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Evaluation the Using of Eco-friendly Materials in Treatment of the Structure of Archaeological Wood

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Abstract

Archaeological wood, particularly architectural wood, is susceptible to numerous deterioration factors due to exposure to environmental conditions, resulting in rapid decay. Therefore, the search for and enhancement of new eco-friendly materials, sourced from renewable and readily available resources, which are cost-effective and low in toxicity, represent a contemporary approach to preserving wood structures. This paper introduces eco-friendly materials such as hydroxypropyl cellulose (HPC), Nano cellulose (NC), and Nano Clay (Halloysite) (NH), derived from renewable natural sources. These materials can be utilized to formulate mixtures compatible with wood, treating its structure at various concentrations. The Klucel nanocomposite is developed to enhance the properties of hydroxypropyl cellulose (HPC) using nanomaterials. Mixtures of (HPC) 1% / (NC) 3-5% and (HPC) 1% / (NH) 3-5% were prepared and applied to naturally aged samples of a hardwood type, specifically beech wood. The efficiency of these materials was assessed by measuring the mechanical and optical properties of the treated samples, along with infrared FTIR, X-ray diffraction (XRD), and scanning electron microscope (SEM) examinations, to determine the optimal mixture. The results indicate the superiority of the mixture (HPC) 1% / (NC) 3% in enhancing properties without altering the chemical composition of the wood even after aging.

Keywords: Structure- Eco-friendly materials - Klucel - Nano-cellulose - Nano-clay (Halloysite)

Introduction

Since the dawn of civilization, man began the process of exploring nature, discovering the significance of trees, which have played a vital role in daily life, particularly through the utilization of wood.[1] Wood is considered one of the basic materials that played a major role in human history. It has been used in arts and architecture since prehistoric times, continuing through historical epochs up to the present day.[2] This may be due to its physical, mechanical, thermal, acoustic, and electrical properties, [3] facilitating the multiple uses of wood in the manufacture of wooden antiques. Wood, derived from organic plant matter, is porous and hygroscopic, possessing the ability to absorb and retain moisture.[4]

Furthermore, it is an easy-to-form material that responds to external influences and undergoes various forms during growth.[5] Wood is composed of several basic compounds: cellulose: 40-50%, hemicellulose: 25-35%, and lignin: 16-33%. [6] These polysaccharide carbohydrates are primarily made up of cellulose fibers, which have the symbol ($C_6H_{10}O_5$) n, and are considered the primary structural component of all plant cell walls. [7] The chemical compounds of wood are distributed within the cell wall (cell wall) in a non-uniform manner throughout the layers of the cell wall. [8] Cellulose is one of the most abundant organic materials in the plant kingdom. Cellulose is the main component of the plant cell wall, that is, it is the nucleus of the wood structure and plays the role of the structural structure of the xylem tissue [9] . It is the main factor affecting the strength of the wood and the tensile strength in the direction of the axis, due to the arrangement of each cell. This arrangement stems from microfibrils and chains of cellulose molecules within the S2 layer of the secondary cell wall, aligned in the direction of the tree axis, [10]

The occurrence of any damage or deterioration to cellulose directly affects wood. Over time, antique woods, particularly architectural elements like ceilings and lintels, are exposed to various environmental factors such as heat, humidity, and light, which gradually degrade the wood.[11] These damage factors affect the chemical composition of the wood, especially cellulose, leading to chain breakage and disintegration, thereby weakening the mechanical and physical properties of the wood. [12] This loss of resilience makes the wood vulnerable to further deterioration, which leads to the collapse of the wood over time as a result of its weak structure. Therefore, it is necessary to find solutions for consolidating and treating weak wood structures[13]of the lintel by using Klucel nanocomposite that can improve the lintel's resistance to environmental conditions.[14] Several studies have explored the use of nanomaterials and their mixtures in treating organic antiques, [15] especially archaeological wood. David, M. E.,et al. 2020 discussed the utilization of nanomaterials in the treatment and restoration of cultural heritage,[16] while Darwish, S. S., et al. 2020 , [17] Afifi, H.et al. 2021 [18] , and Hassan, R. R. et al 2021 [19] used a traditional compound such as hydroxypropyl cellulose loaded with nanomaterials for treating organic antiques. Hamed, S. A. et al 2019 the efficacy of hydroxypropyl cellulose mixed with Nano-cellulose in different concentrations for

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treating archaeological wood, yielding promising results [20] . This study focuses on the use of eco-friendly materials to treat archaeological wood, aiming to address the weak structure of such wood and enhance its ability to withstand surrounding environmental factors. This study utilized the compound hydroxypropyl cellulose, which is derived from cellulose and created by the reaction of alkaline cellulose with perbalene oxide at high temperature and pressure. [21] It is commonly used by a restoration specialist for wood restoration at archaeological sites due to its good properties.[22] Hydroxypropyl cellulose can be enhanced by incorporating materials with nano properties and preparing Klucel nanocomposite,[23-19], which increases the compound's ability to penetration, spread and resist the environmental factors to treat the weak structure of archaeological wood.Klucel nanocomposite is composed of mixing hydroxypropyl cellulose at a fixed concentration of 1% with the addition of a natural and environmentally friendly nanomaterials such as Nano-clay (Halloysite). Nano-Halloysite, a type of Nano-clay containing elements like aluminium silicates, magnesium, iron, calcium, and potassium, is very effective in treating and consolidating archaeological wood.[24],[25] Cavallaro, G., et al 2017 and Cavallaro, G, et al 2018 discussed the use of Nano- Halloysite as a good material in the consolidation of weak wood,[26][27] and also with the use of Nano-Halloysite by adding another substance or compound in the consolidation of wood.[28] Nano Cellulose is also used in the work of Klucel nanocomposite, and Nano-Cellulose is a material produced from cellulose materials and used in the preservation of cultural heritage,[29] especially consisting of cellulose materials such as archaeological wood because of its good features that treat and consolidation weak wood. [30] This study addressed hydroxypropyl cellulose at 1% concentration and added nanomaterial (Halloysite-cellulose) at 3-5% concentration for a homogeneous mixture with high penetration, prevalence and tolerance properties, to find good solutions in treating and consolidation the weak structure of archaeological wood. The efficacy of these materials can be assessed by applying them to old beech wood samples mimicking lintels, followed by various tests to evaluate mechanical, crystal, optical, and surface properties. This rigorous evaluation aims to identify the optimal mixture that delivers positive results without adverse effects, ensuring the safe treatment and consolidation of archaeological wood.

Martials and methods

2.1. Wood Samples

Wood samples were obtained from beech wood, a type of hardwood commonly used in historical lintels dating back to the year 1759. Beech wood samples were prepared, which mimic archaeological lintels, were naturally aged and located in the same place as the archaeological lintel. The samples were meticulously prepared according to British standard specifications, with dimensions of 2 x 2 x 2 cm, in preparation for evaluating the efficiency of the treatment materials on archaeological wood structures.

2.2. Preparing Consolidation Materials

Hydroxypropyl cellulose (HPC) was selected as the primary material at a concentration of 1%, with the addition of nanomaterials to form Klucel nanocomposite. To obtain 3% concentrations of HPC) 1% / (NH) 3% and (HPC) 1% / (NC) 3% nanocomposites, Klucel nanocomposite solutions were prepared by the addition of 0.03 g of each clay or cellulose nanoparticle separately into 100 ml Ethanol. Each solution was mixed for about 10 min; then, 1.0 g of Klucel E powder was added and mixed vigorously and the mixture was sonicated for 15 min to obtain 3% concentrations of (HPC) 1% / (NH) 3% and (HPC) 1% / (NC) 3% nanocomposites.To obtain 5% concentrations of (HPC) 1% / (NH) 5% and (HPC) 1% / (NC) 5% nanocomposites, Klucel nanocomposite solutions were prepared by adding 0.05 g of each clay or cellulose nanoparticle separately into 100 ml Ethanol. Each solution was mixed for about 10 min; then, 1.0 g of Klucel E powder was added and mixed vigorously and the mixture was sonicated for 15 min to obtain 5% concentrations of (HPC) 1% / (NH) 5% and (HPC) 1% / (NC) 5% nanocomposites.

2.3. Application and Ageing of Consolidation Materials

The materials used to treat the structure of the wood were applied using a brush, considering the accuracy of the application on the samples in a good and even manner. Consistency in application was maintained by simultaneously applying materials to the samples under controlled conditions, with a room temperature set at 20°C ± 2°C and relative humidity at 55% ± 5%.[8] Drying for two weeks and exposure to thermal aging at a temperature of 80◦C and 65% relative humidity for 240 hours, and also to photoaging using ultraviolet rays for 240 hours [32] at the National Research Centre, After that, the samples are tested to assess the effectiveness of the materials used to treat and consolidate the structure.

2.4. Evaluation of the Efficiency of the Consolidation Treatment

2.4.1. Measurement of Colour Change

The total colour change (ΔE) of the treated samples was measured using an Optimatch 3100® device from SDL Company at the National Centre for Measurement and Calibration. This measurement was conducted after aging (both thermal and photo) and compared to the total colour change of the standard sample using the CIG lab system for measuring colour change. Colour system, the colour parameters L* (lightness), a* (redness), b* (yellowness), [33] were calculated using the following equation.[34]

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

2.4.2. FTIR Spectroscopy Analysis

Chemical changes in the functional groups of the wood (cellulose, hemicellulose, and lignin) were studied using a Spectrum Two FT-IR Spectrometer from PerkinElmer at Helwan University - Faculty of Science. Spectra were collected in the wavelength range of 400 to 4000 cm[35] .The intensity and areas of the absorption spectrum for different functional groups in the treated samples were compared with standard samples using the Essential FTIR 2023 program.

2.4.3. X-ray Diffraction Analysis

To study the degree of crystallinity of cellulose in the treated wood samples and compare it with the standard sample, a Bruker co D8 Discover Cu target device was utilized at the Metallurgical Research Centre - Tebin - Helwan. The analysis was conducted with a start angle of 10 degrees and an end angle of 40 degrees, using a wavelength of 1.54 Å, and operating conditions set at 40 mA and 40 kV. The data analysis was performed using the program Match 3.14 - 2023. The Segal method was employed for measurement, where the reflection intensity (I_{002}) at an angle of 22.6º and the diffraction height (I_{am}) at an angle of 18º were determined. The crystallinity percentage of cellulose (Cr) in the treated samples was calculated using the equation: $Cr = \times 100\ (I_{002}-I_{am})/I_{002}$. [36].

2.4.4. Scanning Electron Microscopy (SEM)

A scanning electron microscope, model JEOL GSM 5400LV EDX Link ISIS- Oxford, Assiut University, is used to evaluate the surface of the treated samples and the efficiency of the spread and penetration of materials by comparing the treated samples after aging with the standard sample. [37]

2.4.5. Compression Test

The AG-X autograph (Table – Top type – Shimadzu – japan) was used at the Grand Egyptian Museum. All measurements were carried out under specific operating conditions, with a temperature below 23 degrees and relative humidity between 50-60%, to measure the compressive forces on the treated samples after aging and compare them with standard samples

2.4.6. Bending Strength Test

The AG-X autograph Table-Top type device by Shimadzu in Japan was used at the Grand Egyptian Museum. All measurements were carried out under specific operating conditions, including a temperature below 23 degrees Celsius and a relative humidity between 50-60%. Samples with dimensions of 10 x 10 x 110 mm ± 2 mm were utilized to measure the bending resistance of the treated samples after aging and compare them with standard samples

Results and Discussion

3.1. Measurement of Colour Change

The samples treated with the Nano Klucel compound mixtures gave different results for the total colour change, ranging between 1.14 and 4.16 ΔE after aging and compared to the standard sample (Table 1). The sample treated with hydroxypropyl cellulose exhibited a total colour change of 4.16 ΔE, which is a noticeable colour change the naked eye. [38]Meanwhile, the sample treated with Klucel 1% / Nano cellulose 5% showed a total colour change of 3.64 ΔE, which is also a noticeable colour change to the naked eye due to the addition of Nano-cellulose 5% to hydroxypropyl cellulose. The sample treated with Klucel 1% / Nano cellulose 3% showed a total colour change 1.65 ΔE, which is unnoticeable and invisible to the naked eye,[39] due to the addition of Nano-cellulose 3% to hydroxypropyl cellulose, enhancing its penetration. The sample treated with Klucel 1%/Nano clay 3% showed a total colour change of 1.58 ΔE, which is an unnoticeable change and is not visible to the naked eye due to the compound's ability to resist aging. The sample treated with Klucel 1%/Nano clay 5% showed a total colour change of 1.14 ΔE, which is also invisible to the naked eye, indicating its superior effectiveness in minimizing colour change after aging among the tested mixtures.

3.2. FTIR Spectroscopy Analysis

The wavelengths of the functional groups of the standard sample of wood as assigned in (Table 2). We can notice the effect of the samples treated with Klucel nanocomposite on the aging process compared to the standard sample and the extent of the effect of these compounds on the chemical changes of the wood (Fig 1). It can be observed that all samples treated with Klucel nanocomposite were affected in the 3300-3350 cm^{-1} region, where the hydroxyl group was affected. The treated sample hydroxypropyl cellulose had a slight effect on the hemicellulose in the 1733 cm^{-1} wavelength region.[40] The sample treated with hydroxypropyl cellulose had a slight effect on the hemicellulose at the 1733 cm^{-1} wavelength region. Meanwhile, the sample treated with Klucel 1% / Nano cellulose 3% exhibited a slight effect, particularly on the lignin content, as indicated by a decrease in C = C stretching of the aromatic ring at 1506 cm^{-1} (Lignin: syringyl & guaiacyl), offset by a slight increase in C = C stretching of the aromatic ring at 1594 cm^{-1} (Lignin: syringyl & guaiacyl),[41] suggesting that the lignin content in the sample was affected proportionally to the treatment. Klucel 1% / Nano cellulose 5% exhibited oxidation of the hemicellulose at a wavelength of 1730 cm^{-1} compared to the standard sample[42], with a fracture occurring in the cellulose chains at the C-O-C Bridge Oxygen Stretching at 1156 cm^{-1}. [43] Klucel 1% showed water decomposition due to the affected group of the hydroxyl at a length of 3347 cm^{-1} [44] , indicating a noticeable change in the relative density of CH-H at 1423 cm^{-1}.[45] Klucel 1% /Nano clay 5% has a clear oxidation in the hydroxyl group area at a wavelength of 3352-3335 cm^{-1} and also a noticeable change in the relative density of CH-H at 1424 cm^{-1}, which can be attributed to a change in cellulose crystal. [46]

Table 1 Results of measuring the colour change of the samples

Sample .Cod	L	a	b	ΔL	Δa	Δb	ΔE
St	70.53	7.78	20.99	-----	-----	-----	-----
(HPC)1%	66.38	7.51	20.72	-4.15	-0.27	-0.27	4.16
NC 3%	69.25	6.85	20.51	-1.28	-0.93	-0.48	1.65
NC 5%	72.42	5.91	18.50	1.89	-1.87	-2.49	3.64
NH 3%	68.98	7.53	21.21	-1.55	-0.25	0.22	1.58
NH 5%	69.60	7.12	20.99	-0.93	-0.66	0	1.14

Table 2 The functional groups in wood samples

WAVE-NUMBER (CM^{-1})	FUNCTIONAL GROUP BANDS	ASSIGNMENT
3352	OH stretching	Cellulose, lignin and hemicellulose
2899	C-H2 asymmetric stretching as a shoulder	Cellulose, lignin and hemicellulose
1733	Unconjugated C = O stretching	Xylan and hemicellulose
1640	Absorbed O-H and conjugated C = O	Due to oxidation of cellulose
1592	C = C stretching of the aromatic ring	Lignin (syringyl > guaiacyl)
1503	C = C stretching of the aromatic ring	Lignin (syringyl < guaiacyl)
1422	CH2 scissor vibration	Cellulose (crystallized and amorphous)
1371	C-H deformation	In cellulose and hemicellulose
1323	C-H vibration in cellulose and C-O vibration	In syringyl derivatives
1234	Syringyl ring and C-O stretch	In lignin (syringyl) and xylan
1157	C-O-C bridge oxygen stretching	Cellulose
1032	C-O stretching	Cellulose and hemicellulose
897	C-H deformation	Cellulose

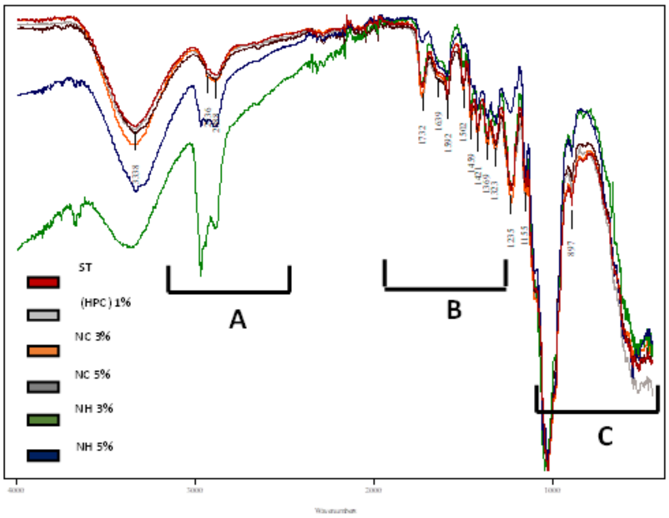

Fig 1. Infrared spectroscopy of the treated samples after aging (thermal - Photo) compared to the standard samples and Signed with three regions (A-B-C) of wavelength from (400-4000 cm^{-1})

1.23.3. X-ray Diffraction Analysis

This technique is used to determine the average width of micro cellulose crystals, [47] cellulose is considered a semi-crystalline material, and the degree of crystallinity of cellulose ranges between (50-90%). [48] X-ray diffraction analysis is the most common method to obtain structural characteristic changes is XRD, as it provides information about crystal size. *Table 3* shows the change in crystallization of cellulose in all samples [49] treated with the Nano cellulose compound were affected after aging compared to the standard sample. Except for the sample treated with Klucel 1% / Nano cellulose 5%, which maintained a degree of crystallinity of 71.2 even after aging, the treated sample Klucel 1% / Nano cellulose 3% exhibited a degree of crystallinity of 66.5, which is comparable to the standard sample's degree of crystallinity of 68.9. The sample hydroxypropyl cellulose showed a degree of crystallinity of 65.7, which is satisfactory. Additionally , the sample treated with Klucel 1% / Nano clay 5% had a degree of crystallinity of 63.8 and the sample Klucel 1%/Nano clay 3% showed the lowest degree of crystallinity of 61.9 after aging. This indicates that some of the merging compounds could improve the crystalline properties or maintain them even after aging.

Table 3 Results of determining the degree of crystallinity of the samples

Sample .Cod	I_{002}		I_{am}		widening $^{o}I_{002}$	Degree of crystallinity %
	I	2Θ°	I	2Θ°		
ST	261.1	22	81.1	18.21	6.8	68.9%
(HPC)1%	500	22.1	171.1	18	7.5	65.7%
NC 3%	278.8	22	93.3	18	6.8	66.5%
NC 5%	366.6	22	105.5	18.1	7.3	71.2%
NH 3%	204.4	22	77.7	18	5.9	61.9%
NH 5%	221.1	22.43	83.3	18	5.2	63.8%

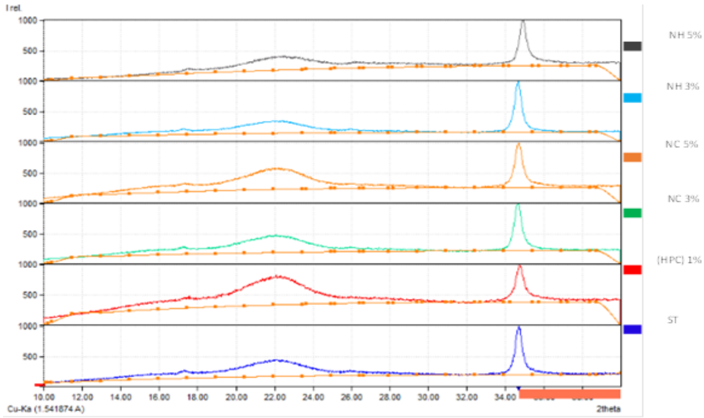

Fig 2. X-ray diffraction pattern of samples treated after aging (thermal-photo) compared to the standard sample to determine the degree of crystallinity in cellulose.

3.4. Scanning Electron Microscopy (SEM)

The scanning electron microscope is used to examine and study the success of the consolidation process by determining changes in the anatomical structure of the treated wooden samples [50][51] using Klucel nanocomposite solutions in the treated samples after accelerated aging. Through examination using SEM of the treated samples after aging, it became clear that the mixture Klucel 1% / Nano cellulose 3% achieved the best results compared to the standard sample and other compounds. This is due to the nanometre grains settling in the internal spaces of the cells, acting as both support and filler. [48] The examination showed good and homogeneous spread, along with effective filling of the spaces between the fibers, resulting in robust bonding and consolidation.[52] Notably, the tangential section displayed optimal results (Fig 3), although the cross-section exhibited suboptimal spreading. Subsequently, the Klucel 1% / Nano clay 5% mixture demonstrated satisfactory spreading but lacked homogeneity in both the tangential and cross-sections. Nevertheless, it effectively filled the joints and voids, achieving commendable fiber bonding (Fig 4). Conversely, the Klucel 1% / Nano clay 3% mixture exhibited inadequate and non-uniform spreading, with concentration in specific areas, resulting in partial fiber connection, particularly evident in the tangential section (Fig 3). Furthermore , the mixture of Klucel 1% / Nano cellulose 5% showed a poor surface morphology and also a lack of good and homogeneous spreading in the tangential section; while the cross-section not only showed clustering of the mixture in certain places, but also its ability to fill and partially connect the fibres (Fig 4). Lastly, the material hydroxypropyl cellulose showed good spreading, but it was not homogeneous. It also produced a layer with fine cracks in the tangential section and also in the cross-section, which showed its inability to spread well (Fig 3).

3.5. Compression Test

Through the results of measuring the compressive forces in (Fig 5) for the treated samples after aging and compared to the standard sample, which recorded (13.631 N/mm2) the standard sample, we can conclude that the Klucel 1%/Nano clay 5% mixture gave the best results after aging and compared to the standard sample and gave an increased value of (4.1%). However, all other formulations were affected by aging. The Klucel 1% / Nano cellulose 3% and Klucel 1% / Nano clay 3% mixtures experienced a decrease in pressure tolerance of (-1.9%) followed by HPC, which was affected by aging and exhibited a lack of strength under pressure at (-8.8%). Meanwhile, the Klucel 1% / Nano cellulose mixture was severely affected by aging, resulting in a lack of pressure strength with a decrease of (-9.6%)

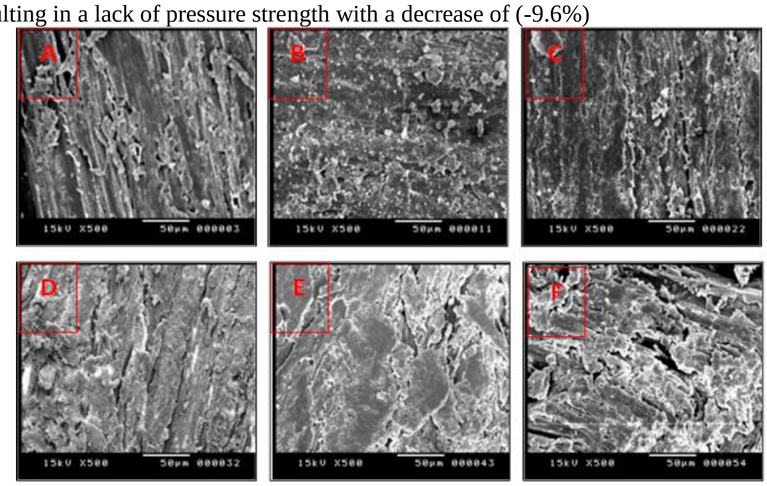

Fig 3. SEM micrographs for the tangential section magnified (×500) of samples treated after aging compared to the standard samples, (A) Standard (B) Klucel E 1% (C) Klucel 1% / Nano cellulose 3% (D) Klucel 1% / Nano cellulose 5% (E) Klucel 1%/Nano clay 3% (3) Klucel 1%/Nano clay 5%

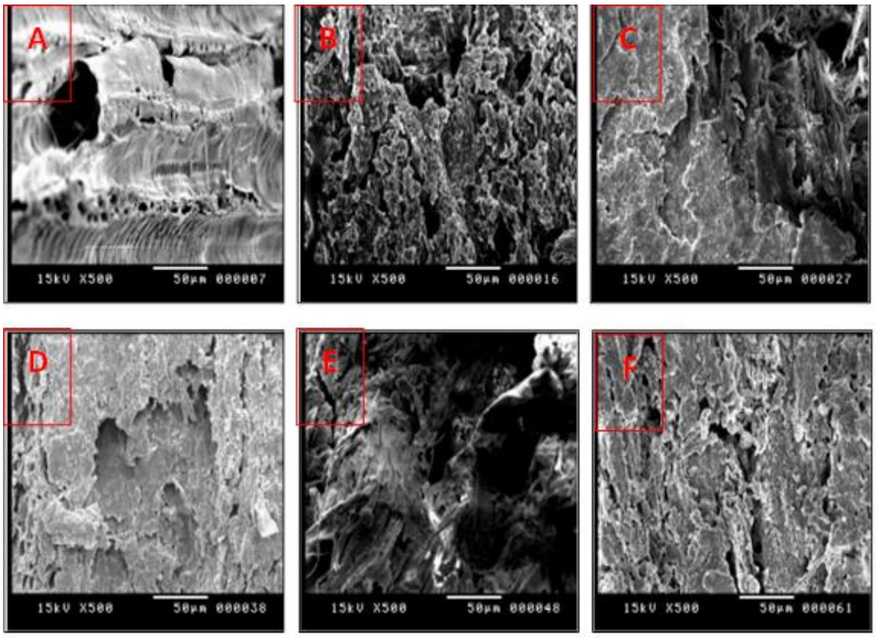

Fig 4. SEM micrographs for the cross-section magnified (×500) of samples treated after aging compared to the standard samples, (A) Standard (B) Klucel E 1% (C) Klucel 1% / Nano cellulose 3% (D) Klucel 1% / Nano cellulose 5% (E) Klucel 1%/Nano clay 3% (3) Klucel 1%/Nano clay 5%

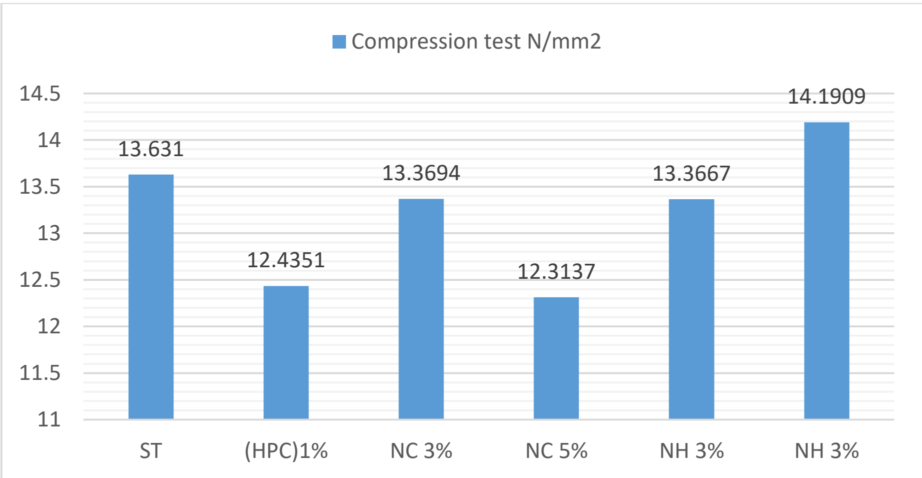

Fig 5. Compression test results show increasing compression strength in the presence of a mixture of Klucel 1%/ Nano clay 5%

3.6. Bending Strength Test

Through the results of measuring the bending endurance forces in (Fig 6) for the treated samples after aging and compared to the standard sample, which recorded (105.885 N/mm^2) the standard sample, we can conclude that the Klucel 1%/Nano clay 5% mixture gave the best results after aging and compared to the standard sample and gave an increased value, we conclude that the Klucel 1%/Nano clay 5% mixture yielded the best results after aging compared to the standard sample, showing an increased value. All samples showed positive results, with the Klucel 1% / Nano cellulose 3% mixture exhibiting the highest increase in bending bearing strength at 31.6%. Then, the mixture Klucel 1% / Nano cellulose 3% had an increase in the bending bearing strength by (31.4%), followed by the mixture Klucel 1% / Nano cellulose 5% which had an increase in the bending bearing strength by (25.3%) . In contrast, the hydroxypropyl cellulose material demonstrated the smallest increase compared to the standard sample, with an increase of (10%).

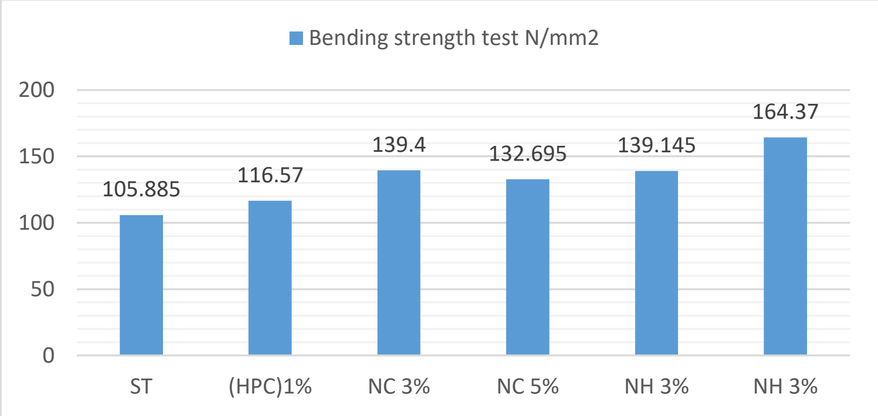

Fig 6. Bending strength test results show increasing bending strength in the presence of a mixture of Klucel 1%/Nano clay 5% presence of Nano cellulose (NC).

4. Conclusions

The exposure of archaeological wood to environmental factors, particularly as lintels, can lead to structural weakening. Therefore, it is obligatory to find solutions employing eco- friendly materials. This study introduced the new concept of Klucel nanocomposite. The findings demonstrated the efficacy of certain materials in the treatment of archaeological wood. Various tests are conducted to evaluate the effectiveness of Klucel nanocomposite, which involves adding either Nano-cellulose 3-5% or Nano-halloysit 3-5% to hydroxypropyl cellulose 1%. The results of the tests on processed samples subjected to accelerated aging indicate that incorporating Nano-cellulose at a concentration of 3% with hydroxypropyl cellulose at 1% yields several advantages. This combination enhances the material's ability to penetrate and spread evenly, while also improving the compression and bending strength of wooden samples. Notably, it has minimal impact on the overall color change of the wood and does not significantly alter its functional groups or crystal properties, even after accelerated aging. Therefore, it is preferable to use hydroxypropyl cellulose 1% with 3% Nano-cellulose in treating the weak structure of archaeological wood. Moreover, adding 5% Nano-halloysit to 1% hydroxypropyl cellulose enhances the properties of the latter, further increasing the compression and bending strength of processed samples. Similar to the Nano-cellulose composite, this mixture demonstrates good penetration and spread with minimal effect on color change. However, it noticeably affects the functional groups and crystal properties of wood. We suggest further experimentation with this mixture on softwood types to optimize treatment strategies for archaeological wood.

5. Conflicts of interest

There are no conflicts to declare.

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