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EVALUATING NEW AGEING TECHNIQUE FOR ARCHAEOLOGICAL TEXTILE CONSERVATION FIELD USING RADIOFREQUENCY AIR AND OXYGEN PLASMA

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ABSTRACT

Artificial ageing is a type of testing that uses variables such as heat, humidity, light, acids, air pollutants, and other factors to mimic ageing in particular materials. This testing is an extremely significant stage in studying the behavior of archaeological and historical materials in different environments (e.g., during storage and display) and evaluating the suitability and efficiency of conservation treatments. In this research, the authors evaluate new ageing technique based on the use of Radio Frequency (RF) plasma, which is an eco-friendly method that requires a very short exposure time when compared with other conventional ageing techniques (i.e., thermal, chemical, and light ageing) which require prolonged exposure time. The results proved the effectiveness of this new ageing technique.

KEYWORDS: Plasma Aging, Thermal Aging, Tensile Strength, Colour, Mimic Samples, Archαeological Restoration, Wool, Contact Angel**.**

1. INTRODUCTION

Artificial ageing techniques are used in archaeological textile conservation field for various purposes, such as preparation of artificially deteriorated dyed textile samples with definite properties as well as evaluation of any new materials suggested to use in conservation of museum textiles (Abdel-Kareem, 2005, Abdel-Kareem, et al, 2015). Three different aging techniques e.g., light, thermal, and chemical were used for the wool fabric. Aged samples were examined for their surface morphology and colour parameters in accordance with Lab-CIE colour measuring system (Elnagar et al., 2013; Elnagar et al., 2005).

Radiofrequency (Rf) plasma was used in textile field in many applications Joanna Pawlat et al., (2016) studied the using RF atmospheric plasma jet to enhance the surface wettability of cellulosic materials with only small changes in morphological changes, the used plasma operated with various gas mixtures. El-Nagar et al., (2006) used Direct current voltage (DC) pseudo plasma discharge treatment of polyester textile surface for disperse dyeing to get good dyeability and UV protection. The surface treatment of polymer textiles using a plasma process has been studied for several years (Biederman, 2000). The pseudo-discharge is a low-pressure gas discharge located in the left branch of the Paschen curve. In this region, the mean free path for ionizing collisions of electrons is comparable or larger than the electrode separation (Urban and Frank, 2001). Zhang et al., (1999), established two electrodes consist of a cathode and a mesh anode to obtain pseudo-discharge. Pseudo-discharge can give excellent results in surface modification of materials and industrial components because of the high-power density concentration of its chemically active particles (mainly atomic oxygen, ozone, and activated nitrogen molecules) (Kropke et al., 2001). The plasma is almost as homogenous as a low-pressure glow discharge. The important parameters which characterize such discharges are the charged particle density, n (electrons or positive ions), and the electron temperature, T°c (or mean kinetic energy). On the other hand, the electrons which acquire their kinetic energy directly from the applied voltage collide with the gas molecules and thereby transfer energy through ionization, bond breakage (molecular fragmentation), and other forms of excitation.

Many authors have studied using the plasma on different materials (Krčma et al., 2014), studied plasma generated in liquids, and applied it to selected original archaeological glass materials. Madani and Dehkordi (2018) overviewed the application of plasma technology to protect cultural-historical objects and to remove corrosion curst on metals, particularly tarnished silver. Decontaminating wood and objects based on cellulose, like paper and cotton, and protein materials (leather, wool) is another application of cold plasma presented in the paper. (Bandt et al., 2000) worked on using the plasma for aging the protein. (De-Graaf et al., 1995) used cascaded arc plasma treatment to clean iron archaeological artifacts and the removal of the dirt crust of the excavated artifacts was greatly facilitated in the treatment. (Bhat et al., 2011) investigated the use of air plasma to modify the surface of cotton materials with dichlorodifluoromethane (DCFM) to improve dyeability with reactive and natural dye considering that air plasma increased the water wicking while DCDFM increased the water-repellence (Naebe et al., 2011). Studied the use of helium gas plasma for 28 days and suggested this technique to remove the lipid layer permanently. This research will introduce air and plasma as a new aging technique for woollen samples for only 60 minutes. Several surface modification methods are employed to modify the polymer surfaces, such as chemical treatments, thermal treatments, mechanical treatment, and electrical treatment under (a) atmospheric pressure plasma (Corona discharge) and (b) low-pressure plasma (glow discharge). Glow discharge under low-pressure plasma is a popular technique, which results in better uniformity in surface modification of the polymer. Moreover, it is a dry treatment method, which is better suited for industrial applications. It is now well established that the glow discharge creates physical and chemical changes such as cross linking, degradation, formation of free radicals, and oxygen functionalization. The temperature of gases in a glow discharge generally remains low and the plasma plays a predominant role in the surface modification of polymers (Bhomik et al., 2004). Since plasma nitriding is conventionally with DC glow discharge at lower pressure, the plasma contains many numbers of active species, which increase the nitriding efficiency (Fewell et al., 2000).

This study aims at evaluating the use of atmospheric and oxygen radiofrequency (Rf) plasma as a fast and eco-friendly aging technique to prepare artificially deteriorated wool textile samples with definite properties to be used as experimental samples in the evaluation and estimation of any new materials, techniques and methods used in conservation and restoration of deteriorated wool textiles before their application on ancient ones. Also, these artificial experimental samples can be used for the conservation of practical training of textile conservators. The performance of aging was characterized by studying mechanical properties, color changes, and hydrophilicity. The aged samples were compared with traditional thermal aging.

2. EXPERIMENTALS

2.1 Materials

The materials used in the research are the following: pure wool fabrics 100% (weight) supplied by Mahalla Co., Wool fabric was scoured and purified by a solution containing 2g/L of nonionic detergent at a liquor ratio 1:50 at 40^{o}C for 15minutes. The samples were thoroughly washed with water and then dried in ambient.

2.2 Accelerated thermal aging

Accelerated heat aging at 90°C for 5 days was applied to the studied samples according to Abdel-Kareem and Nasr, 2010, Abdel-Nasser et al. 2022, with some modifications to increase the stain's stability on the surface. The thermal aging was applied using (Heraeus D.63450 Hanau, Type: VT 6130M), performed in the laboratory inside the reaction oven in a dry atmosphere

2.3 Plasma Aging

The pre-washed samples were exposed to RF plasma discharge treatments oxygen and air, at different times (5, 15, 30 and 60 min). The schematic diagram in Figure 1 shows the experimental set-up of exposing samples surface to oxygen plasma or air gases. Plasma is shown in. Fig. 1, the plasma is created by using high frequency voltages (typically kHz to >MHz) to ionize the low-pressure gas, A radio frequency (RF) oscillating electric field is generated in the gas region through magnetic induction. At sufficiently low pressures, the combined effect of the electric field acceleration of electrons and elastic scattering of the electrons with neutral atoms or field lines leads to heating of the electrons. When electrons gain kinetic energy in excess of the first ionization threshold in the neutral gas species, electron-neutral collisions lead to further ionization, yielding additional free electrons that are heated in turn.

The potential difference is generated by a helical coil of metal surrounding the glass processing chamber V. The discharged gases are pumped to have pressure of 300 m torr before filling in with the oxygen or air mixed gases. The working pressure is controlled by needle valve. The cathode is connected to the negative potential terminal of the power supply, whereas the anode was ground (Elnagar et al. 2005; Essa et al., 2019, Ibrahim et al., 2021).

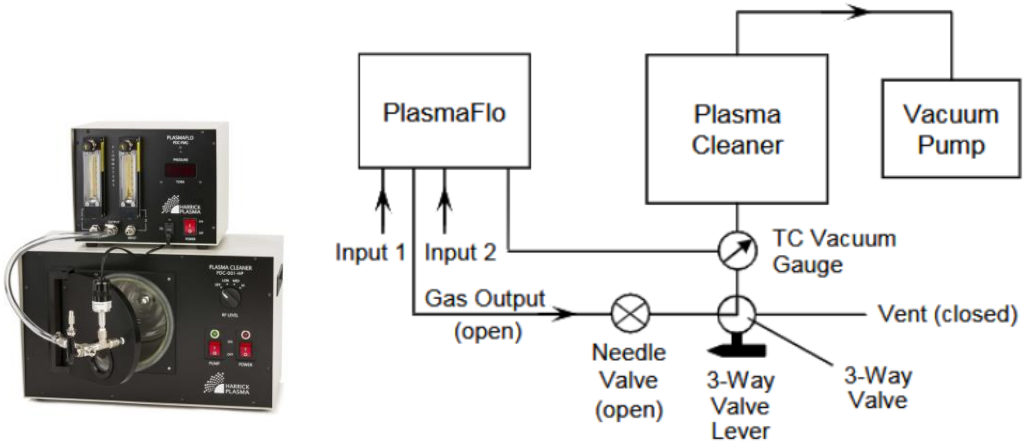


Figure 1. Schematic diagram for Air/ Oxygen Cold Plasma system

2.4 Testing and Analysis

2.4.1 Color Measurement

Effect of aging using air or oxygen plasma through changes in the colors of the samples as a result of exposure at different periods. The CIE Lab color components are denoted ('L' for brightness, 'a' for red-green, 'b' for yellow-green), while the whiteness index is denoted by 'W', and 'Y'. denotes the yellowness index. It was measured using a Macbeth double beam spectrophotometer (SDL-UK) attached with integrating sphere. All measurements were compared to the standard white tile that has traceability to the SI measurement system. The spectrophotometer used in this measurements was calibrated for its photometric and wavelength scales (ASTM standards 2001). The samples were preconditioned before testing at standard environmental conditions of temperature (20±2°C) and relative humidity (65±5%) using a standard conditioning room (SDL-UK 1998).

2.4.2 Contact Angle

Contact angle of the treated fabrics samples was determined by Compact video microscope (CVM) that is manufactured by SDL-UK, contact angle measured by horizontal plate camera perpendicular to liquid droplet plane (Abdel-Maksoud et al 2022; El-Bisi et al., 2016).

2.4.3 Mechanical Behaviour

All treated and untreated samples of polyester and silk were tested for their tensile strength and elongation behaviors using a Shimadzu Universal Tester of (C.R.T) type S-500, Japan. The results listed in this paper are the mean of five times measurements (Ibrahim et al. 2012).

2.4.4 Fourier transform infrared spectroscopic analysis (FTIR)

The IR spectra of the treated and untreated samples were measured by using Nicolet 380 (FTIR) Spectrometer, USA.

2.4.5 Statistical Analysis

All results presented in this paper are the average of three individual results. Regarding the mechanical properties, the measurement uncertainty was evaluated using coverage factor to give 95% confidence level (for both the maximum force and elongation %).

3. RESULTS AND DISCUSSIONS

3.1 Effect of aging with Air and Oxygen cold plasma on the tensile strength and elongation of the wool fabric.

The results in Table (1) show that the value of the tensile strength of the samples exposed to the air plasma increases significantly from 271 Newton at the exposure time of 5 minutes to 324 Newton at 60 minutes; however, it decreases when using oxygen plasma. This increase in air plasma may be due to the fixation of atmospheric nitrogen in wool samples, which increases cross bonds. In contrast, the effect of oxygen plasma is limited by the oxidation and cracking of chemical bonds in wool fibers.

Table 1. Effect of aging with Air and Oxygen cold plasma on the tensile strength and elongation of the wool fabric. Expanded uncertainty of tensile strength was calculated with coverage factor =2 confidence level 95% =5.7%, Expanded uncertainty of Elongation % was calculated with coverage factor =2 confidence level 95% =10.1%

Samples	Maximum Force (N)	Elongation (%)
Blank	280.0	44.45
Air 5 min	271.6	36.70
Air15 min	296.0	40.15
Air 30 min	317.6	47.30
Air 60 min	324.8	43.55
Oxygen 5 min	304.0	41.45
Oxygen 15 min	303.2	35.45
Oxygen 30 min	294.0	38.60
Oxygen 60 min	288.8	25.68
Thermal at 90^{o}C for 5 days	265.7	29.23

By studying the change in elongation, it is clear that the elongation decreases significantly at the beginning of exposure to air plasma. Then, it increases gradually with increasing exposure time up to 30 minutes and then decreases relatively at 60 minutes. However, the change in elongation behavior when exposed to oxygen plasma differs from the values of unexposed samples. The amplitude decreases with increasing exposure time from 41.45 at 5 minutes to 25 at 60 minutes exposure time.

The effect mentioned above of air and oxygen plasma can be attributed to using the air plasm, which contains about 78% N_2 in addition to 20.95%of Oxygen, 0.93% of Ar, 0.04of CO_2, 0.4% of water vapor, and a small number of other gases (Karki et al., 2021). Plasma is an ionized gas, and Nitrogen is the major constituent that causes crosslinking more than the degradative oxidation by oxygen species in addition to the azides on the surface (El-Nagar et al., 2006). Therefore, compared with Oxygen, plasma produces more oxidizing species that degrade the wool fibers. Tensile strength results showed no significant changes, but elongation percent showed that the samples loosed about 43% (in 60 minutes) compared to 34% when using normal thermal aging (at temperature of 95^{o}C for 5 days)

3.2 Effect of Aging with Air and Oxygen cold plasma on the color properties.

Table 2 shows the results of colorimetric measurements of samples exposed to air and oxygen plasma at times from 5 to 60 minutes. The results showed that by increasing the exposure time to air plasma, there was no significant change in L values; however, it increased from 83.41 to 84.13 with increasing exposure time to oxygen plasma. Moreover, when studying the values of a, it was found that the samples decreased in green colour with increasing exposure to oxygen plasma. The green colour component (-a) increases with increasing exposure time to oxygen plasma during the same period. By studying the change in the colour compound b, it becomes clear that the yellow colour component (+b) increases with increased exposure time. It was also found from the results shown in the table that the degree of whiteness W decreases significantly with increasing exposure time to both air and oxygen gas plasma. The degree of yolk increases from 22.78 to 24.08 by increasing the exposure time to air plasma, while it increases from 23.20 to 23.92 by increasing the exposure time to oxygen plasma. Furthermore, by comparing the results of exposure to air plasma and oxygen plasma with conventional thermal ageing, it was found that thermal ageing causes a greater decrease in the degree of whiteness and the degree of yolk compared to the untreated sample.

The reflection spectra (in the wavelength range 400-700 nm) and CIELAB colour components expressed as red-green component (a) and yellow-blue component (b) of the material surface were studied. When light incidents on the material's surface, it is either reflected from the first layer of the surface or enters the material and scattered internally to be either absorbed or reemerged as diffusely reflected light. If the material particles have colour, only certain wavelength(s) will reemerge, giving a specific colour to the viewer (Maupan et al 2004; Nour et al. 2010).

Table 2. Effect of Aging with Air and Oxygen (O2) cold plasma on the color components (L, a &b), whiteness (W) and yellowness (Y) indices.

Samples	L	a	b	W	Y
Blank	84.23	0.17	11.31	7.68	23.46
Air 5 min	83.44	-0.18	11.31	7.12	22.78
Air15 min	83.80	-0.18	11.38	6.27	23.52
Air 30 min	83.49	-0.17	11.36	5.57	23.42
Air 60 min	83.71	- 0.11	11.71	4.36	24.08
O_2, 5 min	83.41	- 0.11	11.47	4.79	23.20
O_2, 15 min	84.07	- 0.17	11.48	6.90	23.24
O_2, 30 min	84.16	- 0.18	11.51	6.46	23.55
O_2, 60 min	84.13	- 0.21	11.71	5.37	23.92
Thermal at 90^{o}C for 5 days	82.58	-0.11	16.20	-21.32	32.71

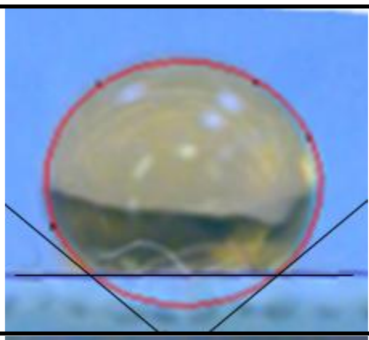
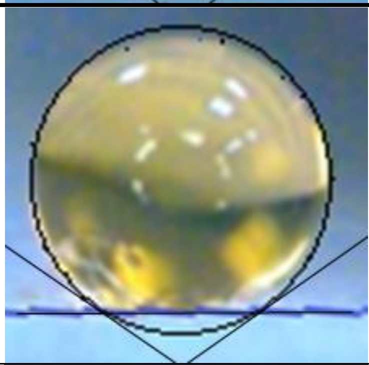
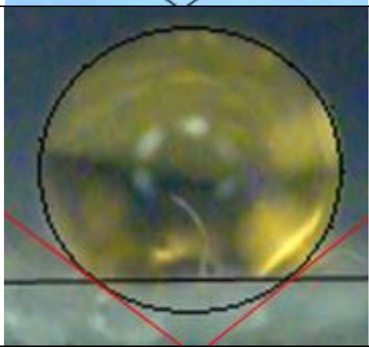
Color components used in this table: Lightness (L); red-green (a); Yellow blue (b). Whiteness index (W) and Yellowness index (Y).

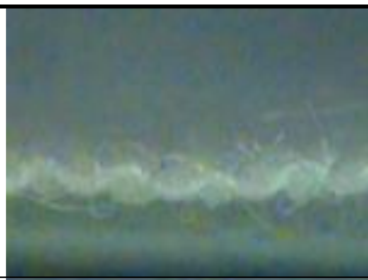
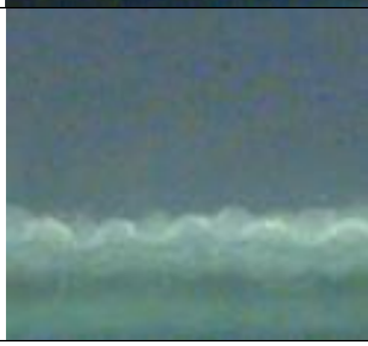
It is known that the raw wool samples are creamy white, and Table (2) indicates that L is very close to the white (L=84.23, W=7.68, and Y=23.46). When wool is exposed to air plasma, the degree of whiteness and brightness is not affected until 5 minutes, then the degree of whiteness decreases with increasing exposure with the relative stability of the red and blue color compounds. Light reflection and scattering on the substrate surface strongly affect the measured coloristic properties. The exposure to Air plasma showed a significant change decrease in the Whiteness index by 43% and 30% for air and plasma, respectively (for 60 minutes only), while the conventional thermal aging caused a high reduction in the whiteness **index** and increased yellowness, These can be attributed to that plasma result in bleaching action (more white and lower yellowness).

3.3 Effect of concentration Aging conditions on the contact angle of aged wool samples.

From Table 3 and Fig.2, by studying the effect of exposure to air plasma from 15 to 60 minutes, it was found that the water contact angle on the wool surface for untreated samples is 145 degrees, and this angle decreases with increasing exposure time (the lower the degree of contact, the greater the water absorption). Oxygen plasma exposure time turned wool samples into water-absorbent compared to the untreated samples. Natural protein wool fiber contains a large number of polar groups in its polypeptide chains (Tian et al., 2020) and exhibits great moisture-absorbing properties. But wool has a hydrophobic surface due to the existence of its special scale structure, which is composed of a fatty layer of 18-methyleicosanoic acid covalently bound to the protein layer of the wool cuticle via a thioester linkage (Hassan et al., 2018; Chen et al., 2010).

Table 3. Effect of Aging at different times (5-60 min) by oxygen and air RF plasma on the contact angle of wool samples.

Samples	Contact Angle	
Blank	140° (Hydrophobic)	
Air 5 min	145°	
Air15 min	142°	
Air 30 min	60° (partially hydrophilic)	
Air 60 min	0° Absorbed	

Oxygen 5 min	0º Absorbed	
Oxygen 15 min	0º Absorbed	
Oxygen 30 min	0º Absorbed	
Oxygen 60 min	0º Absorbed	

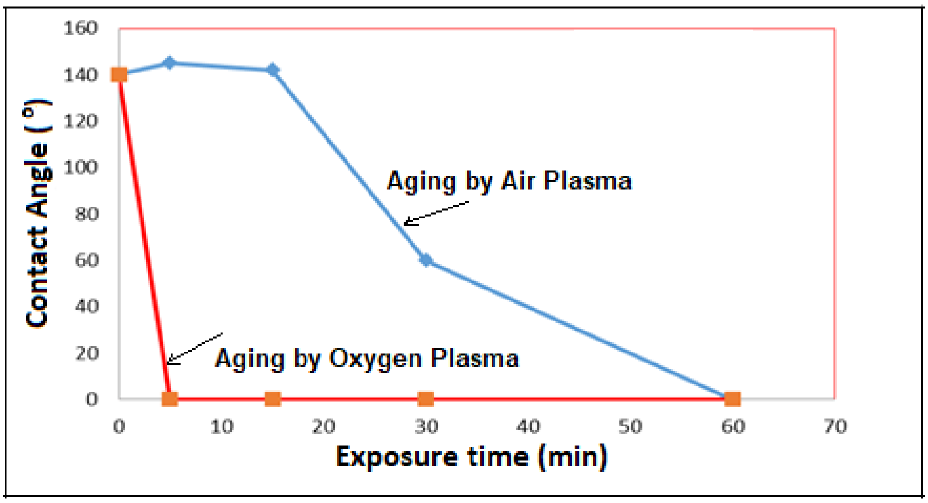


Figure 2. Water contact angle on wool surface treated with air (blue line) and oxygen plasma (red line) at different exposure time (5-60 min).

It is clear in the photos in Table 3 in the table that the hairiness on the surface is removed by increasing the exposure time to the oxygen plasma as a result of the severe oxidation effect caused by the free oxygen species, which contact the water droplet on the surface of the fibers more by increasing the exposure time. In addition, oxygen plasma breaks down the greases on the sample's surface and breaks the cysteine bonds, which greatly increases the degree of water absorption and hydrophilicity for the woolen samples under study (it is worth mentioning the efforts made in conservation to seek appropriate techniques and materials for the hydrophilicity, either desired or unwanted, in material culture objects, see Manoudis et al., 2017).

3.4 FTIR-Spectroscopy of aged wool samples exposed to Rf plasma

Figures (3-5) show typical absorption peaks for wool treated samples. The characteristic peaks of wool are at 3285 cm^{-1} (NH and OH), 2925 cm-1 (CH), 1624−1640 cm^{-1} attributing to amide I (carboxyl stretching), 1230−1240 cm^{-1} corresponding to amide III (stretching CC and bending C=O), and at 1510−1520 cm^{-1} corresponding to amide II (NH bending and CN stretching), (Tian et al., 2020). As shown in Figures 3-5. By increasing the exposure time to Air plasma the amide III and NH peaks increased due to the increment of the azide group from nitrogen in the air and action of free radicals generated by the RF plasma. CO group was increased with Oxygen plasma due to the oxidative action on the wool structure (El-Nagar et al., 2016).

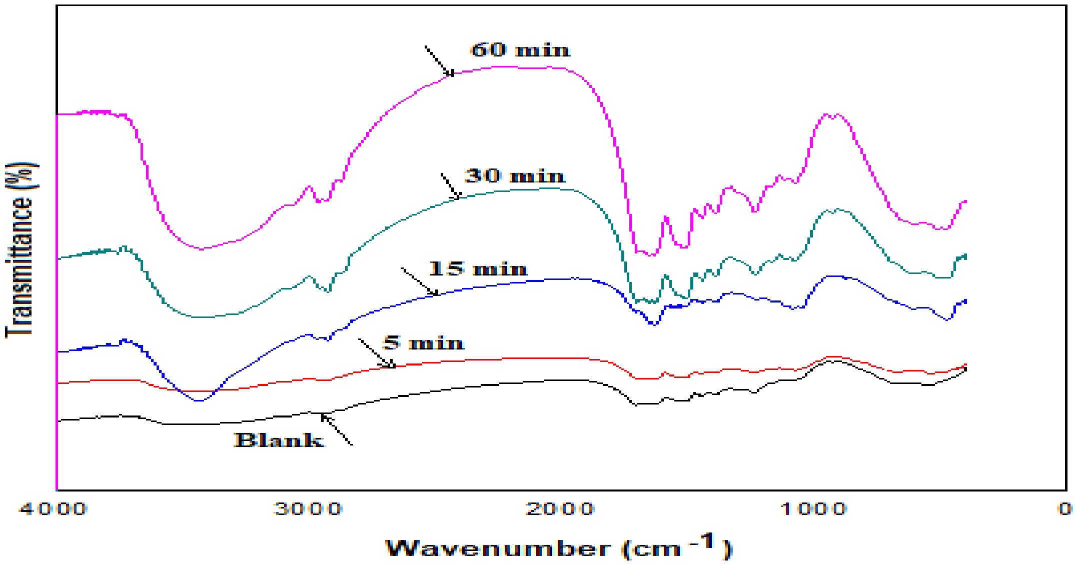


Figure 3. FTIR Diagram of blank wool fabric and exposed samples to air Rf plasma for different times (5-60 min).

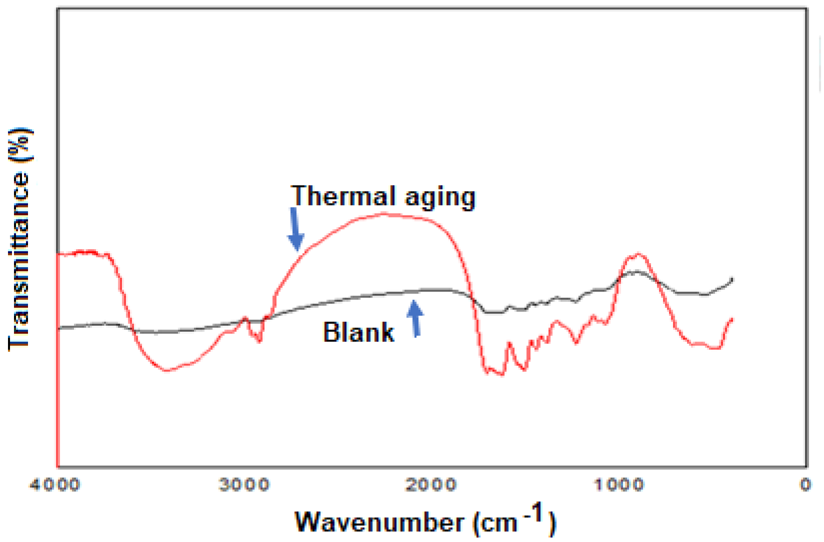


Figure 4. FTIR Diagram of blank wool fabric and thermal treated sample

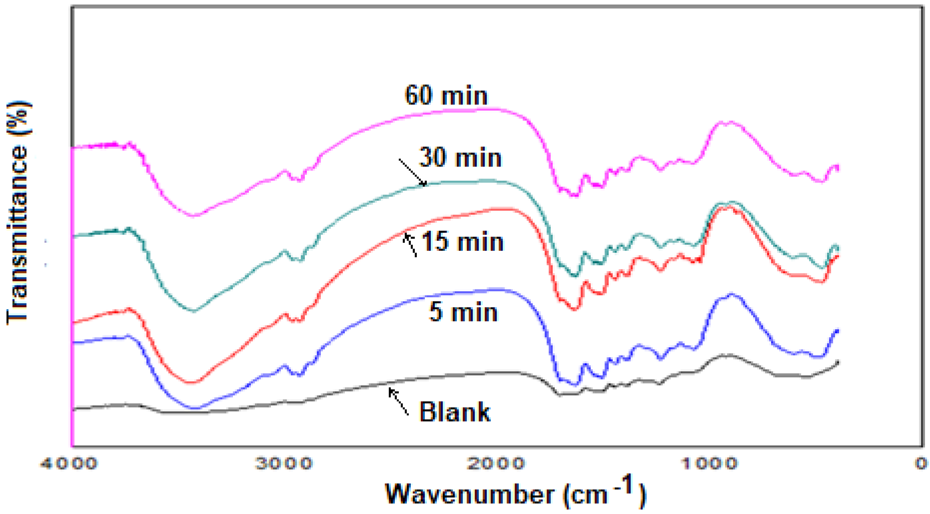


Figure 5. FTIR Diagram of blank wool fabric and exposed samples to oxygen Rf plasma for different times (2-60 min)

4. CONCLUSION

This research presents the eco-friendly plasma technique as an effective and fast method for preparing aging samples that can be used in antiquities conservation. The effectiveness of this new technique was studied by evaluating the effect of aging times that ranged from 0 to 60 minutes for air plasma and oxygen plasma on each of the mechanical properties. Tensile strength results showed no significant changes, but elongation percent showed that the samples loosed about 43% (in 60 minutes) compared to 34% when using normal thermal aging (at a temperature of 95oC for five days). CIE color components showed significant changes in the whiteness index (W) and yellowness (Y). The water contact angle shows a significant change in the degree of hydrophilicity. Samples's hydrophilicity changed by exposing them to air plasm after 60 minutes to partially hydrophilic, while changed to completely hydrophilic by oxygen after only 5 minutes. FTIR absorption peaks showed significant changes in the OH, CO, and S-S groups, indicating the degradation in the studied woolen samples. The results were compared with conventional thermal aging at 90°C for five consecutive days. The results proved that using Oxygen plasma as an aging tool is helpful for archeologists in preparation the mimic samples in a very short time with low energy consumption.

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