

EVALUATION OF STARCH AND CELLULOSE BASED CONSOLIDATION MATERIALS ON THE MECHANICAL PROPERTIES OF PAPYRUS

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In this study, polysaccharides, namely, potato starch, ethyl hydroxyethyl cellulose and cellulose nanocrystals (CNC), were used as consolidation materials for papyrus. The starch solution was applied on papyrus strips by two different methods: by brush and by an ultrasonic humidifier, the latter being a new approach for the application of consolidation materials. The consolidated papyrus was subjected to a thermal ageing study and several measurements were conducted to evaluate the efficiency of the selected consolidation materials and of the two different application methods of starch. The mechanical properties of the consolidated papyrus strips were evaluated by measuring the tensile strength and elongation. FT-IR spectroscopy and scanning electron microscopy of the papyrus strips were performed before and after thermal ageing. The study showed that CNC and ethyl hydroxyethyl cellulose improved the mechanical properties of the tested strips. Applying potato starch using the ultrasonic humidifier improved the mechanical properties of the consolidated strips more significantly than its application by brush.

Keywords: papyrus, CNC, thermal aging, ultrasonic humidifier, tensile strength, elongation

INTRODUCTION

Cyperus papyrus L. is considered one of the most important writing supports used by ancient Egyptians since the dawn of history, followed by other materials, such as animal skin, wood surfaces *etc.* Papyrus continued to be used until the Greco-Roman, Byzantine and early Islamic periods.^{1,2} It has been established that the main component of the papyrus plant pulp is cellulose, with contents of 54-68%, followed by lignin, with 24-32%.^{1,2} Other studies have also found sodium, potassium, silicon and calcium minerals³ in papyrus, in addition to its basic components – hemicelluloses, cellulose and lignin. The papyrus plant has been exploited in ancient Egypt in many and varied ways, in addition to being used as a writing material; one of the most famous uses of papyrus was for manufacturing boats for transport, travel, hunting and fishing, as well as for transporting blocks of heavy stone that were used in building many monuments, including the pyramids.⁴

There is always an urgent need to preserve human history by preserving heritage papyrus artifacts. Various consolidation methods have been used for preserving papyrus, for example, applying a 3% Arabic gum solution by spraying. This method has been found the most suitable to strengthen papyrus writing materials. However, Hassan *et al.* recommended that the strengthening treatment should not be applied more than once to prevent loss of elasticity, suggesting treatments with fish glue solution, Arabic gum or 2% hydroxypropyl cellulose.⁵ Another solution was found for conserving the Ramesseum Papyri in the first part of the twentieth century, combining gelatin and Celluloid (trade name for materials consisting of cellulose nitrate). These materials are characterized by transparency, which allows reading the texts written on both sides of the papyrus sheets.⁶

Research and technological advancements have focused on finding new conservation tools and materials to treat ancient papyrus. Cellulose nanocrystals have been reported as the material of choice for restoring the original properties of paper documents. Considering that nanocrystalline cellulose is

basically a major component of paper, it demonstrates a strong affinity towards the latter. Moreover, a treatment formulation based on cellulose nanocrystals does not require the addition of adhesives. Cellulose nanocrystals (diameters ranging from 5 to 50 nm and lengths between 100 and 500 nm) have a wide range of applications in many fields due to their exceptional mechanical and thermal properties, optical transparency, crystallinity and non-toxicity.⁷⁻⁹ They are commonly used as fillers and reinforcements in nanocomposites, or as pulp modifiers to increase its resistance.^{10,11} It has been established that nanocelluloses of different dimensions and chemical functionalization display different consolidation effects.¹²⁻¹⁵

Considering the aspects discussed above and our previous experience in using natural polymers in several applications,^{8,16-20} this study aimed to investigate different conservation treatments intended to preserve papyrus artifacts. The treatment should preserve the papyrus, while maintaining its historical integrity, as well as mechanical and chemical properties.

EXPERIMENTAL

Materials

Ethyl hydroxyethyl cellulose (MW 100,000) was purchased from Across Chemical Co. The degree of substitution of the ethyl and hydroxyl ethyl was expressed in terms of ethyl (average number of ethyl groups per anhydroglucose unit of the polymer). The molecular substitution (MS_{EO}) refers to the average number of hydroxyl ethyl groups per anhydroglucose unit of the polymer. The following values were provided by the manufacturer: $DS_{ethyl} = 0.6-0.7$ and $MS_{EO} = 1.8$.

Potato starch was purchased from Across Chemical Co. Sodium bromide and sodium hypochlorite were purchased from Sigma-Aldrich (Germany). Sulfuric acid, acetic acid, HCl, calcium chloride and sodium hydroxide were purchased from El Naser Co. (Egypt).

Cellulose was extracted from bagasse pulp delivered by Quena Paper Industry Co., Egypt. Cellulose nanocrystals (CNC) were prepared using the following procedure: pure cellulose fibers (10 g) were dispersed in 200 mL of 65% sulfuric acid at 45 °C under mechanical stirring for 25 min. The hydrolyzed pulp was centrifuged to remove the excess of acid and dialyzed against water for several days until neutral pH (6-7). The obtained CNC were sonicated for 5 min to reduce the aggregate size.^{8,21}

Methods

Preparation of papyrus strips

The strips were made from papyrus stems. The white inner part of the stem was cut into thin strips of the same thickness as much as possible, after the green outer rind was peeled. The strips were dried under pressure. 90 papyrus strips were prepared (1.50 cm width x 10 cm length) according to TAPPI T 220.⁵ The thickness of the strips was 1 mm, as measured using a caliper. Each type of consolidation material was applied on 10 papyrus strips on both sides of the strip, while 10 strips were retained as the control group (unconsolidated papyrus). All the samples were conditioned at 50% RH for 48 h before testing.

Application of consolidation materials on papyrus strips

Ethyl hydroxyethyl cellulose, potato starch and CNCs were tested as consolidation materials and were applied on both sides of the papyrus strips.

Potato starch was used in two concentrations: of 0.5% and 1%, in 100 mL of distilled water. The mixtures were prepared by adding starch to boiling water under stirring to obtain a clear solution. The mixtures were allowed to cool and were then filtered with a piece of gauze. Then, the prepared starch solution was applied on the strips by two different methods: using a brush and an ultrasonic humidifier device (Fig. 1A). The ultrasonic humidifier was used in this study as a new approach to applying consolidation materials. The Boneco U7145 Ultrasonic Humidifier (Fig. 1) uses a high-frequency ultrasonic vibration system to turn the solution into a micro-fine mist (Fig. 1A). We adapted the ultrasonic humidifier to provide micro-fine mist of the starch solution for applying on the papyrus strips (Fig. 1C).

Ethyl hydroxyethyl cellulose and CNC were also used in two concentrations: of 0.5% and 1%, and were applied to the strips using a brush only.

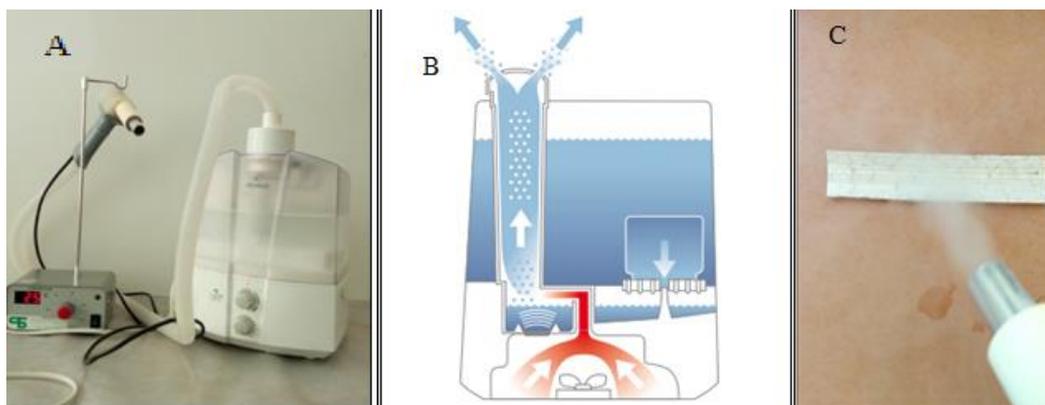


Figure 1: Ultrasonic humidifier device (A); the working technique of the system (B); applying potato starch mist on papyrus strips (C)

Thermal ageing study

Half of the strips (45 strips) were placed into a thermal ageing oven at 100 °C ±5 °C for 144 hours (6 days), which is equivalent to 50 years of normal ageing, and at a temperature of 105 °C for three days, which is equivalent to 25 years of natural ageing.

The tensile strength and elongation of the papyrus strips were measured according to TAPPI T494 om-88,⁵ before and after thermal ageing, using Tinius Olsen H5K apparatus at a speed of 5 mm/min.

Measuring the color changes of papyrus strips

Eleven papyrus strips were prepared for color measurement before and after thermal aging. A standard unconsolidated papyrus strip for each consolidation material was tested separately. Each group of consolidated papyrus samples (for each consolidation material), as well as its corresponding control sample, was cut from a single papyrus piece to ensure that all samples have exactly the same color grade. As papyrus is a heterogeneous plant material, color tones may vary from one strip to another. The color change of the strips was measured before and after thermal aging in order to accurately observe the color changes induced by ageing.

An Optimatch 3100 Spectrophotometer was used for observing the color changes of the papyrus strips. The color changes were determined using the CIELAB system based on the L*, a* and b* values. L* represents the degree of lightness with the range of 0-100, where zero indicates black color and 100 indicates white color. A decrease in the value of L* indicates the darkness of color. Also, a* represents the color values between red and green: when the value is positive, the color is red, and when it is negative, it turns green; while b* represents the yellow/blue scale: when the value is positive, the color is yellow, and if it is negative, the color tends to blue. The color difference (ΔE) between the color of the consolidated strips before aging and after thermal aging was calculated by the following equation:²²

$$\Delta E = \{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2\}^{1/2} \quad (1)$$

Characterization of CNC consolidation material

CNC was characterized by transmission electron microscopy (TEM) using a JEOL 1230 transmission electron microscope (JEOL, Tokyo, Japan), with an acceleration voltage of 100 KV. A drop of nanoparticles suspension was used on a copper grid bearing a carbon film.

Fourier transform infrared (FTIR) spectroscopy was carried out using a Mattson 5000 spectrometer (Unicam, UK) by the KBr technique.

X-ray diffraction patterns (XRD) were recorded using an X-ray diffractometer (PANalytical, Netherlands) at room temperature, with a monochromatic Cu-K α radiation source ($\lambda = 0.154$ nm) in the step-scan mode, with 2 θ angles ranging from 5° to 60°, with a step of 0.04 and a scanning time of 5.0 min.

The crystallinity index was calculated from the height of the (200) peak (I_{200} 2 $\theta = 22^\circ$) and the minimum intensity between the (200) and 110 peaks (I_{am} 2 $\theta = 18^\circ$), using the Segal method,²³ where I_{200} represents the crystalline region and I_{am} represents the amorphous material.

RESULTS AND DISCUSSION

In this study, three materials: potato starch, ethyl hydroxyethyl cellulose and CNC, were used as consolidation materials for treating papyrus strips. Each treatment was applied on a separate group of samples and the effects were compared among the groups, as well as with the control for each group. The starch solution was applied by two methods: the classic method, using a brush, and using an ultrasonic humidifier for applying the treatment as a starch mist. Ethyl hydroxyethyl cellulose and

CNC solutions were applied on the papyrus strips using a brush. Measurements and tests were conducted to evaluate the efficiency of the two methods for applying the starch treatment, as well as that of the consolidation materials used for strengthening the papyrus samples.

Characterization of CNC

Firstly, CNC was prepared from dissolved pulp by controlled acid hydrolysis. This process removes the amorphous region from the cellulose fibers and reduces the particle size from micro-scale to nano-scale. The internal structure of the prepared CNC was examined by TEM, FT-IR and XRD analyses, as shown in Figure 2. The preparation and characterization of CNC were reported and described in detail in our previous work.^{21,24,25}

IR spectroscopy of papyrus strips

IR spectroscopy was carried out on each group of papyrus strips before and after ageing, as well as on their respective control samples. The papyrus mainly consists of cellulose, with 54-60%, lignin, with 23-24%, and hemicelluloses. These proportions depend on the source of the plant and may vary even within a single slice of the papyrus plant.^{26,27}

The carbonyl group and its absorption range ($1600-1850\text{ cm}^{-1}$) can be used for monitoring the degradation of cellulose, as the decomposition of cellulose can occur by oxidation, causing an increase in the number of carbonyl groups. The infrared spectra of the control papyrus strips before and after thermal ageing showed the characteristic peaks of the functional groups of cellulose, hemicelluloses and lignin,²⁸ with slight changes after thermal ageing. The IR spectra of the papyrus consolidated with starch and the control strips after thermal ageing showed slight changes appearing between the two methods of application of the treatment, as a decrease in the intensity of absorption of OH, CH, CH₂ and an increase in the intensity of C=O due to the oxidation process caused by thermal ageing (Fig. 3 (A, B)).

The spectra of the papyrus samples consolidated with ethyl hydroxyethyl cellulose and CNC and their control strips after thermal ageing showed the frequency of the absorption in the two substances approximating that of the control strips after ageing at 1730 cm^{-1} , with a decrease in the intensity of C=O in the strips treated with both consolidation materials (Fig. 4 (A, B)).

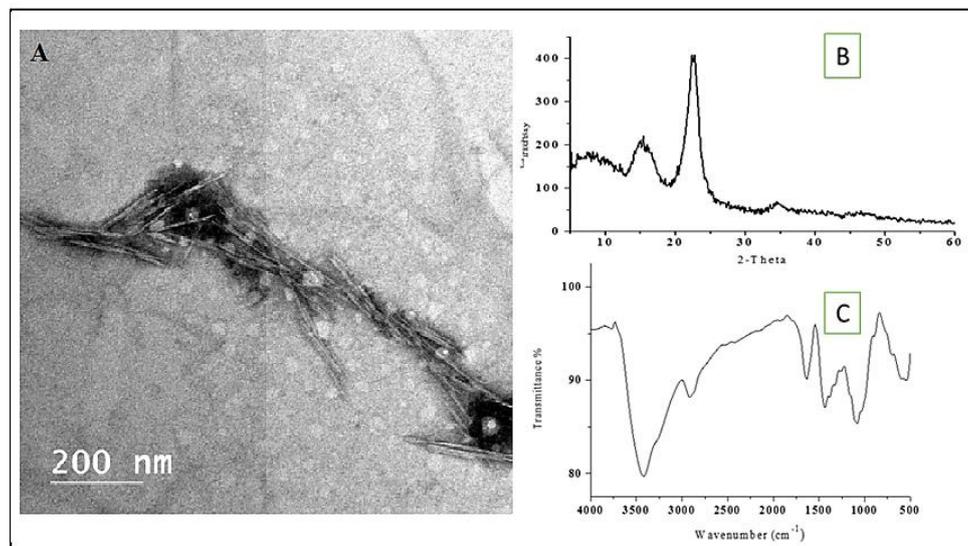


Figure 2: TEM image (A), XRD (B) and FT-IR (C) of the prepared CNC

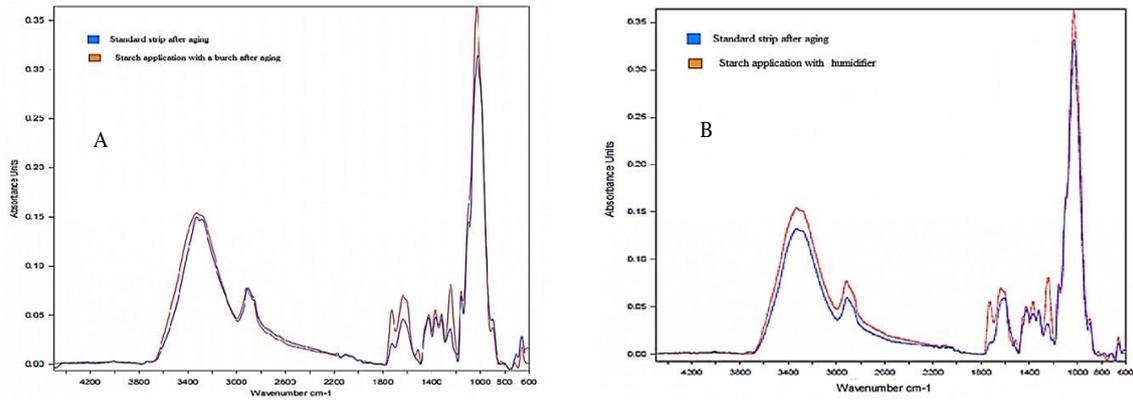


Figure 3: FT-IR spectra of the control and consolidated strips with starch applied by brush (A) and using the ultrasonic humidifier (B) after thermal aging

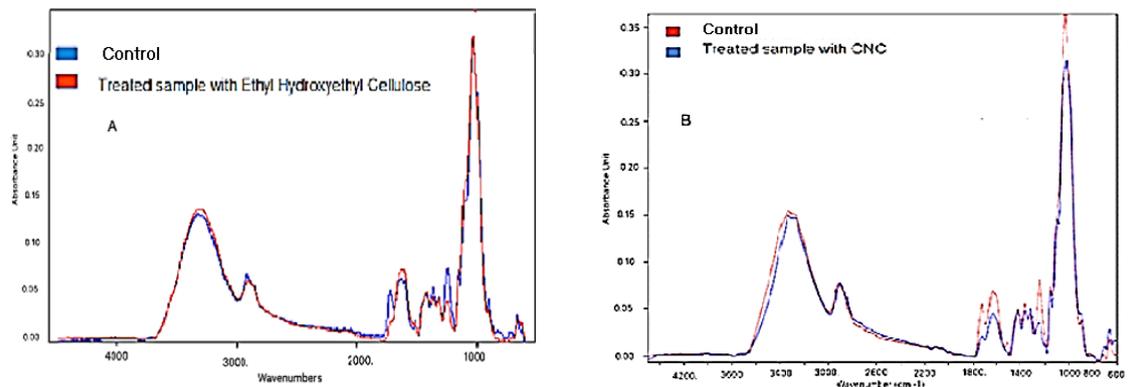


Figure 4: FT-IR spectra of the control and consolidated strips with ethyl hydroxyethyl cellulose (A) and with CNC (B) after thermal aging

Scanning electron microscopy (SEM)

Scanning electron microscopy (SEM) was used for monitoring the effects of thermal ageing on the surface morphology of the control papyrus samples and those consolidated with starch, ethyl hydroxyethyl cellulose and CNC.

The SEM image of the control sample before thermal ageing shows a clear cellular arrangement in the papyrus structure, while after thermal ageing, its surface exhibits cracks in different places and fibers appear weak and irregular because of changes in the cellular arrangement (Fig. 5).

Figure 6 presents the SEM images of the papyrus consolidated with starch applied by brush before and after thermal ageing. The images reveal starch granules between the cells, smooth cell walls before ageing, with some changes in the shape of the fibers after ageing. The changes result from the heating effect and the deposition of starch granules between fibers and on their surfaces.

The SEM of the papyrus consolidated with starch with the help of the ultrasonic humidifier before thermal ageing showed good dispersion of the starch into the fibers, good coverage of the fiber surface, soft and clear cell walls. After thermal ageing, the cell walls were still coated with starch and appeared smooth (Fig. 7).

The image of the papyrus treated with ethyl hydroxyethyl cellulose before ageing showed that the consolidation material penetrated well into the papyrus cells, the cells appeared soft, without asperities, indicating good coating of the cells with the ethyl hydroxyethyl cellulose. After the exposure to thermal ageing, the uniform shape of the cells was significantly maintained and the cell walls appear well coated (Fig. 8).

Before thermal ageing, the CNC formed a thin layer on the surface of the fibers, without interfering with the cellular arrangement, and the cells appeared well-aligned. After thermal ageing, in the SEM of the papyrus sample treated with CNC, the thin layer of CNC may still be seen on the surface of the fibers, still revealing the cellular arrangement, with a regular cell appearance and good cohesion among the cells (Fig. 9).

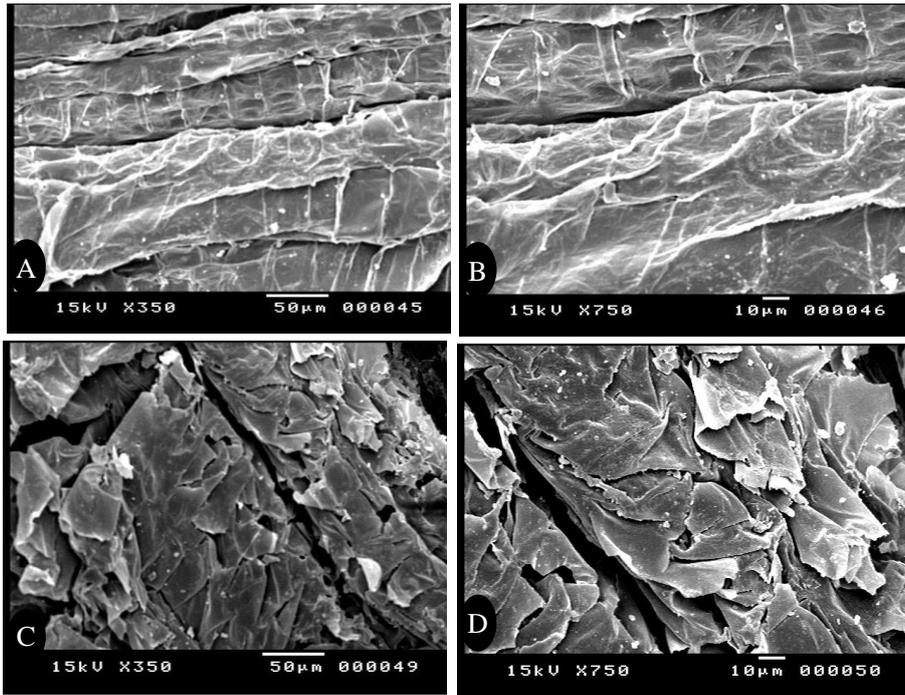


Figure 5: SEM images of control papyrus strips before (A, B) and after (C, D) thermal ageing

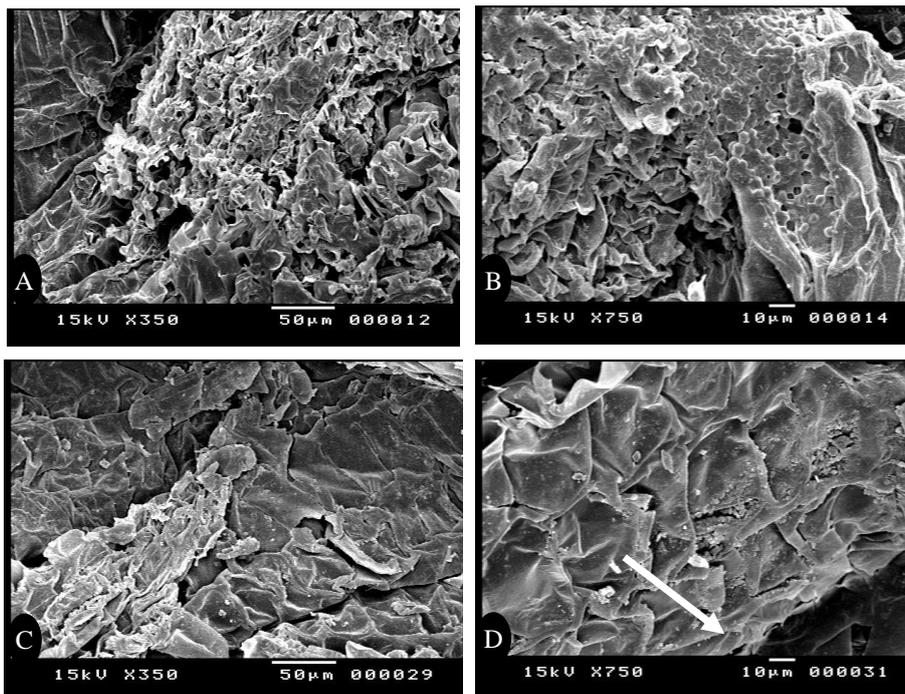


Figure 6: SEM images of papyrus strips consolidated with starch applied by brush before (A, B) and after (C, D) thermal ageing

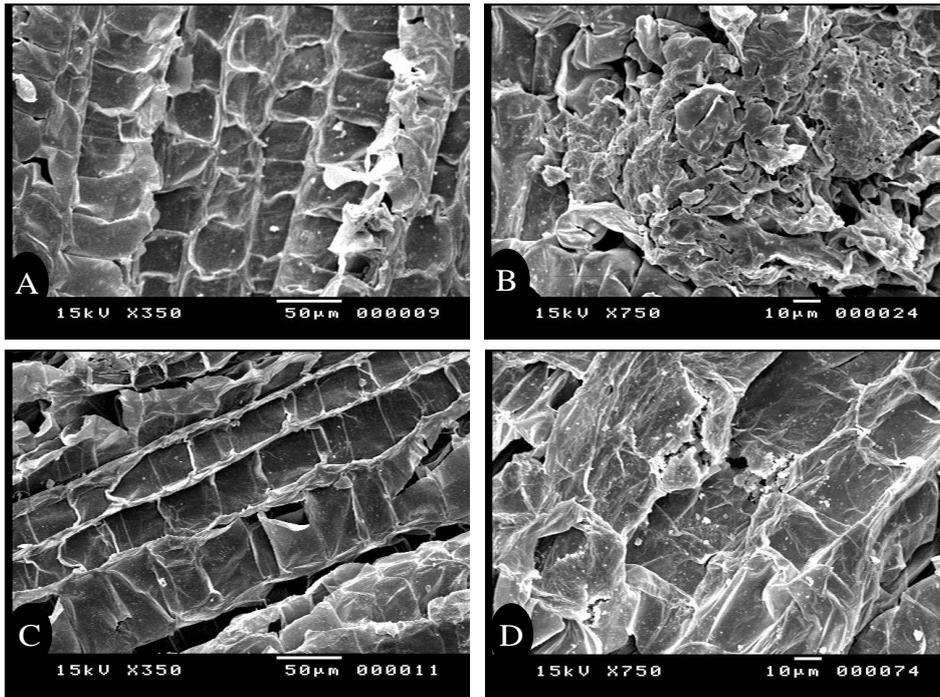


Figure 7: SEM images of papyrus strips consolidated with starch applied by the ultrasonic humidifier before (A, B) and after (C, D) thermal aging

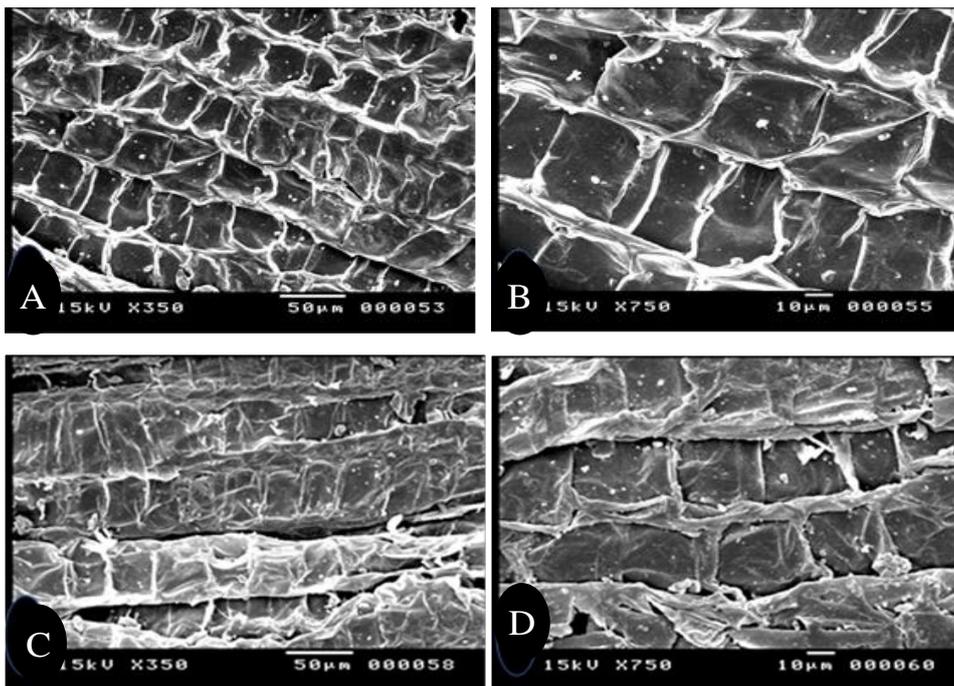


Figure 8: SEM images of papyrus consolidated with ethyl hydroxyethyl cellulose before (A, B) and after (C, D) thermal aging

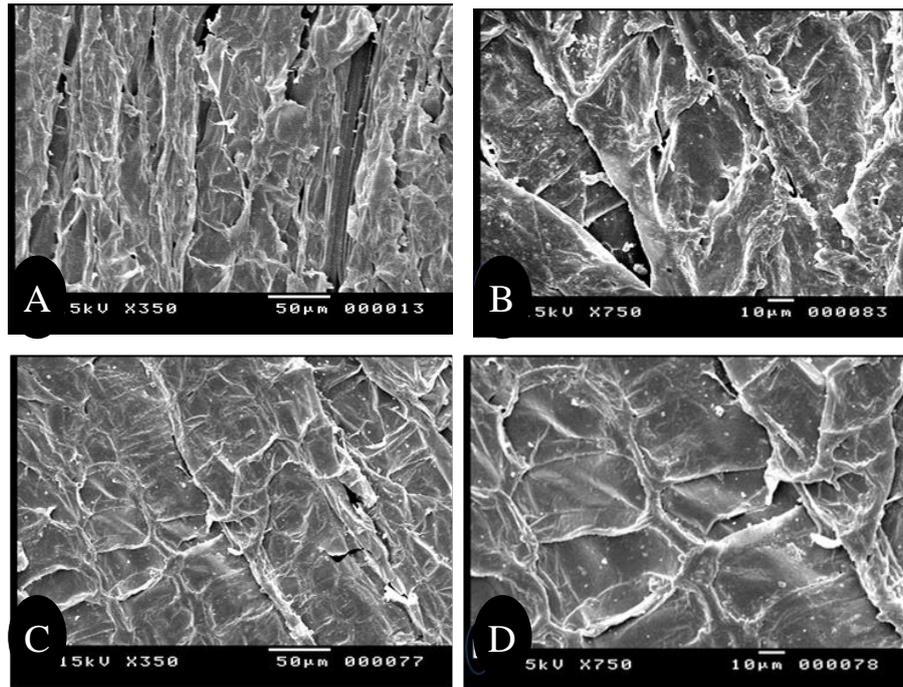


Figure 9: SEM images of papyrus strips consolidated with CNC before (A, B) and after (C, D) thermal aging

Mechanical properties of papyrus strips

The mechanical properties of the papyrus samples were evaluated by measuring the tensile strength and elongation of the treated and untreated papyrus for determining the efficiency of the studied consolidation materials during thermal ageing.

Samples treated with starch

The data presented in Table 1 reveal that, after thermal ageing, the values of tensile strength decreased for all the strips consolidated with starch, by both methods, while the elongation ratios decreased for some of them, but increased for others.

The percentage loss of tensile strength of the samples was calculated as follows: Percentage loss in tensile strength = $\frac{\text{Tensile strength before ageing} - \text{tensile strength after ageing}}{\text{Tensile strength before ageing}} \times 100$. Lower values of percentage loss in tensile strength after thermal ageing indicate a more successful consolidation method.

The measurement of tensile strength and elongation of the control strips before aging gave values of 55.7 N/mm² and 1.32% for tensile strength and elongation, respectively. After conducting the thermal ageing, the tensile strength decreased to 44.35 N/mm², while the elongation ratio – to 1.17%. The percentage loss for these properties after thermal ageing recorded for the control sample was 20.3% and 0.15%, respectively.

Similarly, the tensile strength of the strips consolidated with 0.5% starch (applied by brush) was 60.1 N/mm² and after ageing – 48.7 N/mm², while for the concentration of 1%, the tensile strength was 63.4 N/mm² before thermal ageing and 51.4 N/mm² after thermal ageing. As regards the elongation of the samples treated with 0.5% starch, it reduced by 0.09%, while for those treated with 1% starch, it decreased by 0.12%. The percentages losses in the tensile strength and elongation of the reinforced samples at the two concentrations are lower or close to those of the control sample, which means that the starch treatment applied by brush did not significantly improve the mechanical properties of the reinforced samples.

The other alternative for applying the starch solution on the papyrus investigated here was using the ultrasonic humidifier. For the samples treated in this way with the 0.5% starch solution, the loss in the tensile strength of the strips after the thermal aging was of 10.08%, while for the concentration of 1%, the loss in the strength of the strips after aging was of 26.3%. Also, the elongation percentage of the samples subjected to the 0.5% starch treatment reduced by 0.04%, while for the concentration of 1%, it reduced by 0.07%. These results reveal that the strips consolidated by the 0.5% starch treatment

applied using the ultrasonic humidifier have higher mechanical properties after aging, compared to those to which the treatment was applied by brush and with a higher concentration of the starch solution.

Samples treated with ethyl hydroxyethyl cellulose

The second material used to strengthen the papyrus strips, in this study, is ethyl hydroxyethyl cellulose, with concentrations of 0.5% and 1%. The tensile strength of the papyrus treated with the 0.5% ethyl hydroxyethyl cellulose solution, before and after thermal ageing, was 78.3 N/mm² and 73.4 N/mm², respectively, with a loss in tensile strength of 6.25%. In the case of the 1% ethyl hydroxyethyl cellulose treatment, the tensile strength of the papyrus strips was 90.5 N/mm² and 81.1 N/mm², before and after thermal ageing, respectively, with a loss in tensile strength of 10.38%.

The loss in elongation for the strips consolidated with 0.5% ethyl hydroxyethyl cellulose after ageing was of 0.06%, close to that recorded for the samples treated with 1% ethyl hydroxyethyl cellulose – of 0.04%. As can be remarked, the treatment with ethyl hydroxyethyl cellulose has improved the mechanical properties of the consolidated papyrus strips in terms of tensile strength and elongation. Both concentrations of the consolidation material led loss ratios significantly lower than those determined for the control samples.

Samples treated with cellulose nanocrystals

When cellulose nanocrystals, in a concentration of 0.5%, were applied to strengthen the papyrus samples, the tensile strength of the samples was determined as 73.5 N/mm² before aging and 62.55 N/mm² after aging, with a loss in tensile strength of 14.89%. Further, the application of the 1% CNC solution led to tensile strength values of 80.3 N/mm² and 66.42 N/mm², before and after aging, respectively, with a loss of 17.28%. The elongation values of the strips consolidated with 0.5% CNC solution after aging decreased by 0.06% and in the case of the 1% CNC concentration – by 0.05%.

These results revealed that the CNC improved the mechanical properties of the consolidated strips in terms of both tensile strength and elongation ratio. Thus, according to the percentage loss in tensile strength of the samples, it can be concluded that the reinforcement materials can be arranged in the following order, by their efficiency: 0.5% ethyl hydroxyethyl cellulose > 1% ethyl hydroxyethyl cellulose > 0.5% CNC > 1% CNC > 0.5% starch > 1% starch. Otherwise said, the results demonstrate that the most efficient treatment that successfully strengthened the mechanical properties of papyrus strips was the one with ethyl hydroxyethyl cellulose, followed by those with CNC and starch applied using the ultrasonic humidifier device.

Table 1
Tensile strength and elongation of control and consolidated papyrus strips before and after thermal aging

No	Treatment	Before aging		After aging	
		Tensile strength (N/mm ²)	Elongation ratio (%)	Tensile strength (N/mm ²)	Elongation ratio (%)
1	Control sample	55.7	1.32	44.35	1.17
Starch solution applied by brush					
2	0.5% starch	60.1	1.140	48.7	1.05
3	1% starch	63.4	1.20	51.4	1.08
Starch solution applied using an ultrasonic humidifier					
4	0.5% starch	56.5	1.188	50.8	1.15
5	1% starch	69.9	1.170	51.5	1.10
6	Ethyl hydroxyethyl cellulose (0.5%)	78.3	1.26	73.4	1.20
7	Ethyl hydroxyethyl cellulose (1%)	90.5	1.309	81.1	1.265
8	CNC (0.5%)	73.5	1.27	62.55	1.21
9	CNC (1%)	80.3	1.29	66.42	1.24

Effect of consolidation materials on color changes of papyrus strips

Effect of starch

The treatment with the lower concentration of starch had no significant negative effects on the color changes of the samples, but a change in color was detected with increasing the starch concentration. Similar results in terms of color changes were obtained after the starch treatments, regardless of the application method (Table 2).

Effect of ethyl hydroxyethyl cellulose

While the treatment with ethyl hydroxyethyl cellulose had an excellent impact on the mechanical properties, compared to the control and to the other consolidation materials, it had a similarly positive effect on optical properties, as a very little color change was observed on the samples after ageing, even less than that of the control. Even when a higher concentration of the ethyl hydroxyethyl cellulose was applied, the impact on color change was insignificant (Table 2).

According to the results listed in Table 2, the values of L^* decreased after conducting the thermal aging study, while the values of b increased, *i.e.* the color of the papyrus strips tends to yellow after aging. Considering the ΔE values of the strips strengthened at both concentration levels and comparing them with those of the control, it was noted that the two concentrations gave the same ΔE value, which is lower than that for the control. Considering the high strengthening effect of the ethyl hydroxyethyl cellulose treatment, as well as the low ΔE values, it appears that ethyl hydroxyethyl cellulose can be recommended as an efficient consolidation material for papyrus.

Effect of CNC

While contributing to excellent results in terms of mechanical properties, compared to the control, the treatment with CNC was observed to have a negative effect on optical properties, causing a noticeable chromatic change after the aging process. The thin layer of CNC observed by SEM on the surface of the papyrus fibers may explain the apparent color change (Table 2).

Table 2
Effects of starch, ethyl hydroxyethyl cellulose and CNC on color changes of papyrus samples after thermal ageing

No	Treatment	After			Before			ΔE
		L^*	a^*	b^*	L^*	a^*	b^*	
1	Control	85.37	1.29	15.52	84.39	0.32	14.38	1.79
Starch solution applied by brush								
2	Starch (0.5%)	84.59	1.04	14.26	85.04	-0.59	13.06	2.07
3	Starch (1%)	80.23	2.41	15.94	85.15	0.00	13.81	5.87
Starch solution applied using an ultrasonic humidifier								
4	Starch (0.5%)	85.28	0.65	14.26	85.76	0.03	13.25	1.28
5	Starch (1%)	81.59	0.50	15.26	84.00	0.21	14.85	2.46
6	Ethyl hydroxyethyl cellulose (0.5%)	87.15	1.11	12.49	87.77	0.63	12.23	0.82
7	Ethyl hydroxyethyl cellulose (1%)	86.72	1.02	13.94	87.31	0.55	13.62	0.82
8	CNC (0.5%)	86.43	1.01	13.92	85.03	-0.63	11.02	3.61
9	CNC (1%)	85.56	1.03	14.25	86.70	-0.55	11.19	3.63

CONCLUSION

In the present study, three consolidation materials were investigated for strengthening papyrus strips, namely potato starch, ethyl hydroxyethyl cellulose and cellulose nanocrystals. The color changes and the mechanical properties, *i.e.* tensile strength and elongation, of the samples were evaluated before and after thermal ageing of the samples to determine the efficiency of the proposed solutions and their suitability as consolidation materials for the conservation of papyrus artifacts.

The findings of the study revealed that CNC and ethyl hydroxyethyl cellulose improved the mechanical properties of the tested strips most significantly. Applying potato starch by means of an ultrasonic humidifier device improved the mechanical properties as well, but less efficiently. As

regards the impact of the applied solutions on the optical properties of the samples, the treatment with ethyl hydroxyethyl cellulose had the most negligible color change values. Considering the high strengthening effect of the ethyl hydroxyethyl cellulose treatment, as well as the low ΔE values it produced, it can be concluded that ethyl hydroxyethyl cellulose can be recommended as an efficient consolidation material for papyrus.

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