

# Application of the Fast Aging Technique to Wool Fabric Dyed with Natural Dyes Having Different Stains to Obtain Homogeneous Mimic Samples for Archaeological Measurements.

## Application of the Fast Aging Technique to Wool Fabric Dyed with Natural Dyes Having Different Stains to Obtain Homogeneous Mimic Samples for Archaeological Measurements.

تطبيق تقنية التقادم السريع على نسيج الصوف المصبوغ بأصباغ طبيعية مبقعه ببقع مختلفة للحصول على عينات محاكاة متجانسة للقياسات الأثرية.

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### Abstract:

This research uses a new technique to accelerate the aging of samples prepared to simulate those archaeological woolen fabrics. Natural dyed woolen Fabric with turmeric and mordanted with Alum, copper, and potassium dichromate were stained with three common stains found on archaeological textiles, namely; mud, oil, and rust, then exposed to UV/ozone for five different periods ranging from 5 min to 120 min. The effect of artificial fast aging on surface morphology was studied by scanning electron microscope (SEM). In addition, the mechanical behavior was studied concerning testing tensile strength and elongation percentage. Also, the changes in the CIE  $L^*a^*b^*$  color values and the total change in the color ( $\Delta E^*$ ) were studied before and after exposure to the UV/Ozone. The detrimental deterioration effect of old stains on mimic samples had the same appearance as those cases of old stains on archeological objects in museums<sup>1</sup>. It gave conservators valuable data for research and experiments about using materials like detergents, resins, consolidations substances, and others to provide results that are used as guidelines to decide whether to use it on actual old pieces or not. SEM images also showed the penetration of mud, oil and rust stains into the interstitial spaces of the hairs inside the woollen cloth dyed with different mordants. The results obtained proved with increasing the exposure time, the colour change and elongations % were increased while the tensile strength decreased.

<sup>1</sup> Abdel-Kareem O et al., Scientific Culture, 2022, 77-88.

Oily stained Aluminum mordanted dyed fabric and muddy stained chromium mordanted dyed fabric increased the susceptibility to aging.

**Key Words:** Natural dyes, Stains, Wool, Fast aging, UV/ozone, Mechanical properties, Color, SEM, Turmeric, Archaeology.

#### الملخص:

يستخدم هذا البحث تقنية جديدة لتسريع تقادم العينات المعدة لمحاكاة الأقمشة الصوفية الأثرية. تم صبغ نسيج صوفي طبيعياً مع الكرم ومرسخ مع الشبه والنحاس وثنائي كرومات البوتاسيوم وتم تبقيعه بثلاث بقع شائعة موجودة على المنسوجات الأثرية، وهي الطين والزيت والصدأ، ثم تم تعريضهم للأشعة فوق البنفسجية والأوزون لمدة خمس فترات مختلفة تتراوح من 5 دقائق إلى 120 دقيقة. تمت دراسة تأثير التقادم الاصطناعي السريع على تضاريس السطح عن طريق مسح المجهر الإلكتروني (SEM) بالإضافة إلى ذلك، تمت دراسة السلوك الميكانيكي فيما يتعلق باختبار قوة الشد ونسبة الاستطالة. أيضاً، تمت دراسة التغييرات في القيم اللونية  $L * a * b$  والتغير الكلي في اللون ( $\Delta E^*$ ) قبل وبعد التعرض للأشعة فوق البنفسجية والأوزون. كان لتأثير التقادم على البقع على العينات المقلدة نفس مظهر حالات البقع القديمة على القطع الأثرية في المتاحف. أعطت للآخرين بيانات قيمة للبحوث والتجارب حول استخدام مواد مثل المنظفات والراتجات ومواد التجميع وغيرها لتقديم النتائج التي يتم استخدامها كإرشادات لتقرير ما إذا كان سيتم استخدامها على القطع الأثرية الفعلية أم لا. كما أظهرت صور الميكروسكوب الإلكتروني الماسح اختراق بقع الطين والزيت والصدأ في المساحات الخلالية للشعر داخل القماش الصوفي المصبوغ بمواد مختلفة. أثبتت النتائج التي تم الحصول عليها أنه مع زيادة وقت التعرض تغير اللون وزادت نسبة الاستطالة بينما انخفضت قوة الشد وزاد القماش المصبوغ بالألومنيوم الملطخ بالزيت والكروم الملون الموحد من قابلية التقادم.

الكلمات الدالة: أصباغ طبيعية، البقع، الصوف، التقادم السريع، الأشعة فوق البنفسجية والأوزون، الخصائص الميكانيكية، اللون، المسكروسكوب الإلكتروني الماسح، الكرم، علم الآثار

## 1. Introduction

Treating any valuable item to conserve ancient historical textiles is thought to be highly risky. To prevent damage to museum artifacts and to extend the lifespan of valuable pieces for coming generations to protect their cultural heritage, conservators explored an idea that would enable them to study and experiment with various proposed conservation plans or proposed solutions. Even though textiles are delicate organic materials highly susceptible to atmospheric influences, working with them in museum laboratories is already difficult. For the conservation and restoration, artificially accelerated aging for simulated samples of those real ones is a good option to test and experiment with remedies and find the best to relate to the original piece after ensuring its adequacy over time. Understanding the nature and type of these stains is crucial to remove those from historically dyed samples. Various stains' detrimental effects were discussed<sup>2,3</sup>. According

<sup>2</sup>Timir-Balazsyet al., Chemical principles of textile conservation, London: Butterworth-Heinemann, 1998

<sup>3</sup>Singhal D. et al., International Journal of Applied Home Science, 2015, 248.

to descriptions of the ageing of oil stains on textiles<sup>4,5</sup> colored humic materials like dyes, inks, and pigments render fibers more sensitive to light<sup>2</sup>.

Microorganisms because biomedical deterioration involves bacteria and microfuge, as described<sup>6</sup>. describes how a textile artifact's stiffness from synthetic resins may speed up ageing and cause mechanical damage. Some stains can be removed with a simple wash, but harsh archaeological stains might need special care because they are covalently tied to the fabric's fiber and engage chemically and physically with time. A conservatory can learn useful information from this study about the effects of actual old stains on textiles<sup>7</sup>. Between 380 and 780 nanometers, a small amount of energy constitutes the visible spectrum. U.V. energy was available in the 100–400 nm range. The I.R energy spans 0.78 m to 103 m. A narrow range of wavelengths, at around 380 to 780 nanometers, illuminate an object. Light is accompanied by trace amounts of infrared (I.R.) and ultraviolet (U.V.); In contrast to non-visible I.R. and U.V., which contribute to damage but also not vision, light contributes to both vision and damage. The effects of exposure are considered by a straightforward formula. Light, radiant energy, and object damage Radiant heating and/or photolysis action are two processes that are enhanced when energy is incident on a material's surface<sup>8,9,10</sup>.

Ultraviolet light has been noted as one of the degradation agents affecting lithic artifacts and functional residues. UV patinates flint. The UV concept also clarified instant resin coating. Although this is best described as arising from the air-water exchange between the environment and also the clastic artifact. However, the influence of UV on flint continues to be debated and is a topic of concern. Although UV light has been involved in the deterioration of glues and debris, research on its effects is missing. From other areas of research, like those focusing on polymerization, disinfection, skin cancer, degradation of organic matter (in the environment as well as in museum environments and the food industry), pollutants, and construction materials, it is known how UV affects various materials. However, controlled trials or syntheses have yet to be written to recognize and repeat these phenomena in archaeology. The interpretation of altered material is enhanced by learning from earlier research about how ancient historical material and functional residues may be hurt and altered by UV. Here, we synthesize earlier<sup>11,12</sup>.

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<sup>5</sup> Moreland, B., IFI Fabricare News ,1987, 14.

<sup>6</sup> Caneva, G.et al., Biology in the Conservation of Works of Art, ICCROM,1991,60-1.

<sup>7</sup> Ballard. M.W., the 10th Triennial Meeting of the ICOM Committee for Conservation, 1993, 331-8.

<sup>8</sup>ElnagarKh. etal., Fibers and Polymers 2013,1581-1585.

<sup>9</sup>Gotti, M.B et al.,Light Sources and Dye Fading G.E. Lighting, Retrieved Friday, 2009.

<sup>10</sup>Ibrahim S.F. et al., International Journal of Conservation Science, 2015,15-22.

<sup>11</sup>Hebeish A.et al., Egypt. Journal of Chem.,2015, 415-430.

<sup>12</sup>Hebeish A.et al., Materials Sciences and Applications, 2014, 698-707.

Artifacts of cultural heritage from a wide variety and made of various materials are kept in museums. Special storage conditions for each type of object through historic sites impede the decay of materials, an essential component of the life cycle. UV light, high humidity, temperature, and altering these two factors are all variables that speed up material changes. Living things, particularly fungi, are some of the worst material decomposers<sup>13,14</sup>.

Physical influences typically cause the materials to deteriorate. However, first, that enables (micro)organisms to denature them faster<sup>15,16</sup>. These factors carry on the depolymerization of molecules, hydrolysis, and instability of intra- and intermolecular bonds<sup>13,17,18</sup>.

To define the deleterious effects of aged stains on dyed woolen textiles, a new rapid procedure is used to speed up the ageing of samples of woolen textiles dyed with turmeric and stained with three common stains, such as mud, oil, and rust. It has also been noted that the UV/ozone technique can be applied safely and efficiently like an intensifying technique, particularly for specimens of silk and wool. Last but not least, UV/ozone treatment is beneficial and practical for creating replica archaeological samples that can be used in conservation and restoration work to give conservators valuable information that truly benefits them<sup>19</sup>. However, it is a quick and simple method that saves time, money, and the environment.

## **2. Experimental Work:**

### **2.1 Materials and Methods:**

#### **2.1.1 Wool fabric**

Golden Text Factory in Egypt supplied 100% pure wool fabric, which was scoured and bleached (200 g/m<sup>2</sup>). This fabric had 25 yarns in the warp direction and 34 yarns in the weft direction. An aqueous solution containing non-ionic detergent (2 g/l) was prepared to scrub wool fabric for 15 min at 25 ° C. Then, the fabric was thoroughly cleaned with tap water and dried at room temperature. After that, samples (30×30 cm<sup>2</sup>) were cut from the divided fabric.

#### **Dyes**

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<sup>13</sup>Szostak-Kotowa J., *International Biodeterioration and Biodegradation*, 2004,53:165-70.

<sup>14</sup>Seves A, et al., *International Biodeterioration and Biodegradation*, 1998, 42:203-11.

<sup>15</sup>Zotti M. et al., *Mycological and FTIR analysis of biotic foxing on paper substrates*. ITOG, 2023, 2-15.

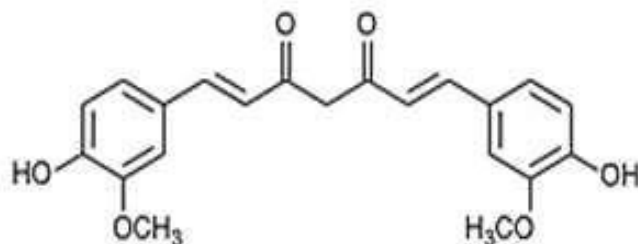
<sup>16</sup>Valentin N. et al., *Molecular biology and cultural heritage*,2003, 287.

<sup>17</sup>Proniewicz LMet al.,*Journal of Molecular Structure*, 2001,596: 163-9.

<sup>18</sup>Montegut D. et al., *International Biodeterioration*,1991, 209-26.

<sup>19</sup>Chakravarty T. et al., *Applied and Environmental Microbiology*,1962, 441-7.

The natural dyestuffs used in this work were turmeric obtained from "Heraaz's Supplier", Cairo, Egypt. Turmeric *Curcuma longa*. Its principal component is curcumin with chemical structure [(1E,6E)-1,7-bis(4hydroxy-3-methoxyphenyl) hepta-1,6-diene-3,5-dione].



Curcumin chemical structure

### 2.1.2 Chemical Mordants

2.1.2.1 Alum Aluminum sulfate,  $KAl(SO_4)_2 \cdot 12H_2O$ .

2.1.2.2 Potassium dichromate,  $K_2Cr_2O_7$ .

2.1.3.3 Coppersulfate,  $CuSO_4$ .

### 2.2 Mordanting procedures

The samples were mordanted by introducing L: R 1:20 of the mordants, which were dissolved in distilled water and heated to simmering heating on a hot plate for 1 hour. Next, the scoured wool fabrics were added, maintained at this temperature for 30 min, and allowed to cool in the dark overnight. They were then rinsed with distilled water and left to dry.

### 2.3 Extracting the Dyes

Distilled water was used to extract the turmeric. Additionally, the mixture was allowed to soak for 30 minutes until boiled for 60 minutes. The content was then filtered and cooled. For dyeing, the filtrate was employed. The method of exhaustion used to carry out the dyeing. (acetic acid is used to adjust pH to 4) The temperature was increased to 91–93 C (the simmering point) and held there for one hour. The dyed samples were soaked in distilled water and then room temperature dried.

### 2.4 Preparing different stains

Figure 1 shows three different stains used to test the fabric strips: mud soils, iron rust, and corn oil stains. On the mordanted dyed samples, a known quantity of each stain was applied separately and allowed to dry at ambient temperature before ageing.










	(a) Mud	(b) Oil	(c) Rust
<b>Cu</b>			
<b>Alum</b>			
<b>Cr</b>			

Figure (1) Wool fabric samples dyed with turmeric dye mordanted with Cu, Al, and Cr salts and the application of (a) mud, (b) oil, and (c) rust stains on them.

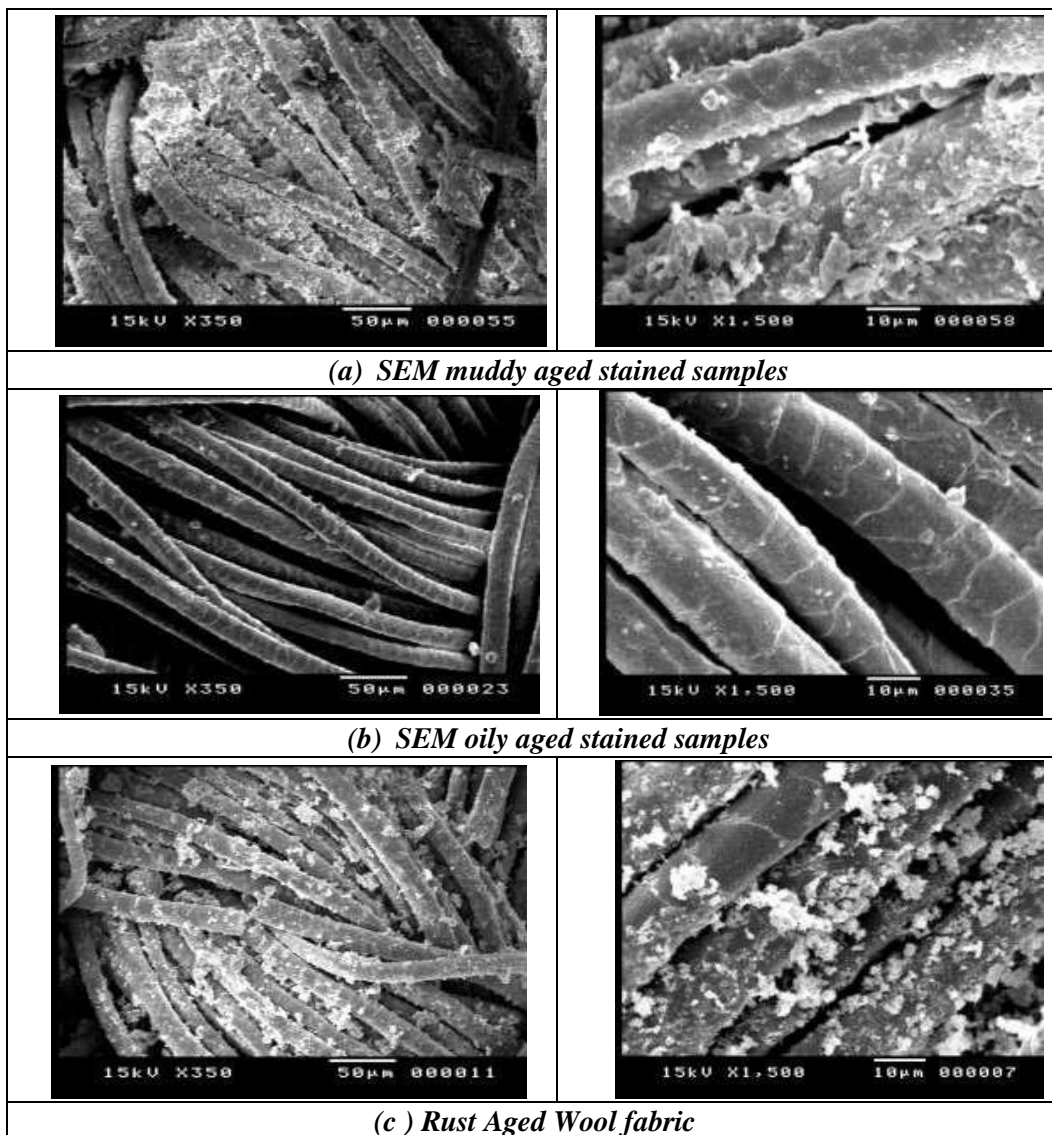


Figure 2: SEM images for stained, dyed samples with two magnification powers (X350 and X1500). (a) Mud, (b) Oil and (d) Rust stains of the blank samples.

### **2.5 UV/Ozone Treatment.**

A low-pressure mercury lamp (LRF 02971, 200 watts, 220 volts, made in Poland) was placed in a cubic box with sides that measured 60 cm in length that provided high-intensity UV/O<sub>3</sub> irradiation. Strips of samples were hung around the source for various times 5, 20, 50, 110, and 120 min at a distance of around 20 cm<sup>8</sup>.

### **2.6 Testing and Analysis:**

#### **2.6.1 Color measurement:**

Using an Optimatch 3100 ® SDL spectrometer, color readings for all test samples were carried out in line with the CIELAB system. Due to the color and hue changes caused by staining and cleanup,  $\Delta E$  was used to calculate the color change between the standard and sample in the visible spectrum (400-700 nm). The freshly stained and dyed sample was provided as the standard, and the stained, dyed, aged samples served as the samples. The determination of the L (White-Black), a (Red-Green), and b (Yellow-Blue) coordinates of the CIELAB space results in the tracing of the colors triplicates for every sample<sup>20,21, 22</sup>.

#### **2.6.2 Mechanical measurements**

Tensile strength and elongation (%): Using a Shimadzu Universal Tester of (C.R.T)-type S-500 Japan, the tensile strength and elongation percent at break of both untreated and treated samples for various eras were tested in consonance with the ASTM Standards (ASTM, 2000) <sup>22</sup>.

#### **2.6.3 Morphological study with SEM:**

The stains within wool fibers and how they adhere to them were examined using SEM or scanning electron microscopy. SEM evaluation. Using an SEM model Philips XL 30 with an accelerating voltage of 30 kV, a magnification range of 10x to 400.000x, and a resolution for wavelength, Sem micrograph of the samples used in our study were taken (3.5 nm). Gold is used to coat the samples. Staining samples are examined under SEM to ascertain their morphology.

## **3 Results and discussion:**

### **3.1 Color measurement:**

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<sup>20</sup>Christie R.M.et al., The Chemistry of Color Application, Blackwell Science Ltd. London, 2000.

<sup>21</sup>Abdel-Maksoud G.et al., Journal of Cultural Heritage, 2022, 128-137.

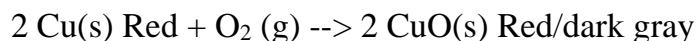
<sup>22</sup>El-Nagar Kh. et al., Mediterranean Archaeology and Archaeometry, 2022, 203-213

### **3.1.1 Effect of mordants on the color of wool samples and their aging at different times**

It is well known that natural dyes need metal ions from suitable mordants in order to bond with the fiber surface and form a stable composition. Every ion has a distinct color and behavior with regard to how turmeric dyes age.<sup>23,24,25</sup>

Table 1 shows a study of the effect of using different mordants such as aluminum, chromium, and copper on wool samples dyed with turmeric dye. It is clear from the results that the samples fixed with aluminum ions have the most brightness, 'L' (74.63), followed by copper (67.59) and then chromium (60.41). On the other hand, we find that aluminum gives a degree of redness 'a' (3.67) greater than chromium (2.52), while the copper ion tends to form a color with a degree of greenness '-a' (-1.72). When observing the yellow-green color component 'b', we find that aluminum tends to give a yellow color (46.92) greater than copper (33.50), then chromium (20.68).

By examining the effects of aging on samples dyed with turmeric, we discover that the L value increases significantly with longer exposure times due to the relative oxidation of aluminum compounds, which are prone to turning white. Due to the strength of the bonding between the chromium element, the dye, and the fibers, when studying the effect of aging time on the samples fixed with chromium ions, we find some stability in the degree of L. While the percentage of L decreases significantly as exposure time increases due to the oxidation of copper to form a dark grey copper oxide and the tendency to form hydrated green iron in the presence of sulfur or carbon oxides,  $\text{Cu}_4\text{SO}_4(\text{OH})_6$  and  $\text{Cu}_2\text{CO}_3(\text{OH})_3$ .



By observing the change in the values of a, we find that aluminum causes an increase in the degree of redness by increasing the exposure time, while chromium works to reduce the degree of redness by increasing the exposure time. Similarly, copper reduces the degree of redness and increases the degree of greenness, as it is known to increase the formation of green copper oxide by increasing the exposure time.

Table 1 also shows that, when using aluminum, the yellow-blue color compound decreases with exposure time (from 46.92 to 33.98), just like it does with chromium (from 20.86 to 16.27) and copper (from 33.50 to 27.59).

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<sup>23</sup>El-Nagar Kh. et al., *PolymerPlastics Technology and Engineering*, 2005, 1269-1279.

<sup>24</sup>Adeel, S. et al., "Dyeing of Cotton Fabric Using UV Irradiated Turmeric (*Curcuma longa* L.) as Natural Dye", *Research Journal of Textile and Apparel*, 2011, 71-76.

<sup>25</sup>TeraFerial Met al., *Journal of Textile & Apparel Technology & Management (JTATM)*, 2012, 1-6.



Table (1) effect of mordant types on color and its aging.

Exposure Time (Min.)	Alum			Potassium Di-chromate			Copper sulfate		
	L	a*	b*	L	a*	b*	L	a*	b*
0	74.63	3.67	46.92	60.41	2.52	20.86	67.59	-1.72	33.50
15	72.89	6.10	39.89	60.44	1.73	18.22	66.29	-0.74	30.62
30	73.68	5.91	37.94	60.39	1.56	17.42	65.86	-0.28	29.58
60	73.86	5.59	35.44	60.04	1.09	16.29	66.58	-0.08	28.80
120	74.09	5.86	33.98	60.64	0.85	16.27	65.98	0.10	27.59

### 3.1.2 Effect of aging time on the color change ( $\Delta E$ ) of dyed samples stained with mud, oil, and rust

Figure 3a depicts the effects of aging time on samples of turmeric stained with mud, oil, and rust stains. As a result of their high fatty acid content, which speeds up the oxidation of both the dye and the used mordant, the samples with the oil stain in the existence of the aluminum mordant are the most discolored, according to the results. However, by leaving the surface of the fabric samples and acting as scarifies to protect the substrate and the dye, rust stains can be flaked away by oxidation.<sup>23,26</sup>

Figure 3b shows that samples with mud and chrome mordant stains are most affected by aging, whilst samples with oil and rust stains exhibit relatively stable color change. Rust is less sensitive to moisture, and chrome tends to form crosslinks with the elements of the mud slick to a large extent than fatty acids.

Figure 2c's findings show that all stains act uniformly, which may be described by the protective covering created by ageing's oxidation effect. Also, mud and ageing can reduce Cr (IV), which results in a weaker crosslinking of the dye and fibers<sup>27</sup>.

<sup>26</sup>Tera F. M.et al.,Journal of Textile & Apparel Technology& Management (JTATM),2011, 1-10

<sup>27</sup>Tzou M., Loeppert R. H., Wang M. K. (2003), Light-Catalyzed Chromium (VI) Reduction by Organic Compounds and Soil Minerals J. Environ. Qual. 32:2076–2084.

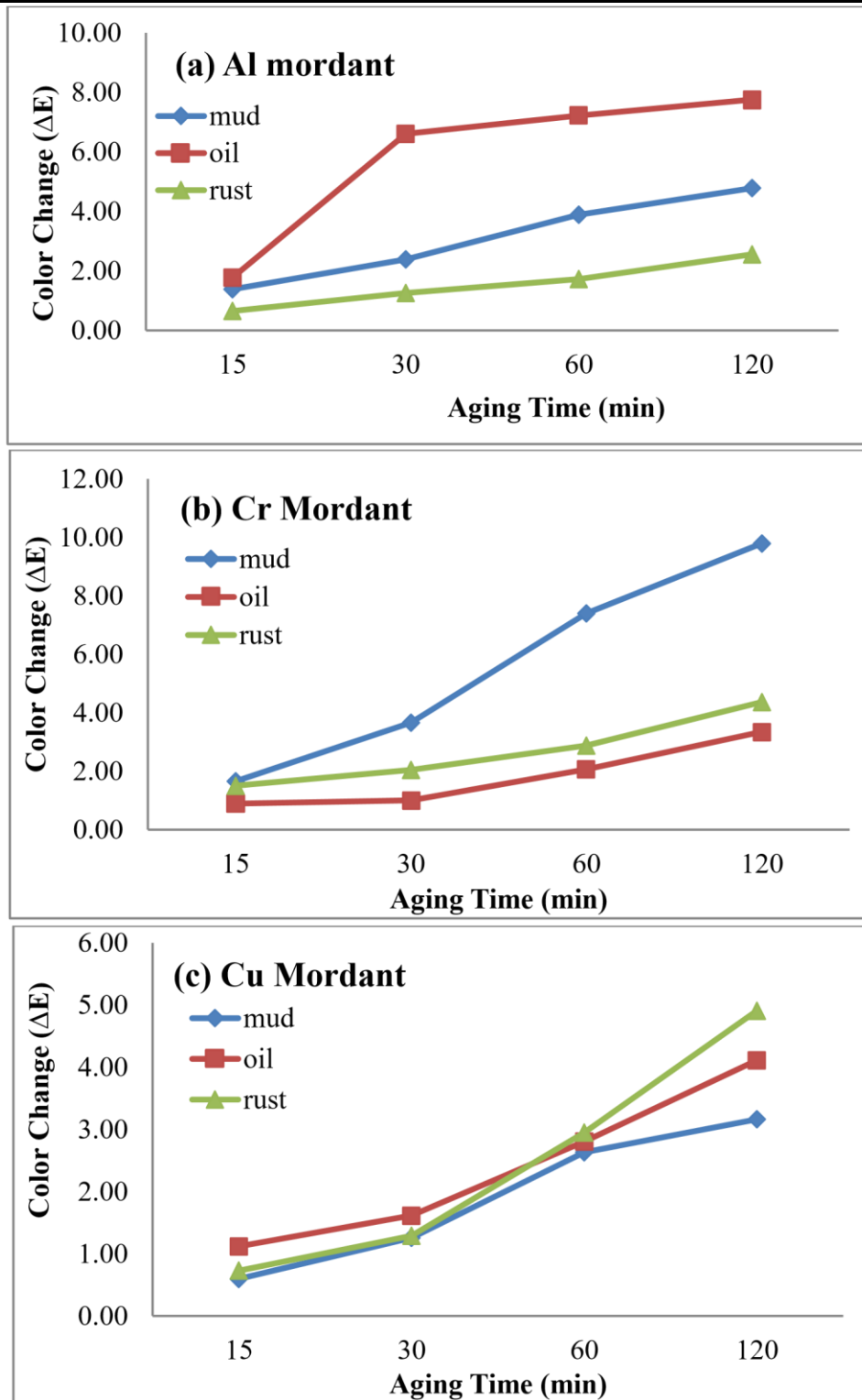


Figure 3: Effect of aging time on color change( $\Delta E$ ) of turmeric dyed samples stained with (mud, oil, and rust) and mordanted with a) Alum; b) Chromium, and c) Copper.

### 3.2. Mechanical measurements

On woolen samples that were dyed with turmeric dye and fixed with three different mordants such as aluminum, chromium, and copper, as well as staining them with three different stains like mud, oils, and rust, Table 2 demonstrates the connection between mechanical characteristics (tensile and elongation%) and the duration of exposure to ultraviolet/ozone radiation as a method for rapid aging<sup>28</sup>. Table 1 indicates that the tensile strength of all samples decreases as the radiation exposure period with the studied mordants and stains. The cross-linking between the copper and the dye could explain why the aluminum mordant of the clay-stained clay had a relatively lower tensile strength and why the wool fiber surface had a smaller surface area between the fibers. The likelihood of crosslinks forming between dyes and the fibers is greater when chromium is used as a mordant for turmeric dye because of its several coordination and crosslinking bonding. The main cause of the decrement in tensile strength is the acidic effect of oils, which increases the effect of ultraviolet radiation in the presence of ozone, an effective oxidizing agent. Another factor is the temperature, which may arise due to affecting the temperature of the light source.<sup>23</sup>.

Table (2) Effect of Aging time on tensile strength (Maximum force, N) of the wool samples dyed with turmeric Mordants dyes using Al, Cr, and Cu mordants in addition to the application of mud, oil, and rust.

stains Mordants Time (min)	Mud			Oil			Rust		
	Al	Cr	Cu	Al	Cr	Cu	Al	Cr	Cu
0	299.6	405.0	350	345.2	372	333.6	335.6	366.0	332.4
15	293.2	378.8	312.0	340.4	348	330.0	322.0	339.6	316.8
30	286.4	350.4	308.4	327.6	338	320.4	303.6	333.6	307.6
60	282.8	326.8	300.4	323.6	336.8	294.8	302.0	328.8	296.0
120	267.6	277.2	296.8	300	316.4	290.4	254.4	320.0	213.6

Figure 4a-c shows the effect of aging time on wool samples dyed with turmeric and fixed with three mordants (aluminum, chromium, and copper), each stained with three different spots (mud, oil, and rust). The results showed in Figure 2 that the rust-stained samples were not affected by the nature of the mordant, while the mud-stained samples were the most affected, followed by the oil-stained samples; This can be explained in light of the dislike of water to oil stains, followed by rust, followed by mud. Also, as a result of the exposure of the rust spot to aging, it results in slippery sheets that increase the amounts of elongation corresponding to the different samples, while the element chromium and

<sup>28</sup>Smejda-Krzewicka A. et al., The Effect of Filler Type on the Properties of CR ProductsMaterials,2021.

aluminum are water compounds due to their reduction by the action of organic compounds and water <sup>27,29</sup>.

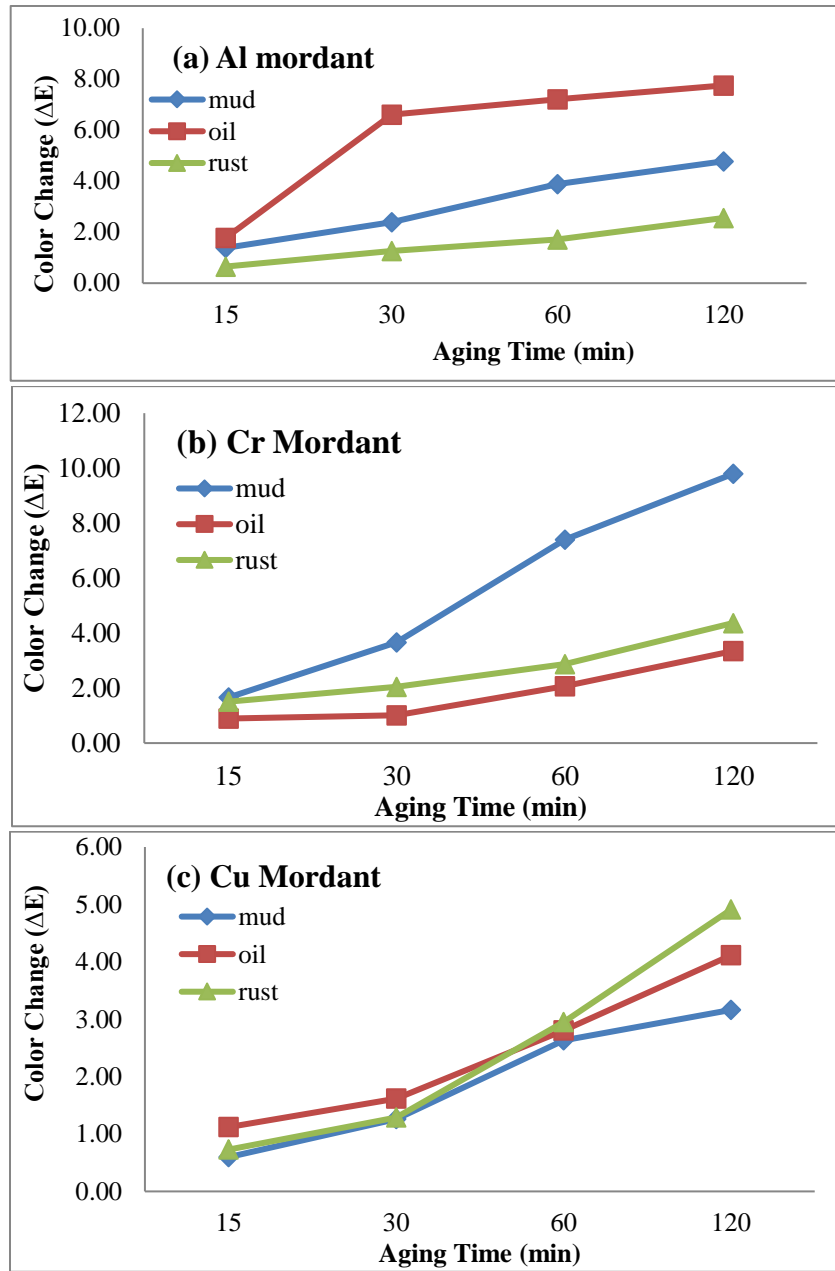


Figure 4: Effect of aging time on Elongation % of woolen fabric dyed with turmeric dye and mordanted (with Al, Cr, and Cu). Stained with a) mud, b) oil, and c) rust.

<sup>29</sup>Touzi Het al., Journal of Inorganic and Organometallic Polymers and Materials, 2021, 3011–3026.

#### **4 Conclusions:**

This study examined the impact of accelerated UV/Ozone aging on woolen samples dyed with turmeric dye and fixed with three distinct mordants: aluminum, chromium, and copper. Additionally, each sample was stained with three various stains, including mud, oil, and rust. The findings demonstrated that, in addition to the color change, the color compounds represented in L, a, and b also changed significantly in response to the differences in mordant, stain, and aging time because chromium is more sensitive to copper than aluminum. Additionally, the mechanical characteristics of the samples varied due to exposure to the dissolved aging because of the variations in the color of the minerals used as supplies (from 0 to 120 minutes). In light of the results of the CIE color components (L, a, and b), total color change ( $\Delta E$ ), mechanical properties (tensile strength and elongation%), as well as the study of morphological properties, it was determined that care must be taken in choosing the mordant when conducting studies on archaeological mimic samples and designing treatment and maintenance protocols and its applications.

#### **5 Acknowledgments**

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#### **6 Conflict of Interests**

The authors declare that there is no conflict of interest regarding the publication of this paper.

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