

FAST PRODUCTION OF ARTIFICIAL MIMIC TEXTILE SAMPLES USING UV/OZONE TREATMENT APPLICATION IN CONSERVATION AND CONSOLIDATION

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Abstract

The present work deals with evaluating the possibility and the efficiency of applying UV/ozone treatment as a tool for aging three natural fabrics namely; silk, wool and cotton using different exposure times. The work is extended to compare this method with two other techniques of aging by acid hydrolysis and thermal aging taken cotton samples as an example of cellulosic fabrics. The produced aging samples were followed through evaluation of the change in the overall samples surface morphology via x-ray diffraction, tensile strength and air permeability. The obtained results showed that UV/ozone technique can be used successfully as an accelerating technique and applied safely especially for silk and wool samples. Finally, the application of UV/ozone treatment is very useful and practical for the production of mimic archaeological samples that can be applied in conservation and restoration work. Never the less, it is an easy and accelerated method, time saving, economic and environmental friendly technique.

Keywords: *Chemical Aging; Thermal Aging; UV/Ozone Treatment; -Tensile Strength; Air Permeability; X-ray Diffraction..*

Introduction

All textiles are deteriorated by light, insects, microorganisms, and air pollution which, alone or together, cause considerable loss of tensile strength and pliability. The oxygen in the atmosphere affects all organic substances to varying degrees. Prolonged exposure to normal atmospheric conditions will cause textiles to weaken and disintegrate. The speed of the deterioration varies according to environment and the nature of the fibers. The main factors that promote the decay of textiles can be categorized into three groups [1]: a) Organic: because textiles are organic, they are subject to attack by molds and bacteria. Decomposition is greatest in situations that favor the growth of these organisms, such as damp heat, and contact of the material with vegetable matter. Attack by destructive insects may also be encountered. b) Physical: excessive heat causes desiccation and embrittlement; exposure to ultra-violet light causes a type of deterioration known as 'tendering,' as well as the photochemical degradation of susceptible dyes. c) Chemical: exposure to noxious gases can also cause tendering. In some cases, these gases are converted to acids, which are the main cause for the deterioration of some

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textiles. Thus, the damage of the fabric fibers can be divided into physical and chemical damage, including light, heat, chemical, biological, mechanical damage and other factors [2].

Natural fibers are aged in the process of wearing, washing, drying as the influence of the climate. After the fabric is aged, the strength decreases, and then the appearance and the quality are changed, and they will affect the fabric wearing comfort [3].

Cultural heritage is unique and irreplaceable and this makes the conservation and restoration of museums collections an important issue and places the responsibility of preservation on the museum's conservators and a lot of scientific work was done in this field of research [4-6]. The degradation of textile art work in museums is a result of being exposed to human intervention, but those affecting textiles, but above all, environmental variations. Many factors contribute to textiles degradations [7]. But those affecting textiles collections the most are: light in the visible and UV ranges, temperature, relative humidity and pollutants [8]. Conservation of textile artifacts go through many processes including cleaning stains removal and strength caring. Since artifacts must not be used as a field of research or for trials because any mistake will cause damage to the art work and they will be lost. So, artificial mimic archaeological textile samples are very useful to conservators [9] as they can apply them in the preliminary conservation processes and when they succeed in that trials they can be applied on the artifacts safety and surely [1, 2, 8].

Many scholars summarized the efficiency of the different methods used to accelerate the natural fiber like silk, cotton and wool aging by thermal, light aging and chemical acid hydrolysis aging methods [10]. The influence of UV on wool, silk fabric strength is larger. It indicates that protein fiber is easy to be damaged by ultraviolet radiation, especially for silk fabric. After UV aging treatment, not only the strength decreases rapidly, but there is also a significant change in color [11, 12].

The main goal of this work is to represent UV/ozone technique using different exposure times as an accelerated method for aging silk, wool and cotton fabric samples to be used in the conservation field as alternatives to the archaeological ones. Also, the work is extended to compare this method with two other techniques of aging by acid hydrolysis and thermal aging taken cotton samples as an example of cellulosic fabrics. The aged samples were analyzed by testing surface morphology via X-ray diffraction, tensile strength and air permeability.

Experimental

Materials

The materials used in the research are the following: pure wool fabrics 100% (weight 176,71g/m²) supplied by Golden-Tex Co., Egypt; pure silk fabrics 100% (weight 100g/m²) supplied by El-Khateeb Factory, Akhmeem, Egypt. Wool and silk fabrics were scoured and purified by a solution containing 2g/L of nonionic detergent at a liquor ratio 1:50 at 40°C for 15 minutes. The samples were thoroughly washed with water and then dried in ambient conditions [13]. Also pure cotton fabrics 100% of 150g/m² was kindly supplied by El-Shorbagy Company treated with a solution containing, 0.5g/L Egyptol non ionic detergent and 0.5g/L Na₂CO₃ at a temperature of 70°C for 1h, thoroughly washed and air dried at room temperature.

UV/Ozone Treatment

A high intensity, low-pressure mercury lamp without an outer envelope (LRF 02971, 200watt, 220volt, made in Poland) was placed in a cubic box 60cm in length. Strips of the samples were hanged around the source at a distance (~ 20cm) for periods of 5, 10, 15, 20, 40 and 60 minutes. The Theoretical basis for ozone production via UV radiation was clearly discussed before in published work [14-17].

Chemical Aging for Cotton by Acid Hydrolysis [6]

Three groups of cotton samples were immersed in three concentrations of sulfuric acid (10, 20, and 30%) with liquor ratio (1:50) for different time periods (2, 5, and 9 hours). Then the samples were removed from the acid solution and rinsed with water and were left for drying at room temperature then were put samples in a conditional cabinet at $23 \pm 2^\circ\text{C}$ and humidity $65 \pm 5\%$ for 24 hours before testing.

Thermal Aging for Cotton

The tested samples were exposed inside an oven with an upper hole for entrance of atmospheric oxygen to oxidize the samples at different temperatures (150, 160 and 170°C) for different exposure times (1, 2, 5.5, 7.5 hours) then they were put in a conditional cabinet at $23 \pm 2^\circ\text{C}$ and humidity $65 \pm 5\%$ for 24 hours before testing [6].

X-Ray Diffraction Studies on the UV/Ozone Treated Cotton Fabrics

The change in the overall shape of the UV/ozone treated cotton samples was tested by X-ray diffraction analysis using X-ray diffractometer (Pert Pro. PAN analytical with graphite monochromatic Cu X-ray source).

Tensile Strength Measurements

Tensile strength of untreated and treated samples for the different periods were measured and evaluated using a Shimadzu Universal Tester of (C.R.T) - type S-500 Japan. The measurements were carried out according to - Standard Method [18].

Air Permeability Measurement

All of the examined fabric samples were subjected to air permeability measurement according to the ASTM Standard Method [19] by SDL-UK Air Permeability Tester.

Results and Discussion

Effect of UV/Ozone Treatment on the Physical Properties of the Fabric Samples

In this part we will study the effect of UV/ozone treatment on the physical properties of the examined fabric samples.

Tensile Strength

The effect of UV/ozone treatment for different periods on the tensile strength values of the different treated fabrics is represented in figure 1. Wool samples showed a considerable decrease tensile strength values after five minutes reached a loss of (23.53%) followed by a continuous gradual decrease till the end of exposed periods recording a loss of (41.17%), While for silk samples, the decrement in tensile strength values was a gradual slow during the whole experiment periods reached a loss of (52.6%) at the end. This decrease in the tensile strength values of the exposed fabrics is due to the effect of UV/ozone irradiation in reducing elasticity and also the average molecular chain of the treated fabrics, which may be a result of photo-oxidation [15]. Also, these results imply that protein fabric samples had lost part of their strength due to the UV/ozone treatment as the treatment might cause a removal of surface layer of wool protein. But, for cotton fabrics, it was observed that the tensile strength values showed a gradual decrease up to 10 minutes recording a loss of (18.15%), and it increased again reaching the tensile strength of the unexposed samples approximately. Such decrease may be explained in view of photo-oxidation effect caused by the UV/ozone as stated before, while prolonging exposure time lead to a thermal effect on the exposed fabrics which may cause solidification of these fabrics resulting in an increasing their tensile strength again [20, 21].

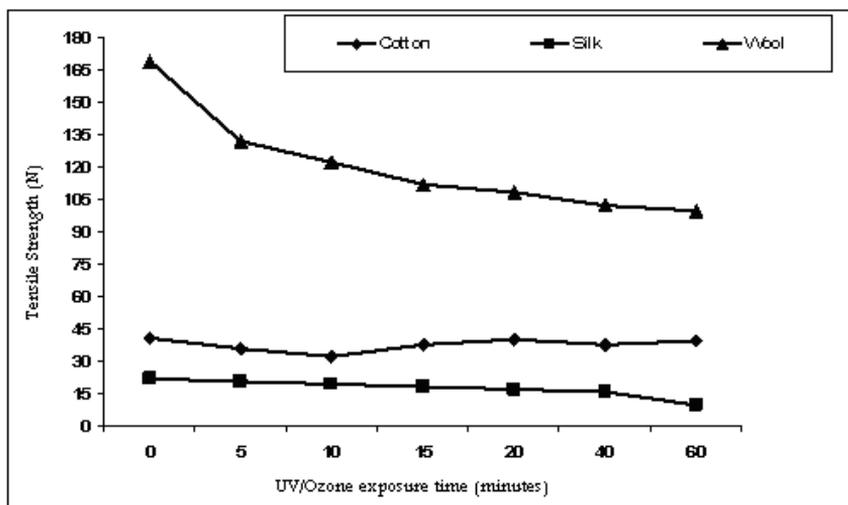


Fig. 1. Effect of UV/ozone treatment on tensile strength of different examined fabrics.

Air Permeability

The results recorded in Table 1 show the effect of UV/ozone on the air permeability values of the different examined fabrics. Generally, it is clear that the air permeability values increase appreciably at the beginning of exposure time for all of the examined fabrics. These results verify the effect of UV/ozone exposure on weakening of the irradiated samples i.e., their ability to pass air through their structure increasing their air permeability values [22].

Both cotton and wool fabrics, showed a continuous gradual increase in the air permeability reaching maximum value at the end of exposure. In case of wool fabrics this result due to the surface oxidation occurred on the exposed wool surface that creating polar groups and modifying or volatilizing surface lipids and the photo-oxidation that mainly modifies C_{21} fatty acid components of wool surface lipids and oxidizes disulphide [23]. But, for cotton fabrics, the increase in their air permeability values with UV/ozone can be explained on view of the previous work [24] which showed that, this exposure leads to the increase in the polarity of the cotton cellulosic by increasing the peak intensity values of hydroxyl OH-stretching group which are responsible for the polarity of cotton fabrics and the increase in their intensity with the increase in exposure time resulting in an increase in polarity [25].

In case of silk fabric samples, there is an increase in the air permeability values at the beginning time exposure period (5-15) minutes, and then it decreased considerably. Such decrease may be explained by the photo-oxidation effect caused by the UV/ozone as stated before. The continuous exposure time may lead to a thermal effect of the exposed fabric which may cause solidification of the silk fabrics resulting in an increase of the air permeability values again [20, 21].

Table 1. Effect of UV/ozone irradiation on air- permeability of cotton, wool and silk fabrics

Time of Exposure (minutes)	Air-Permeability ($\text{cm}^3/\text{cm}^2/\text{S}$)		
	Cotton	Wool	Silk
(0) Blank	17.12	15.1	47.3
5	19.4	15.3	48.63
10	20.6	15.6	58.7
15	19.5	16.0	57.5
20	22.36	16.6	53.9
40	22.9	17.5	47.3
60	21.3	19.8	41.13

Comparison between UV/Ozone Treatment and other Aging Techniques

In this part we make a comparison between UV/ozone treatment and other aging techniques on cotton fabric.

Thermal Aging of Cotton Samples

When heating the samples at (150, 160 and 170°C) a noticeable change in the color of the tested samples, to dark beige by increasing the exposure time (1, 2, 5.5 and 7.5 hours). Also, the tested samples becomes more solid, rough and brittle as a consequence off loss of part of the moisture content [6], that was related to the oxidation of cotton. The results in the Table 2 show that the rate of loss in the tensile strength increases by increasing exposure time and temperatures which was attributed to the fact, the amount of thermal energy absorbed. This amount of energy was enough to break the links between the atoms making up the molecules of cellulose and leading to a decrease in tensile strength.

Table 2. Percent loss in tensile strength and elongation of thermally aged cotton fabric.

Time (hours)	% loss in tensile strength			% loss in Elongation		
	150°C	160°C	170°C	150°C	160°C	170°C
1.0	13	18	23	12	18	20
2.0	21	25	39	14	20	23
5.5	24	37	50	24	28	30
7.5	28	51	68	30	33	40

Chemical Aging of Cotton Samples by Acid Hydrolysis

After examining the samples treated with a solution of sulfuric acid at concentrations (10, 20 and 30%) respectively and times (2, 5 and 9 hours) it was observed that the samples had become whiter and lost part of their strength. The data presented in Table 3 showed that increasing the acid concentration and prolonging the immersion times was accompanied by a decrease in the tensile strength values and elongation i.e. the % loss in tensile strength and elongation increased. This may be due to the fact that part of cellulose fabrics had dissolved in the acid solution and also a decomposition of the cellulose chain to shorter units had occurred by acid hydrolysis. Cotton samples had lost (69%) of their strength and (31%) of their elongation after immersion in (30%) concentrated solution of sulfuric acid for 9 hours [6].

Table 3. Percent loss in tensile strength and elongation of chemically aged cotton fabric.

Immersion Time (hours)	Acid Concentration (%)	% loss in tensile strength			% loss in Elongation		
		10 %	20%	30%	10%	20%	30%
2.0		17	28	41	12	15	20
5.0		25	39	55	17	20	28
9.0		36	49	69	22	26	31

X-ray Spectroscopy of Different UV/Ozone Treated Cotton Fabric

This measurement was taken as a verification of the tensile strength behavior of the different UV/ozone treated cotton fabrics. Figure 2 shows the X-ray diffractograms of the different examined cotton fabrics. On considering the major band that characterizes blank cotton centered at $2\theta \sim 22^\circ$ its values are assigned in Table 4 showing the effect of the different UV/ozone exposure times ranging from 10-60 minutes on the values of, d-spacing [\AA] and peak height [cps]. It was noticed that, there is a decrease in the peak intensity expressed as the height by count per cycle with the highest decrease after 10minutes exposure with a percentage decrease of (6.94%) relating to blank unexposed sample. This result verified the decrease in the

mechanical strength after 10 minutes, which indicating the decrease in the crystalline/amorphous ratio resulting in more brittle fabric. After this time the intensity was increased again with continuous exposure, causing a solidification of the fibers, resulting in an increase in the crystalline again and an irregular change in peak intensity values up to 60 minutes UV/ozone exposure; this means that, an irregular change in the crystalline/ amorphous ratio. As the amorphous areas were damaged upon exposing to UV/ozone, rearrangement of associated chains may occur, leading to an increase in the crystalline/ amorphous ratio [27]. Also, exposure ensures the decrease in length and increase the mobility of polymer chains and hence encouraging the formation of-more ordered crystalline regions. Generally, the increase in crystal structure is the main factor in the stiffer, more brittle character of these materials and the reverse holds true for the increase in amorphous. These results should be taken into consideration in studying both the mechanical properties and dyeing characteristics [27]. Also, the increase in the height of the characteristic band means the increase in sharpness and the differences in the sharpness of X-ray diffractograms, from fabric to another is due almost to [28] the differences in the crystalline amorphous character of these fabrics.

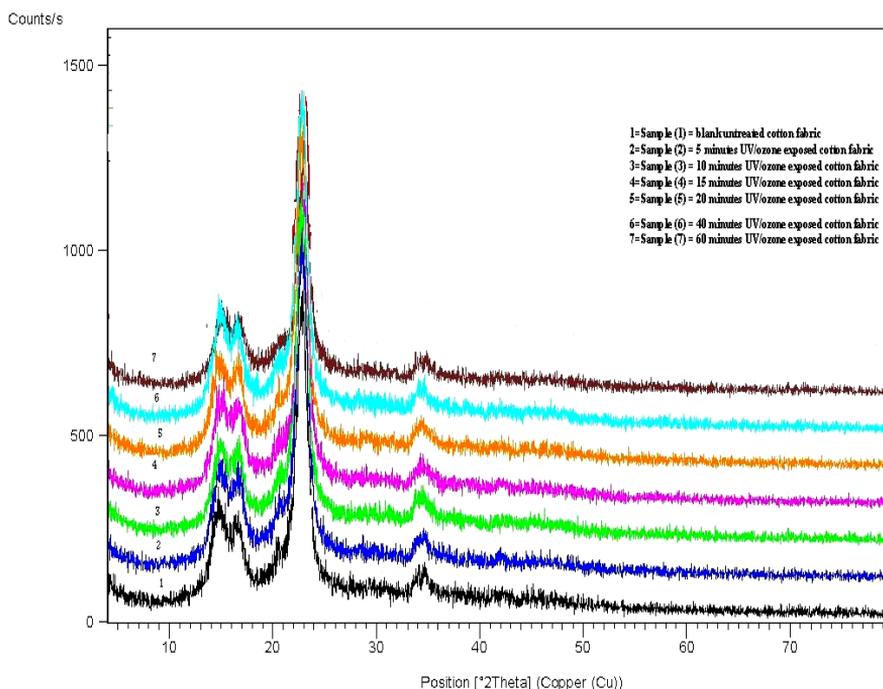


Fig. 2. X-ray diffractogram of different examined cotton fabrics treated with different UV/ozone exposure time, from 5 to 60 minutes.

Table 4. The changes in peak intensity values with different UV/ozone exposure times for cotton fabric.

<i>Exposure Time (minutes)</i>	<i>d-spacing [Å]</i>	<i>Count per cycle [cpc]*</i>
0.0(blank)	3.88684	706.48
5.0	3.83485	606.21
10.0	3.87252	657.43
15.0	3.89029	702.27
20.0	3.89989	761.20
40.0	3.88663	797.41
60.0	3.85659	514.14

* CPC = Count per Cycle.

Conclusions

Summing up the overall results and verifying the effect of uv/ozone treatments for different periods on the physical properties of the different examined fabric samples and its efficiency as a new accelerating environmental aging technique applied safely especially for silk and wool samples and is very practical for the production of mimic archaeological samples that can be applied in conservation and restoration work.

The tensile strength of wool and silk fabrics decreases gradually by prolonging UV/ozone exposure time to a maximum percentage after 60 minutes. But, for cotton fabrics, these values decrease gradually up to 10 minutes, and then it increased again reaching the value of the blank samples approximately. Where on considering the major band that characterizes blank cotton centered at ($2\theta \sim 22^\circ$) observed that a decrease in the peak intensity expressed as the height by count per cycle with the highest decrease after (5) minutes exposure with a percentage decrease of (6.94%) relating to blank unexposed sample. These results verified the decrease in mechanical strength after 10 minutes, which indicates the decrease in the crystalline/ amorphous ratio, thus more brittle fabric was obtained.

Generally, the air permeability increases appreciably at the beginning of exposure time for all of the examined fabrics.

By comparing UV/ozone treatment and other aging techniques on cotton fabrics it was concluded that: in case of the thermal aging for cotton samples, the results showed that the rate of loss in their tensile strength increased by increasing exposure time and the temperature of heating.

The chemical aging for cotton samples by acid hydrolysis: examining the samples treated with a solution of sulfuric acid at different concentrations and immersion times showed that the samples had become whiter and samples lost part of its strength and that the rate of loss in tensile strength increased by increasing the acid concentration.

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