9	23.7	24.6

4. Conclusion

Studying the effect of uncontrolled museum conditions on the characteristics of historical textiles is an important step in the creation of a standard museum display conditions. This process will preserve the historical textiles from damage and increases the lifetime of these textiles. It is noticed that the effect of heat on the surface morphology of the wool fiber in terms of breakage and bombardment in the wool fiber. This type of damage causes a reduction in the strength of the wool fiber. This hypothesis is consistent with the results of the T.S and E% of aged wool fabric. In addition, the color change study showed that the dyed linen fabric became darker ($-\Delta L$), more red ($+\Delta a$), and more blue ($-\Delta b$) in character upon aging. The thermally aging causes a change in chemical structure of the aged wool fibers. Especially, the bands refer to the peptide bonds ($-\text{CONH}-$) that indicate as amide I, amide II and amide III. It is highly recommended to control the environmental conditions in the museums display especially the temperature.

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