



## Consolidation of Archaeological Papyrus: Evaluation of Selected Materials

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### Abstract

Ancient papyrus sheets contain valuable information from old civilizations. Consolidation process is a vital step in restoration process of archaeological papyri to strengthen the papyrus fibers and saves archaeological papyri for the next generations. This research assessed the effect of Funori, Isinglass, Starch, Hydroxypropyl cellulose, and Methyl cellulose in treatment of modern papyrus samples. The evaluation was conducted through applying the selected materials on papyrus sheets surface by brush and examine the sheets before and after artificial accelerated ageing. The effectiveness was investigated by measuring the mechanical properties of papyrus fibers such as tensile strength and elongation as well as study the color change using CIELAB system. Moreover, Scanning Electron Microscope was used to investigate the surface morphology of treated samples before and after ageing. The results revealed that Hydroxypropyl cellulose and Methyl cellulose achieved a better penetration and distribution in papyrus structure leading to fibres protection after thermal ageing. Moreover, only a slight colour change in papyrus surface was observed comparing with the other materials. However, starch has the lowest efficacy in papyrus consolidation.

Keywords: Papyrus; consolidation; SEM; colour change; mechanical properties; artificial ageing.

### 1. Introduction

Papyrus plant stem was used in making writing support using peeling [1], [2] or strips [2]–[4] methods. The oldest blank papyrus sheet was found in tomb of Hemaka at Saqqarah, dates back to the first dynasty, and it is about 2900 B.C., while the earliest inscribed one dates to 2500 B.C. was found at the Red Sea port of Wadi el-Jarf that dates to the fourth dynasty [5], [6].

Papyrus sheets stored in unsuitable conditions are exposed to oxidation, hydrolysis, photo-deterioration and biodeterioration [7]–[9], as a result, papyrus sheets need to be consolidated to prevent further damage to the papyrus sheets and their inks. The consolidation material has to be chemically stable, does not discolor with ageing, does not affect the object's colour, non-toxic [10]–[12], affordable and readily available.

Different materials were tested previously to be used as a papyrus consolidant; a comparative study carried out by Brooklyn Museum to select the suitable material for papyrus consolidation revealed that Funori can consolidate paints or papyrus support without causing darkening on both of them [4]. Leach,

B and Green, L 1995 claimed that Isinglass is a successful consolidant for painted areas on papyrus during removal unsuitable backing from archaeological papyri [13]. The biodegradability of some papyrus consolidants was evaluated, and the result showed that a biocide has to be added to the consolidants in order to inhibit biodegrading ability [14]. Conducting consolidation treatment under magnification saves weak parts and avoids losing it, sodium carboxymethyl cellulose (SCMC) was also used for consolidation of two fragile papyri at Trinity College Library Dublin, Ireland [15]. Recently, Hydroxypropyl cellulose loaded with Zinc Oxide nanoparticles (ZnO) was tested as a consolidant and it protected the papyrus structure from degradation of artificial ageing [16].

This research aims to assess the efficiency of Funori, Isinglass, Hydroxypropyl cellulose, Methyl cellulose, and starch in consolidation of modern papyrus, in order to select the most appropriate materials for treating archaeological papyri.

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## 2. Experimental

### 2.1. Materials

2.1.1. Papyrus samples: Consolidation treatment was carried out on blank modern papyrus samples from Al-Qaramous, Abu-Kbir, Alshrqia, Egypt as this village has become a commercial centre for making papyrus for tourism purposes.

2.1.2. Funori (0.5 %): Is a polysaccharide mucilage made from the seaweed *gloiopeltis*, which is harvested from natural populations in Japan [17], was purchased from TALAS, USA. 1.5 g Funori was cut from dried seaweed web (Fig.1) and was soaked in 300 ml distilled water overnight. After that, the mixture was put in a double boiler pan over low heat until the seaweed is dissolved without boiling. The resulted solution was left to cool at room temperature and was strained through a cotton cloth.

2.1.3. Isinglass (2 %): Is a pure collagen from dried sturgeon bladders of the highest quality, blood and rust free was obtained from TALAS, USA (Fig.1) 4 g Isinglass was cut and soaked in 200 ml distilled water overnight to soften and swell and the same steps of preparing Funori were followed.

2.1.4. Hydroxypropyl Cellulose HPC (2 %): Is also known as Klucel G delivered from Sigma (Sigma–Aldrich, Germany). 4g Klucel G was dissolved in 200 ml ethanol and the mixture was stirred until obtaining a homogeneous solution.

2.1.5. Methyl Cellulose MC (0.5 %): obtained from (Sigma–Aldrich, Germany). 1 g of Methyl cellulose was soaked in 100 ml distilled water overnight, and after that 100 ml ethanol was added to the mixture with stirring.

2.1.6. Starch (1 %): was purchased from El-Gomhouria Co. For Trading Drugs, Chemicals & Medical Supplies, Cairo, Egypt. 1 g starch was added to 100 ml water, and the mixture was stirred and also was put on over heat until bubbling and left to cool. Brush was used to apply the tested consolidants on papyrus samples surfaces.

### 2.2. Methods

Two groups of papyrus samples were prepared. The first one was subjected to an accelerated thermal ageing at 105 °C, for 216 h that equivalent to about 75 years of ageing under normal conditions according to ISO 5630-1:1991 [18], [19]. The aim of heat ageing is to estimate the long-term effect of the consolidation, accelerated heat ageing was used for untreated and treated samples.

The second group was subjected to light ageing by exposure to artificial daylight lamp (MLphilips500 w, 220-230 v) for 200 h [8], one strip was set aside as a control sample to monitor the colour change of the samples after light ageing. The assessment process was conducted using the following methods;

2.2.1. Scanning Electron Microscopy (SEM): Philips Environmental Scanning Electron Microscope XL30 (ESEM) at the central lab of the National Research Centre in Giza, Egypt, was used to examine the surface morphology of papyrus samples before and after applying each resin, as well as after thermal ageing in order to follow any changes in the surface of the tested samples. The samples were coated with gold to avoid charging.

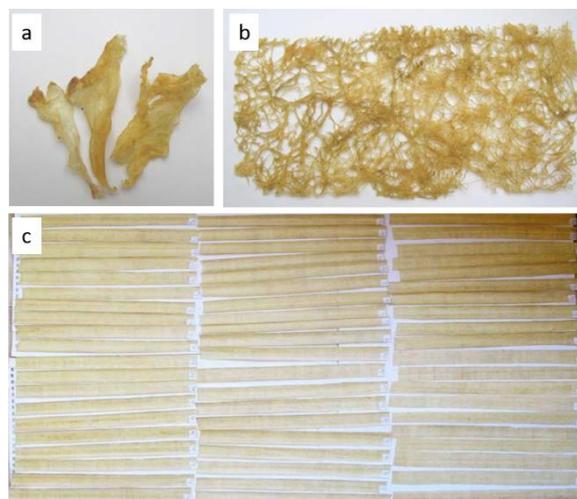


Fig.1. (a) Isinglass, (b) Funori, (c) Papyrus strips.

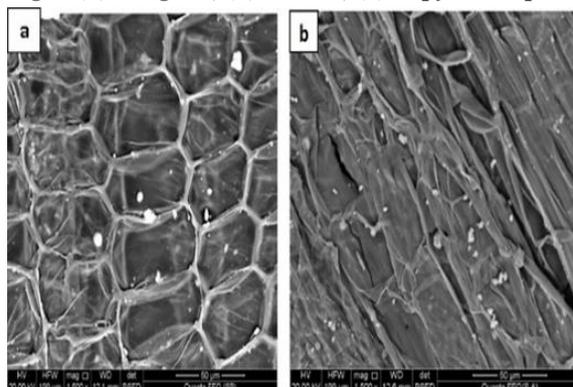
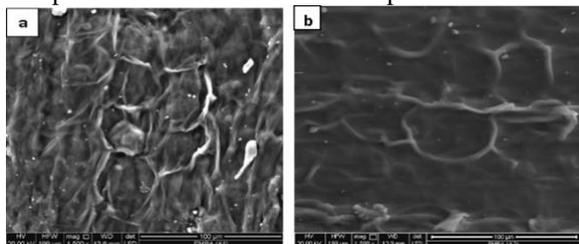


Fig.2. SEM pictures of controlled sample of papyrus surface without any treatment before (a) and after (b) thermal ageing

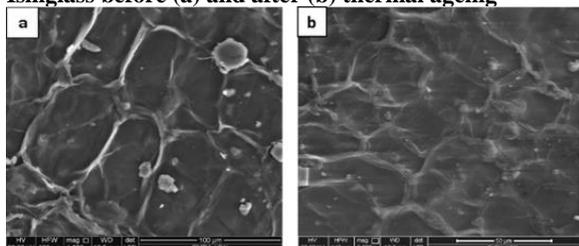
2.2.2. Color measurements: The color changes of papyrus samples with size 8 × 10 cm were measured using Optimatch spectrophotometer 3100® in the National Institute of Standards (NIS), Giza, Egypt. Results were expressed in CIELAB colour space L, a, and b; L index of color represents black-to-white color, a index represents green-to-red color, and b index represents blue-to-yellow color. Three measurements were performed for each specimen and the average value was taken.

2.2.3. Mechanical properties: Mechanical parameters including tensile strength and elongation were measured at the National Institute of Standards (NIS), Giza, Egypt according to the ASTM.D828-97 using Tinius Olsen H5KT machine. The papyrus sheets were

cut in the machine direction to strips with dimensions 25.4 cm × 2.55 cm and the measurements were made before and after treatment and after heat aging compared to that of the control sample.



**Fig.3.** SEM pictures of the papyrus surface with: Isinglass before (a) and after (b) thermal ageing



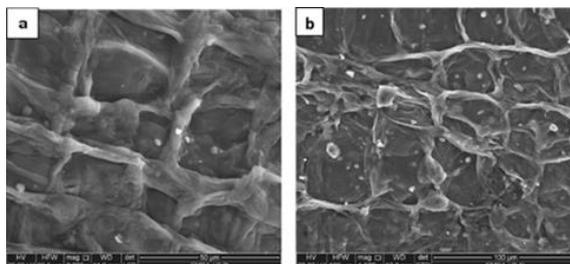
**Fig.4.** SEM pictures of the papyrus surface with: Funori before (a) and after (b) thermal ageing

*Results and discussion*

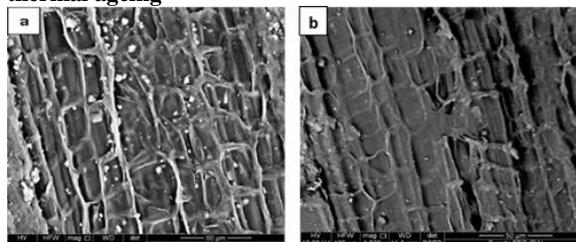
2.3. Visual examination: The tested samples were examined by naked eye before and after treatment, and also after light and thermal ageing. It is observed that no marked change in colour has been observed after applying the materials on papyrus surface, However, a gloss has been observed on samples treated with Isinglass after ageing, and samples treated with starch became brittle and dry after ageing and this result agreed with Menei, E 2015, who claimed that wheat starch cannot be used on consolidation because it does not have sufficient qualities of flexibility [20].

2.4. Scanning Electron Microscopy (SEM) observations: Examination by Scanning Electron Microscope was used to study the penetration and distribution of the tested materials within the papyrus structure, as well as the ability of these materials to consolidate and protect papyrus samples from degradation after heat ageing. It can be seen that heat ageing caused breakages in papyrus fibers of untreated papyrus and samples appeared to be very fragile. SEM showed the greatest effectiveness of consolidation by HPC (Fig.5); it covered papyrus with a higher efficiency and penetrated easily into the papyrus fibers structure. Isinglass showed low penetration inside papyrus fibres, and after ageing, it caused darkening the papyrus surface and covered the cells shape. Funori achieved more penetration than Isinglass, and saved the cells shape. However, there is breakage on some cell walls. Samples treated with Methyl Cellulose showed high penetration inside the cellulose structure and it was noticed limited degradation on the cell walls in some parts. Although Starch grains exist

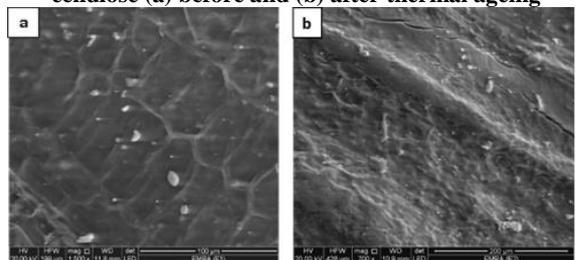
naturally in papyrus plant and was used as adhesive of strips during making papyrus sheets [20], it did not give acceptable result in our experimental study. The fiber structure completely disappeared after coating with starch and the samples became cockled after ageing and had cracks.



**Fig. 5.** SEM pictures of the papyrus surface with: Hydroxypropyl cellulose before (a) and after (b) thermal ageing



**Fig.6.** SEM pictures of the papyrus surface with Methyl cellulose (a) before and (b) after thermal ageing



**Fig.7.** SEM pictures of the papyrus surface with Starch (a) before and (b) after thermal ageing

2.5. Colour change measurements

Color parameters L, a, and b of the treated samples before and after light ageing were measured and the total color differences ( $\Delta E$ ) was calculated according to the following relation[21], [22].

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

The measurement of color values indicated that the  $\Delta E$  values of untreated samples are higher than  $\Delta E$  of the blank ones due to light fading of the samples, and the role of the consolidant in preventing degradation of fibers. It is observed that L values (lightness) of samples treated with Funori, Starch, MC and HPC decreased after light ageing, in contract these values increased in samples treated with Isinglass. The lowest degree of colour change was noticed in the sample treated with Isinglass followed by MC, Starch, Funori, while the highest colour change value was in sample

treated with HPC. Isinglass and Starch have clear drawbacks in treated samples; Isinglass caused shining in the samples and the starch lead to sever dryness and shrinkage. MC achieved the best result for saving samples from discoloration and did not cause any dry for the samples (Table.1), (Fig.8).

## 2.6. Mechanical properties

The values of tensile strength and elongation are shown on (Table. 2).

It is noticeable that samples treated with MC gave the highest value of tensile strength followed by Isinglass, HPC, and Funori. On the other hand, the lowest value of tensile strength was recorded by starch. After thermal ageing, the mechanical properties of all treated and untreated samples had decreased. The percentage of loss of tensile strength and elongation can be calculated from the equation below.

$$\text{Loss in tensile strength (\%)} = \frac{(\text{Tensile strength before aging} - \text{Tensile strength after aging})}{(\text{Tensile strength before aging})} \times 100$$

Funori gave a loss in tensile strength 51.42 %, while Isinglass showed a loss in tensile strength 37.83 %. MC, HPC and starch recorded 35.98 %, 24.61 %, 41.05 % respectively. The ascending order of the samples according to the loss in tensile strength as follows:

hydroxypropyl cellulose < standard sample < methyl cellulose < Isinglass < starch < Funori. Important observation was noticed, the standard sample tensile strength was 238.9 N/mm<sup>2</sup>, and changed to 157.9

**Table (1) Color parameters data of papyrus samples before and after light ageing.**

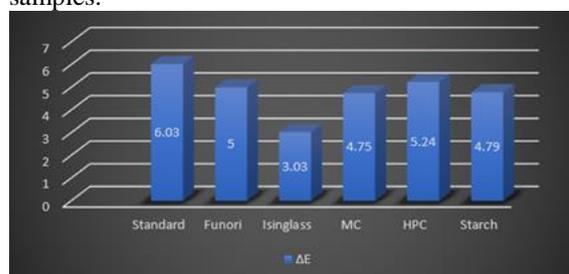
Selected materials	Before ageing			After ageing		
	<i>l</i>	<i>a</i>	<i>b</i>	$\Delta l$	$\Delta a$	$\Delta b$
Standard	78.08	-0.38	23.49	78.96	1.15	17.77
Funori	77.83	1.72	26.94	77.07	1.72	26.94
Isinglass	76.36	2.81	29.69	76.78	2.83	26.69
Methyl cellulose	77.89	-0.08	21.84	75.87	3.26	19.14
Hydroxypropyl cellulose	78.62	0.59	18.81	65.75	2.95	15.19
Starch	75.30	0.36	25.87	73.44	3.34	22.61

**Table (2) Mechanical properties of papyrus samples**

Selected materials	Before ageing		After ageing		
	Tensile strength (N/mm <sup>2</sup> )	Elongation ratio (%)	Tensile strength (N/mm <sup>2</sup> )	Elongation ratio (%)	
Standard	238.9	5.06	157.9	4.36	
Funori	161.2	4.76	78.3	1.9	
Isinglass	235	6.25	146.09	4.44	
Methyl cellulose	241.8	6.56	154.8	3.81	
Hydroxypropyl cellulose	175.5	4.74	132.3	3.25	
Starch		142.5	4.12	84	2.78

N/mm<sup>2</sup> after aging with an average loss in tensile strength 33.9 %, which guided us to avoid consolidation treatment as we can and use it if it is necessary and the case of the object is required.

Regarding the elongation, the results revealed that all aged consolidated samples showed a significant decrease in elongation after aging. Treated sample with Isinglass gave the lowest loss on elongation 28 % compared to the other samples, while treated sample with Funori gave the highest values for loss in elongation. starch, and HPC gave values 41.92 %, 32.52 %, 31.43 %. It is observed that Funori recorded the highest loss in tensile strength and elongation, and it did not improve the mechanical properties or protect papyrus fibres from degradation, so it should not be used in papyrus consolidation. Although Isinglass showed the lowest loss in tensile strength after aging, it caused shining to papyrus surface. We can conclude that HPC followed by MC recorded the best values in improving the mechanical properties of papyrus samples.



**Fig. 8. Colour difference (ΔE) of selected consolidants before and after exposure to light ageing.**

### 3. Conclusions

This work presented results of an experimental comparative study of using five materials: Funori, Isinglass, HPC, MC, and Starch in papyrus consolidation. Analytical techniques including (Color measurement, Scanning Electron Microscope (SEM), and mechanical properties) were used to evaluate the effect of selected consolidants of aged and non-aged modern papyrus samples. The results proved that starch did not give any acceptable and the treated samples lost its flexibility while cellulose derivatives, which have chemical similarity with papyrus, proved to be very efficient for the consolidation of papyrus samples as the mechanical properties of the tested samples were improved and no marked change in colour has been observed.

### 4. Conflicts of interest

“There are no conflicts to declare”

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