ردمد: ٤٥٨٦-٢٥٢١

الخزانة

مجلة علمية نصف سنوية تعنى بالتراث المخطوط والوثائق

تصدر عن مركز إحياء التراث التابع لدار مخطوطات العتبة العباسية المقدسة

العدد الثاني، السنة الأولى، ربيع الأول ١٤٣٩هـ / كانون الأول ٢٠١٧م

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4. Conclusions

The study has proved that samples treated with Klucel G, particularly 3%, gave better results in terms of mechanical behavior, compared to 1% and 2%.

As for cellulose crystallinity index measurements, Klucel G (2%) gave the best results, while Klucel G (3%) caused a significant change in cellulose crystallinity after artificial aging.

Chemically speaking, Klucel G (2%) gave the best results since no change was detected in the functional groups of cellulose before and after accelerated aging using Fourier transform infrared spectroscopy.

Color measurements detected slight darkening and yellowing in all aged samples.

Klucel E gave moderated results in terms of mechanical behavior and excellent results in cellulose crystallinity index measurements, particularly Klucel E (2%). Excellent results were also obtained using FTIR spectroscopy in the case of the concentrations 1-2% since no change was detected in the functional groups of cellulose before and after accelerated aging.

Color measurements revealed slight darkening and yellowing of aged samples.

MC samples gave the least values in mechanical behavior measurements. Additionally, it also caused significant changes in cellulose crystallinity. However, good results were obtained using FTIR spectroscopy.

Excellent results were obtained in color change measurements, before and after aging, since only slight change in color was detected.

Crystallinity index = 45.5% showing a large decrease in the crystallinity index of cellulose, indicating that a significant change has occurred in the chemical and mechanical properties of cellulose.

linity index of cellulose, indicating that only a minor change has occurred in the chemical and mechanical properties of cellulose.

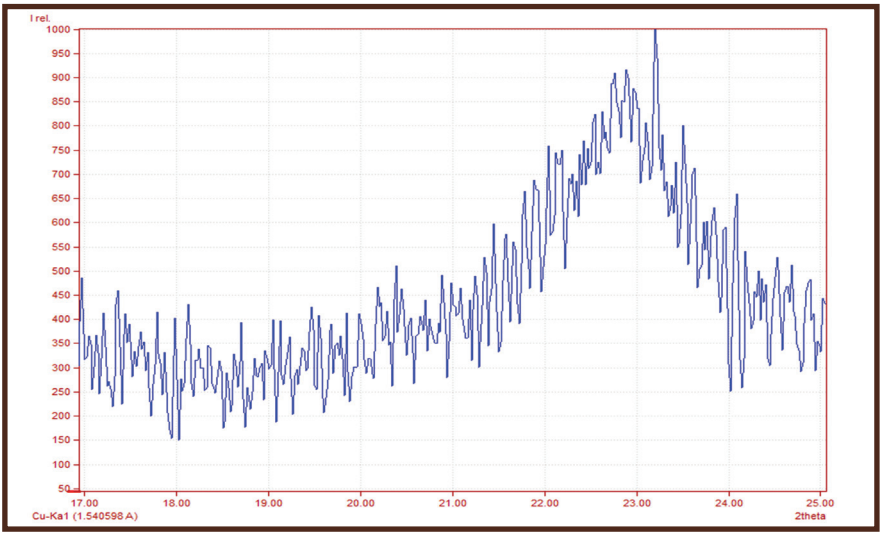

Figure (22) shows XRD pattern for the sample MC2

Crystallinity index = 51.2% showing a large decrease in the crystallinity index of cellulose, indicating that a significant change has occurred in the chemical and mechanical properties of cellulose.

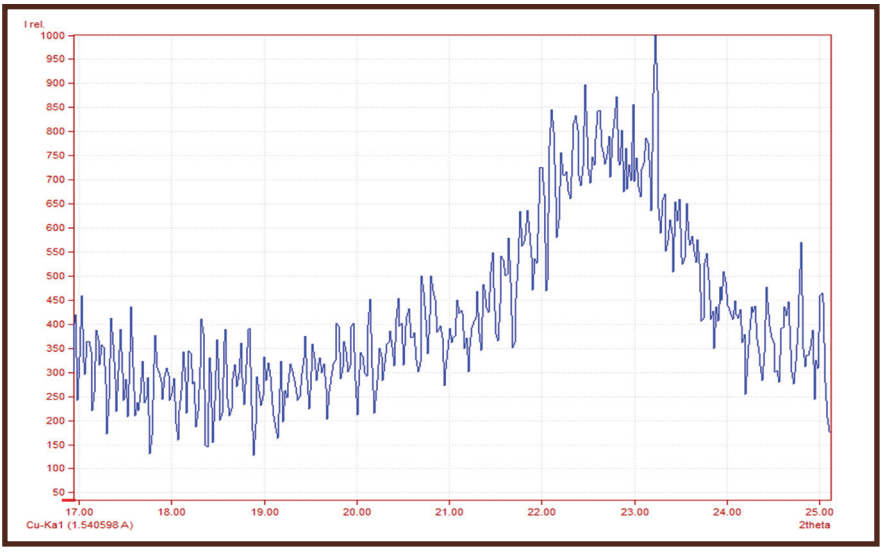

Figure (23) shows XRD pattern for the sample MC3

Crystallinity index = 61.7% showing no change in the crystallinity index of cellulose.

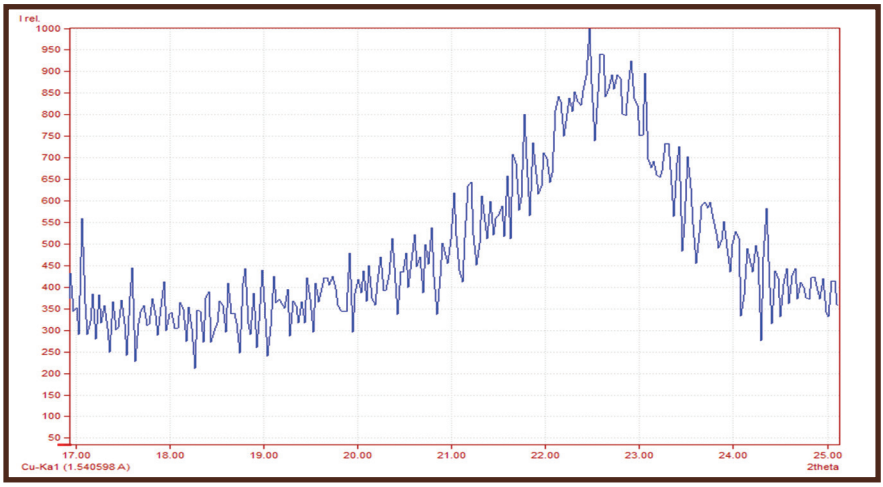

Figure (20) shows XRD pattern for the sample KE3

Crystallinity index = 56.1% showing an average decrease in the crystallinity index of cellulose, indicating that only a minor change has occurred in the chemical and mechanical properties of cellulose.

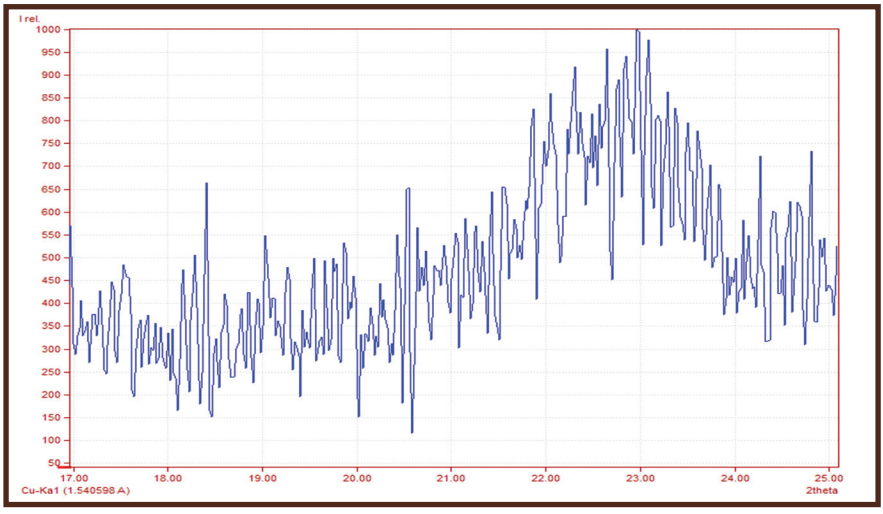

Figure (21) shows XRD pattern for the sample MC1

Crystallinity index = 53.7% showing an average decrease in the crystal-

Crystallinity index = 50.6% showing a large decrease in the crystallinity index in the cellulose crystallinity compared to the control sample, which means that a change has occurred in the chemical and mechanical properties of cellulose.

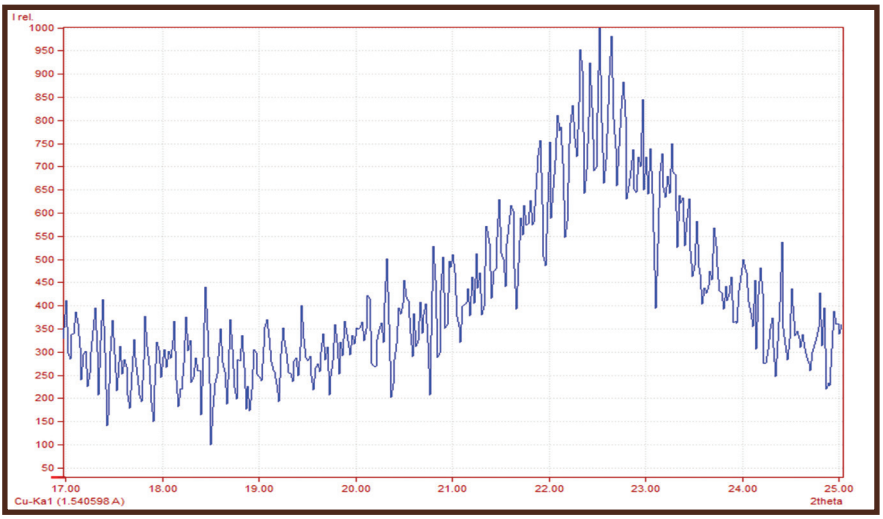

Figure (18) shows XRD pattern for the sample KE1

Crystallinity index = 63.4% showing a slight increase in the crystallinity index, which means that a change has occurred in the chemical and mechanical properties of cellulose.

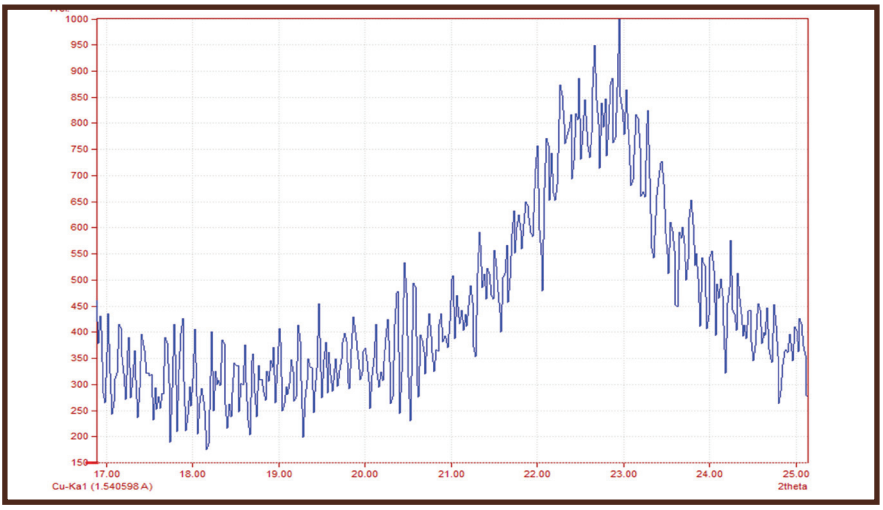

Figure (19) shows XRD pattern for the sample KE2

tallinity compared to the control sample, which means that a change has occurred in the chemical and mechanical properties of cellulose.

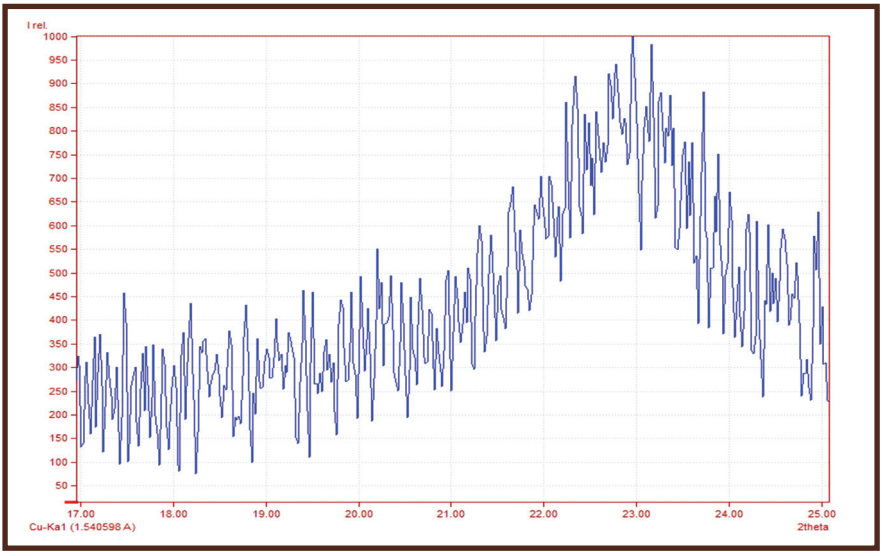

Figure (16) shows XRD pattern for the sample KG2

Crystallinity index = 58.5% showing a slight decrease in the crystallinity index compared to that of the control sample.

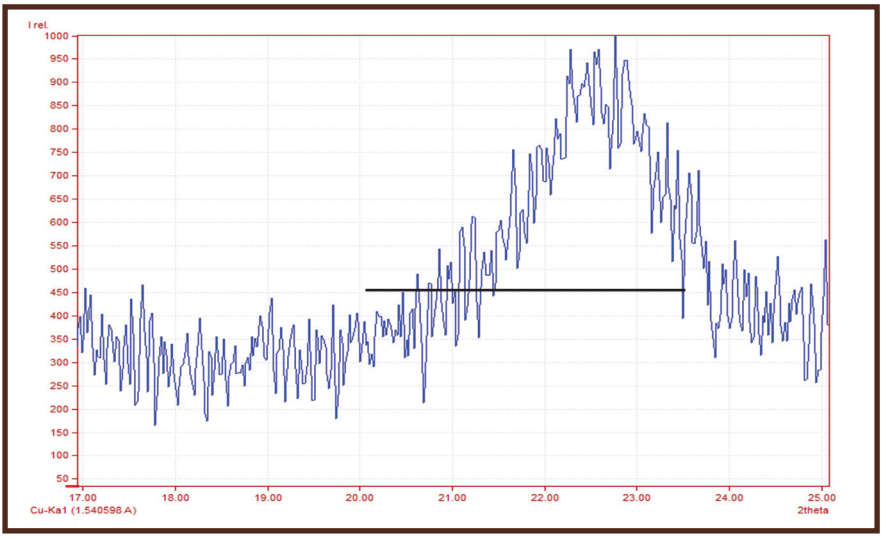

Figure (17) shows XRD pattern for the sample KG3

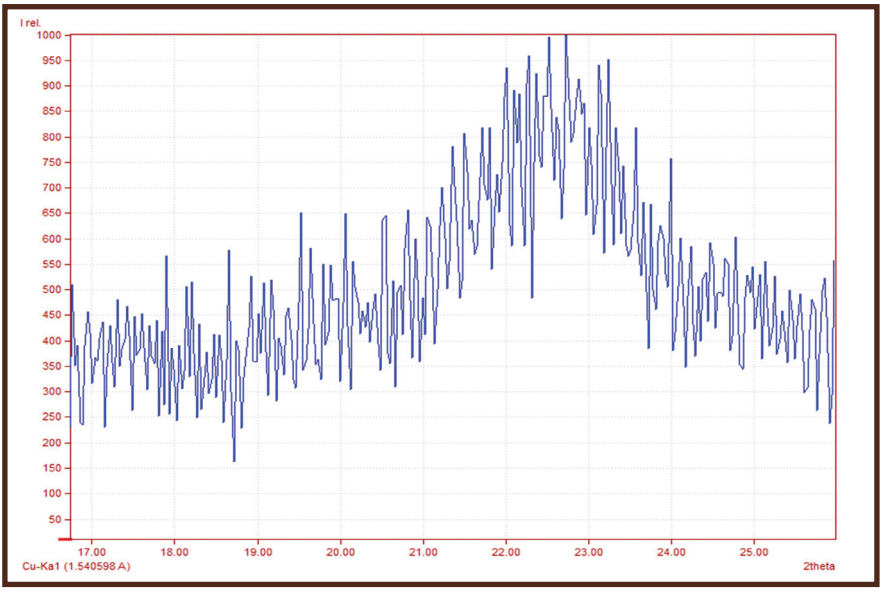

Figure (14) shows XRD pattern for the untreated control sample

Crystallinity index = 61.4%

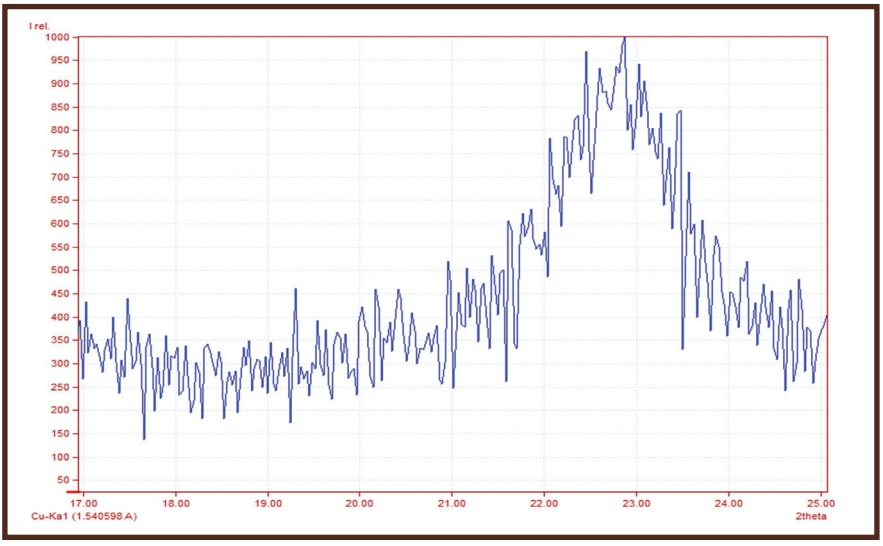

Figure (15) shows XRD pattern for the sample KG1

Crystallinity index = 66.5% showing an increase in the cellulose crys-

The two spectra are to a large extent identical indicating that no changes have occurred.

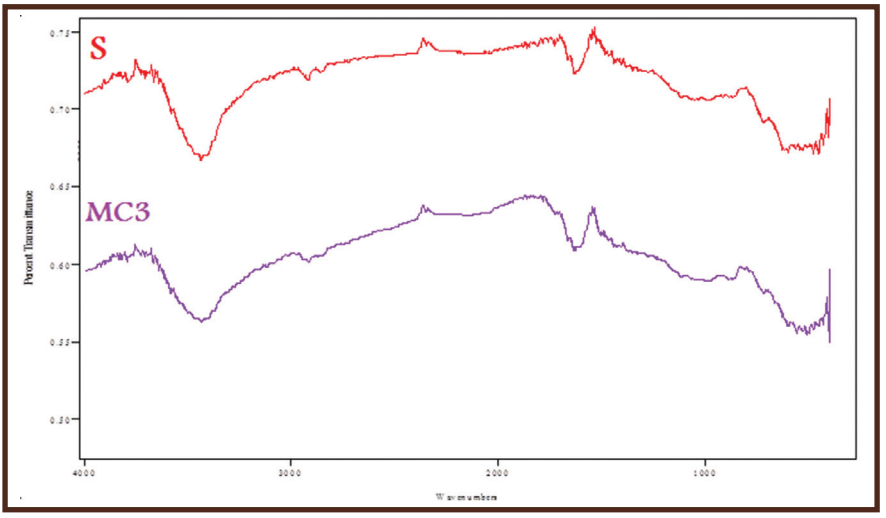

Figure (13) FTIR spectra of control sample and MC3

Compared to the control sample, no changes are observed in the functional groups characteristic of cellulose.

3.4. X-ray Diffraction Analysis (XRD)

The cellulose crystallinity for the control sample and the treated sample were calculated according to the following equation:

$$\text{Cr. I. \%} = \frac{(\text{I (002)} - \text{I } 18^{o}) \times 100}{\text{I}_{(002)}}$$

Where $\text{I}_{(002)}$ and I 18° are the maximum scattering intensities of the diffraction from the (002) plane at $2\theta = 2.26°$ and the diffraction intensity of the background scatter measured at $2\theta = 18°$, respectively, and the latter value being attributed to the non-crystalline cellulose form.

A slight increase in the OH group at around 3300 - 3400 cm^{-1} is observed and this indicates the occurrence of hydrolysis. There is also a slight increase in the absorption band at around 1620 – 1650 cm^{-1} as a result of cellulose oxidation.

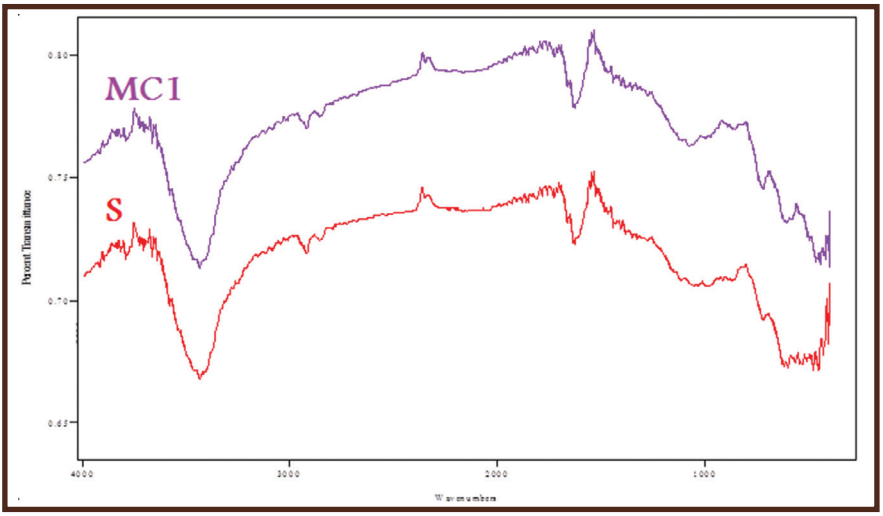

Figure (11) FTIR spectra of control sample and MC1

There is also an increase in the absorption band at around 1620 – 1650 cm^{-1} as a result of cellulose oxidation.

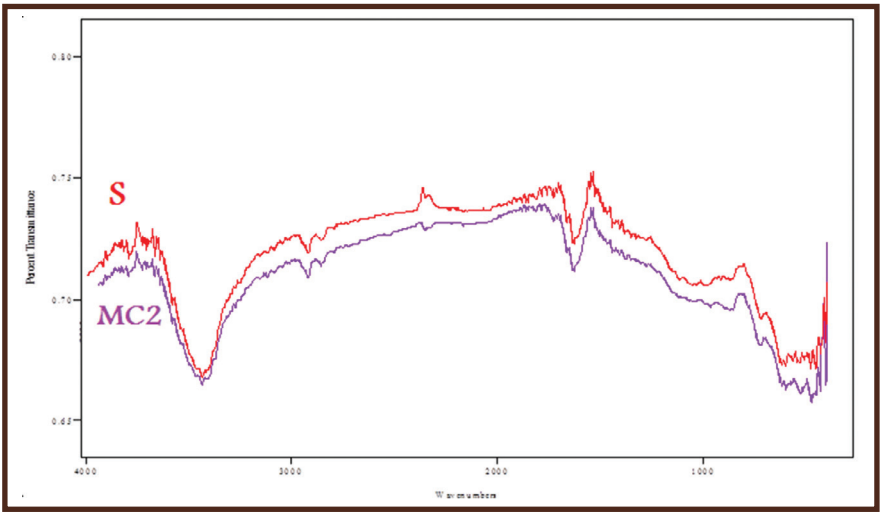

Figure (12) FTIR spectra of control sample and MC2

Compared to the control sample, no changes are observed in the functional groups characteristic of cellulose.

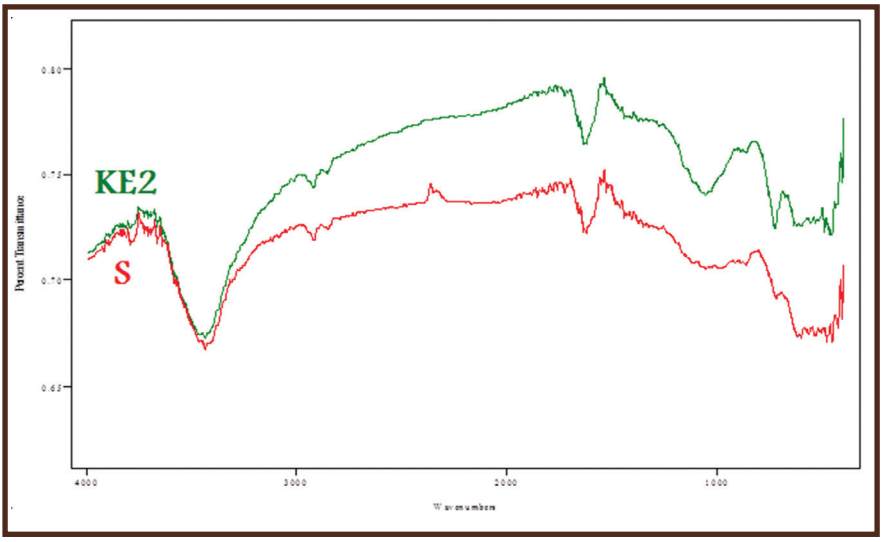

Figure (9) FTIR spectra of control sample and KE2

The two spectra are to a large extent identical indicating that no changes have occurred.

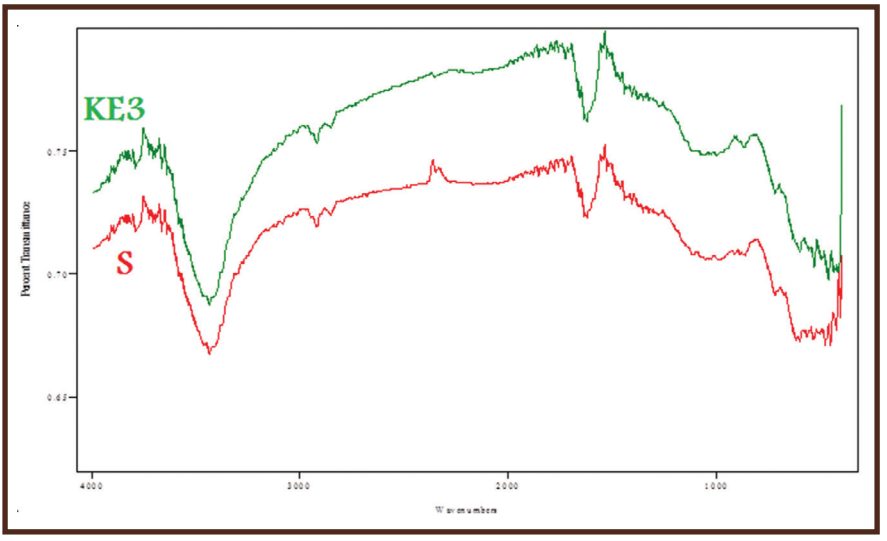

Figure (10) FTIR spectra of control sample and KE3

Compared to the control sample, no changes are observed in the functional groups characteristic of cellulose.

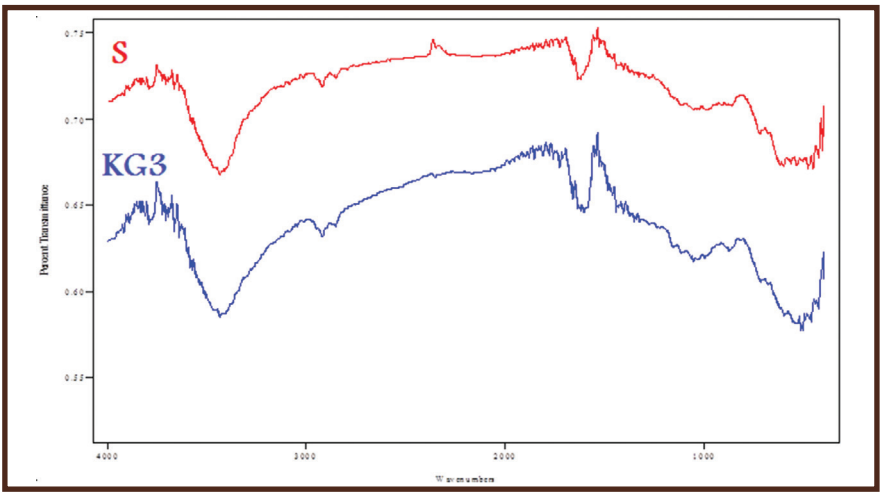

Figure (7) FTIR spectra of control sample and KG3

A slight increase in the OH group at around 3300 - 3400 cm^{-1} is observed and this indicates the occurrence of hydrolysis. There is also an increase in the absorption band at around 1620 – 1650 cm^{-1} as a result of cellulose oxidation.

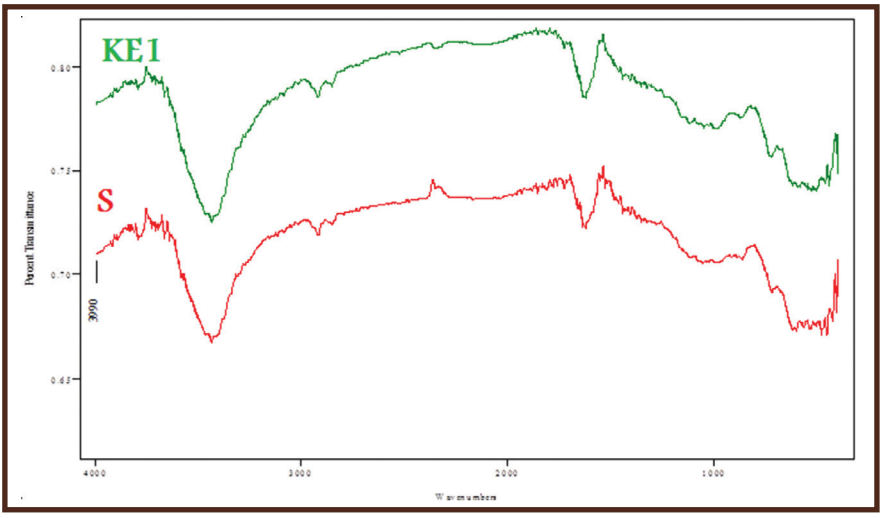

Figure (8) FTIR spectra of control sample and KE1

FTIR results of the control sample shows the presence of C=O group, with absorption bands at around 1620 – 1664 cm^{-1}, and it is assigned to the oxidation of the cellulose in the accelerated aging ovens. Comparison between treated samples and the control sample has been carried out and the results were as follows:

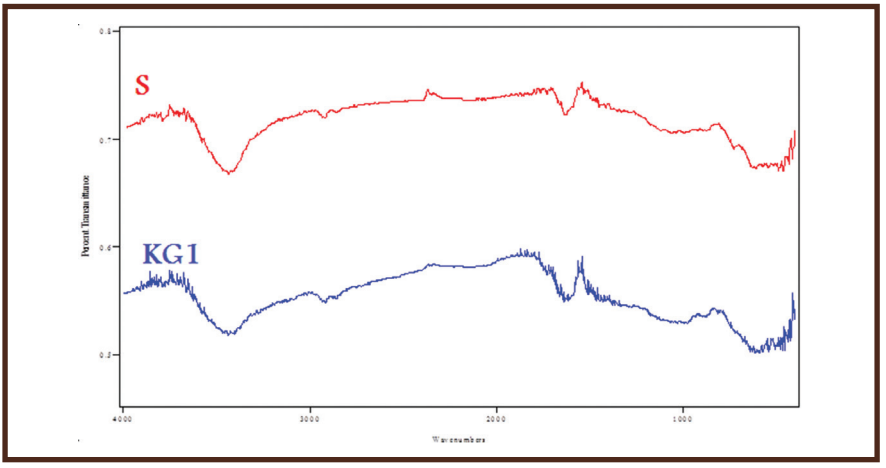

Figure (5) FTIR spectra of control sample and KG1

The decrease in the OH band at around 3300 – 3400 is a result of the loss of water molecules. An increase is also noticed in the C=O absorption band at (1620-1650) indicates that the cellulose has been oxidized.

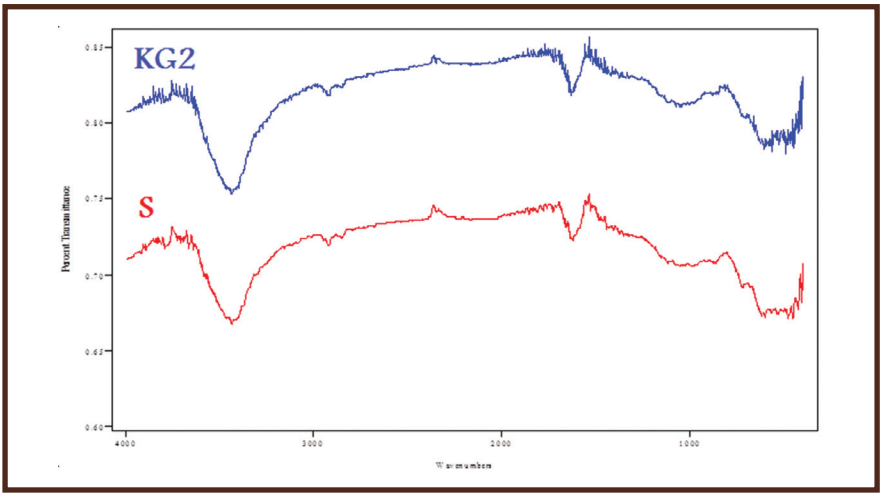

Figure (6) FTIR spectra of control sample and KG2

3.3. Fourier Transform Infrared Spectroscopy (FTIR)

Figure (4) FTIR spectrum for the control paper sample

Table (4) shows the function groups for the control sample

Functional group	Wavelength (cm^{-1})	Interpretation
-OH stretching	3433 cm^{-1}	Assigned to the hydroxyl groups in cellulose.
-CH stretching	2881-2920 cm^{-1}	Assigned to the hydrocarbon group in cellulose, hemicellulose and lignin.
-C=O stretching	1628-1664 cm^{-1}	resulted from cellulose oxidation and its transformation to carbonyl and carboxyl groups.
-C=C stretching (Aromatic)	1530 cm^{-1}	Assigned to the presence of lignin.
-C-O stretching	1035 cm^{-1}	Assigned to cellulose and hemicellulose.
-CH	873 cm^{-1}	Assigned to cellulose.

MC2 after treatment	89.55	- 0.02	11.22	
MC3 before treatment	90.18	0.06	9.19	1.97
MC3 after treatment	89.79	-0.07	11.12	

Results revealed the darkening of all samples compared to the aged control sample. Sample MC3 showed the least darkening with a L* value of 89.79. The increase in the value of b* indicates that all samples have become yellow except for KG3 which has become blue.

3.2. Mechanical Behavior Measurements

The values of tensile strength and elongation (%) are shown in table (3). The results reported are the average of five measurements with standard deviation.

Table (3) Tensile strength and elongation values of treated aged samples

Sample No.	Elongation (%)	Tensile Strength
S	9.94	35.70
KG1	1.29	84.1
KG2	1.75	97.0
KG3	3.19	91.0
KE1	1.44	80.4
KE2	1.40	85.3
KE3	1.43	81.3
MC1	1.23	82.0
MC2	1.14	83.7
MC3	1.27	80.0

KG3 gave the highest increase in tensile strength and elongation compared to the other samples, while MC samples gave the lowest values for tensile strength and elongation. Results for KE samples indicate average values.

3. Results and Discussion

3.1. Color Measurements

The colorimetric coordinates L*, a*, and b*of the CIE L*a*b* color space were used to express color change. The CIELAB color space is organized in cube form. The L* axis runs from top to bottom. The maximum for L* is 100, which represents white. The minimum for L* is zero, which represents black. The a* and b* axes have no specific numerical limits. Positive a* is red. Negative a* is green. Positive b * is yellow. Negative b* is blue.

Color measurements were carried out before and after aging and results are shown in table (2):

Table (2) shows color measurements for treated samples before and after aging

Samples	L*	a*	b*	ΔE
S	89.85	- 0.08	10.89	
KG1 before treatment	90.30	0.07	9.64	6.95
KG1 after treatment	86.90	0.04	15.70	
KG2 before treatment	90.12	- 0.01	9.53	6.24
KG2 after treatment	88.09	0.02	15.43	
KG3 before treatment	90.26	0.02	9.90	5.26
KG3 after treatment	87.42	- 0.40	- 0.40	
KE1 before treatment	90.42	0.12	9.46	5.27
KE1 after treatment	88.77	0.13	14.46	
KE2 before treatment	90.32	0.11	9.41	6.25
KE2 after treatment	88.35	0.25	15.62	
KE3 before treatment	90.21	0.17	9.64	5.42
KE3 after treatment	88.01	- 0.26	14.58	
MC1 before treatment	90.32	0.13	9.45	1.81
MC1 after treatment	89.56	- 0.04	11.08	
MC2 before treatment	90.24	0.22	9.45	1.91

ology, Cairo University using Diffractometer PW 1480, Netherland and a program analysis: PDF4 2015 + Match2[1][2][3].

(1) C.M. Popescu, P.T. Larsson, C.M. Tibirna, C. Vasile, Characterization of fungaldegraded-lime wood by X-ray diffraction and cross-polarization magic-angle-spinning 13Cnucle-armagnetic resonance spectroscopy, Applied Spectroscopy, 64(9), 2010, pp. 1054-1060.

(2) Park, Sunkyu, John O Baker, and Michael E Himmel, . 2010. «RCeseealrlcuh lose crys-tallinity index: measurement techniques and their impact on interpreting cellulase per-formance.» Biotechnology for Biofuels 3:10.

(3) Zugenmaier, Peter. 2008. Crystalline Cellulose and Derivatives. Characterization and Structures. Verlag Berlin Heidelberg: Springer.

measurements were made before and after treatment and compared to that of the control sample[(1)(2)(3)]. The procedure was carried out in aging ovens at the National Institute of Standards (NIS) in Cairo, Egypt.

2.3.3. Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectroscopy was used to study the functional groups present in paper and the changes that have occurred due to treatments compared to the control paper sample. FTIR spectra of paper samples were measured on a Nicolet 380 FT-IR Spectrometer, in the frequency range of 4000 - 400 cm^{-1}, in reflectance mode. The procedure was also carried out in aging ovens at the National Institute of Standards (NIS) in Cairo, Egypt[(4)(5)].

2.3.4. X- Ray Diffraction Analysis (XRD)

This technique was employed to study the crystallinity degree of cellulose for the treated samples compared to the control sample. This procedure was done at the Conservation Department at the Faculty of Archae-

(1) Jablonský, M., Botková, M. (2012) Accelerated Ageing of Wood-Containing Papers: Formation of Weak Acids and Deterioration of Tensile Strength. Wood Research, Vol. 57, Issue 3.

(2) Zervos, S. (2010) Natural and Artificial Ageing of Cellulose and Paper: A Literature Review. In Cellulose: Structure and Properties, Derivatives, and Industrial Uses, Nova Science Publishers, Inc., NY.

(3) Zeng, X., Vishtal, A., Retulainen, E., Sivonen, E., Fu, S. (2013) The Elongation Potential of Paper-How Should Fibres be Deformed to Make Paper Extensible, BioResources, Vol. 8, Issue 1.

(4) Jiangtao Shi, Dong Xing, and Jian Lia. 2012. «FTIR Studies of the Changes in Wood Chemistry from Wood Forming Tissue under Inclined Treatment.» International Conference on Future Energy, Environment, and Materials. by Elsevier. 758-762.

(5) J. Lojewska, P. Mis´kowiec, T. Lojewski, and L.M. Proniewicz. 2005. «Cellulose oxidative and hydrolytic degradation In situ FTIR approach.» Polymer Degradation and Stability 88 512-520.

Figure (3) shows the paper samples treated with methyl cellulose

2.3. Analysis Techniques

2.3.1. Color Change Measurements

The color of the samples was measured with a Optimatch 3100® from the SDL Company. All samples were measured in a visible region[(1)(2)(3)]. All measurements were made before and after treatment and compared to that of the control sample. The procedure was carried out in aging ovens at the National Institute of Standards (NIS) in Cairo, Egypt.

2.3.2. Mechanical Behavior Measurements

Mechanical behavior of the samples (i.e. tensile strength and elongation%) were studied using the dynamometer produced by SDL ATLAS, H5KT. The samples were cut in the machine direction to strips of 2 cm × 10 cm. All

(1) Pellizzi, E., Lattuati-Derieux, Lavédrine, A., and Cheradame, H., Flexible Polyurethane Easter Foam Consolidation: Preliminary Study of Aminopropylmethyldiethoxysilane Reinforcement Treatment, Proceedings Adhesives and Consolidants for Conservation: Research and Applications, CCI Symposium 2011, Ottawa, Canada.

(2) Hunter L, a, b color space, Applications Notes: Insight on Color, Vol. 8, No. 7, 1996.

(3) Limbo, S., and Piergiovanni, Shelf life of minimally Processed Potatoes: Part 1. Effects of High Oxygen Partial Pressures in Combination with Ascorbic and Citric Acids on Enzymatic Browning, Postharvest Biology and Technology, Vol. 39, 2006.

2.2. Artificial Aging

Samples were exposed to moist heat aging at a temperature of 80 °C and a relative humidity of 65% for a period of 120 hours, which is equivalent to aging of paper under natural conditions for 25 years. The aging procedure was in conformance with the ISO 5630-3:1996 standard(1). The procedure was carried out in aging ovens at the National Institute of Standards (NIS) in Cairo, Egypt.

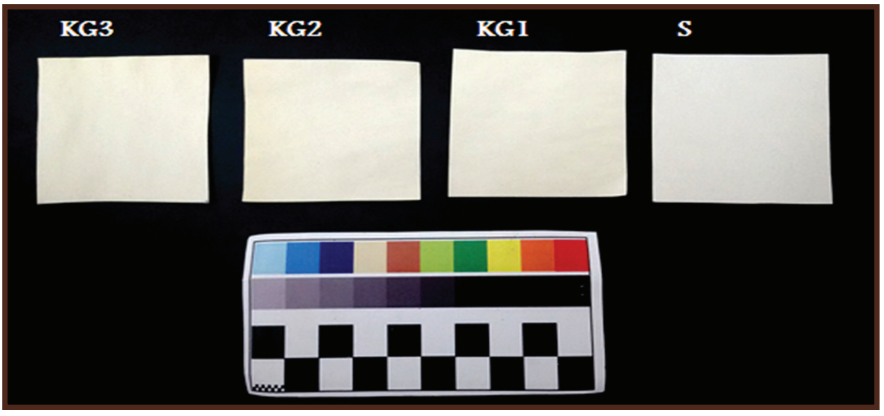

Figure (1) shows the paper samples treated with Klucel G

Figure (2) shows the paper samples treated with Klucel E

(1) Kamińska, A., Sawczak, M., Ciepliński, M., Śliwiński, G., Kosmowski, B. Colorimetric Study of the Post-Processing Effect due to Pulsed Laser Cleaning of Paper, Optica Applicata, 2004: Vol. 34, Issue 1.

- Methyl cellulose (1%, 2%, 3%).
- Klucel G and Klucel E (0.5, 1, and 1.5).

These concentrations were selected since they are commonly used in paper consolidation[1][2][3]. The consolidants were applied to the paper samples using a soft brush. Samples were each given a number as shown in the table below:

Table (1) Sample numbers and applied treatments

Sample No.	Treatment
S	Control sample (untreated)
KG1	Sample consolidated with 0.5% of Klucel G
KG2	Sample consolidated with 1% of Klucel G
KG3	Sample consolidated with 1.5% of Klucel G
KE1	Sample consolidated with 0.5% of Klucel E
KE2	Sample consolidated with 1% of Klucel E
KE3	Sample consolidated with 1.5% of Klucel G
MC1	Sample consolidated with 1% of Methyl cellulose
MC2	Sample consolidated with 2% of Methyl cellulose
MC3	Sample consolidated with 3% of Methyl cellulose

(1) Hofmann, Christa , Andreas Hartl, Kyujin Ahn, Laura Völkel, and Ina Faerber. 2013. «Verdigris I: Compromises in Conservation.» Paper Conservation: Decisions & Compromises. Vienna,: International Council of Museums (ICOM). 34-35.

(2) Wheeler , Michael, Nicholas Barnard, and Karine Bovagn. 2013. «The Conservation and Digitization of Jain Manuscripts at the Victoria and Albert Museum.» Paper Conservation: Decisions & Compromises. Vienna: International Council of Museums (ICOM). 101-104.

(3) Laaser, Tilly, Karolina Soppa, and Christoph Krekel. 2013. «The Migration of Hydroxy Propyl Cellulose During Consolidation of a Painted Wallpaper: A Case Study Using a Fluorescent - Labelled Consolidant.» Paper Conservation: Decisions & Compromises. Vienna: International Council of Museums (ICOM). 88-90.

However, cellulose derivatives are yet the most common materials for consolidation of paper manuscripts such carboxy methyl cellulose, methyl cellulose, hydroxy propyl cellulose [(1)(2)(3)].

2. Materials and Methods

2.1. Materials

2.1.1. Paper Samples

The experimental studies were carried out on wood pulp paper samples since their structure is similar to that of old paper manuscripts.

2.1.2. Consolidants

- Klucel G, hydroxyl propyl cellulose.
- Klucel E, hydroxyl propyl cellulose.
- Methyl cellulose.

These were all supplied by CTS imported by Andalus Company for Conservation and Restoration supplies. The solvent used is ethanol and was supplied by DEWK.

2.1.2.1. Preparation of Consolidants:

Consolidants were all dissolved in ethyl alcohol (ethanol) to the following concentrations:

(1) Ardelean, Elena, Doina Asandei, Mihaela Tanase, and Elena Bobu. «Study on some resizing and consolidation methods of old paper support.» European Journal of Science and Theology, 2007: Vol.3, No.3, 53-61.

(2) Petrea, Puiu, Florin Ciolacu, and Sorin Ciovică. «The evaluation of some consolidation agents applied in the conservation of graphical documents.» European Journal of Science and Theology, 2010: Vol.6, No.1, 67-75.

(3) Ardelean, Elena, Raluca Nicu, Doina Asandei, and Elena Bobu. «Carboxymethyl-chitosan as consolidation agent for old documents on paper support.» European Journal of Science and Theology, 2009: Vol.5, No.4, 67-75.

1. Introduction

The issue of consolidation of paper manuscripts has been growing an interest among many researchers and restoration centers worldwide, and this is mainly due to the severe embrittlement of paper manuscripts which is caused by the effect of different deterioration factors such as temperature and relative humidity changes and pollutants [(1)]. Paper is also vulnerable to microbiological damage [(2)(3)].

Many natural materials were used to consolidate paper manuscripts such as Funori[(4)]. In the recent years, nanomaterials have also become very popular in conservation of historical and cultural heritage[(5)(6)(7)], and several were used to consolidate paper manuscripts such as hydroxy apatite.

(1) Usman, Zainab, and Hauwa Mari. «Deterioration of Library Resources and its Causes: Theoretical Review.» International Journal of Basic and Applied Science, 2013: 773-778.

(2) Silva, Manuela da, et al. «Inactivation of fungi from deteriorated paper materials by radiation.» International Biodeterioration & Biodegradation. Elsevier Ltd, 2006. 163–167.

(3) Sato, Yoshinori, Mutsumi Aoki, and Rika Kigawa. «Microbial deterioration of tsunami-affected paper-based objects.» Microbial biodeterioration of cultural property, Proceedings of the International Symposium. Tokyo, 2014. 51-65.

(4) Geiger, Thomas, and Françoise Michel. «Studies on the Polysaccharide JunFunori Used to Consolidate Matt Paint.» Studies in Conservation (Studies in Conservation) 50, no. 3 (2005): 193-204.

(5) O'Brien, Paul O'Brien, Harry Kroto, and Ralph Nuzzo. Nanoscience for the Conservation of Works of Art. Edited by Piero Baglioni and David Chelazzi. Cambridge: The Royal Society of Chemistry, 2013.

(6) Piero Baglioni, David Chelazzi, Rodorico Giorgi. Nanotechnologies in the conservation of cultural heritage: A compendium of materials and techniques. Springer Science + Business Media Dordrecht, 2015.

(7) Iona, Rodica-Mariana, Sanda Maria Donceaa, Mihaela-Lucia Ionc, Valentin R˘adit¸oiub, and Viorica Am˘ariutei. «Surface investigations of old book paper treated with hydroxyapatite.» Applied Surface Science (Elsevier B.V.), 2013: 27– 32.

الملخّص

تعتبر هشاشية الورق أحد أكثر مظاهر التلف شيوعا بين مجموعات المخطوطات. وينتج هذا المظهر جراء تأثير عدة عوامل، ومنها الطبيعية الفيزيائية للورق، وهذا ما يفسر تأثره الشديد بعوامل التلف المختلفة مثل المستويات الغير مناسبة من درجات الحرارة والرطوبة النسبية، والملوثات الجوية، والعرض والتخزين السيء، والترميم الغير ملائم.

هذه العوامل السابقة تحفز حدوث تغيرات فى التركيب الكيميائي للورق فضلا عن انخفاض خصائصه الميكانيكية، مما يتسبب فى هشاشية الورق بشكل كبير. وتعد مشتقات السليلوز واحدة من أهم المواد المستخدمة في تقوية المخطوطات الورقية. ومن هنا فإن هذا البحث يهدف إلي تقييم كفاءة بعض مشتقات السليلوز في تقوية الورق ، حيث تم اختيار مواد التقوية التالية:

- كلوسل G (هيدروكسي بروبايل سليلوز).
- كلوسل E.
- ميثيل سليلوز.

وتمت دراسة تأثير هذه المعالجات على الخواص الفيزيائية والبصرية للورق باستخدام تحليل حيود الأشعة السينية لقياس درجة التبلور، كما تم استخدام التحليل الطيفي بالأشعة تحت الحمراء لدراسة التغير في المجموعات الوظيفية للورق.

Abstract

Embrittlement is considered one of the most common deterioration aspects among paper manuscript collection. This is caused by several factors such as the physical nature of paper, and this explains why it is highly affected by deterioration factors (i.e. improper temperature and relative humidity levels, pollutants, improper storage and display, and inadequate restoration).

These previous factors promote changes in the chemical structure of paper as well as loss in its mechanical properties, eventually causing the paper to become extremely brittle.

Cellulose derivatives are considered one of the most significant materials used in the consolidation of paper manuscripts. The main aim of this paper was to evaluating the efficiency of several cellulose derivatives in consolidating paper, and the following consolidants were selected:

- Klucel G, hydroxyl propyl cellulose.
- Klucel E, hydroxyl propyl cellulose.
- Methyl cellulose.

The effect of these treatments on the physical and optical properties of paper was studied using X-ray diffraction technique to measure the crystallinity degree and also Fourier transform infrared spectroscopy was employed to detect the change in the functional group of paper.

A Comparative Study to Evaluate Consolidation of Paper Manuscripts Using Cellulose Derivatives

دراسة مقارنة لتقييم تقوية المخطوطات الورقية باستخدام مشتقات السليلوز

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