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Evaluation of Physical, Mechanical and Chemical Properties of Cedar and Sycamore Woods after Heat Treatment

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THIS WORK is concerned with the study of the impact of heat (160, 180, 200 and 220°C), in addition to the time of exposure on cedar and sycamore woods for different times (2, 4 and 6 hours). The change in the color of the wood was studied. The mechanical properties, rate of mass loss, and decreases on the density of the heat treated woods were evaluated. The surface roughness and the fiber separation were also studied using scanning electron microscopy (SEM). The obtained results showed that, both types of wood were affected by heat treatment (160 and 180°C), where the surface smoothness increased for both also it was detected as temperatures rose to (200 – 220°C), there is a high roughness in the outer surface of the wood with increased separation of fibers. The anatomical structure of the wood was examined via (SEM), where the cedar samples showed higher quality in heat resistance than sycamore. The heat treated wood was investigated using FT-IR spectroscopy to study the changes in the wood spectra structure, as well as X-ray diffraction pattern (XRD) analysis were also used to study the extent of change in the crystallization of cellulosic wood.

Keywords: Cedar, Sycamore, Heat treatment, TGA, FTIR, SEM, XRD.

Introduction

Throughout history, wood has been one of the most important renewable natural components, possessing unique physical and chemical properties, making it one of the most widely used materials in all works of art [1]. Wood as cellulosic material is exposed, as other materials, for many damage factors, such as influencing the deterioration of wood material, is high temperature [2, 3]. The heat impacts on all components of wood, and develops severe changes in the basic components of the wood, from the declination of the proportion of cellulose, lignin and hemicellulose, which is reflected in the weakness of mechanical properties and the occurrence of physical changes through a complex damage mechanism, and the study of such mechanism of these changes is important to preserve the wooden antique pieces [4]. Wood has been used for the production of various artifacts, since the dawn of history due to its enormous properties (strength, durability, elasticity information, etc.) [5], which are part of the cultural heritage [6]. However, being a biological material, the cellulosic materials of wood suffers from the same fate of natural materials in terms of exposure to many damage factors [7, 8].

The most dangerous factors wood monuments are exposed to high temperatures [9] which lead to the phenomenon of thermal degradation of wood [10]. The research addresses the mechanism of thermal degradation of wood and many scientists have attempted to explain this phenomenon, including Florian [11], where he defined the thermal degradation as the temperature and time period in which wood is subjected to irreversible changes. Miyuki [12] also defined thermal degradation as the absorption by organic matter, including wood, of thermal energy, which leads to molecule agitation, consequently would affect

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the chemical components of the wood (cellulose, hemicellulose and Lignin). Hemicellulose is the most heat affected component, and it composed the bulk of the supporting plant cell structure, followed by cellulose, the main component of the cell wall [13].

The least affected component is the lignin, which is the most important component in wood tissue formation, as it gives stiffness to the cell wall, and acts as the defense line against the microorganisms and prevents water leakage from the cell wall [15]. The thermal degradation of wood greatly weakens wood structure, and the wood becomes fragile, breakable and vulnerable to lose with decreasing in weight through complex damage mechanism. In addition to the color change of wood and the change in the outer texture of the wood surface [16]. As the temperature continues to rise, the stiffness of the wood decreases accompanied with changing in its dimensions which eventually leads to the occurrence of cracks and breakouts, while the wood itself becomes fragile and cannot be handled by hand, this damage is final and irreversible [17, 18]. The process of thermal degradation of wood is not done at a similar and constant rate in all types of wood [19].

Therefore this work is interested in studying the impact of thermal treatment of wood at different temperatures (160, 180, 200 and 220°C) for three time periods on the chemical, physical and mechanical properties in two types of wood. Two types of wood were selected; one of them is soft wood (Cedar), which is considered as a good type of wood, and resistant to fungal infection because of its content of high concentrations of chemicals (resins, waxes and oils) which represent a natural fungicide [20]. The sycamore wood has been selected as a hardwood, with its habitat in Egypt, where the ancient Egyptian had used it since the fifth dynasty, in the manufacture of coffins [21].

Materials and Methods

Materials

Cedar and Sycamore woods were delivered from Lebanon& Egypt, respectively.

Experiments of thermal degradation of wood

Samples of cedar and sycamore woods were prepared of 10 x 1 x 1 cm in dimensions for bending tests. And the preparation of samples of 2 × 2 × 2 cm in dimensions for compression tests as in the physical and chemical tests were conducted on the same samples of the above dimensions.

All samples were weighed before and after heat treatment and by applying the law of mass reduction rate.

$$\text{Percentage of loss in mass} = \frac{\text{Sample mass after final limitation- Sample mass before final limitation}}{\text{Sample mass before final limitation}}$$

The percentage of loss in the mass has been calculated.

By calculating the mass and size of the samples before and after the heat treatment, and recording the results, the rate of loss in the density of both cedar and sycamore wood was calculated by applying the density law.

$$\text{Density} = \frac{\text{Massin Kg}}{\text{Volume in } M^3}$$

Heat treatment has taken place in LABTECHkiln oven at the National Research Center, Giza, Egypt

Characterization

Mechanical properties

The bending resistance and compressive resistance were measured when the samples were subjected to different temperatures and conducting a comparison between those samples and standard samples according to the ASTM D638-91 standard using a universal testing machine LK10k (Hants, UK) fitted with a 5 kN load cell, and operated at a rate of 5 mm/min on the samples that were cut to suit the operating conditions of the machine for both bending and compression tests. The unit of measurement is (Newton).

The color measurements

Ultra Scan PRO Hunter lab mead in U.S.A. was used to register the rate of color changes for both sycamore and cedar at the above mentioned temperatures.

Morphological properties

Scanning Electron Microscope for samples was examined using SEM Model Quanta 250 FEG (Field Emission Gun) attached with EDX

Unit (Energy Dispersive X-ray Analyzes), with accelerating voltage 30 K.V., magnification14x up to 1000000 and resolution for Gun.1n).was used for the examination of the external surface of wood and anatomical structure of thermally treated wood

Thermal processing

The study dealt with heat processing of samples at different temperatures (160,180, 200 and 220°C) that for three time periods (2, 4 and 6 hours) for each temperature.

Results and Discussions

Visual examination

By continuous heat treatment of the wood, severe physical changes have taken place in all samples, as the color of the wood gradually changed from yellowish brown to dark black with cross shrinkage of the samples and the appearance of capillary cracks at the beginning of the heat treatment, then developed into deep and wide cracks through the granules and spread all over the surface, reaching the occurrence of breaking and crashing in the samples. The samples became, at the end of the heat treatment, very weak and fragile. Tables 1 and 2 display the precise visual description of both types of woods after thermal treatment. Cedar wood differed from sycamore wood in terms of the degree of color change and the severity of the cracks and the increase in their number, where the Cedar wood has appeared more resistant than sycamore wood. The rapid collapse of sycamore wood was observed at the beginning of heat treatment.

Color change

The thermally treated wood is characterized by a significant change in color degree compared to standard samples. The degree of color change depends on temperature more than the period of heat exposure, as well as color change is associated with the type of wood [22, 23]. Color change is a reflection of the chemical changes that occur, where hemicellulose and cellulose are accountable for the degree of change in color of wood [12, 24 and 26]. The result of heat treatment was clearly visible to the naked eye as shown in Tables 1 & 2, showing the color change of sycamore and cedar woods after heat treatment. A significant change in (ΔE) registering the rate of color change for both sycamore and cedar woods (Fig. 1 & 2). By comparing the results of color change for both woods, the color change was appearing high in case of cedar wood.

Surface roughness

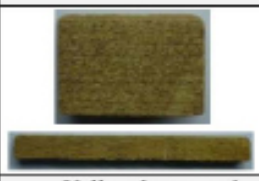
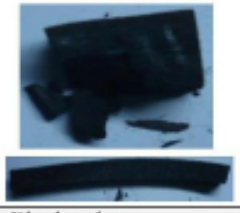
The phenomenon of surface roughness is a complex phenomenon is due to several factors such as (anatomical structure of wood, the characteristics of growth, machines, treatments, vascular, cell cavity, width of secondary rings and hardness) [26]. Studies have shown that in good types of wood, surface roughness constantly decreases with temperatures rising [27] and as temperature increases, the surface roughness decreases; however, continuous heat rise is counterproductive [28]. The rate of change in the physical and mechanical properties of the wood begins at 150°C, as the temperature rises higher than 150°C, surface roughness increases [29].

The examination of the external surface shows that the type of wood played a large role in terms of temperature sensitivity, as in the sycamore wood, which is characterized with the roughness of the surface in the standard sample, the surface softness increased at the onset of the exposure to 180°C, however, soon the surface showed more roughness as the temperature increased from 180 to 200°C, with the increase in the rate of fiber shattering and the separation with the loss of large parts of the wood fiber, until the wood reached an advanced stage of damage and surface roughness at 200°C. The outer surface of the standard sample of cedar wood was characterized by a smooth surface compared to sycamore wood. It retained the characteristics of the surface and the bonding of the fibers while increasing the smoothness of the surface at low temperatures (160-180°C). Getting affected by the temperature rise gradually began to appear in the roughness of the outer surface, fiber shattering and separation began to show at high temperatures of 200-220°C. Thus, cedar wood is one of the most heat resistant woods than sycamore wood in terms of the sensitivity of the external surface and fibers at higher temperatures (Fig. 3 &4).

Loss in Mass

The wood loses a large part of their mass when exposed to heat and the rate of loss is high at the beginning and then gradually decreases [30]. This is due to the loss of the water content of the wood and thus loses its weight [31]. This depends directly on the chemical composition of the wood, especially on hemicelluloses [32- 34]. The percentage of loss in the mass has been calculated as shown in Fig. 5 for sycamore and cedar woods. The results showed that the loss rates in the mass of sycamore wood increased by increasing the

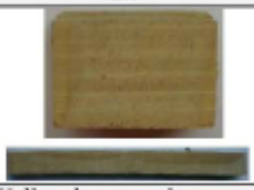
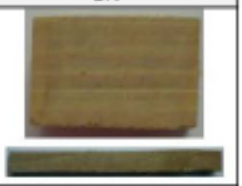
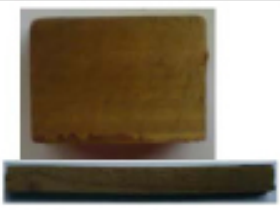
TABLE 1. Descriptions of sycamore wood specimens after thermal treatment at (160, 180, 200& 220°C) in (2,4& 6 hours).

sycamore wood specimens before thermal treatment 			
Temperatures	2h	4h	6h
Sycamore at160°C 			
Descriptions	Yellow brown colour-	-brown colour	Dark-brown colour-
Sycamore at 180°C 			
Descriptions	-brown colour	-Dark brown colour Surface roughness-	-Dark brown colour -Surface roughness
Sycamore at 200°C 			
Descriptions	Dark brown colour	Darker-brown colour -Wider cracks along the longitudinal grain	Darker-brown colour -Wider cracks along the longitudinal grain -Appearance of cross-grain fissures
Sycamore at 220°C 			
Descriptions	Black colour -Shrinkage -Longitudinal cracks -Some hair-line cross-grain fissures	-Black colour -Very well-burnt char with a lot of cracks and fissures -Very fragile (broke when touched)	-Black colour -Very well-burnt char with a lot of cracks and fissures -Very fragile (broke when touched)

temperature from 160 to 180°C at a constant rate and by increasing the exposure time, the mass loss rates increased, and this continued to increase with increasing the temperature up to 200°C. With the continuous increasing in heat exposure time, the mass loss rate gradually declined again, although the increase in heat exposure time continued with rising the temperature up to 220°C, the rate of loss in the mass increased, but the continuous increase in the heat exposure time led to decrease in the rate of loss in mass once again.

In respect of cedar wood, the rate of loss in mass increased slowly when samples were exposed to temperatures 160 and 180°C. The

TABLE 2. Descriptions of Cedar wood specimens after thermal treatment at (160, 180, 200& 220°C) in (2, 4& 6 hours).

Cedar wood specimens before thermal treatment		
Temperatures	4h	2h	6h
Cedar at 160°C			
Descriptions	-Yellow brown colour	-Yellow brown colour	-Dark-brown colour
Cedar at 180°C			
Descriptions	-brown colour	Dark-brown colour-	-Dark brown colour
Cedar at 180°C			
Descriptions	-Dark-brown colour	Dark brown colour Surface roughness	Dark brown colour Surface roughness
Cedar at 200°C			
Descriptions	Black colour -Longitudinal cracks -Some hair-line cross-grain fissures	-Black colour -Shrinkage -Very well-burnt char with a lot of cracks and fissures -Very fragile)	-Black colour -Very well-burnt char with a lot of cracks and fissures -Very fragile (broke when touched)

mass loss increased in the first hours when samples were exposed to temperatures 200 and 220°C at a constant rate, and by increasing the heat exposure time from (2 to 4) hours and then to 6 hours, the rate of loss in the mass decreased. By comparing the mass loss rates for both sycamore and cedar woods, it was found that the mass loss rates for sycamore wood are higher than that of cedar wood.

Density

The thermal degradation over time leads to the loss of percentage of cellulose, the main component of the wood. This results in the occurrence of cross cracks, large and deep voids, and by the continuity of thermal degradation,

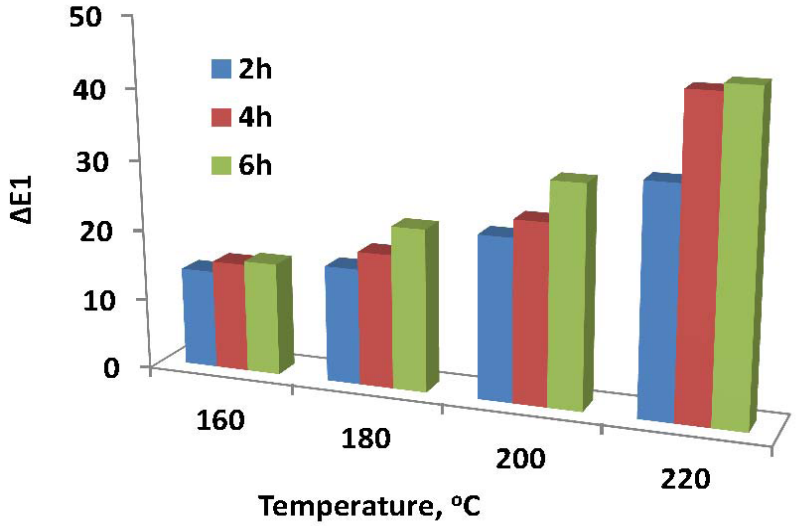


Fig. 1. Color change rates of Sycamore.

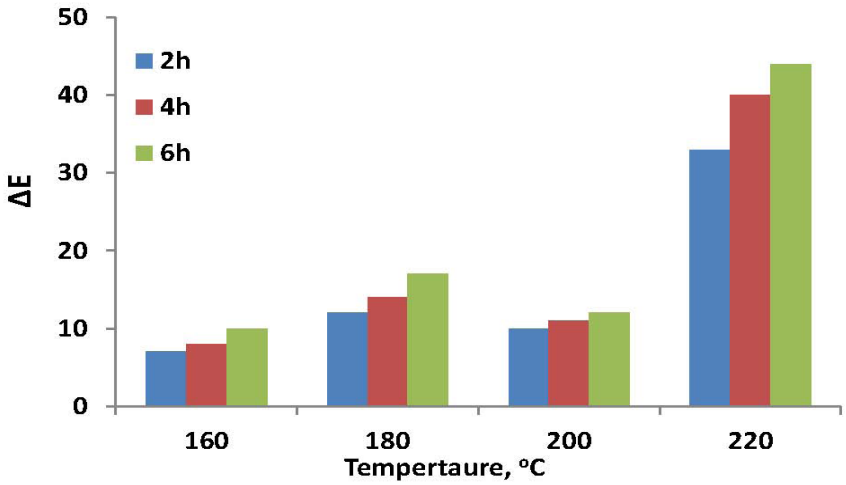


Fig. 2. Color change rates of Cedar wood.

cross cracks increase with the absence of cellulose, the essential asset of wood, which leads to decline of density of wood [35, 36]. By comparing between the changes in the density rate of reduction of sycamore and cedar woods at the same temperature for the same heat exposure time it shows that. The reduction of density rate of sycamore wood was higher than that of cedar wood as shown in Fig. 6.

Mechanical properties of wood

Mechanical properties are the properties related to the behavior of wood material when subjected to loads such as pressure and bending [37]. Figures 7&8 display the bending and compression resistance of both sycamore and cedar wood. By comparing the rate of reduction of the bending and compression resistance between sycamore and cedar woods at the same temperature and for the same heat exposure time, it is apparent that the bending and compression resistance of sycamore wood in the standard sample is higher than the bending and compression resistance in the standard cedar wood sample where that is explained due to the vessels in the hard wood (Sycamore), and the reduction rate of bending and compression

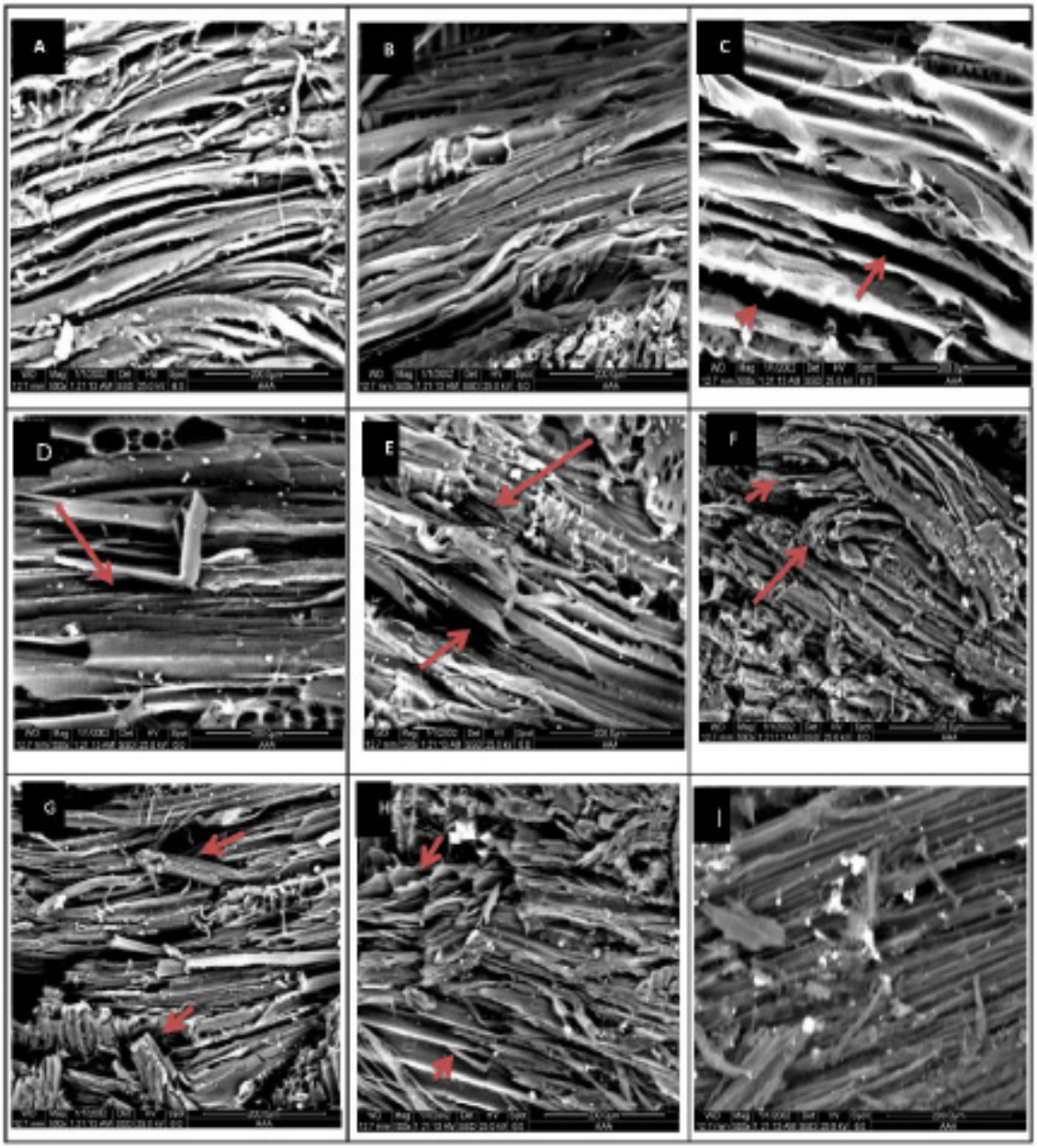


Fig. 3. Sycamore wood sample surface after its exposure to heat treatment.

resistance of sycamore wood after heat treatment was rapid, unlike the cedar wood, which has been regular gradual after heat treatment.

Therefore, the Cedar wood is considered one of the high quality woods, retains its mechanical properties such as (bending resistance and compression resistance) in the high temperature atmosphere circumstances relative to the sycamore wood, which is high mechanically affected by high temperatures due to the presence of water vessels [38].

Anatomical structure of thermally treated wood

The internal deformities resulting from the exposure of the studied samples to heat for different periods of time were examined. Modification of the cell wall structure occurs as a result of the rise in temperature [39] and the emergence of plasticity in the external surface of the samples compared to the standard samples and the cell loss of its strength, accompanied by a breakdown in most of the cell walls after heat treatment. A radial cross section in the sycamore wood and a longitudinal cross section

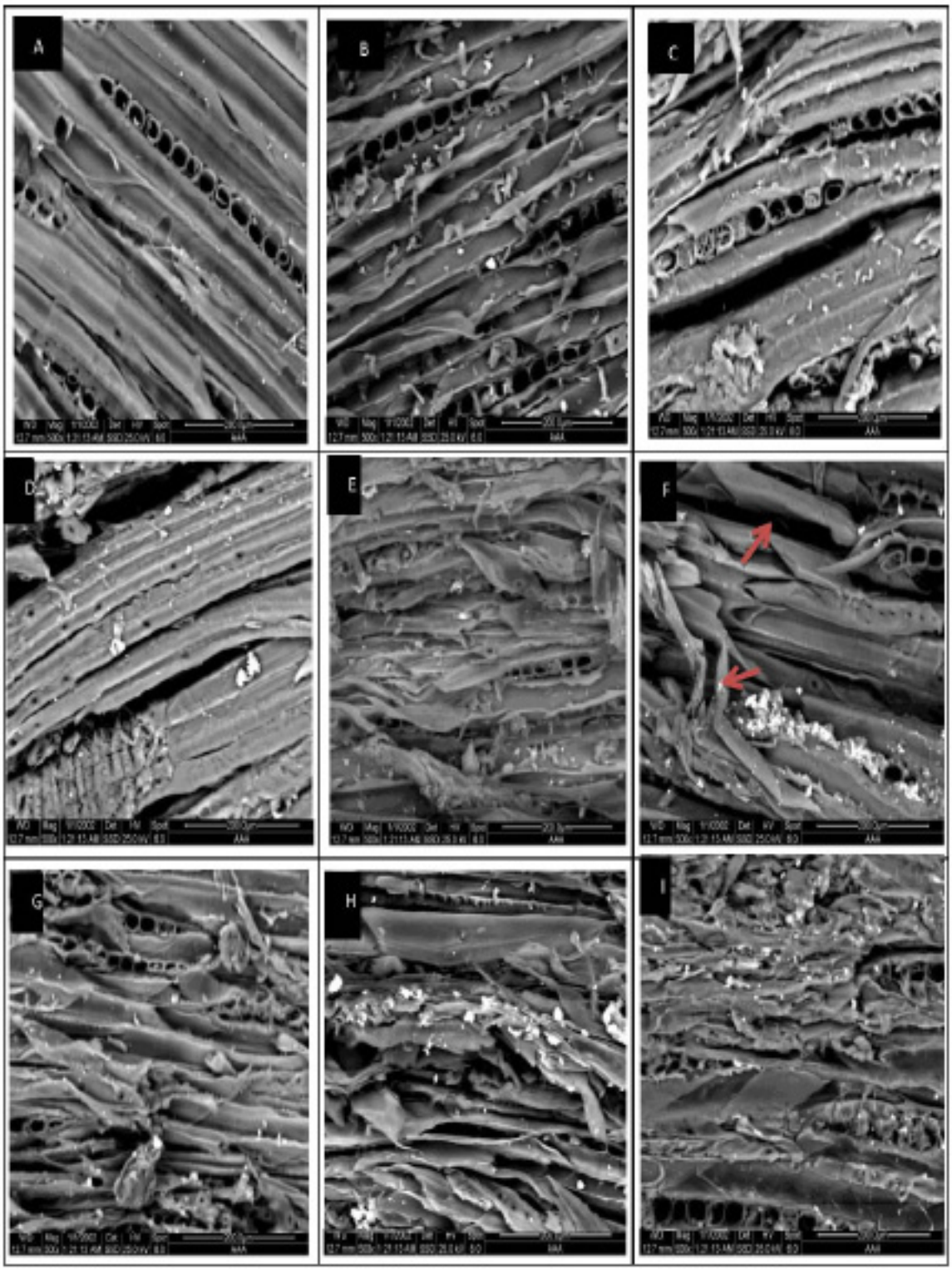


Fig. 4. Cedar wood sample surface after its exposure to heat treatment.

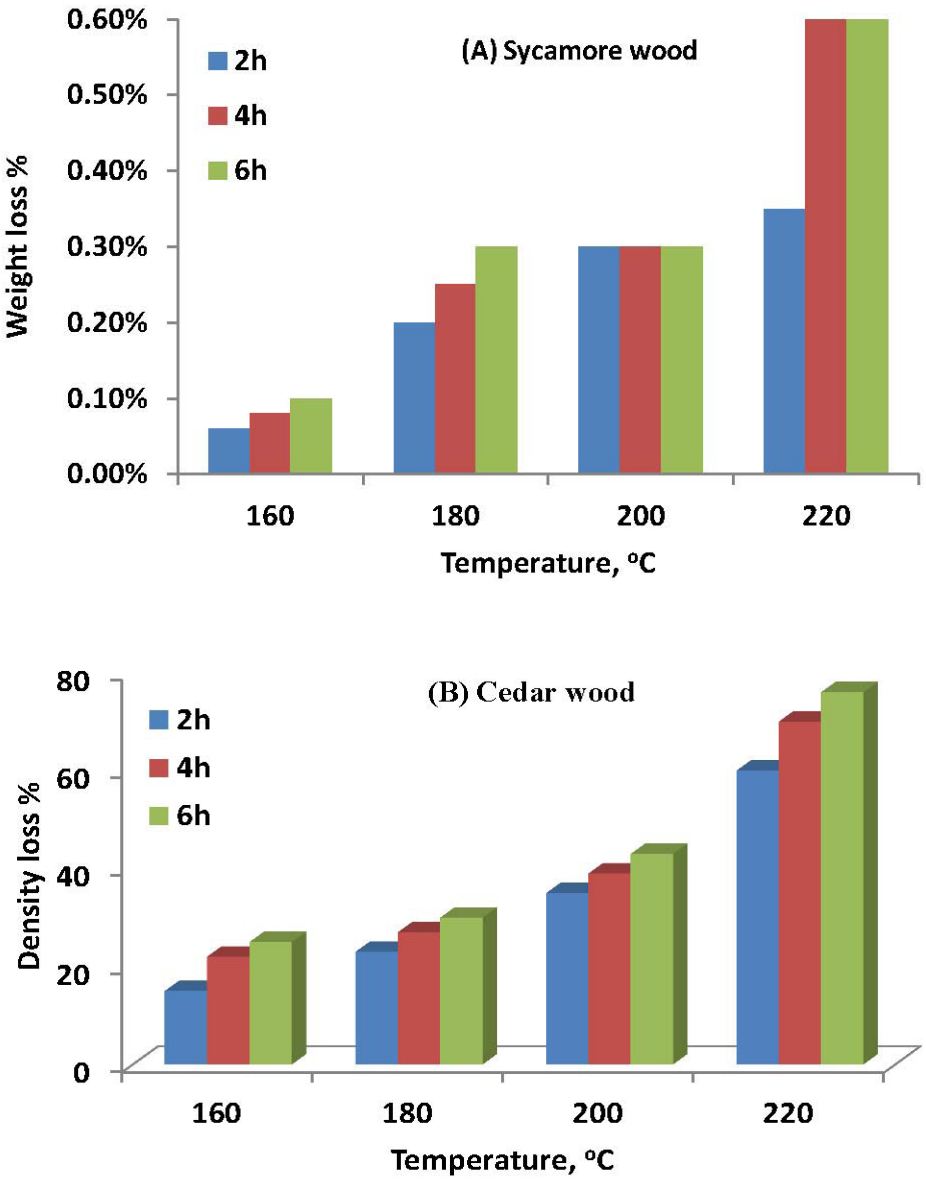


Fig. 5. (A&B): The rate of weight loss for sycamore and cedar wood.

in the cedar wood, which are the most affected sections by heat. This was evident when bending tests were carried out, where breakage took place in the radial section of sycamore wood and the longitudinal section of cedar wood. The samples were examined at temperatures (160, 180, 200 & 220°C) for 2 and 6 hours.

Figures 9 and 10 demonstrated that the internal deformations of the cell walls as a result of the exposure of the samples of both sycamore and cedar woods. Both types of wood were affected by heat beginning from temperature 160°C, with the increase in the rate of damage by the increase in temperature, till 220°C. It is may be attributable to the rates of damage to sycamore wood were higher than that of cedar wood at different temperatures, as severe deterioration has occurred to the sycamore wood at different temperatures compared to cedar wood.

Thermal analysis

Non-isothermal degradation in static air atmosphere, for all wood species, occurs through three successive processes accompanied by mass losses [40]. Figure 11 revealed that the mass losses of the samples of both sycamore and cedar woods. Each wood species shows: (1) an endothermic

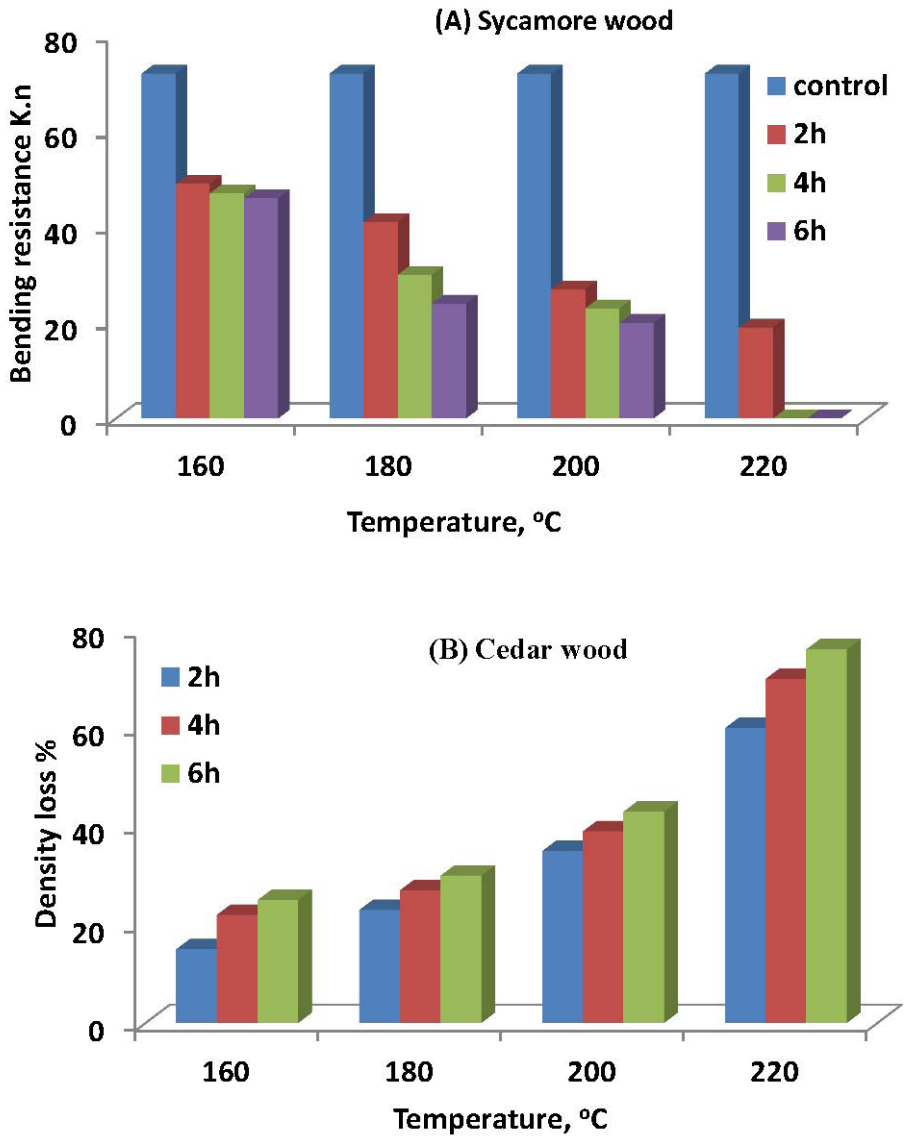


Fig. 6. (A& B): Rate of density loss for Sycamore and Cedar wood.

process, denoted as, when water loss takes place, (2) an exothermic process, denoted as consisting of thermal degradation assigned to cellulose and hemicelluloses, over the 250-380 °C, (3) an exothermic process, denoted as, assigned to lignin, over the 400-500. By comparing the change in the mass loss ratios of sycamore and cedar woods shows that the mass loss ratios of hemicelluloses and cellulose to lignin of sycamore wood was higher than that of hemicelluloses and cellulose to lignin in cedar wood.

X-ray diffraction analysis (XRD)

The XRD pattern was used to calculate the degree of crystallization of cellulose in wood samples [41, 42]. The degree of crystallization of thermally treated wood was calculated through the diffraction curve at (160, 180, 200 & 220°C) at two time periods; 2 and 6 hours. Figure 10 exhibited that the low degree of crystallization in both of sycamore and cedar samples with the continuity of heat treatment, with the sycamore wood more affected by the heat treatment, as it showed a rapid decrease in the crystallization

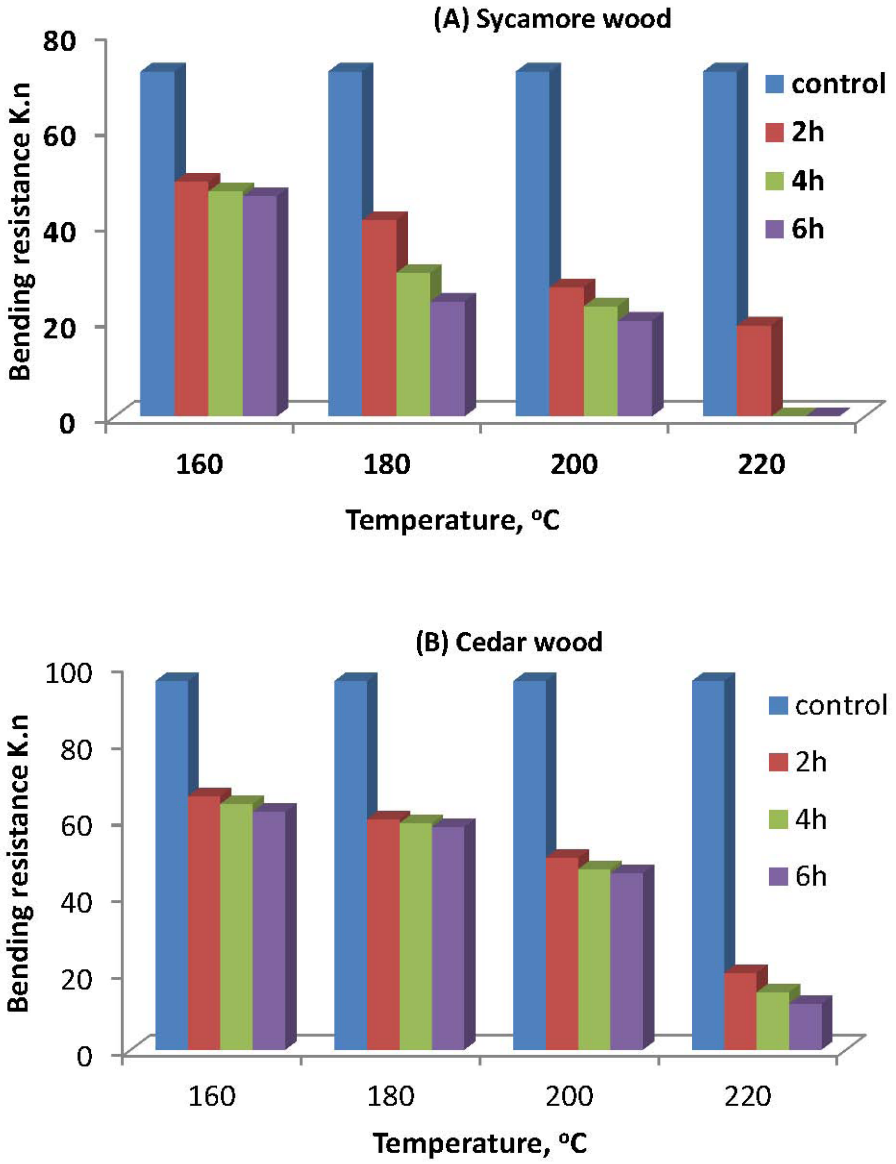


Fig. 7. (A&B). Bending resistance for Cedar and Sycamore wood

from (63 to 6%) as shown in Table 3, and the cedar wood showed more heat resistance, as its decrease in the crystallization from (56 to 10%) after the heat treatment as revealed in Fig. 12.

Infrared analysis (FTIR)

FTIR is a method of qualitative analysis used to identify functional groups in molecules of organic matter [43-45], by identifying changes in peaks of absorbance [46], Each type of wood has its pattern of infrared rays characteristic thereof, with some common features [47, 48]. When exposed to heat, severe changes occur in absorption areas, which differ from one type of wood to another, confirming chemical changes due to heat treatment [36, 49]. Figure 13 demonstrations spectral analysis of both sycamore and cedar woods at 160, 180, 200 & 220°C in two time periods of exposure (2 & 6 hours), where the change in the general structure of cellulose

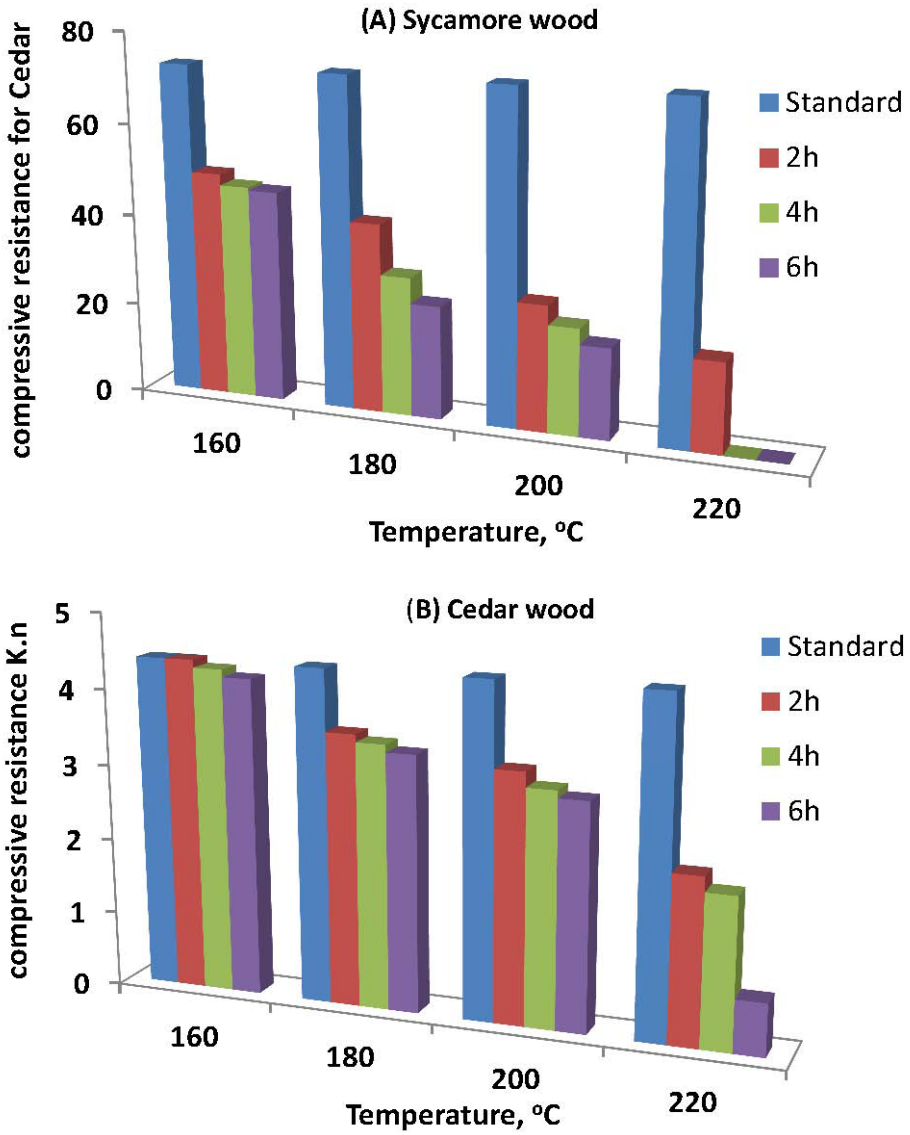


Fig. 8. (A&B): Compressive resistance for Cedar and Sycamore wood.

through an increase in some of the absorption characteristic of a group of cellulose areas and shortage in other areas and the formation of new groups.

Conclusion

Cedar wood was found more stable in heat resistance than sycamore wood. By comparing the mass loss rates for both sycamore and cedar woods, it was found that the mass loss rates for sycamore wood are higher than that of cedar wood. After conducting mechanical tests on the wood, the sycamore showed that it is more stable than the cedar wood in the onset of the heat treatment, due to the water vascular in sycamore wood, and with continuous heat treatment, the rates of mechanical characteristics for sycamore wood has declined rapidly, on the contrary to the cedar wood, where it has been more stable than sycamore wood post heat treatment. FTIR and XRD analyses proved that cedar wood is one of the woods of higher quality than sycamore wood, as it showed higher chemical stability than sycamore wood.

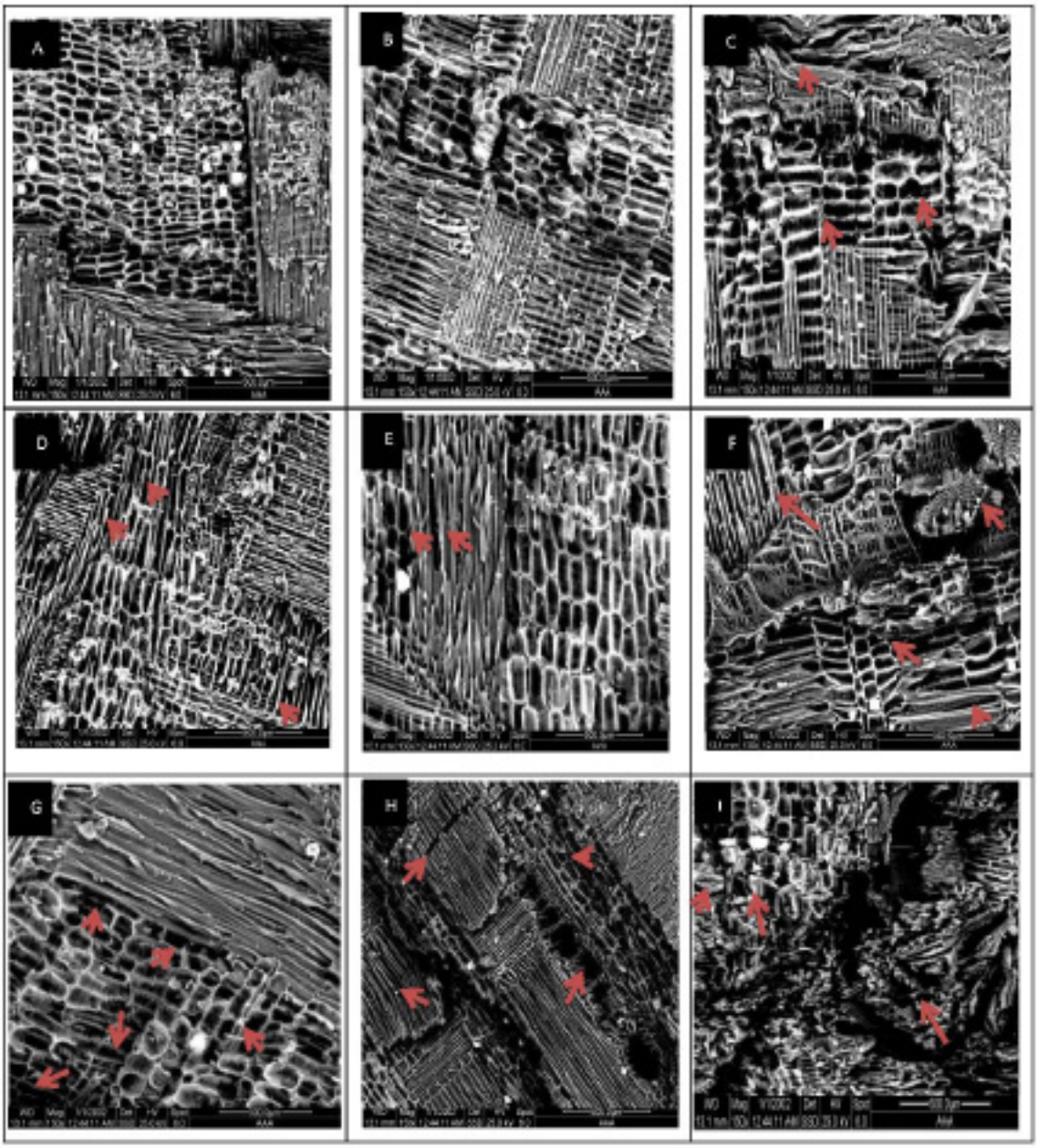


Fig. 9. Radial cross section in a sample of Sycamore wood, after exposure to heat.

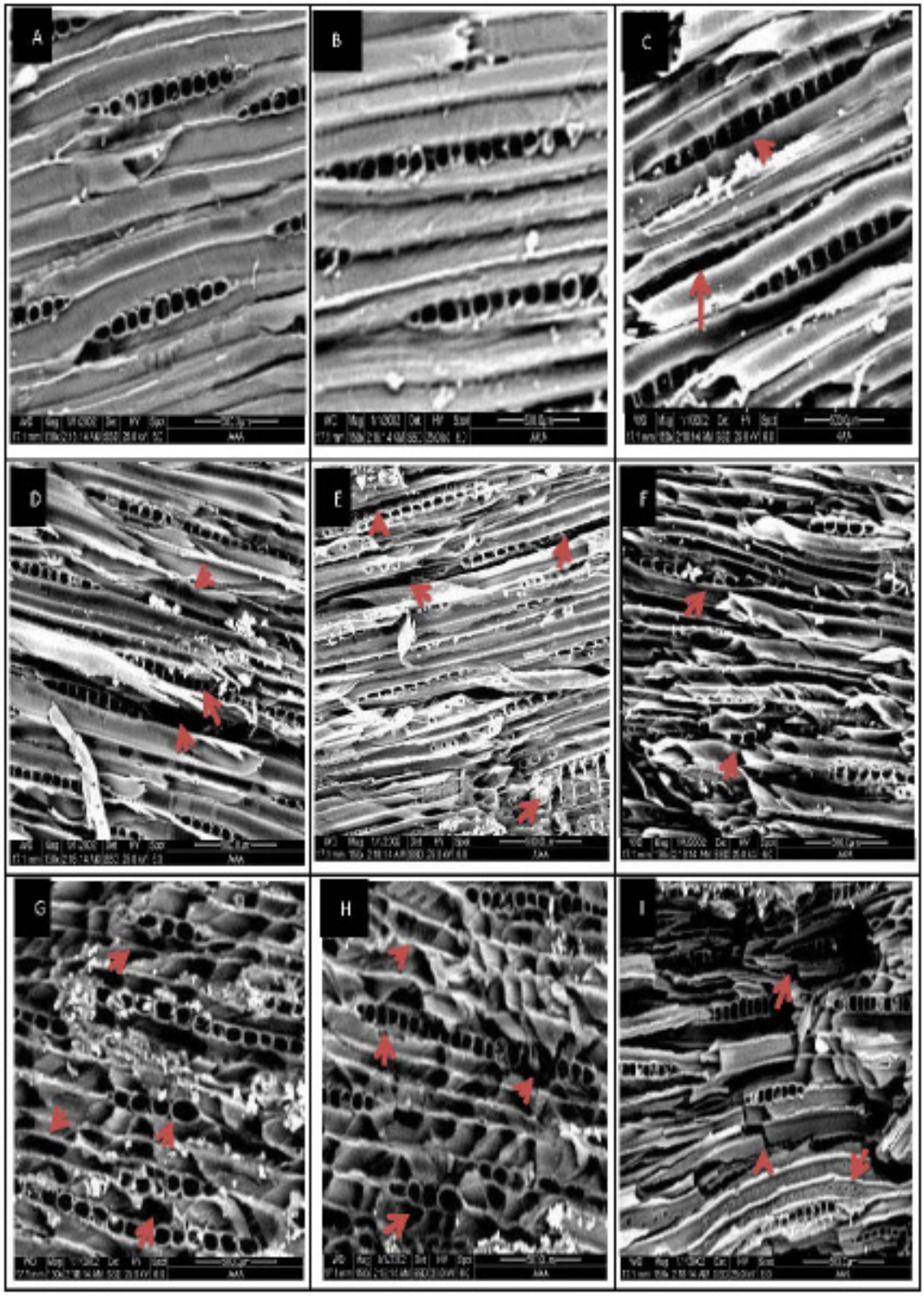


Fig. 10. Longitudinal cross section in a sample of Sycamore wood, after exposure to heat.

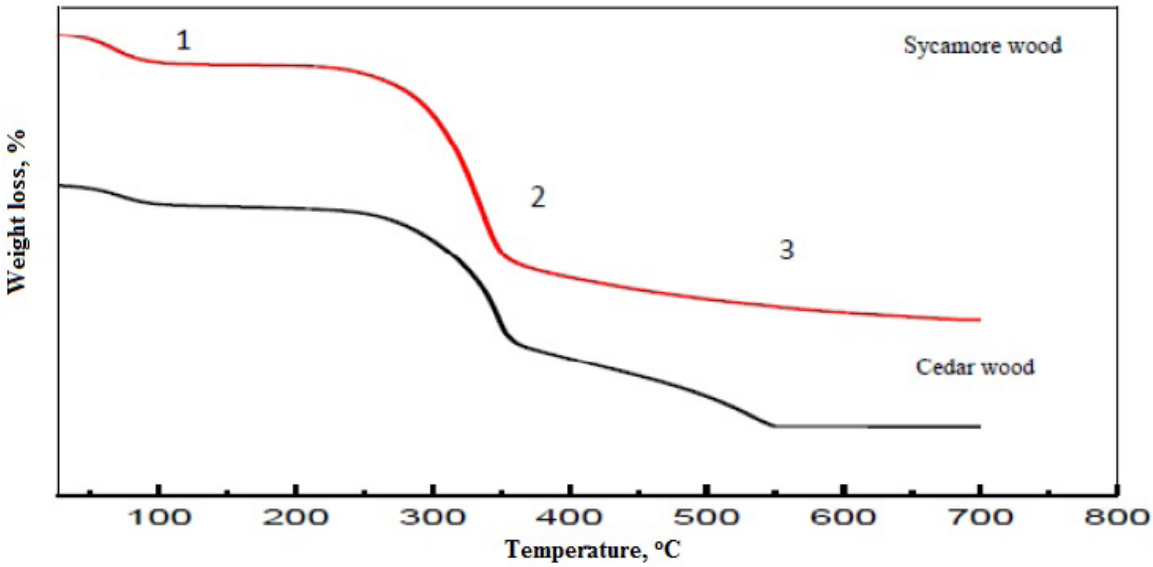


Fig. 11. TGA of Sycamore and Cedar wood

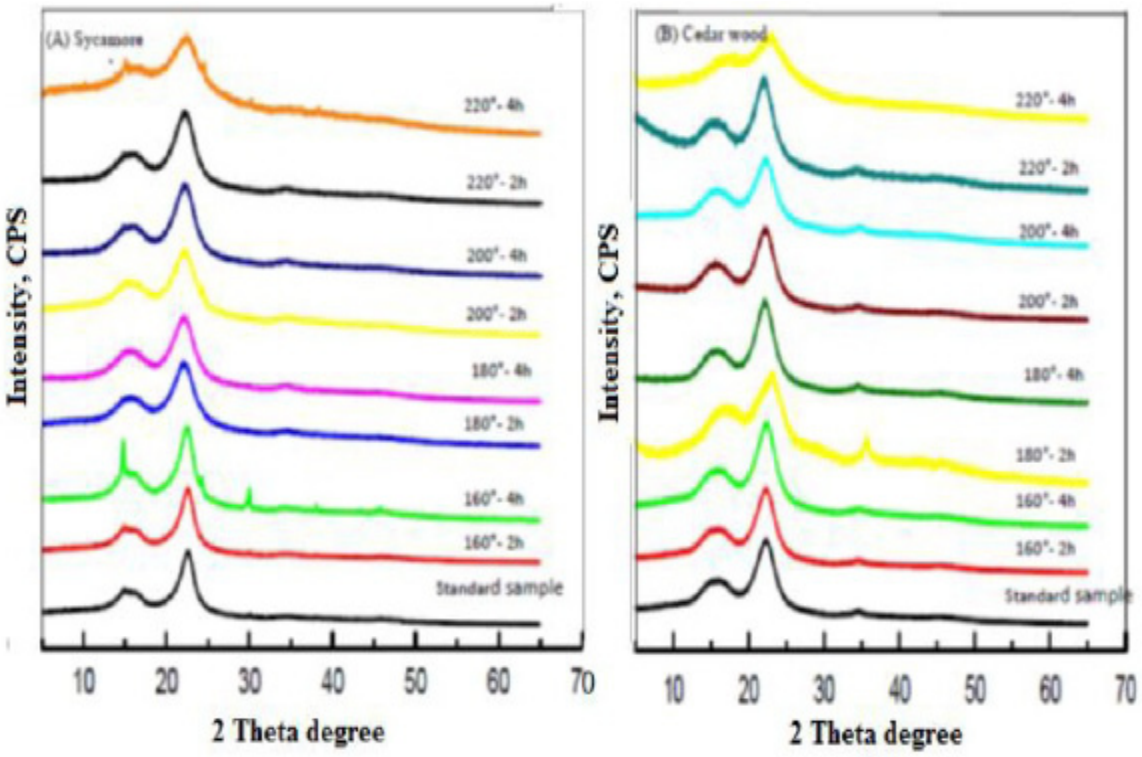


Fig. 12. Cellulose analysis by X-ray deviation of the Sycamore and Cedar wood

TABLE 3. Degree of crystallization of thermally treated Sycamore and Cedar wood

Temp. (°C)	160		180		200		220	
Time (hour)	2	6	2	6	2	6	2	6
Sycamore	63.0	55.9	42.0	31.0	27.0	26.0	11.0	06.0
Cedar	56.0	52.0	54.0	48.5	30.5	29.9	14.0	10.0

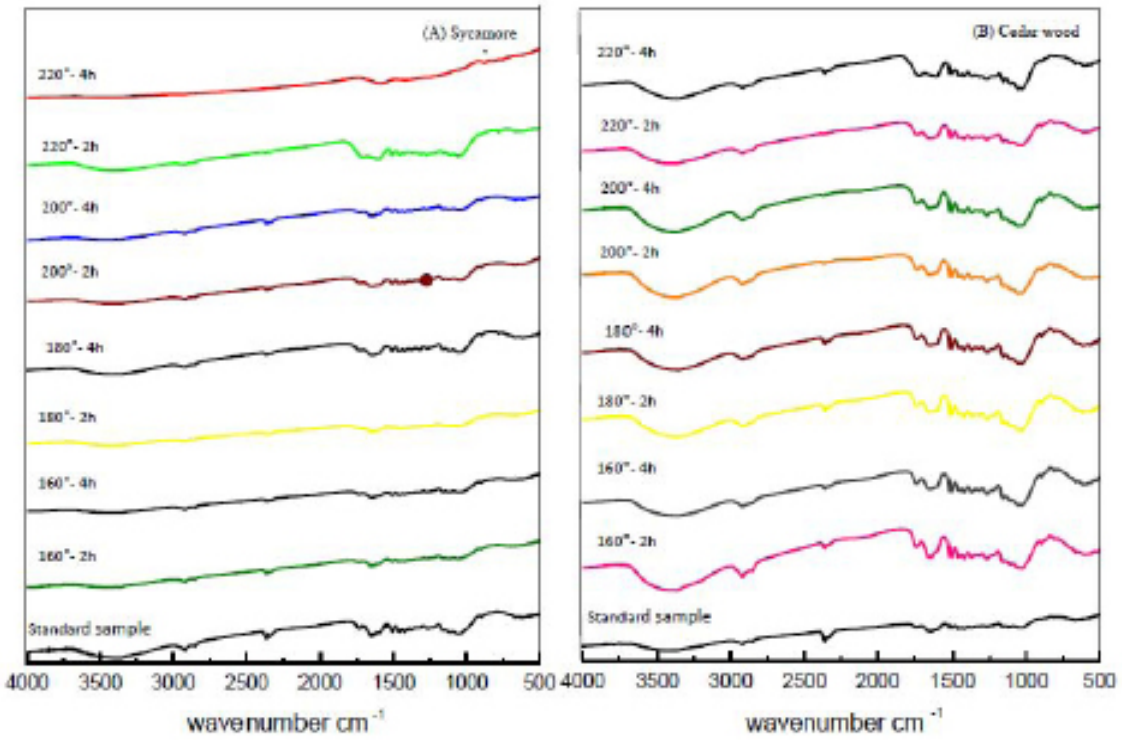


Fig. 13. Change in the general structure of cellulose of Sycamore and Crdar wood

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تقييم الخواص الفيزيائية والميكانيكية والكيميائية لخشب الارز والجميز بعد المعالجة الحرارية

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يهدف هذا البحث إلى دراسة تأثير الحرارة على نوعين من الخشب (الأرز– الجميز) فى محاولة لمحاكات تأثير الحرارة على الخشب الأثرى. حيث تم تعريض الخشب لدرجات حرارة مختلفة (١٦٠–١٨٠–٢٠٠–٢٢٠°) لمدة (٢–٤–٦ ساعة) ودراسة تأثير الحرارة على الخصائص الفزيائية والميكانيكية والكيميائية للخشب. وقد إستخدم الميكروسكوب الإلكترونى الماسح (SEM) لدراسة تأثير الحرارة على الخصائص التشريحية والتركيبية للخشب من انفصال للألياف وخشونة السطح. والتحليل بالأشعة تحت الحمراء (FTIR) لدراسة التغيرات بالتركيب الكيميائى للخشب. بينما إستخدم التحليل بالأشعة السينية (XRD) لدراسة تأثير الحرارة على درجة تبلور السليلوز. وقد أوضحت الدراسات أن كلا نوعين الخشب قد تأثر بدرجات الحرارة حيث حدث إنخفاض بخشونة السطح للخشب فى درجات الحرارة (١٦٠–١٨٠°) وبإرتفاع درجات الحرارة إلى (١٨٠–٢٠٠°) زادة خشونة السطح مع زيادة انفصال الألياف. وبشكل عام أظهرت عينات الأرز جودة أعلى فى مقاومة الحرارة عن خشب الجميز.