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Using UV/Ozone Accelerated Aging Technique to Prepare Artificial Deteriorated Standard Dyed Woolen Samples for Textile Conservation Purposes

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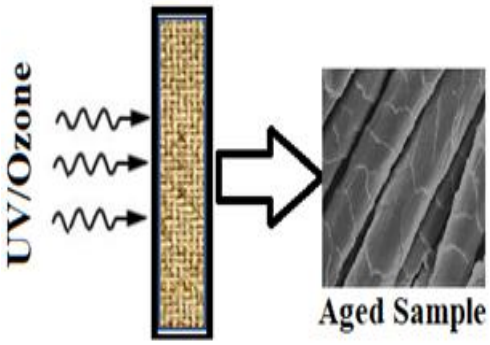
HIGHLIGHTS

- This research offers a new eco-friendly and fast accelerated aging technique.
- UV/Ozone showed high performance aging effect on wool fabric samples.
- Simulated samples to historical textiles are achieved throughout this work
- Morphological, mechanical, and color parameters proved the efficiency of the ageing process.
- Three natural dyes mordanted with three mordants were chosen to achieve different chroma and hues.

GRAPHICAL ABSTRACT



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ABSTRACT

Before the introduction of synthetic dyes in 1856, natural dyes from plants and insects were commonly used to color fabrics. However, these dyes are prone to fading and damage, posing challenges for conservators when cleaning, preserving, or displaying historical textiles. To address this issue, creating mimic samples that accurately replicate the conditions of these valuable artifacts provides a valuable opportunity for conservation research and testing of materials. This study aims to explore the feasibility of using the UV/Ozone technique to create artificial

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samples of faded dyed woolen fabric that resembles ancient textiles, without damaging the original artifacts. These samples could be used as models in conservation laboratories. Three natural dyes (Turmeric, madder, and cochineal) were used to dye new wool fabrics, with different mordants (alum, potassium dichromate, and copper sulfate) to achieve various hues. The dyed wool samples were then artificially aged using UV/Ozone for different durations. The color of the samples gradually faded as they aged, with changes in the CIE L*a*b* parameters and overall color difference (ΔE*) analyzed. The morphology and mechanical properties, including tensile strength and elongation, were also studied. Yellow dyes were found to be the most light-sensitive, while red dyes aged faster. The results suggest that artificial aged wool textile samples can be quickly created for experimental purposes, providing valuable insights for conservation research.

1. Introduction

Wool is classified as an animal fiber because it is made of protein. It is highly absorbent compared to other textile fibers. Wool is composed of keratin, a protein that consists of amino acids linked by peptide bonds. Wool has unique characteristics that differentiate it from hair or fur, such as its crimped texture, elasticity, and tendency to grow in clusters. People have been using wool as a textile for thousands of years.

Natural dyes have been used since ancient times for various purposes, including dyeing natural fabrics such as linen, wool, cotton, silk, fur, and leather. They were also used to color cosmetics, inks, watercolors, and artist paints. The practice of using natural dyes for textile coloring was common until the introduction of synthetic dyes by Perkin in 1856 [1, 2]. The presence of oriental rugs in museums around the world is a testament to the historical significance and prestige of using vegetable, animal, and mineral-based dyes.

Natural dyes require the use of mordants, which are metal salts that help bind the dye to the yarn. Mordant metal ions act as electron acceptors to form coordination bonds with the dye molecules [2].

Chemical linkages between the dye molecule and the mordant create unique colors in the woolen yarn. Alum, chromium, stannous chloride, copper sulfate, and ferrous sulfate are common mordants used in textile dyeing.

Museum collections typically include items with natural colors, which can be delicate and prone to fading. This presents challenges for conservators who must decide how to handle, clean, preserve, and display these items. Fading is a major concern for museum staff tasked with protecting antique textiles for future generations. Textiles dyed with natural substances are especially vulnerable to light exposure, making long-term preservation difficult. Fading tests are often done under accelerated conditions using artificial light sources like carbon arc lamps and xenon arc lamps, rather than natural sunlight [2].

Artificial aging techniques are used in archaeological textile conservation for various purposes. These techniques are employed to create artificially deteriorated dyed textile samples with specific characteristics and to evaluate new materials for preserving museum textiles [3].

The UV/Ozone technique is recognized as a safe and efficient method for accelerating changes in silk and wool samples. Factors such as ultraviolet radiation, high humidity, and temperature contribute to the acceleration of material changes. Living organisms, especially fungi, play a significant role in material decomposition. The use of UV/Ozone treatment is beneficial for creating replica excavation specimens that can be used in conservation and restoration projects. This method is simple, efficient, cost-effective, and environmentally friendly [3].

The aim of this study is to introduce the use of UV/Ozone aging technique to create artificially deteriorated wool textile samples with specific properties. These samples will be used for evaluating new materials and techniques for conserving and restoring deteriorated wool textiles before applying them to historical textiles. They can also be used for training textile conservators. The study involved wool fabric samples dyed with natural turmeric, cochineal, and madder, and mordanted with alum, chromium, and copper. The samples were subjected to light ag-

ing using a UV-ozone lamp, a method for efficiently creating synthetic samples as substitutes for historical textiles. A comparative analysis of the aged samples was conducted, assessing parameters such as tensile strength, elongation, and color difference (ΔE) values [4].

2. Materials and Methods

2.1. Materials

The fabric used in this study was 100% pure wool with a plain 1/1 weave and a weight of 200 g/m^2. It was purchased from Golden Text Factory in Egypt. The fabric had a yarn count of 25 yarns in the warp direction and 34 yarns in the weft direction [5].

2.2. Methods

2.2.1. Samples preparation

The wool fabrics were cleaned using a solution with 2g/L of nonionic detergent at a ratio of 1:20 at 40°C for 15 minutes. They were then rinsed with water and air-dried. The fabric was cut into 30cm x 30cm samples.

2.2.2. Mordanting procedures

Chemical Mordants

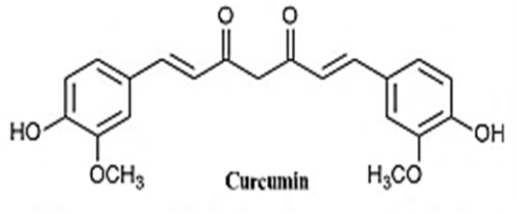
- Alum; Aluminum sulfate, KAl $(SO_4)_2.12H_2O$. (as source of Al mordant).
- Potassium dichromate, $K_2Cr_2O_7$. (as source of Cr mordant).
- Copper sulfate, $CuSO_4$. (as source of Cu mordant).

These chemicals were purchased from Sigma Aldrich agent, Cairo, Egypt. The mordants were dissolved in distilled water at a fabric to liquor ratio of 1:20. The solution was heated to a simmering temperature on a hot plate for 1 hour. The scoured wool fabrics were then added to the bath and maintained at this temperature for 30 minutes. The bath was allowed to cool overnight before the fabrics were rinsed with distilled water and left to dry [6].

2.3. Dyes

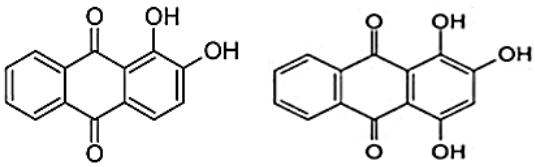
The natural dyestuffs were purchased from "Heraaz's Supplier", Cairo, Egypt. They were as follows:

- **Turmeric *Curcuma longa*.** Its principal component is curcumin.



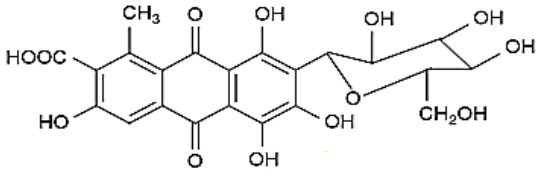
Chemical structure of curcumin [7]

- **Madder** (*Rubia tinctorum L.),* its principal components are alizarin and purpurin.



Chemical structure of alizarin and purpurin (the madder's dyestuffs) [8].

- **Cochineal (*Dactylopius coccus.*):** It was prepared from dried cochineal insect (its principal components are carminic).



Chemical structure of carminic acid the cochineal's dyestuff [6].

2.4. Preparation of dyes solutions

Turmeric, madder, and cochineal were all dissolved in distilled water. After a 30-minute soaking period, the mixture was boiled for 60 minutes. The resulting solutions were then cooled, filtered, and used for dyeing. The dyeing process employed the exhaustion method, with the pH adjusted to 4 using acetic acid. The temperature was raised to 91-93°C and maintained for 1 hour. After dyeing, the samples were rinsed with distilled water and dried at room temperature [9].

2.5. Aging procedure

Aging is the gradual and permanent changes that occur over time. A team from the National Institute of Standards (NIS-Egypt) used artificial aging techniques to study aging. They developed a new method using UV with Ozone to quickly age samples for conservation and restoration purposes.

In the experiment, a high-intensity, low-pressure mercury lamp (LRF 02971, 200 watts, 220 volts, made in Poland) was placed in a 60 cm long cubic box. Samples were hung 20 cm away from the lamp for 0, 15, 30, 60, and 120 minutes [4].

The products were exposed to atomic oxygen, leading to the creation of simpler and volatile molecules that were then released from the surfaces. The presence of both wavelengths caused a continuous production of atomic oxygen and the continuous cycle of ozone formation and destruction [10].

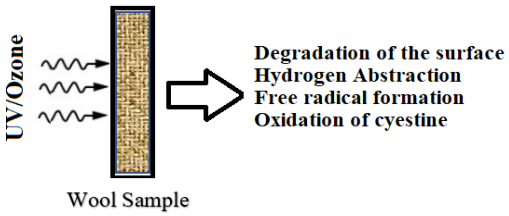


Fig.1. UV-Ozone lamp used for artificial acceleration aging in this study. It is a high-intensity, low-pressure mercury lamp without an outer envelope (LRF 02971, 200 watt, 220 volts, made in Poland).

2.6. Testing and Analysis

2.6.1. Tensile strength and elongation

The tensile strength of dyed aged samples was measured and evaluated over different time periods using a Tinus Olsen H5KT dynamometer, following ISO 2062 standards. The experiments were carried out according to the Standard Method [11].

2.6.2. Color change

The objective is to compare the effects of exposure time on the extent of color change by measuring the ΔE value for each dye across five distinct time periods. The Color Eye 3100 Spectrophotometer SDL, manufactured in England, will be utilized for this purpose. The equation is utilized to determine the overall color difference [12].

$$\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2}$$

Where 'L* scale measures lightness and varies from 0 (black) to 100 (perfect white). a* scale measures red-green; + a is redder; -a is greener. The b ∗scale measures yellow blue, + b is more yellow, -b is bluer.

2.6.3. Morphological Study

An investigation was conducted on the surface structure of untreated and aged fabric samples using a Scanning Electron Microscope (SEM) Model Quanta 250 FEG (Field Emission Gun) in the Central Laboratories Sector of the Egyptian Mineral Resources Authority [13].

2.6.4. Infra-red spectroscopy

The effect of UV/Ozone on the chemical composition of woolen fabrics. was studied using Fourier Transform Infrared (FTIR) spectroscopic analysis. The IR spectra of the KBr disk loaded with fixed quantitative volume of the dye solution (ranged from 0.001 to 0.05 ppm) were measured by using Nicolet 380 (FTIR) Spectrometer, USA, in the wavelength range 4000-500 cm^{-1}. Metrological traceability was achieved by the calibration of the spectrophotometer by using standard polystyrene film traceable to NIST [14].

2.6.5. Statistical Analysis

All results in this research are the average of three individual readings. The uncertainty of analytical and testing techniques was: FTIR measurements ± 3.3 cm^{-1} (calculated from standard polystyrene film, repeatability and spectrophotometer used), tensile strength ± 3%, elongation ± 1% (calculated from repeatability and tensile strength testing machine), color measurements 0.6% (calculated from standard white tile and color matching spectrophotometer).

3. Results and Discussion

3.1. Effect of UV/Ozone exposure on tensile strength and elongation

The results in Figs. 2 to 4 show how wool samples dyed with turmeric, cochineal, and madder dyes, which were treated with aluminum, chromium, and copper mordants affect tensile strength. The tests were conducted under standard conditions of 25°C and 65% RH. Fig. 2 indicates that turmeric dye fixed with aluminum had the highest tensile strength, followed by chromium and then copper. This increase in strength is likely due to the formation of cross-links caused by the heavy metal compounds present in the dyes, enhancing the tensile strength of the samples [15].

The results shown in Fig. 3 of the madder dye analysis reveal that Cu had the highest tensile strength, followed by Al and then Cr. The increased tensile strength can be linked to the formation of cross bonds due to heavy metal compounds present, enhancing the strength of the samples. This effect is influenced by the interaction between the wool fibers, heavy elements in the mordant, and the dye molecules.

Fig. 4 shows the tensile strength of samples dyed with cochineal dye. The samples fixed with aluminum exhibit the highest tensile strength, followed by Cu and Cr.

The study found that exposure to ultraviolet rays from ozone gas reduces the tensile strength of materials over time. Aluminum showed the highest rate of decrease in tensile strength, followed by chromium and then copper when exposed to turmeric dye. For madder dye, copper exhibited the highest rate of reduction, followed by chromium and then aluminum. In the case of cochineal dye, chromium had the highest decrease rate, followed by aluminum and then copper. This variation in tensile strength reduction can be attributed to the interactions between dyes, mordants, and wool fibers.

Previous research has shown that dyes with different mordants can help protect wool fibers from damage caused by exposure to ultraviolet rays and ozone gas. The decrease in tensile strength of dyed samples is likely due to factors such as surface degradation, hydrogen abstraction, free radical formation, and oxidation of cysteine [16].

Figs. 5-7 depict the effect of radiation exposure on the elongation percentage in wool samples dyed with turmeric, madder, and cochineal colors. The results show that the influence of aluminum mordant on turmeric-dyed samples is less significant compared to copper and iron mordants, which have similar effects. Fig. 5 shows the elongation impact on cochineal-dyed samples, with aluminum mordant providing the highest resistance, followed by copper and chromium. Fig. 6 illustrates the interconnected relationship between madder color and various mordants. The observed patterns in the impact of UV/Ozone exposure are attributed to the interactions among natural dyes, mordants, and wool fibers [16].

The UV/Ozone interaction can be due to the behavior of hexavalent chromium Cr(VI), mainly produced in industrial activities and existing as $Cr_2O_7^{2-}$, $HCrO_4^-$, and CrO_4^{2-}, the readily mobile presence of humidity and the radiation induces the photo electron reduction of chemical chelates between studied dye and the fibers [17]. The aluminum containing compounds behave as a good barrier towards the UV radiation [18]. The UV light induced the formation of Cu_2O on Cu compounds, which is more stable and compacted than naturally Cu compounds [19].

3.2. Color change measurements

The results in figures 8 to 10 show how exposure durations of aging affect wool specimens dyed with turmeric, cochineal, and madder dyes fixed with aluminum, chromium, and copper mordants. The impact on color changes is examined. Ultraviolet radiation from ozone gas was found to increase color alteration as exposure time increased from 0 to 120 minutes. The rate of color change varied based on dye and mordant types.

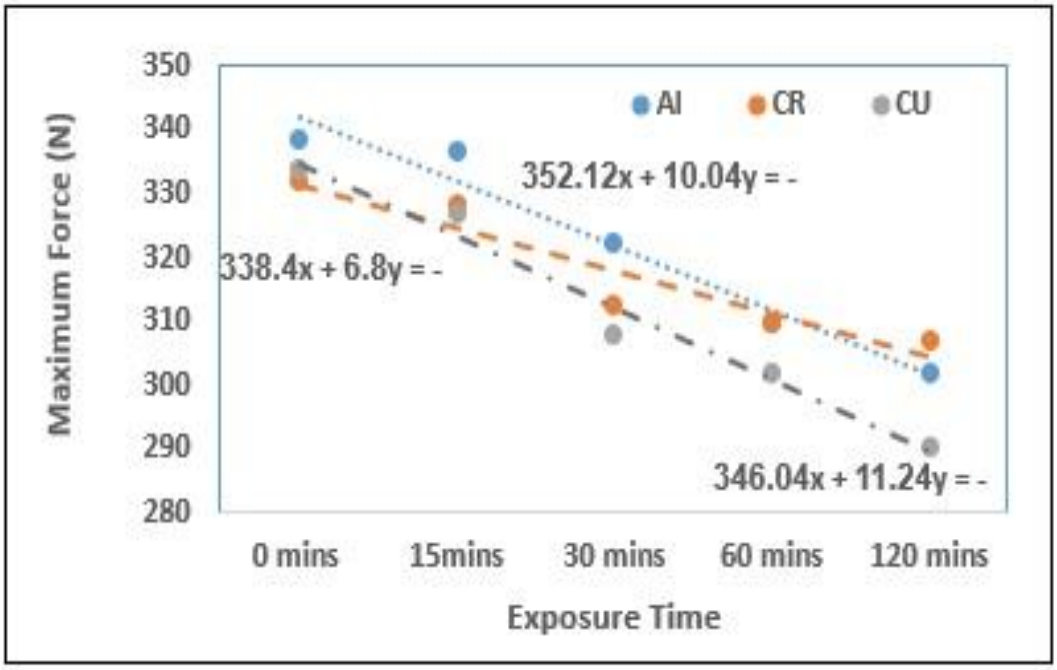


Fig. 2. Effect of exposure time on the tensile strength of woolen fabric dyed with turmeric using different mordants.

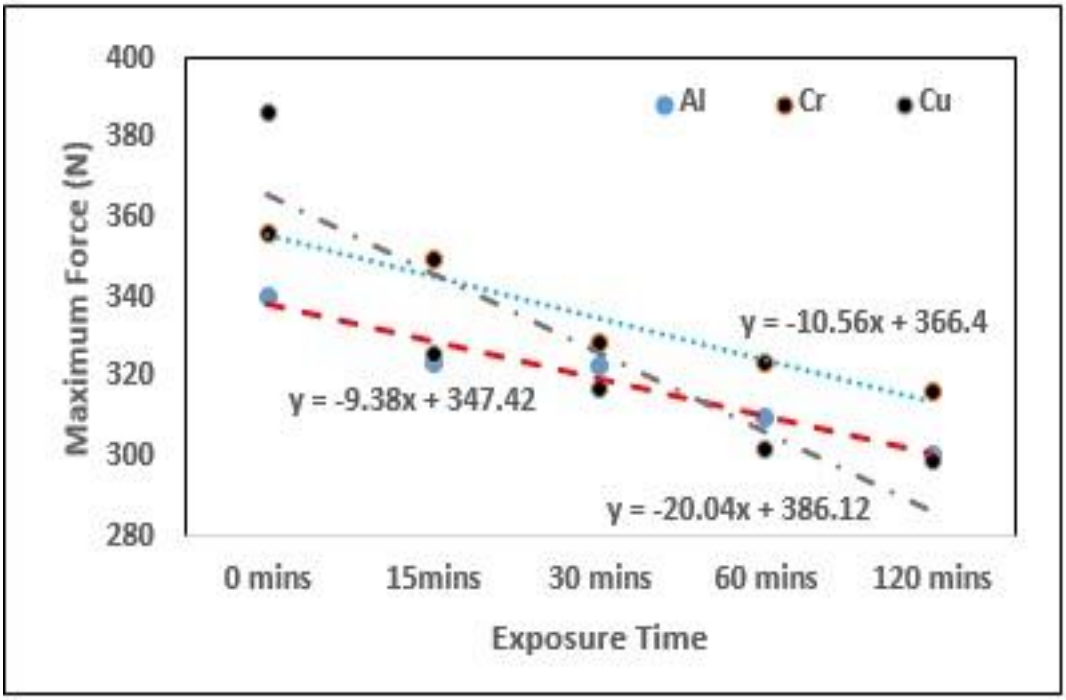


Fig. 3. Effect of exposure time on the tensile strength of woolen fabric dyed with madder using various mordants.

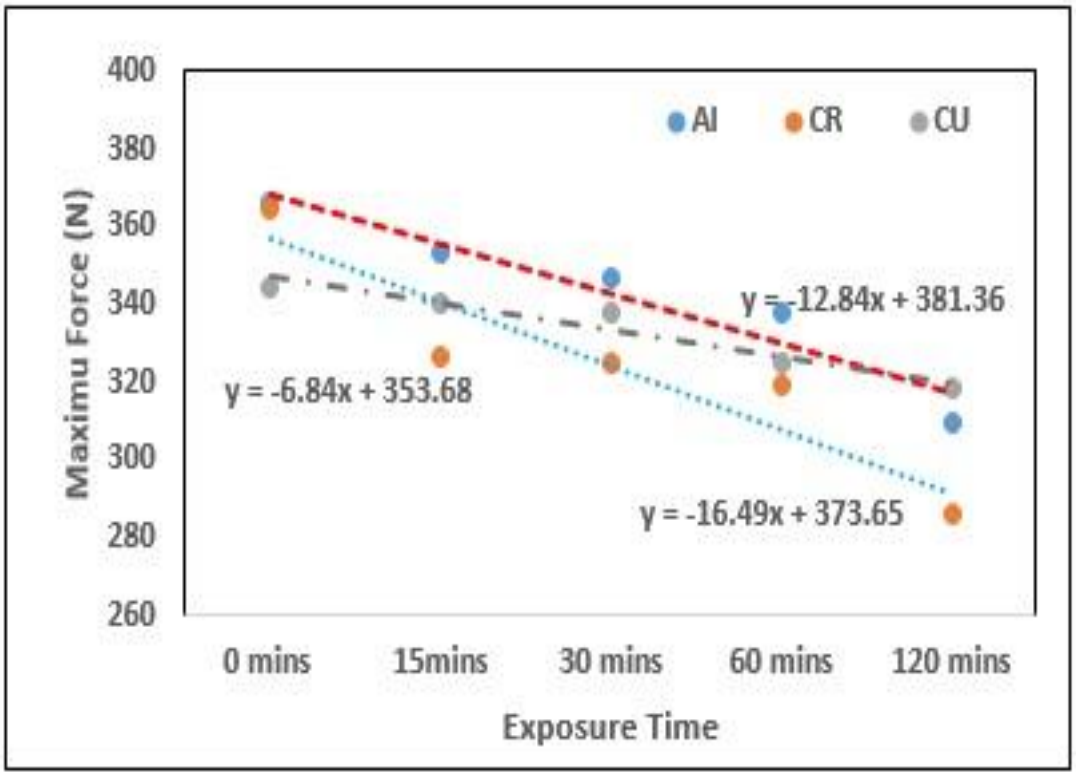


Fig. 4. Effect of exposure time on the tensile strength of woolen fabric dyed with cochineal using different mordants.

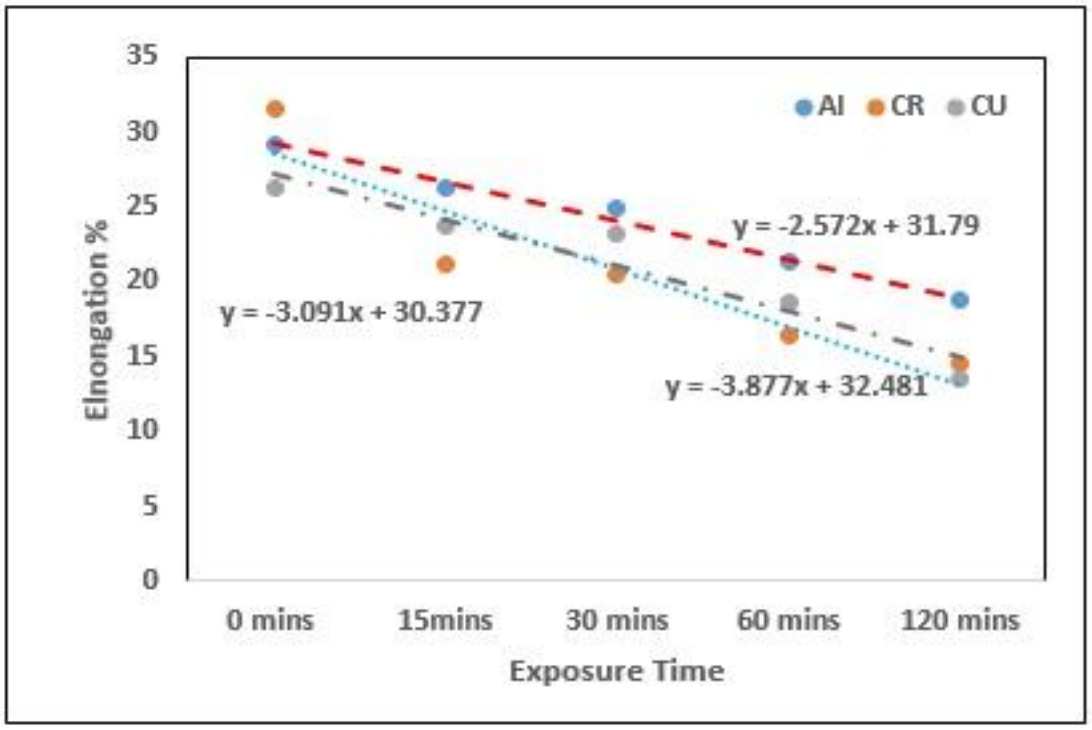


Fig. 5. Effect of exposure time on elongation of woolen fabric dyed with turmeric using various mordants.

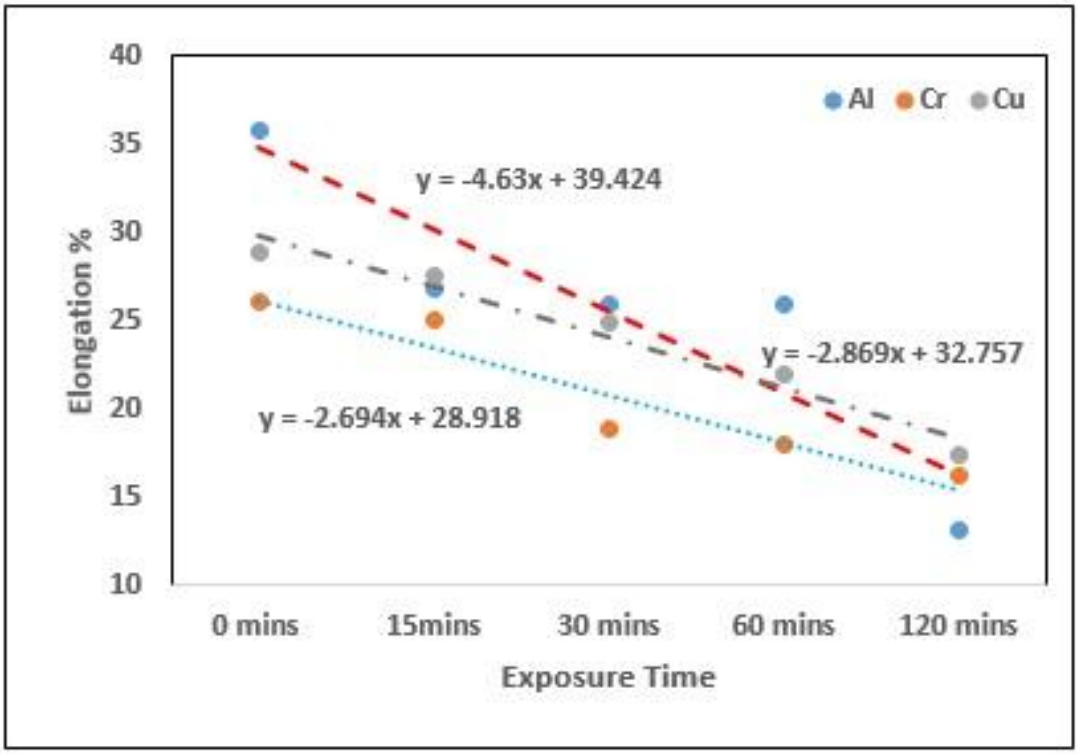


Fig. 6. Effect of exposure time on elongation of woolen fabric dyed with cochineal using different mordants.

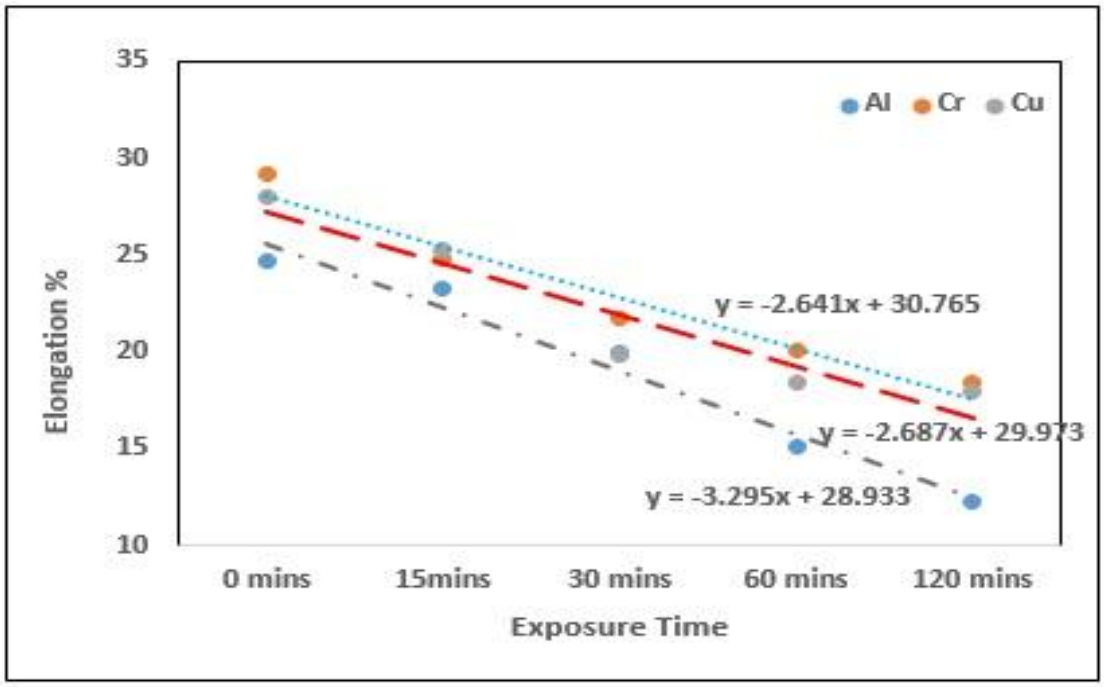


Fig. 7. Effect of exposure time on elongation of woolen fabric dyed with madder using various mordants.

Wool samples dyed with turmeric and fixed with Al showed the most significant color alteration. Cochineal and madder dyes showed less color change compared to turmeric. Al mordant accelerated turmeric dye degradation. Wool samples treated with Cr and Cu mordants showed smaller color changes compared to Al-treated samples. This suggests that Cr and Cu mordants reduce wool degradation. These findings support previous research on the protective effects of different mordants in preventing wool fiber degradation from UV radiation induced by ozone gas [15-16].

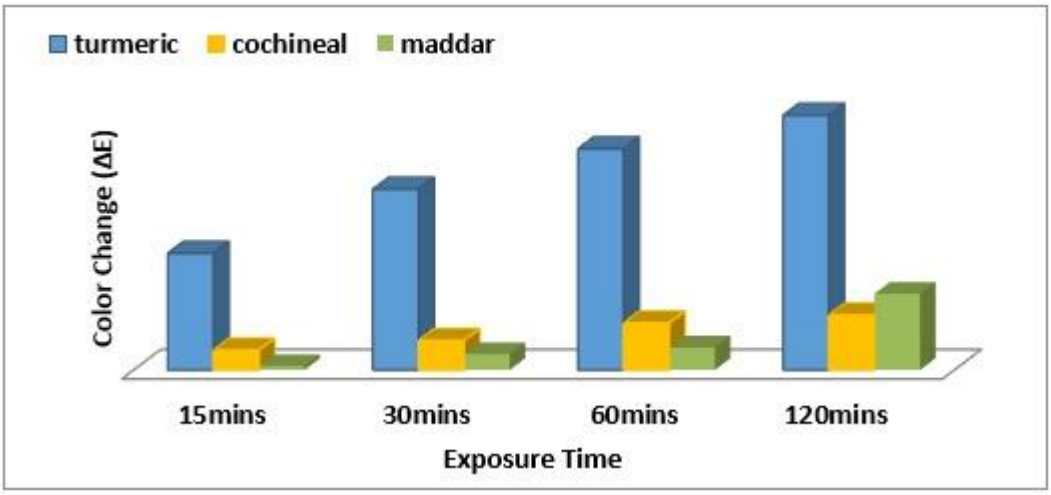


Fig. 8. ΔE of alum mordant dyed wool aged at different exposure periods.

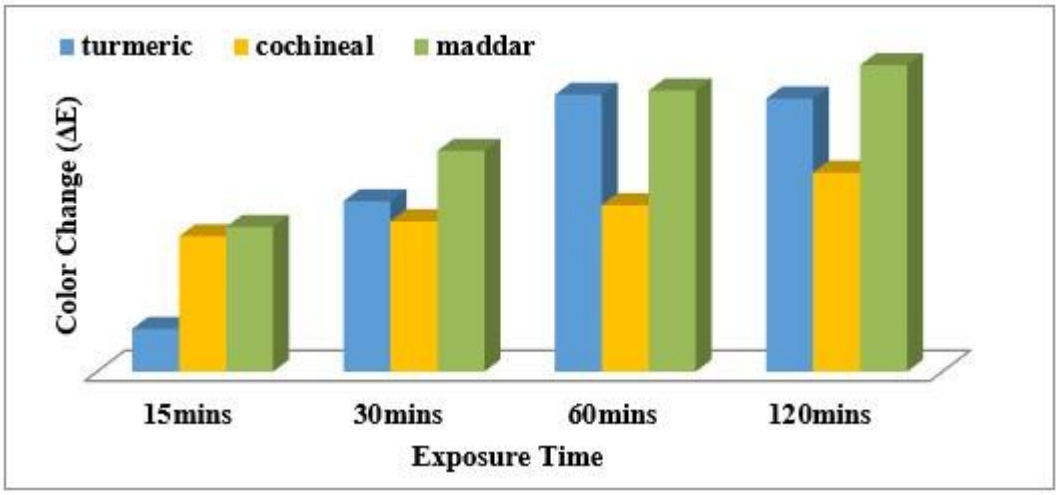


Fig. 9. ΔE of chrome mordant dyed wool aged at different exposure periods

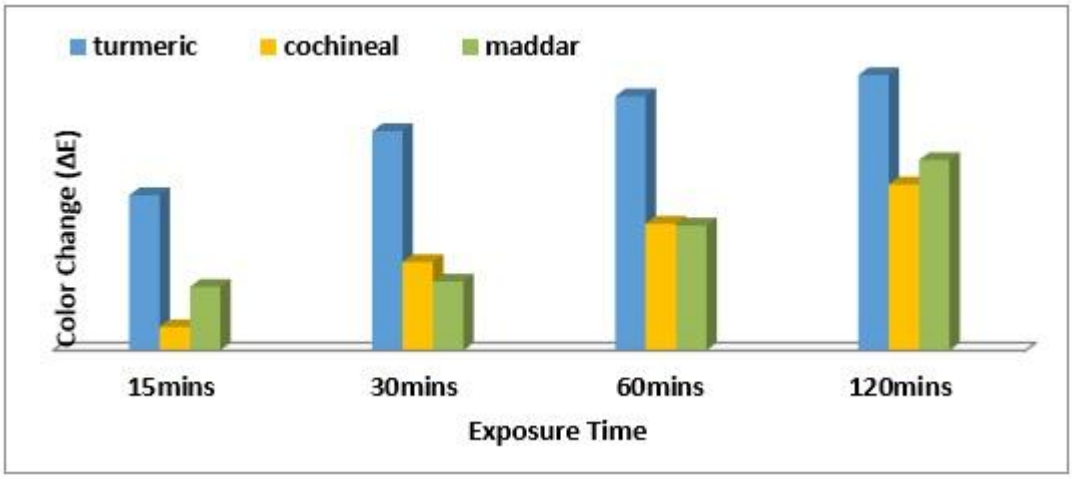


Fig. 10. ΔE of copper mordant dyed wool aged at different exposure periods.

3.3. Effect of aging on the morphology of textiles using SEM

Fig. 11 shows electron microscope images of uncolored wool samples before and after a 120-minute exposure to radiation. The images reveal a partial removal of scales on the wool fibers' surface and cracking in superficial areas. This indicates that UV radiation and ozone gas exposure cause surface effects and cracking [20].

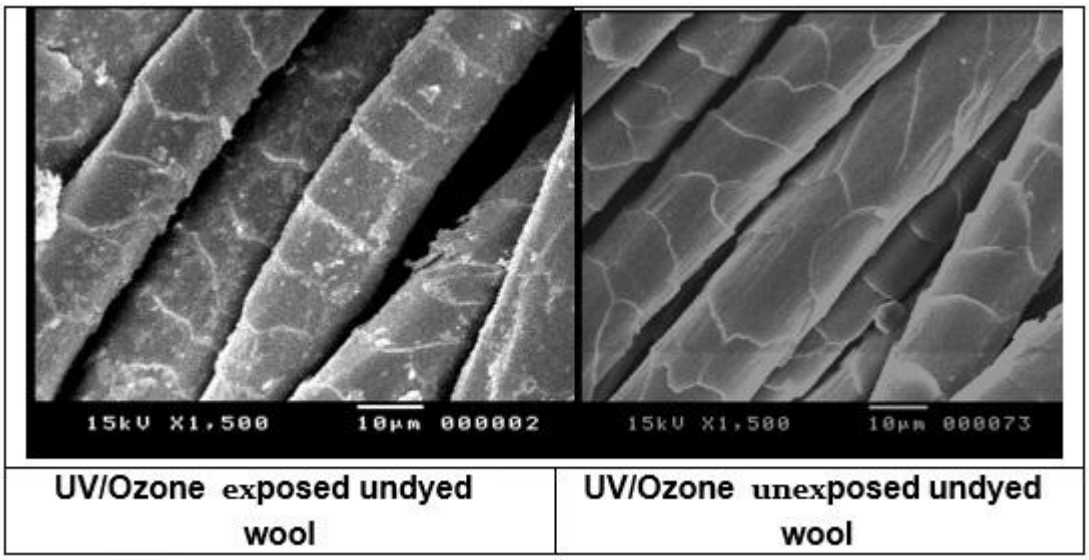


Fig. 11. SEM photomicrographs for unexposed and exposed woolen fabrics to UV/Ozone at 120 min.

3.4. FTIR analysis

The impact of ultraviolet radiation exposure linked to ozone on the active chemical groups of wool is illustrated in Fig. 12, comparing samples exposed to radiation with those that were not. In the spectrum ranging from wave number (400 to 4000) cm^{-1}, the distinct chemical groups of unexposed natural wool are evident. These include the amide group I at 1650 cm^{-1}, associated with the α-helical structure and C=O-stretch vibrations [13-21], amide group II at 1538 cm^{-1}, representing N-H bending and C-N-str. vibrations, and amide group III at 1230 cm^{-1}, indicating

a combination of C-N-str, N-H-bend, C-C-str, and C=O-bending vibrations.

The UV/Ozone-aged samples show noticeable changes in the Amide I and Amide II regions, as shown in Fig. 11. With aging, there is an increase in structure development in Amide I around 1670 cm^{-1} and 1720 cm^{-1}, indicating disorganized formations and potential fatty acid production through lipid oxidation in wool. Fatty acids with a carbonyl group can absorb infrared radiation in the range of 1715 to 1725 cm^{-1}. The decrease in Amide I band intensity is attributed to peptide and disulfide bond breakdown [22].

After 120 minutes of UV/Ozone exposure, a decrease in Amide II band absorbance at 1538 cm^{-1} is observed, along with a broadening of the Amide III peak towards shorter wave numbers. This broadening may result from changes in hydrogen bonding due to keratin protein denaturation, affecting the morphology of the Amide III peak. The appearance of a peak at 1170 cm^{-1} indicates the formation of ester bonds within amino acid residues, suggesting COOC stretching [23-24-25].

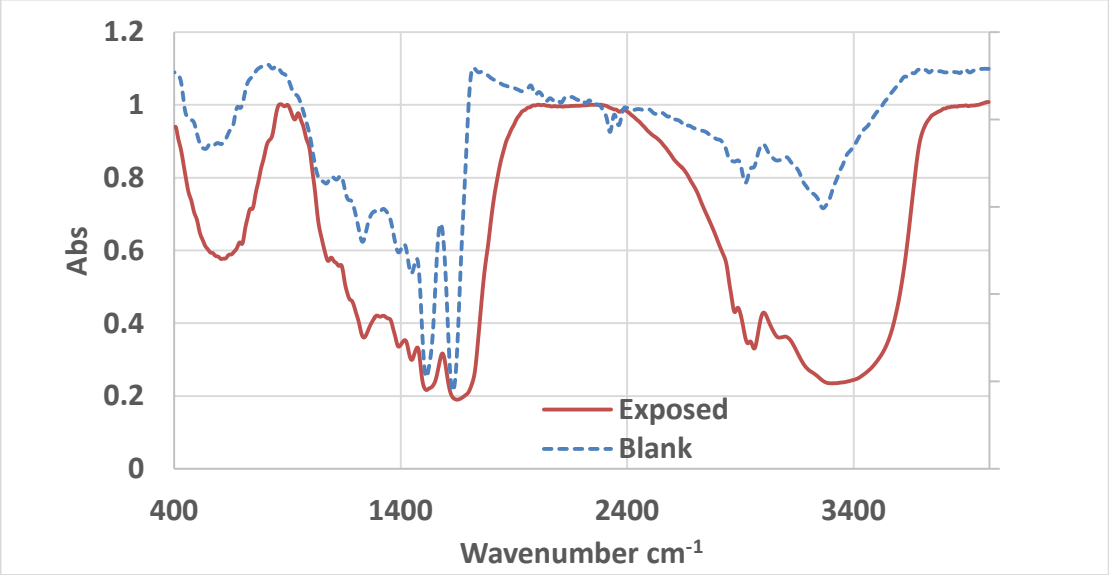


Fig. 12. FTIR analysis for unexposed and exposed wool fabrics to UV/Ozone for 120 minutes.

4. Conclusions

This research showed that wool is susceptible to UV/Ozone exposure. There is a rapid reduction in strength and a notable alteration in colour.

Through comparative analysis and validation of the impact of UV/Ozone treatments over varying durations on the physical characteristics of diverse dyed woollen fabric samples, as well as its efficacy as a novel method for expediting aging in a safe manner on wool samples, this study has showcased the practicality of producing simulated samples of archaeological artifacts that can be utilized in the field of conservation researches. The tensile strength of coloured wool materials diminishes progressively as UV/Ozone exposure is prolonged, reaching its peak after 60 minutes. These findings confirmed the observed reduction in mechanical strength after 10 minutes.

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