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Evaluation of Hydroxypropyl Cellulose, Zinc Oxide Nanoparticles and Nanocellulose for Tracing Papers Consolidation

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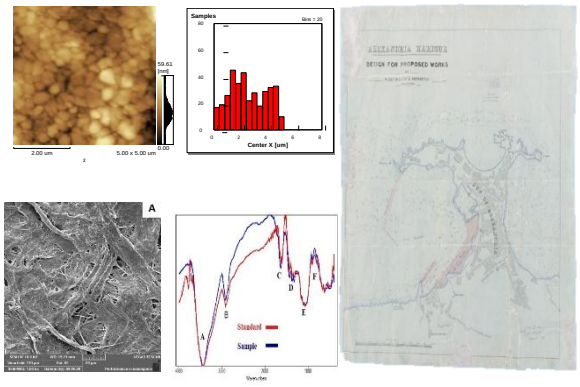
HIGHLIGHTS

- Evaluating the effects of consolidation materials on tracing paper.
- The evaluation method used was by SEM, tensile strength, elongation and FTIR spectroscopy.
- The hybrid mixture of hydroxypropyl cellulose and nanocellulose was one of the best materials to consolidate tracing paper.
- The hybrid mixture gave the best results to treated tracing paper.

GRAPHICAL ABSTRACT



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ABSTRACT

The research aims to evaluate the efficiency of some consolidation materials for tracing paper; hydroxypropyl cellulose (Klucel-E), zinc oxide nanoparticles and nanocellulose. The consolidated materials were used independently, and as hybrid mixtures to improve their properties.

The prepared nanomaterials were examined with an Atomic Force Microscope (AFM), and the materials used to consolidate the tracing paper samples after artificial aging were evaluated. Evaluation methods included investigation of the surface morphology by Scanning Electron Microscope (SEM), measurement of mechanical properties (tensile strength and elongation), and detection of chemical changes of the treated samples before and after thermal ageing at 80°C and 65% relative humidity by Fourier transform infrared spectroscopy (FTIR) analysis.

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Based on the results of the examinations, consolidation by the hybrid mixture consisting of hydroxylpropyl cellulose and nanocellulose proved to be successful; giving the best results when this hybrid mixture was applied on tracing paper samples. This mixture was distinguished by its ability to spread easily inside the paper, giving greater strength to the binding, without forming a film on the surface of the paper and causing stiffness or opacity to treated tracing paper. Therefore, this hybrid mixture was used to consolidate an archaeological tracing map at the Egyptian Geographical Society dating back to (1807 AD).

1. Introduction

The term "tracing paper" is currently used as a general label to designate transparent papers. It has been used in technical and engineering drawings, architectural plans and geographic maps, embroidery as well as jewelry [1]. This paper was available soon after the production of machined plain paper, to investigate a new way to produce paper [2]. At the beginning of the nineteenth century, tracing paper was manufactured by the fibers of cotton and linen and then the wood pulp was utilized. This was before the wax paper appeared; where the paper had been coated with oils such as linseed, almond and poppy seed, varnish such as dammar and sandarac or glue prior to thinning and beating, which led to render this paper transparent [3]. In the middle of the nineteenth century, tracing paper called “vegetable parchment paper” appeared and was produced by the acidic treatment of the paper with sulfuric acid to separate the thick fibers and then treat them physically by using the diluted ammonium hydroxide bath [4], in order to remove air bubbles inside the pulp, which causes opacity, composition and calendaring in the paper to make the paper surface delicate and smooth [5]. At the end of the nineteenth century a new type of the tracing paper appeared called “Paper from overbeaten pulp”. All of the above-mentioned types of paper differ according to the refraction of light to achieve the transparency of paper and depend on the diffusion of light on the surface of paper, which leads to the refraction of light of the paper fibers and the transparency of paper appears. Paper has been impregnated with resins and oils to achieve another additional transparency when exposed to air [6]. Due to the fragility and transparency of the tracing paper, they are subjected to more damaging factors in comparison to synchronized non-transparent paper [7]. The resin materials and the added oils contribute during the process of manufacturing of transparent paper to deterioration, when the factors that assist deterioration are present. These materials begin to function as fatty carboxylic acids and increase acidity and acidic dissolution. The acids make paper affected by treatments. These acids react with alcohols. As a result, important esters are formed to react with oils and other resins and oils are shaped. Thus, paper becomes brittle with time. Moreover, overall discoloration can vary from a pale yellow to a dark brown [8]. Transparent papers are subject to biological deterioration because of a main factor related to tracing paper is called mechanical preparation of paper pulp. There is a relationship between the potent of micro-organisms to digest enzymatically cellulose and overbeating. As paper is more vulnerable whenever vegetable cellulosic fibers are overbeaten, this makes a large area of these fibers exposed to damage [9]. Biological damage of tracing paper results in spots deforming the paper surface because of the secretion of colored pigments on the paper surface. These spots do not result in the compositional deterioration of paper. Additionally, there is another type of biological damage that leads to physical changes due to the chemical transfer that occurs as a result of the secretion of enzymes capable of digesting paper cellulose. This transfer affects the chemical and physical properties of paper, including reduction of mechanical factors [10]. Although these factors vary between mechanical, chemical, physical and biological factors; they can change the properties of this paper, whether separately or combined, limiting its capability of permanence and stability [11]. Therefore, the consolidation processes are key treatments that preserve tracing papers as they increase the survival and sustainability

of the paper [12]. They also improve the physical and mechanical properties of paper without causing any changes in its chemical composition whether during paper treatment or after aging. Moreover, consolidation does not cause any changes in the morphology, degree of opacity, luster or loss of the transparency of paper [13]. This treatment depends on selecting the consolidation material. Therefore, the aim of this paper is to examine the effect of consolidation materials on transparent papers which is achieved by applying hydroxypropyl cellulose and comparing it with nanomaterials such as zinc oxide nanoparticles and nanocellulose, and by using a hybrid mixture of hydroxypropyl cellulose and zinc oxide nanoparticles, and hydroxypropyl cellulose and nanocellulose in order to improve the properties of both materials in the hybrid mixture.

2. Materials and Methods

2.1. Materials

2.1.1. The Tracing map at the "Egyptian Geographical Society" in Cairo

dates back to 1807 AD, its dimensions are 70 cm width X 77 cm length. Due to its large size, it has been poorly archived and was folded several times, which caused a lot of damage to the map, especially creases, wrinkles and deterioration.

2.1.2. Zinc Oxide Nanoparticles

were prepared of dissolving 10,975 g of zinc acetate in 90 ml ethanol in a magnetic stirrer (500 RPM) at room temperature and dissolving 6,335 g of oxalic acid dehydrate in 100 ml ethanol in a magnetic stirrer. Followed by adding zinc acetate dissolved in a water bath and the oxalic acid drop-by-drop, mixed by magnetic stirring (500 RPM) at room temperature. This was followed by adding two drops of dilute hydrochloric acid composed of 1.6 ml of HCl diluted in 50 ml of distilled water, to raise the pH of the composed solution, and heated up to 80°C for three hours [14]. A gelatinous solution was formed and left to cool, and then it was put in a crucible at the temperature of 550°C for three hours. Finally, a white powder of zinc oxide nanoparticles is formed after they were roasted and crushed.[15]

2.1.3. Nanocellulose

was composed by adding 5 g of cotton linter cellulose to 100 ml sulfuric acid (concentration 60%), followed by acid hydrolysis at 50°C with continuous magnetic stirring for 90 minutes [16], after which the reaction was stopped by adding distilled water on the above-mentioned liquid then left to cool at room temperature.

A centrifuge was used to remove the excessive acid from the disintegrated core and extract the nanocellulose by washing the core with water several times to reach a pH of 7. Afterwards, the ultrasonic vibrator was used on the extracted nanocellulose for five minutes to turn it into the form of nanofibrils [17].

2.2. Methods

2.2.1. Examination of Nanomaterials

Zinc oxide nanoparticles and nanocellulose were examined by Atomic Force microscope at the Micro Analytical Center, Faculty of Science, Cairo University, Egypt. Device specifications Model SpM9600- Wet (Scanning Probe microscope Shimadzu made in Japan.

It was revealed through the examination that the size of nanoparticles of zinc oxide was 59.61 nm and the size of nanocellulose particles was 26.78 nm; and the particles were similar and distributed uniformly.

2.2.2. Preparation of the used consolidation materials

The 1% hydroxypropyl cellulose was prepared by weighing 1 g of the material to 100cm³ of ethyl alcohol. The nanocellulose and the zinc oxide nanoparticles were prepared by weighing 1 g of each material to 100cm³ of isopropyl alcohol. The hybrid materials were prepared by mixing hydroxypropyl cellulose with the nanocellulose with a ratio of (1:1) and mixing the hydroxypropyl cellulose with the zinc oxide nanoparticles (1:1) using a magnetic stirrer to properly distribute and mix the composed materials.

2.2.3. Application of the different consolidation materials

Tracing paper samples (3 X 15 cm²) were prepared and placed put on a flat surface. The consolidation materials were applied with a clean soft brush on the surface of the samples according to the above mentioned previously-prepared materials.

2.2.4. Accelerated artificial aging

The aging process was conducted in an oven "BINDER 924030000200 / code: NIS-pMTL-NIS-11" at the "National Institute for Standards – NRC" Haram – Giza – Egypt. The aging standard specification conducted was according to CIE Standard [18]. Moist heat treatment at 80 °C and 65% relative humidity paperback was performed for three days. Paper samples without aging were kept aside for comparison studies with the aged samples that had not been treated. Consolidation processes were performed on some aged samples by using the previously-prepared materials.

2.2.5. Analytical techniques

2.2.5.1. Tensile strength and elongation

The test was conducted by (H5KT) device at the "National Institute for Standards – NRC" Haram – Giza – Egypt at an average speed of 25 mm/minute, and the space between the device's jaws was 5 cm; the width of the sample measured 3 cm and the length was 15 cm. An average of five measurements of each sample were taken for tensile strength and elongation.

2.2.5.2. Scanning Electron Microscope (SEM) investigation

The samples were coated with gold and then examined by using the Scanning Electron Microscope equipped with an EDX unit (JEOL JSM S400 LV EDX Lin ISIS – OXFORD High Vacuum) at the "National Research Center" Dokki, Giza. The aim of the investigation by SEM was to monitor the changes in the surface morphology of the treated samples before and after artificial accelerated ageing, and the objective of this examination was to assess the changes in the fibers which occurred as a result of the accelerated artificial aging process.

2.5.3. Fourier Transform Infrared Spectroscopy (FTIR) examination

Paper samples were analyzed by using the "Nicolet 380 FT-IR" device at the "National Institute of Standards" Haram – Giza– Egypt. The aim of this analysis was to study the chemical changes of the cellulose functional groups, by comparing between the intensity and absorption spectrum bands of the cellulose functional groups in both of the standard and treated samples at the wavenumber of (400-4000 cm^{-1}) [19].

3. Results and Discussion

3.5. Mechanical properties (tensile strength and elongation)

It is the crunch force at which a strip of paper breaks off and is wide 1.5 cm, and the tensile strength in the longitudinal direction is greater than in the transverse direction, due to the arrangement of the fibers in the longitudinal direction with a density greater than the transverse direction [20].

Tensile strength (N/mm 2)

-The results of the tracing paper samples treated with hydroxypropyl cellulose (Klucel-E) showed an increase in the tensile strength compared to the control sample. The average tensile strength of the standard sample was (3.04 N/mm2), while the average tensile of the treated samples increased to (12.92N/mm2); and hardening of the treated samples was observed. After aging it showed a decrease in the tensile strength (11.34 N/mm^2) with the occurrence of hardening as well.

-Samples treated with the hybrid mixture of hydroxypropyl cellulose and zinc oxide nanoparticles before aging recorded average values of tensile strength (8.18 N/mm^2)، and after aging a slight increase with the occurrence of hardening of the treated paper samples was recorded (8.64 N/mm^2).

-The samples treated with nanocellulose did not record any increase in the average values of tensile strength compared to the standard sample, where it was recorded (3.04 N/mm^2) before aging, and after aging (3.01 N/mm^2).

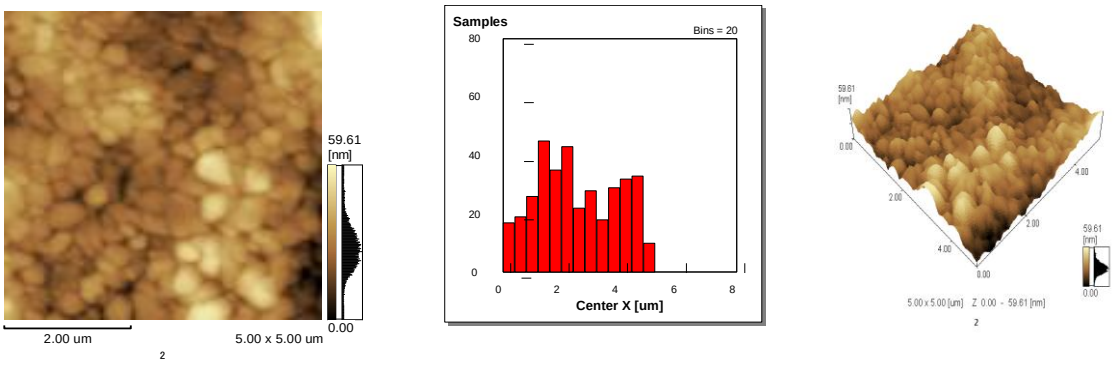


Fig 1: 2D and 3D images particles of zinc oxide

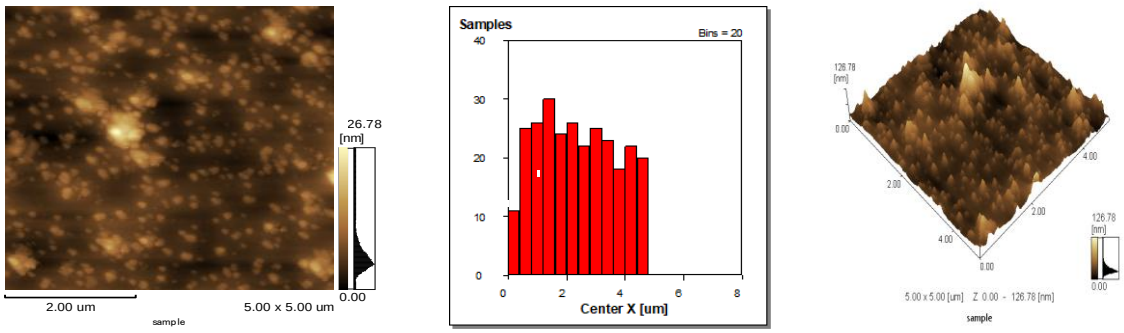


Fig 2: 2D and 3D images particles of nanocellulose

Table. 1: Tensile strength and elongation of treated tracing paper samples with consolidants used before and after artificial accelerated ageing

Samples	Tensile strength (N/mm^2)	Elongation (%)
control sample	3.04	0.307
(Untreated Samples)	2.41	0.269
Aged Control Sample	12.92	0.511
(Untreated Samples)	11.34	0.39
Treated samples with hydroxypropyl cellulose	3.04	0.065
Aged Treated samples with hydroxypropyl cellulose	3.01	0.381
Treated samples with nanocellulose	4.19	0.838
Aged treated samples with nanocellulose	3.66	0.844
Treated samples with zinc oxide nanoparticles	8.18	0.980
Aged treated samples with oxide nanoparticles	8.64	0.994
Treated samples with a mixture of hydroxypropyl cellulose & zinc oxide nanoparticles	5.21	0.936
Aged treated samples with a mixture of hydroxypropyl cellulose & zinc oxide nanoparticles	5.85	0.938

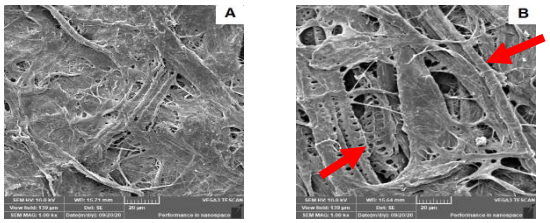
-Samples treated with zinc oxide nanoparticles showed a slight increase in tensile strength at (4.19 N/mm^2) before aging, and after aging (3.66 N/mm^2) was recorded.
-Samples treated with the hybrid mixture of hydroxypropyl cellulose and nanocellulose recorded the best results with an increase in tensile strength before aging (5.21 N/mm^2) and after aging (5.85 N/mm^2)، while the treated papers retained their elasticity and no stiffness occurred compared to the tracing papers treated with other materials .

Elongation (%)

-The results of the tracing paper samples treated with a mixture of hydroxypropyl cellulose and zinc oxide nanoparticles showed an increase in the average elongation before aging (0.980%) and after aging (0.994%) in comparison with the control samples before aging (0.307%)..
-The results of the tracing paper treated samples with a mixture of hydroxypropyl cellulose and nanocellulose showed an increase in the average elongation before aging (0.936%) and after aging (0.938%).
-The tracing paper treated samples with zinc oxide nanoparticles before aging recorded (0.838%) and after aging (0.844%).
-The treated samples with nanocellulose recorded before aging (0.065%) and after aging (0.381%)
-The results of the treated samples with hydroxypropyl cellulose showed an average elongation before aging (0.511%) and after aging (0.390%).
It is noticed that the treated samples with hydroxypropyl cellulose and treated samples with nanocellulose after aging were affected by heat due to the loss of the fibers' water content causing a decrease in the elongation; while the treated samples with zinc oxide nanoparticles and treated samples with a mixture of hydroxypropyl cellulose and nanocellulose or hydroxypropyl cellulose and zinc oxide nanoparticles the ratio of elongation was approximately constant.

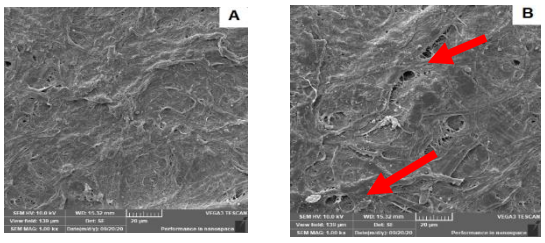
3.6. Investigation of the surface morphology by SEM

The Investigation of the tracing paper samples treated with consolidation materials has shown an improvement in the surface morphology of the cellulose fibers in comparison with the control sample, as there were spaces and gaps between their fibers, especially after the aging process (Fig. 3).



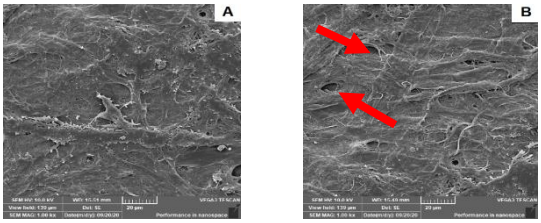
(Fig. 3): SEM micrographs for the surface morphology of the control

(A) The untreated standard sample before aging (1000 x); the fibers appear incoherent with gaps between them. (B) The untreated standard samples after aging (1000 x); the arrows indicate the separation of fibers due to the water loss and exposure to high temperatures, which cause the fibers to separate and form gaps. The examination of the samples treated with hydroxypropyl cellulose before aging has shown a good material coverage and fiber coherence, but after the aging process; the examination has shown embrittlement in the treated samples.



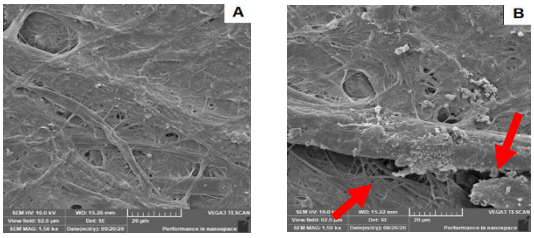
(Fig. 4): SEM micrographs for the surface morphology of the tracing paper samples treated with hydroxypropyl cellulose

(A) The treated sample before aging (1000 x) shows good material coverage and fibers coherence and consistency. (B) The treated samples after aging (1000 x) show gaps between the fibers in the treated samples.
The examination of the samples treated with the nano materials zinc oxide nanoparticles and nanocellulose has shown good results; as the fibers were supported because the nano particles settled in the gaps, causing an increased coherence of the fibers.



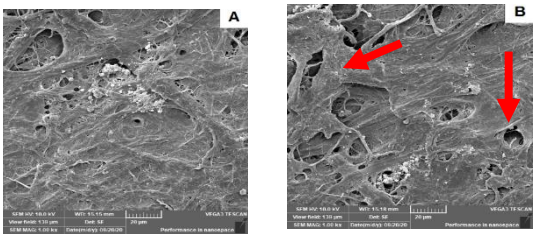
(Fig. 5): SEM micrographs for the surface morphology of the tracing paper samples treated with zinc oxide nanoparticles

(A) The treated sample before aging (1000 x) shows a very good material coverage and an increase in fibers coherence, which indicates a high level of consolidation. (B) The treated samples after aging (1000 x) show that the treatment material has maintained the fibers consolidation and consistency, which indicates a high level of fibers coating and coherence.



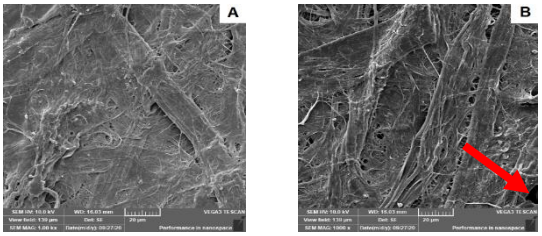
(Fig. 6): SEM micrographs for the surface morphology of the tracing paper samples treated with nanocel-

(A) The treated sample before aging (1500 x) shows a good surface coverage and a high level of fibers coherence and consistency. (B) The treated samples after aging (1500 x) show that the treatment material has maintained the fibers coherence.



(Fig. 7) SEM micrographs for the surface morphology of the tracing paper samples treated with a mixture of hydroxypropyl cellulose and zinc oxide nanoparticles

(A) The treated sample before aging (1000 x) shows fibers coherence and consistency with some small gaps. (B) The treated samples after aging (1000 x); the arrows indicate some gaps between the fibers.



(Fig. 8): SEM micrographs for the surface morphology of the tracing paper samples treated with a mixture of hydroxypropyl cellulose and nanocellulose

(A) The treated sample before aging (1000 x) shows a very good surface coverage. (B) The treated samples after aging (1000 x) show very good results; as the fibers are coherent and bonding.

3.7. Fourier Transform Infrared Spectroscopy (FTIR) Analysis

The results of FTIR analysis for tracing paper samples treated with consolidation materials compared to the standard sample is shown in (Fig .9) (table 2).

1) The treatment with hydroxypropyl cellulose (Fig. b) and also nanocellulose (Fig. c) caused decrease in the absorption spectrum bending C-H (1425 cm^{-1}), and stretching C-O (1150-1160 cm^{-1}) which indicates a change in the degree of crystallization and polymerization of cellulose .

2) No change occurred in the cellulose functional groups in the samples treated with zinc oxide nanoparticles (Fig. d).

3) The treatment with the hybrid mixture of hydroxypropyl cellulose and zinc oxide nanoparticles caused very slight changes that occurred in the cellulose water content, polymerization and crystallization levels. (Fig. e). A slight change occurred:

• in the spectrum breadth of stretching O-H (3300-3400 cm^{-1})، due to the effect of the used solvent on the cellulose water content .

• in the absorption intensity of the C-H stretching (2800-3000 cm^{-1})، due to the slight change of the water content of the paper cellulose .

(Table. 2) FTIR analysis for tracing paper standard sample

Symbol	Functional group	Wave number (cm^{-1})	Assignment
A	Stretching O-H	3300-3400	Characteristic of water content of cellulose
B	Stretching C-H	2800-3000	
C	Stretching C=O	1640-1660	Characteristic of water content and oxidization of cellulose
D	Bending C-H	1425	Characteristic of crystallization of cellulose
E	Stretching C-O	1000-1300	Characteristic of the polymerization of cellulose
F	Stretching C-O-C	820-850	Characteristic of cellulose

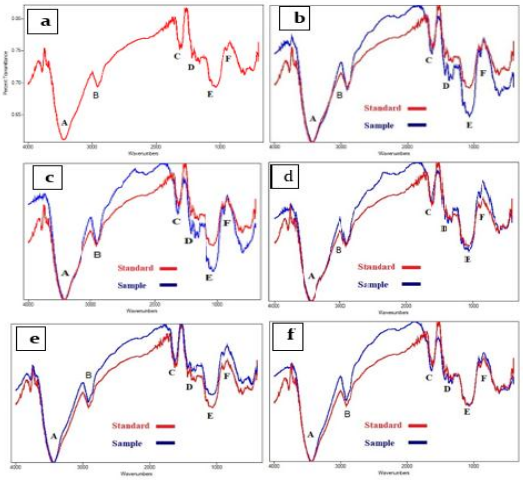
• in absorption spectrum of the C=O stretching (1650 cm^{-1})، C-H bending (1425 cm^{-1})، C-O stretching (1150-1160 cm^{-1}) and stretching C-O-C (856 cm^{-1}) .

4) The treatment with the hybrid mixture of hydroxypropyl cellulose and nanocellulose caused very slight change in the cellulose water content and polymerization level (Fig. f) as follows:

• a slight change in absorption spectrum at the C=O stretching (1652 cm^{-1})، C-H bending (1425 cm^{-1})

• C-O Stretching (1150-1160 cm^{-1}) .

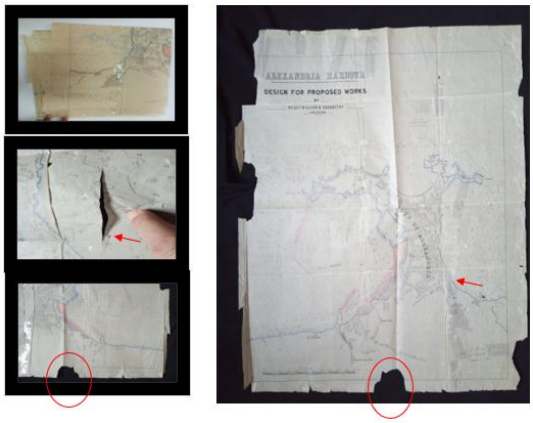
• No change in C-O-C the stretching (856 cm^{-1}).



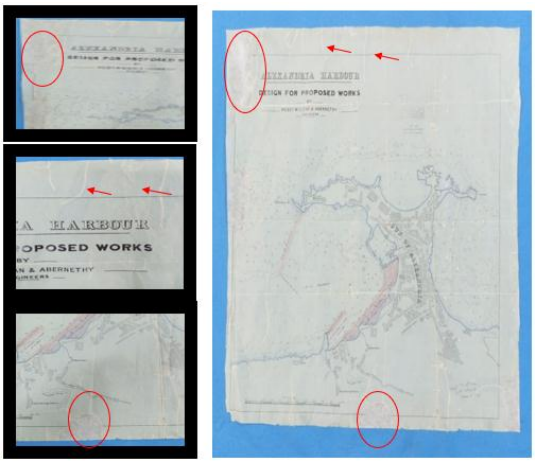
(Fig. 9) FTIR analysis for tracing paper sample, (a) standard, (b) sample treated with hydroxypropyl cellulose, (c) sample treated with nanocellulose, (d) treated with zinc oxide nanoparticles, (e) treated with hydroxypropyl cellulose and zinc oxide nanoparticles, (f) hydroxypropyl cellulose and nanocellulose.

4. Application of the selected consolidation treatment material on an archeological map:

The archeological map was damaged, had folds, cuts, lost parts and inappropriate previous conservation with transparent adhesive tape. According to the previously mentioned results that proved the success of using the mixture consisting of hydroxypropyl cellulose and nanocellulose, this treatment material was used to consolidate an archeological tracing map at the "Egyptian Geographical Society" in Cairo (fig.10). The treatment was performed by putting the map between two insulation organza sheets to support the map paper; then the consolidation material with 1% of the hybrid mixture of hydroxypropyl cellulose and nanocellulose was applied on the surface using a soft brush. The map was left in room temperature and under light pressure until it was completely dry for 1 day; then the organza sheets were removed. (fig.11)



(Fig. 10) The archeological map (before consolidation)



(Fig. 11) The archeological map after consolidation

Conclusion

• The results of examining the mechanical properties (tensile strength & elongation) of the treated tracing paper samples showed that hydroxypropyl cellulose gives the consolidated tracing paper good mechanical properties, but after aging the paper became dry, started splitting and gained a degree of opacity .

• The results of the scanning electron microscope (SEM) showed that the treatment with the hybrid mixture of hydroxypropyl cellulose and nanocellulose is characterized by its ability to easily distribute within the paper; giving it more binding force and improvement of the flexibility degree of the treated samples .

• The selected hybrid consolidation material hydroxypropyl cellulose and nanocellulose did not form a film on the paper surface. Thus, it does not cause any loss of the paper flexibility or transparency.

• The consolidation process helps provide this type of tracing paper with the sufficient ability to be handled and prepared for exhibition or storage .

• The mixture of hydroxypropyl cellulose and nanocellulose (Klucel-E) was one the best materials applied to consolidate tracing paper because it is characterized by spreading fast inside the tracing paper, which resulted in a heavy-duty link between the fibers of the paper.

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