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PREPARATION OF EXPERIMENTAL DETERIORATED DYED TEXTILE SAMPLES SIMULATED TO ANCIENT ONES

Omar ABDEL-KAREEM*

Cairo University, Faculty of Archaeology, Conservation Department, 12613 Orman, Giza, Egypt

Abstract

*This study is carried out to investigate the possibility of preparing artificial deteriorated dyed textile samples simulated to deteriorated dyed ancient textiles, to be used as experimental samples in historical textile conservation researches. In this study new wool textiles were dyed with 10 natural dyes common used for dyeing of textiles in Egypt in different historical periods. Different mordant were used in this study to produce different colours from each dye. The dyed wool samples were artificially aged by light for various periods. The changes in the colour of dyed wool samples after aged by light were observed visually. Also the changes in the CIE L*a*b* parameters L*, a*, and b *values (ΔL*, Δa*, and Δb*) and the total change in the colour (ΔE*) were calculated. The results show that yellow dyes are the most sensitive tested dyes to light aging. Madder is most tested dyes fastness to light. This study informs that the colours that we see on historical textiles in the museum may be different than their original colours in the past in the moment of their production. The obtained results show that it is possible to prepare artificial experimental wool textile samples simulated to faded ancient ones. These artificial experimental wool textile samples can be used for conservation researches, and the practical training of textile conservators. However the exposure time required for preparing these samples depend on type of the dyes. Some of tested dyes such as indigo, cochineal, Lac need about 80 hours and other dyes such as turmeric needs about 5 hours only.*

Keywords: *Wool textile fabric; natural dyes; artificial light aging; CIEL*a*b* measurements; colour change.*

Introduction

The use of dyes in colouring textile fabrics reaches back into history. Dyed fabric from thousand years B.C has been found in Egyptian tombs [1]. Ancient dyed textiles are natural organic substances and hence they are liable to deterioration [2-4]. Textiles deteriorate naturally by oxidation, heat, mechanical stress, radiation, moisture, and microbiological attack [5, 6]. There is no doubt that fading of dyes caused by light radiation is one the most serious damages of dyed textile museums [7-9]. As dyed textiles remain one of the most sensitive materials to light among museum objects. Light is always found in display and storage conditions of museums, as it is not possible to present any object without light [10]. The knowledge of light fastness characteristics of dyed textiles is very important in order to understand the nature of fading. And it helps in looking at the future preservation of these dyed textiles. Light fastness has been investigated widely and intensively in developing new dyestuffs. Most of fading

* Corresponding author: Omaa67@yahoo.com

experiments were made under accelerated conditions with artificial light sources [8]. Dyed textiles are considered one of the most important archaeological materials, as they provide historical evidence on civilizations and development of their periods. They reflect progress societies of their industrial and technological developments. For this reason, every effort should be made to preserve these textiles from light radiation and to pass them to the next generation. In order to select new materials for the preservation and restoration of these textile materials, investigations done on several samples are required, to determine the deteriorating and degradation effects. These experimental samples will be used in evaluation and estimation of any new material; technique and methods can be used in conservation and restoration of deteriorated textiles before their application on ancient ones. Natural dyed historical textiles are characterized by a high degree of variability of properties and therefore, are rarely suitable for experimental research because variations in results can be attributed to the nature of specimens rather than the particular conservation treatment under investigation [11]. In addition, the main aim of conservation researches is to preserve textile objects, so that the usage of ancient ones as experimental samples is prevented [6]. For that this study is carried out to investigate the possibility of preparation of artificially deteriorated dyed textile samples with definite properties. In order to be used as experimental samples in the evaluation and the estimation of any new materials, techniques and methods can be used in conservation and restoration of deteriorated dyed textiles before their application on ancient ones. Also these artificial experimental samples can be used for conservation practical training of textile conservators.

Material and Methods

Fabrics. Scoured, plain wool textile fabric was used (from WOOLTEX manufactory, Egypt).Wool textile fabrics were used in this study.

Dyes. 10 natural dyes common used in historical periods in Egypt were used in this study [1, 12-22]. Dyes used in this study are Cochineal, Cutch, Henna, Indigo, Lac, Madder, Safflower, Saffron, Sumac and Turmeric.

Mordant. Three mordant salts that are common used in mordanting textile fabrics were used in this study [12-17]. The mordant salts used in this study are potassium aluminium sulphate (Alum), ferrous sulphate (Iron) and copper sulphate (copper).

Preparation for dyeing. Almost all fabrics will require some preparation before dyeing can be undertaken. Wool needs to be scoured to remove natural oils or substances added to ease the spinning or weaving process. The liquor was used in ratio 100:1 (100 ml of water to 1g of fabric). The liquor was a 0.05% w/v solution of Synperonic N, (non-ionic surfactant). The wool was immersed in the liquor and raised the temperature to 40°C; apply gentle agitation for 30 minutes. The wool was rinsed in heated softened water lowering the temperature gradually. Never subject it to sudden changes in temperature, as this can cause felting. The final rinse was done in de-ionized water.

Mordanting. Wool textile fabrics were mordanted by metal salts as the following. The mordant was weighted according to Abdel-Kareem [9]. The mordant salt was added to bath of water. The soft water was heated to approximately 30ºC. The mordant salt was dissolved in water bath. The clean wetted wool textile fabric was entered to the bath. The mordant bath was heated with the fabric gradually to boil point. When boiling point has been reached, the heat was lowered and simmered for about 1 hour at 95ºC. The bath was allowed to cool for 1 hr, and then the fabric was rinsed and hung up to dry [14].

Dyeing process. The amount of liquor of dye bath needed is calculated according to the dry weight of the textile fabric, expressed as a ratio between the two, e.g. 50:1, meaning that 50ml of water are required for every one gram of textile fabric. The necessary quantities of dyes were weighing according to Abdel-Kareem [9]. In regards to all dyes except indigo were dyed as the following. The exact weight of each one of used dyes was added to the dye bath. The dye

bath was left overnight. The dye bath was gently heated, and the clean wetted textile fabric was entered in the dye bath. The dye bath was heated with the fabric gradually to boil point except madder was to 60ºC. When required point has been reached, the heat was simmered for about 1 hour. The bath was allowed to cool for 1 hr. After then the dyed fabric, it was washed in soapy water. After then the fabric was rinsed in distilled water and hung up to dry for at least 24 hrs.

Dyeing of samples with Indigo (Blue Colour). The natural indigo powder was ground with 10 ml water until a creamy, even consistency was obtained. 50 ml of warm water was added into a bottle and mixed with 1gr of sodium hydroxide which was slowly applied with continues stirring until dissolved. 50 ml of warm water was also added into another bottle and gently slowly mixed with 1gr of sodium dithionite. All were added, but small amounts of the sodium hydroxide solution and the sodium dithionite solution were reserved. After 1 hour, it was tested by dipping in a glass rod according to Dalby [15]. After preparing the vat dye according to Abdel-Kareem [9], the clean wetted fabric was lowered gently into both sides of the vat. It was kept there and moved gently for 1 to 5 minutes. The fabric was taken out of the vat with a faint yellow colour, which later turned blue in the air within a few minutes. After the dyed fabric dried, it was washed in soapy water, rinsed very thoroughly, and dried.

Light Ageing. For ageing by exposure to light, tests were carried according to international standard tests for colour fastness (ISO 105-B02:1994) [23]. Dyed wool samples were mounted in standard specimen holders and were exposed to light irradiation for 5, 10, 20, 40 and 80 hours without turning of samples holders. Irradiation of the samples was carried out using the Atlas Light Fastness Tester. Type of Atlas Fade-Ometer used in this study is XENOTEST®150S^{+}). A light filter was used to simulate light in museums. Exposure conditions were 50ºC and 55% of RH.

Evaluation of Colour Changes. Changes in colour of tested samples were evaluated visually and using Optimacth 3100 colour Spectrophotometer using the CIE L*a*b* colour system. CIE L*a*b* colour coordinates for L* (lightness), a* (red/green axis), and b* (yellow/blue axis) values were recorded. Five colour readings were made and averaged for each sample. Colour changes are the differences between unaged and aged dyed wool textile sample after aging for each control one, are expressed as ΔL*, Δa*, Δb*. Calculation of total colour change (ΔE*) is achieved by the use of the following equations [24-28]:

$$\Delta E^* = [(\Delta L^*)^2 + \Delta a^*)^2 + (\Delta b^*)^2]^{0.5}. \quad (1)$$

The positive and negative nature of ΔL*, Δa* and Δb* values also gives information about other changes which have occurred to the samples as a result of treatment. A positive ΔL* value indicates that the sample is lighter than the control sample, and a negative value indicates that it is darker. If Δa* is positive then the sample has a greater degree of redness, and if negative then it has a greater degree of greenness or a decrease in redness. Lastly, a positive Δb* value signifies an increase in yellowness, and a negative value signifies an increase in blueness or a decrease in yellowness [4].

Results and discussion

The colour values of wool textile samples that experimentally dyed with selected tested dyes were measured and shown in table 1. These values are used as control samples for the same dyed samples after aged by light for various periods. The results in table 1 show that there are various colours and shades due to using various mordant. The visual observation of wool samples dyed with indigo dyes after aged by light show that indigo dyes are not sensitive to light in museum. The results show that the changes in the colour of wool samples dyed with indigo after aged with light for 80 hours are too little compared with other tested samples (see

Fig. 1). The data of the changes in the brightness and the total colour changes of wool samples dyed with indigo after aged by light for various periods was presented in Figure 2.

The results show that the change in the brightness is too little. As it is not noticeable change in the brightness of tested samples before 20 hours as the value of L was less than 1 unit after 10 hours. After 20 hours of light exposure the samples became little lighter than unaged samples as the changes in L* value were about 4, 6, and 7 units after the exposure to light for 20, 40 and 80 hours respectively. The results show that the change in the total colour of samples dyed with indigo after light ageing is little too. As the total colour changes of samples are about 4, 4, 7 and 9 units after the exposure to light for 10, 20, 40, and 80 hours respectively. This result is in agreement with the results obtained by Crews [29] and Abdel-Kareem [9], who confirm that indigo dye is one of the most light fastness natural dye among ancient dyes in textiles. The results confirm that 80 hours of time exposure is required to prepare aged wool samples dyed with indigo.

Table 1. Colour values of wool textile samples after dyed with tested dyes

Mordant	**Cochineal**			**Cutch**			**Henna**		
	L	**a**	**b**	**L**	**a**	**b**	**L**	**a**	**b**
Alum	46.71	37.46	4.17	54.29	21.79	30.61	36.91	15.25	25.40
Iron	32.26	5.13	1.46	25.23	6.60	4.86	20.48	2.70	6.34
Copper	35.65	14.90	5.03	28.95	15.04	14.45	25.98	9.97	15.60
without	48.16	24.64	-0.59	50.31	22.78	24.08	41.92	16.01	27.04
	Lac			**Madder**			**Safflower**		
Alum	41.17	53.12	27.95	39.46	33.43	28.69	69.40	4.15	55.83
Iron	33.65	41.11	23.42	19.27	7.58	1.97	33.37	2.05	17.97
Copper	33.07	27.74	20.40	31.90	19.68	17.10	49.14	4.39	38.37
without	37.54	51.37	29.19	-	-	-	73.59	-0.16	47.78
	Saffron			**Sumac**			**Turmeric**		
Alum	80.89	-2.73	41.24	64.52	3.48	21.84	75.76	0.38	64.68
Iron	56.96	6.55	28.21	46.16	4.42	25.95	51.24	8.57	32.29
Copper	58.88	-2.46	26.12	26.42	2.89	2.67	53.17	1.50	36.64
without	78.89	-1.33	31.87	60.44	5.46	20.39	73.30	-0.29	64.62
	Indigo								
without	52.52	-3.63	-15.75						

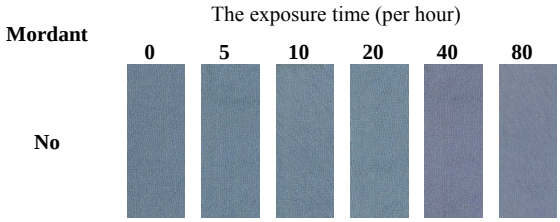

Fig. 1. Visual observation of the effect of the exposure time of light on the colour of wool samples dyed with indigo.

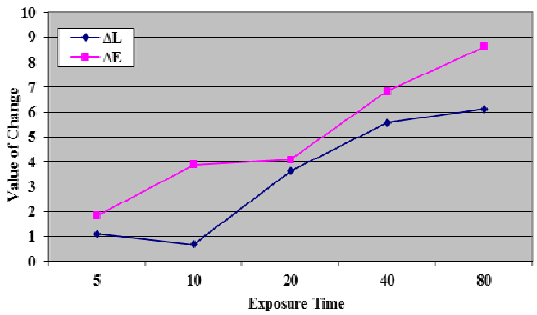

Fig. 2. The colour change of samples dyed with indigo after light aging for various times

The results of visual observation of wool samples dyed with madder after light ageing for various periods show that there are noticeable changes in the colour of dyed samples after light ageing (figure 3). The changes in the brightness of wool samples dyed with madder after light ageing for various periods are shown in figure 4. The results show that the samples became lighter after light ageing, specially the samples mordanted with iron and copper. As the change in values of L* after 10 hours exposure are about 4, 16 and 20 for samples mordanted with alum, copper and iron respectively. The results show that the most changes in the brightness of dyed samples were in the first periods, while after 20 hours, the changes in the brightness were too little.

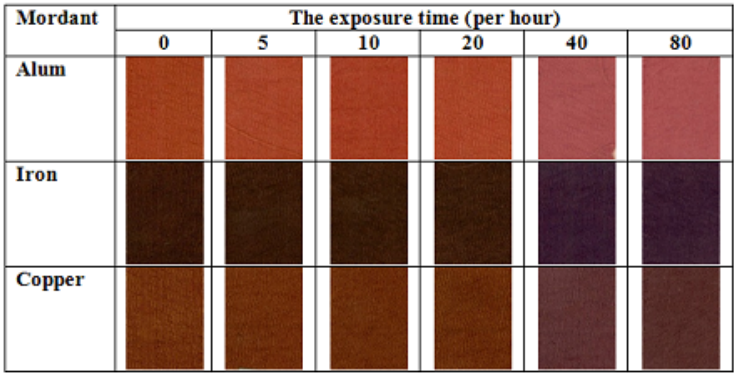

Fig. 3. Visual observation of the effect of the exposure time of light on the colour of wool samples dyed with madder

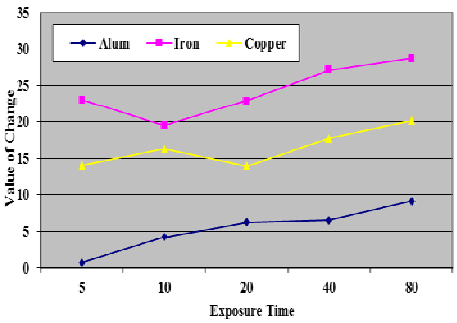

Fig. 4. The change in the brightness (ΔL*) of samples dyed with madder after light aging for various times

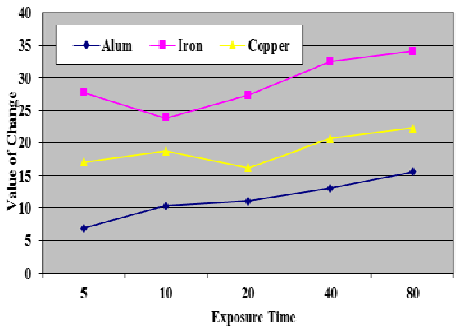

Fig. 5. The total colour change (ΔE*) of samples dyed with madder after light aging for various times

The results in figure 5 show that after light ageing there are noticeable changes in the total colour of wool samples dyed with madder especially samples mordant with iron. The change in total colour after 10 hours exposure are about 10, 19 and 23 for samples mordant with alum, copper and iron respectively. The results show that the most changes in the total colour of dyed samples were in the first periods of the exposure time. As the results show that the changes in the total colour of all samples after 40 and 80 hours were compared with the changes happened after 20 hours only. The results show that the least changes in the total colour of samples dyed with madder is in samples dyed with alum. This result is different than the results obtained by Crews [29], who confirmed that the greatest amount of colour change were attributed to alum mordant.

The results of visual observation of wool samples dyed with cochineal after light ageing for various periods were presented in figure 6. The results show that there are moderate changes in the colour after light ageing. The most changes in the colour were in unmordant samples and that mordant with alum. While the least change in the colour were in samples mordant with iron and copper. The results of the changes in the brightness of samples dyed with cochineal were presented in figure 7. The results show that there are moderate changes in the brightness after light ageing as all samples became moderate lighter than unaged samples. The most changes in the brightness were in unmordant samples and that mordant with alum. While the least change in the brightness were in samples mordant with iron and copper. The results of the changes in the total colour of samples dyed with cochineal were presented in figure 8. The results show that there are moderate changes in the total colour after light ageing. The most changes in the brightness were in unmordant samples and that mordant with alum. While the least change in the brightness were in samples mordant with iron and copper. The results confirm that 80 hours of time exposure is required to prepare aged wool samples dyed with cochineal.

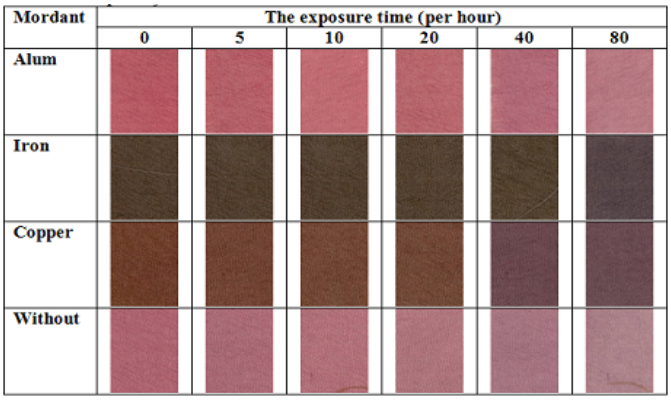

Fig. 6. Visual observation of the effect of the exposure time of light on the colour of wool samples dyed with cochineal

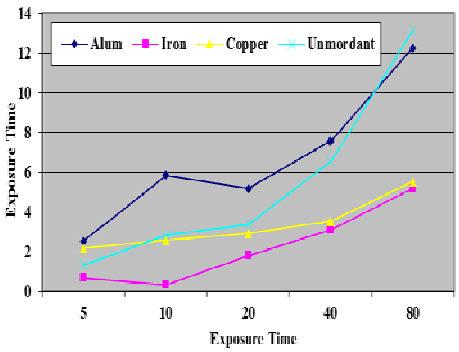

Fig. 7. The changes in the brightness (ΔL*) of samples dyed with cochineal after light aging for various times

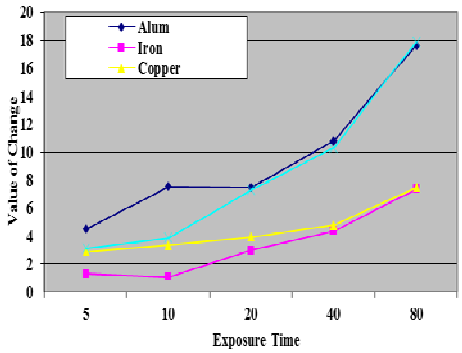

Fig. 8. The total colour change (ΔE*) of samples dyed with cochineal after light aging for various times

The visual observation of wool samples dyed with cutch show that after only 5 hours of time exposure there are noticeable change in colour of all tested samples except that mordant with alum (see figure 9). The results of the changes in the brightness of samples dyed with cutch in figure 10 show that only after 5 hours of exposure time all samples became more lightness except those mordant with alum became little dark. After 10 hours of exposure time the changes in the brightness (L value) were about 4, 11, 14 and 20 for samples dyed with alum, copper, unmordant, and iron respectively.

The results of the changes in the total colour of samples dyed with cutch in figure 11 showed that after 5 hours of exposure time there were high noticeable changes in the colour of all samples except those mordant with alum was little. After 10 hours of exposure time the changes in the total colour were about 8, 17, 18 and 26 for samples dyed with alum, unmordant, copper and iron respectively. These results confirm that 10 hours of exposure time is enough to prepare aged wool samples dyed with cutch.

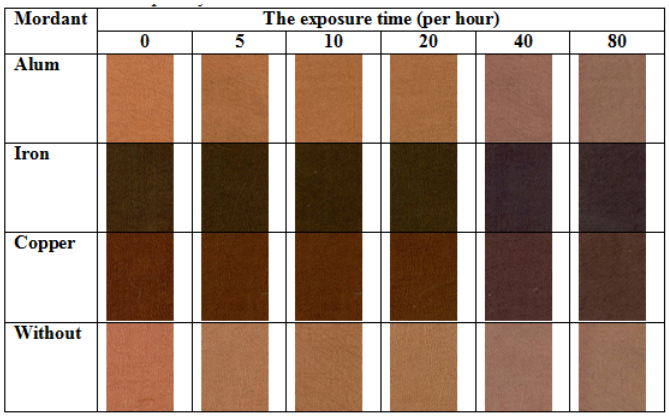

Fig. 9. Visual observation of the effect of the exposure time of light on the colour of wool samples dyed with cutch

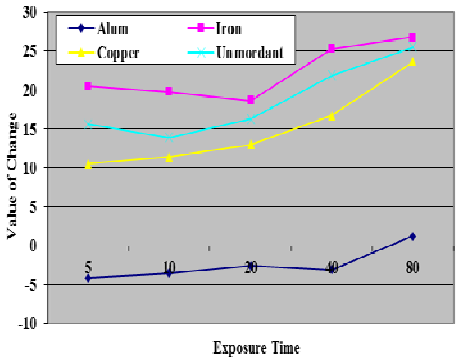

Fig. 10. The changes in the brightness (ΔL*) of samples dyed with cutch after light aging for various

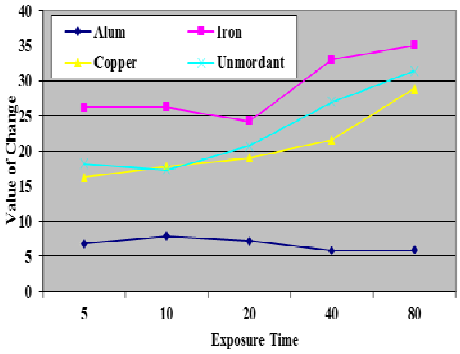

Fig. 11. The total colour change (ΔE*) of samples dyed with cutch after light aging for various times

The visual observation of wool samples dyed with henna show that only after 40 hours of time exposure there are noticeable change in colour of all tested samples especially unmordant one and that mordant with alum (see figure 12). The results of the changes in the brightness of samples dyed with henna in figure 13 show that only after 40 hours of exposure time all samples became more lightness especially unmordant one and that mordant with alum. After 40 hours of exposure time the changes in the brightness (L value) were about 10, 10, 5 and 2 for samples dyed with alum, unmordant, iron and copper respectively.

The results of the changes in the total colour of samples dyed with henna in figure 14 showed that only after 40 hours of exposure time it is noticed changes in the colour of all samples except those mordant with iron and copper. After 40 hours of exposure time the changes in the total colour were about 10, 10, 5 and 2 for samples dyed with alum, unmordant, iron and copper respectively. These results confirm that 40 hours of exposure time is enough to prepare aged wool samples dyed with henna.

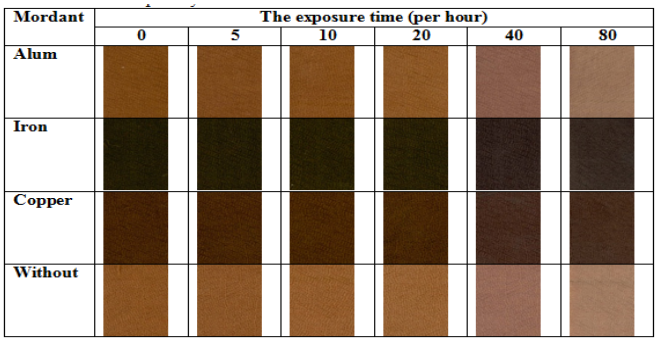

Fig. 12. Visual observation of the effect of the exposure time of light on the colour of wool samples dyed with henna

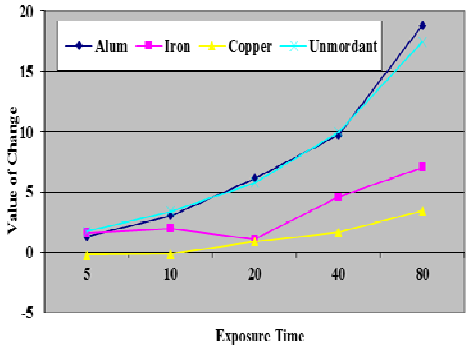

Fig. 13. The changes in the brightness (ΔL*) of samples dyed with henna after light aging for various times

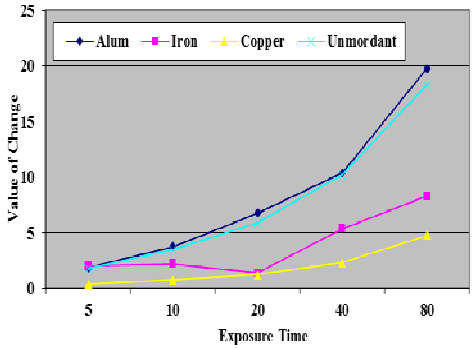

Fig. 14. The total colour change (ΔE*) of samples dyed with henna after light aging for various times

The visual observation of wool samples dyed with Lac show that only after 80 hours of time exposure it can be noticed high change in colour of all tested samples (see figure 15). The results of the changes in the brightness of samples dyed with Lac in figure 16 show that only after 80 hours of exposure time it can be noticed change in the brightness of all samples as they became more lightness as the changes in L value were about 8-10 units. The results of the changes in the total colour of samples dyed with Lac in figure 17 show that only after 80 hours of exposure time it can be noticed change in the colour of all samples as the changes in the total colour of all samples were about 11-17 units. These results confirm that 80 hours of exposure time is enough to prepare aged wool samples dyed with Lac.

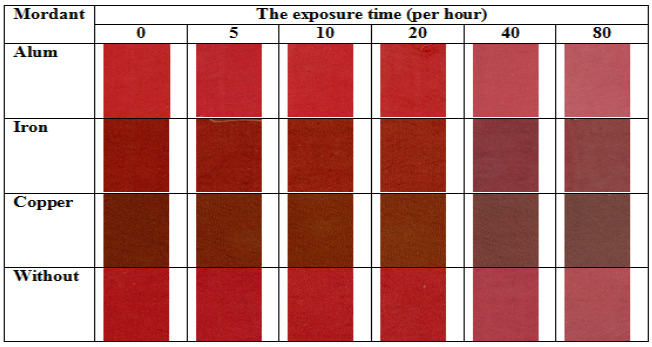

Fig. 15. Visual observation of the effect of the exposure time of light on the colour of wool samples dyed with lac

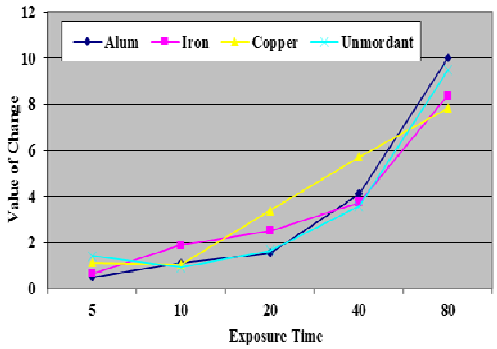

Fig. 16. The changes in the brightness (ΔL*) of samples dyed with lac after light aging for various times

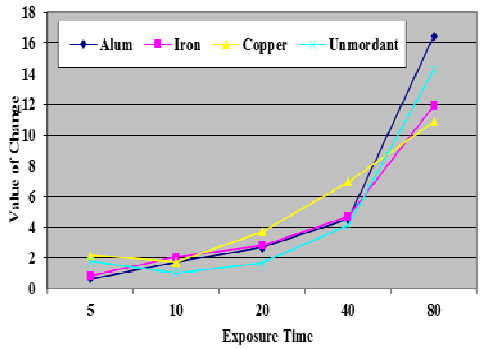

Fig. 17. The total colour change (ΔE*) of samples dyed with lac after light aging for various times

The visual observation of wool samples dyed with safflower show that after 20 hours of time exposure there are noticeable changes in colour of all tested samples (see figure 18). The results of the changes in the brightness of samples dyed with safflower in figure 19 show that after 20 hours of exposure time all samples became more lightness. After 20 hours of exposure time the changes in the brightness (L value) were about 2-6 units. The results of the changes in the total colour of samples dyed with safflower in figure 20 showed that after 20 hours of exposure time there were noticeable changes in the colour of all samples especially unmordant one and those mordant with alum. After 20 hours of exposure time the changes in the total colour were about 27,17,4 and 3 for samples dyed with alum, unmordant, copper and iron respectively. These results confirm that samples dyed mordant with Alum and unmordant one are more sensitive to light. While samples dyed with iron and copper more stable to light. It may be as results to the effect of the produced colour shade. These results are in agreement with results obtained by Abdel-Kareem [9] that confirm that dyes with dark colour shades are less sensitive to the light than dyes with bright colour shades. The results confirm that 20 hours of exposure time is enough to prepare aged wool samples dyed with safflower.

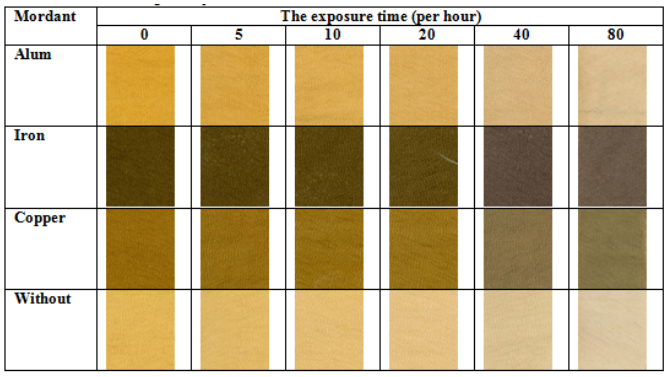

Fig. 18. Visual observation of the effect of the exposure time of light on the colour of wool samples dyed with safflower

The visual observation of wool samples dyed with saffron show that after only 5 hours of time exposure there are noticeable change in colour of all tested samples specially samples mordant with alum and unmordant one (figure 21). The results of the changes in the brightness of samples dyed with saffron in figure 22 show that after only 5 hours of exposure time all

samples became lighter than control ones. After 10 hours of exposure time the changes in the brightness (L value) were about 2- 5 units.

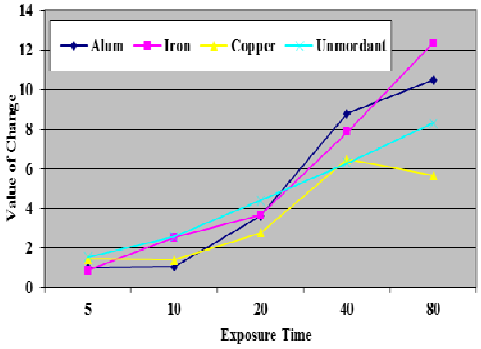

Fig. 19. The changes in the brightness (ΔL*) of samples dyed with safflower after light aging for various times

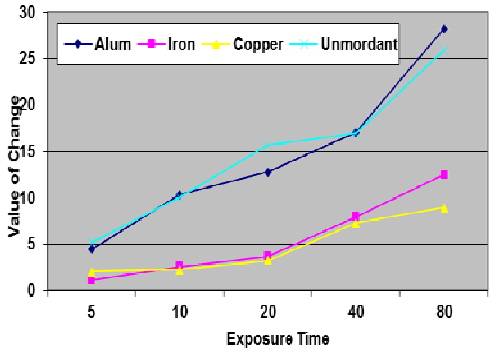

Fig. 20. The total colour change (ΔE*) of samples dyed with safflower after light aging for various times

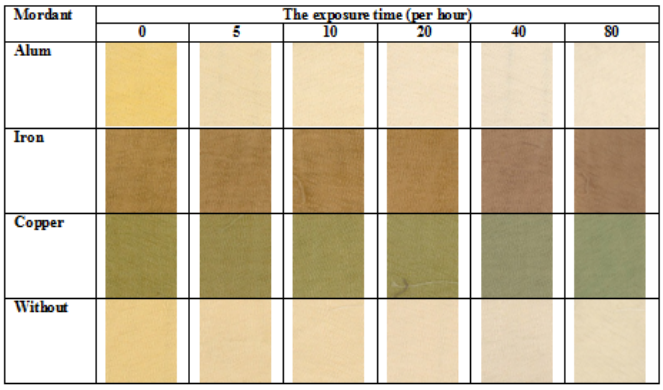

Fig. 21. Visual observation of the effect of the exposure time (per hour) of light on the colour of wool samples dyed with saffron

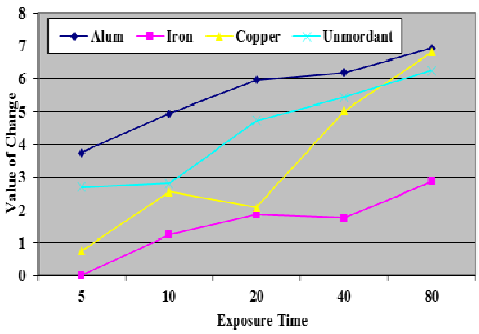

Fig. 22. The changes in the brightness (ΔL*) of samples dyed with saffron after light aging for various times

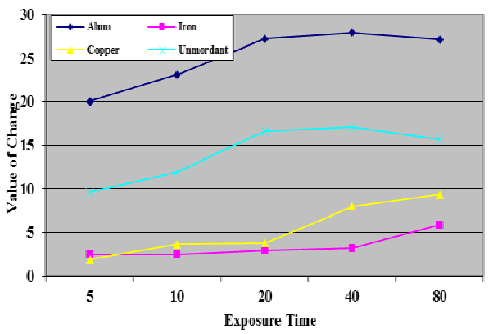

Fig. 23. The total colour change (ΔE*) of samples dyed with saffron after light aging for various times

The results of the changes in the total colour of samples dyed with saffron in figure 23 showed that after only 5 hours of exposure time there were noticeable changes in the colour of all samples especially unmordant one and those mordant with alum. After 10 hours of exposure time the changes in the total colour were about 23, 12, 4 and 3 for dyed samples dyed

unmordant, mordant with alum, copper and iron respectively. These results confirm that samples dyed mordant with alum and unmordant one are more sensitive to light. While samples dyed with iron and copper more stable to light. It may be as results to the effect of the produced colour shade. This results are in agreement with results obtained by Abdel-Kareem [9] that confirm that dyes with dark colour shades are less sensitive to the light than dyes with bright colour shades. The results confirm that 10 hours of exposure time is enough to prepare aged wool samples dyed with saffron.

The visual observation of wool samples dyed with sumac show that only after 80 hours of time exposure it can be noticed change in colour of all tested samples especially unmordant samples and those mordant with alum (see figure 24). The results of the changes in the brightness of samples dyed with sumac in figure 25 show that only after 80 hours of exposure time it can be noticed change in the brightness of all samples especially unmordant samples and those mordant with alum as the changes in L value were about 2-9 units.

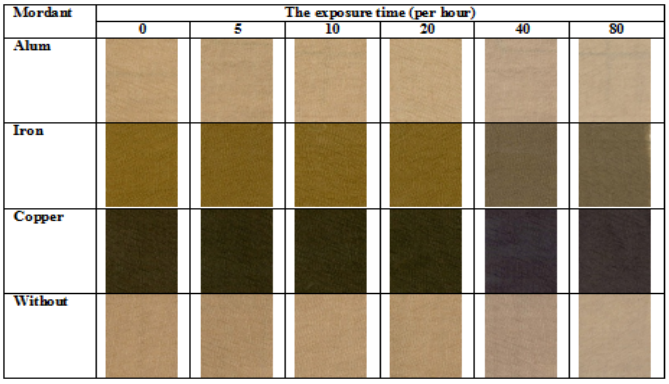

Fig. 24. Visual observation of the effect of the exposure time of light on the colour of wool samples dyed with sumac

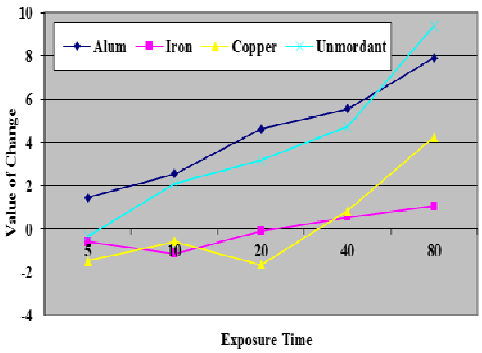

Fig. 25. The changes in the brightness (ΔL*) of samples dyed with sumac after light aging for various times

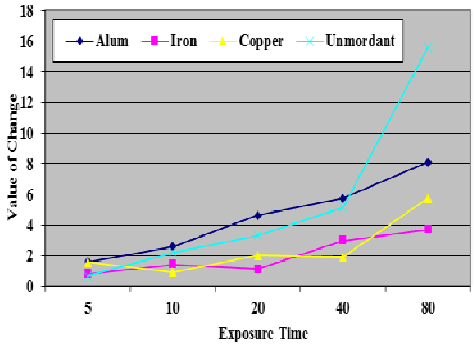

Fig. 26. The total colour change (ΔE*) of samples dyed with sumac after light aging for various times

The results of the changes in the total colour of samples dyed with sumac in figure 26 show that only after 80 hours of exposure time it can be noticed change in the colour of all samples especially unmordant samples and those mordant with alum as the changes in the total colour of all samples were about 4-16 units. These results confirm that 80 hours of exposure time is enough to prepare aged wool samples dyed with sumac.

The visual observation of wool samples dyed with turmeric show that after only 5 hours of time exposure there are noticeable change in colour of all tested samples specially samples mordant with copper (see figure 27). The results of the changes in the brightness of samples

dyed with turmeric in figure 28 show that after only 5 hours of exposure time all samples became lighter than control ones especially. After 5 hours of exposure time the changes in the brightness (L value) were about 11- 46 units. The results of the changes in the total colour of samples dyed with turmeric in figure 29 showed that after only 5 hours of exposure time there were noticeable changes in the colour of all samples especially unmordant one and those mordant with alum. After 5 hours of exposure time the changes in the total colour were about 46, 45, 26 and 11 for dyed samples dyed unmordant, mordant with alum, copper and iron respectively. These results confirm that samples dyed mordant with alum and unmordant one are more sensitive to light. While samples dyed with iron and copper more stable to light. It may be as results to the effect of the produced colour shade. These results are in agreement with results obtained by Abdel-Kareem [9] that confirm that dyes with dark colour shades are less sensitive to the light than dyes with bright colour shades. The results confirm that 5 hours of exposure time is enough to prepare aged wool samples dyed with turmeric.

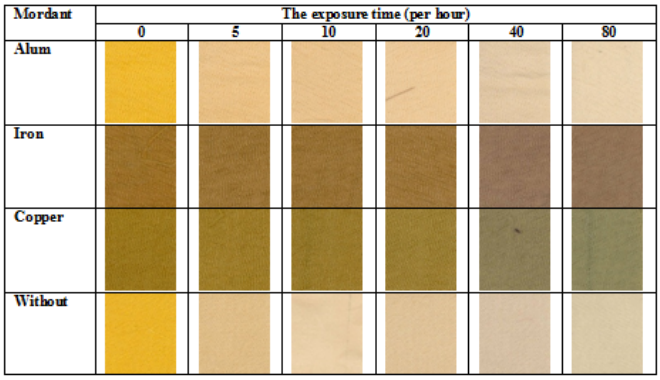

Fig. 27. Visual observation of the effect of the exposure time of light on the colour of samples dyed with turmeric

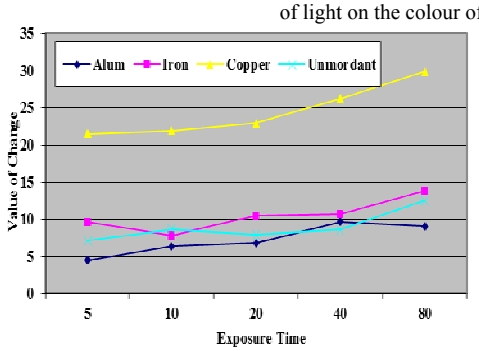

Fig. 28. The changes in the brightness (ΔL*) of samples dyed with turmeric after light aging for various times

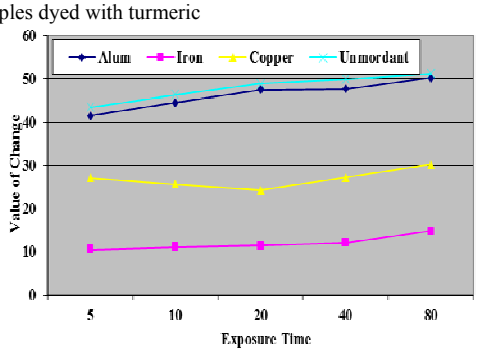

Fig. 29. The total colour change (ΔE*) of samples dyed with turmeric after light aging for various times

Conclusion

This study shows that yellow dyes are the most sensitive tested dyes to light aging while the indigo dye is the most tested dyes fastness to light. The order of fading susceptibility of dyes to light is as follows: turmeric > safflower > saffron > cutch > madder > henna > cochineal > lac > sumac > indigo. The sensitivity of tested dyes to light aging depends on the type of mordant, as the data show that the most changes in the colour of most of tested dyes were in the samples with alum mordant and unmordant samples, while the least changes in the colour were in the samples mordant with Iron and copper. This study confirms that the colour shades that we

see on historical textiles in the museum may be different than their original colour shades in the past in the moment of their production.

This study shows that it is possible to prepare artificial experimental textile samples with definite of colour shade. These artificial experimental samples can be used for conservation researches, and for the practical training of textile conservators. The exposure time required for preparing these samples depends on type of the dye. Some of tested dyes such as indigo needs about 80 hours and other dyes such as turmeric needs about 5 hours only.

The exposure time required for preparing artificial aged wool textile samples dyed with indigo, cochineal, Lac and sumac is about 80 hours. The exposure time required for preparing artificial aged wool textile samples dyed with henna and madder is about 40 hours. The exposure time required for preparing artificial aged wool textile samples dyed with cutch and safflower is about 20 hours. The exposure time required for preparing artificial aged wool textile samples dyed with saffron is about 10 hours while 5 hours only enough to turmeric .

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