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Experimental Study On Using Extracted Dye From Blood Wood For Restoration Insects Holes In Archaeological Wood Applied On SabilAl Kazlar, Ottoman Period, Cairo,Egypt.

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Abstract: *Restoration process of the holes resulting from insects damage is one of the most difficult thingsin front of the conservators during the restoration process. Conservators were either replace the damaged wood or restore it with a mixture of polymer, filler and mineral Oxides. However, first part of this paper is using a new mixture consisted of (Beva 371, medical cotton filler, and Haematoxylin dye) to complete holes which caused by insects and were found in sabil Al Kazlar in Cairo. So this paper used methods of examination and different analysis which represented in (naked eye - Optical microscope - scanning electron microscope)to identify the type of archaeological wood and to study the deterioration phenomena of the Archeological wood. This paper also has been used a number of methods of analysis to identify the structure of both archaeological wood and blood wood (such as chemical analysis - X-ray diffraction-analysis and infrared analysis) through examination and various analyzes the researcher can conclude that the wood used in the Sabil Al Kazlar is Lebanese cedar, Blood wood is the commonname of Haematoxylumcampechianum L.The vegetable dye which extracted from that tree is haematoxylin dye. One of these trees is in Saqqara. The second part of this study is the preparation of a double form for completing materials to compare between a new material and old material of completing insects holes,before and after the thermal ageing .This comparative study was by FTIR analysis. changing in color has been measured by using the device of Light meter which has declared that New mixture is more successful than old mixture which was used in the past.*

Key Words:*Dye, Blood wood, Restoration, Sabil Al Kazlar, Ottoman Period*

I. Introduction

Sabil Al Kazlar considered one of the most Beautiful architectural in Ottoman period. Which was built by Mustafa Agha Alkazlarthis monument carries number (265) located in Alsuffia Street,and built in 1618 A.D - 1028 H. It is one of the Ottoman Monuments(Mohie,M.,2011). This Sabil was built by stone and wood.This wood was used in ceiling, windows and doors As shown in fig. (1)

Figure 1 Shows Sabil Al Kazlar Ottoman period

The aim of this paper is making treatment of one of the most important aspects of archaeological wood. This is insects deterioration as shown in fig. (2)

Figure 2 Shows insects damaged on archaeological wood from Sabil Al Kazlar

the insect infection was a result of closing the sabil all time without conservation. The holes in the wood are holes of wood beetles. Supreme Council of Antiquities has sent specialists to sterilize wood in 2005, with Cidial 50, which killed insects in that period, but insect holes remained as a weak point insects can become again after the finishing the pesticide effect and distorted the archaeological wood. The main objective of this research is to restore damage by insecticide property and antifungal which found in vegetable dye of blood wood (El Sonpaty,A.(1997) in this research completing materials mixed with extracted dye from blood wood (Abdel Magid, 2004) dye instead of metal oxides (hematite - and geothite) which restorers used to complete with it . (Nabil,E., 2015) The properties of the organic material are different from the properties of the metal oxides, so there is heterogeneity between filler materials lead to separation, but the best method forcoloring wood was vegetable dye.

II. Materials And Methods

2-1. Materials

This paper was focused on **1**- damaged wood as an important archaeological material in sabil Al-Kazlar. Restoration was mad by using many substances of completing represented in **2**-hematoxylin dye. **3**- beva 371, **4**-medical cotton fibers. The previous materials are comparing with metal oxides (such as hematite-and Geothite)

2-2-Methods

These methods are as the followings:

2-2-1. Examination

2-2-1-1. Naked eye

The naked eye examination is the most important test methods before the beginning of restoration process, because it acts as the compass which determine the rest of the required methods of examination and analysis for diagnosis and restoration process.The case study in this paper is archaeological wood from sabil Alkazlar in Cairo, which has different insects holes in wooden parts such as doors, the wooden roof and wooden windows.

2-2-1-2.Optical Microscope

German Licka microscope connected with camera is used to identify the type of archaeological wood. (in central laboratories of Ministry of petroleum).

2-2-1-3. Scanning Electron Microscope SEM

SEM Model (Quanta 250 FEG (Field Gun) attached with EDX Unit (Energy Dispersive X-ray Analyses), with Emission accelerating voltage 30 K.V., magnification14x up to 1000000 and resolution for Gun.1n FEI company, Netherlands) is used to examine archaeological wood to identify the damage which the archaeological wood suffered from it . The Blood wood also was examined to know the regions where the vegetable dye concentrate on it. This examination made by using

2-3.Analytical Methods

The archaeological wood of sabil Al-kazlar and the Blood wood were analyzed by different methods to identify the compounds in both of archaeological wood and Blood wood to compare the results. These methods which used are as follows:

2-3-1.(C,H,N) analysis

Carbon, Hydrogen and Nitrogen, analysis is used to know some of the organic compounds such as C,H and N which can not be identified by other analytical method.(Fuk, J.2012)

2-3-2.EDX

The Same device which used in SEM has used in the analysis of archaeological wood from Sabil Al kazlar and the Blood wood which analyzed also to know its main elements.

2-3-3.X- Ray Diffraction

X-ray Diffraction equipment model X´ Pert PRO with Monochromator, Cu-radiation (λ =1.542 ºA) at 45 K.V.,35M.A.and scanning speed 0.03/ sec. were used. The reflection peaks between 2 θ =20 and 60º, corresponding spacing (d, ºA) and relative intensities(1/1º) were obtained. The diffraction charts and relative intensities are obtained and compared with ICDD files. This device used to determine the changes that have occurred in archaeological wood compounds. a Philips X-ray Diffractometer with Cu Kαradiation in faculty of archaeology – Cairo University has been used also to analyze Blood wood to identify the crystalline compounds which did not appear in (C, H, N) analysis.

2-3-4.FTIR

It is used for interpretation samples of archaeological wood and completing mixture. FTIR helped to identify the functional groups which appeared or disappeared before and after exposure to thermal ageing. (Derrick, M.1999)

2-3-5. Light meter

Light meter Color matching system (Color eye 3100) Using uniform CIEL*A*B*(Where color lightness L*, color coordinates+a*reddish /green+b*is the yellowish /bluish parameters). The color Difference ΔE between the sample before and aftertreatment is given by: $\Delta E=\sqrt{(\Delta L*)^2+(\Delta a*)^2+\Delta b*)^2}$

Where: ΔL*= L* (sample) – L* (standard) spectrophotometer, SDL, England.(Ibrahim,S.,2011)

2-3-6.Extraction dye from blood wood

The experimental part in this paper as follows:

First: extraction thevegetable dye by immersing a Blood wood in hot water and leave them for two days, as shown in fig.(3) and the same thing happened with the mineral oxides.

Second: The two pieces of medical cotton immersing one in vegetable dye solution (A) and the other in solution of mineral oxides (B) after that the two pieces lift to dry for two days.

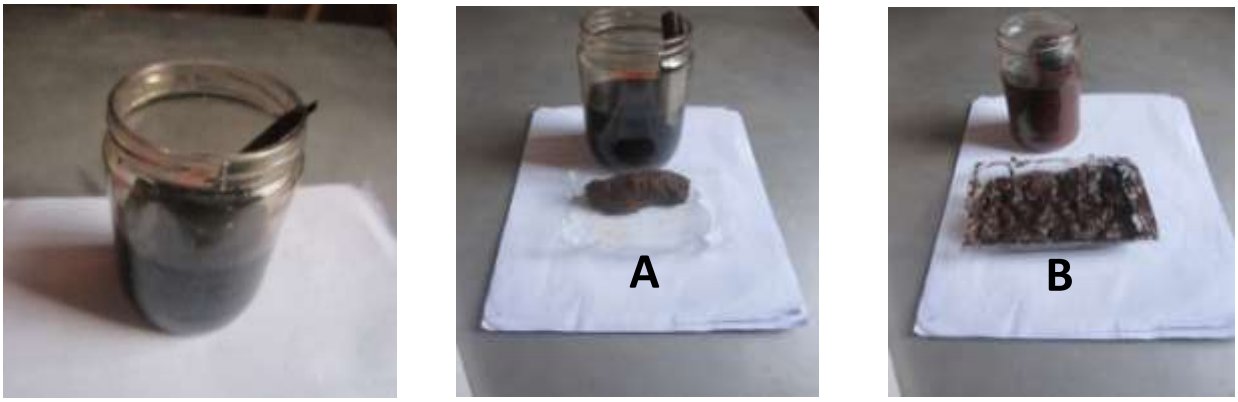


Figure (3) Shows extracting dye from blood wood, A, dyed cotton with haematoxylin dye, and B, colord cotton with mineral oxides

Third: The Experimental samples have prepared to apply thermal ageing on it. These specimens consisted of two types of samples (A) areconsisting of (Beva 371 10%,medical cotton, and vegetable dye) (B) consists of (Beva 371 10% ,medical cotton, and metal oxides) after mixing the materials, placed it in two double mould (A, B) put in nature air as shown in fig.(4). and (Ah, Bh) were the same mixture but it put in thermal oven under 100 ° C for 100 hours. After 100 hour there are some changes.
light meter has been using to measure the intensity of color and differences in samples before and after those steps.

Figure (4) Shows completing mixtures inside double mould before artificial thermal ageing
A/ B mould lift in nature air , and AH/ BH mould will put in an thermal oven

III. Results and Discusstion

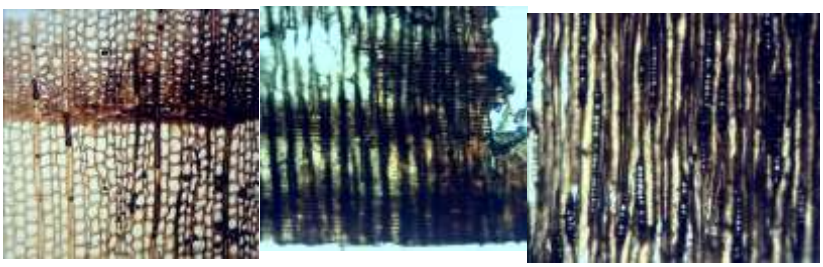
3-1-1.Nacked eye
It shows that there are different holes on archaeological wood surface, As a result of insects infection

These holes represent a source of future e damage.

The Blood wood was* (Identified in the botany department Faculty of Science, Cairo University) heart wood in this tree is dark brown color. Hematoxylin dye is concentrate in heart wood of this tree (.2004 Abdel Magid, A). As shown in fig. (5)

Figure (5) Shows blood wood tree containing dye

3-1-2.Optical Microscope
This examination shows that archaeological wood is Lebanese cedar as shown in Fig. (6,A,B,C)



(A) (B) (C)

Figure (6) Shows Cedar wood sections , A. cross section, B. tangential section, C. radial section
Growth rings distinct, cross section from early- to latewood gradual

3-1-3. Scanning Electron Microscope

(SEM) examination shows the insect holes on the surface of archaeological wood as shown in fig. (7) .
The interior parts are in a good condition as shown in fig.(8,A,B,C) SEM micrographs shows cedar wood sections, A. cross section, B. tangential section, C. radial section and there are not any hole under microscope.
While Figure (9) Shows SEM micrographs of blood wood and the cells of dye from heart wood where there is the haematoxylin dye.

Figure (7) Shows SEM micrograph of insect holes on surface of archaeological wood.

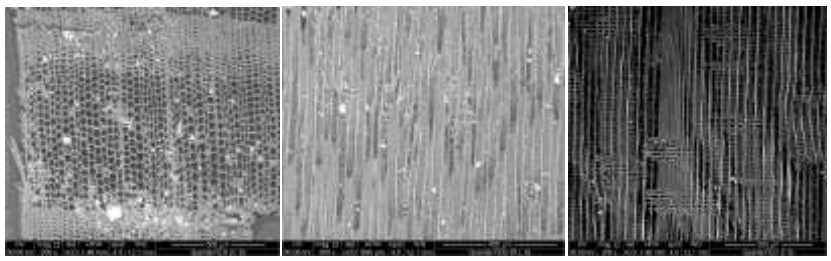


Figure (8) Shows SEM micrographs of cedar wood sections, A. cross section, B. tangential section, C. radial section and there are not any hole. Pit apertures circular to elliptic in the earlywood, slit in the latewood.

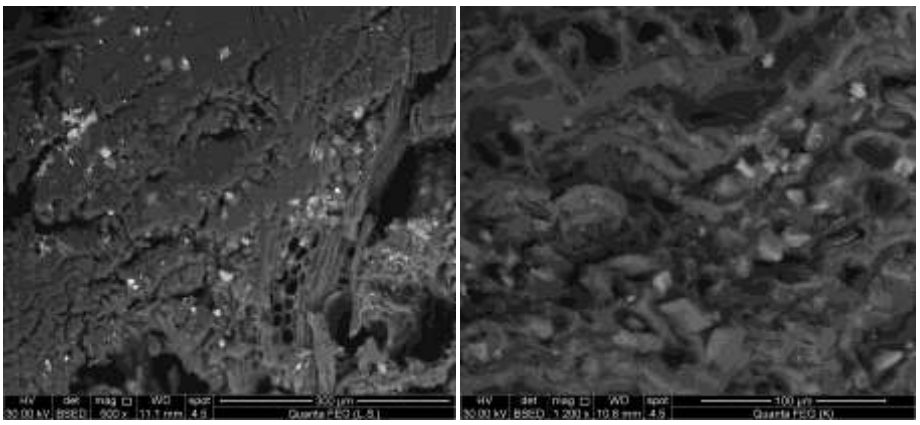


Figure (9) Shows SEM micrographs of blood wood and the cells of dye

3-2.Analysis

The results of different analysis are as follows:

3-2-1. (C,H,N) analysis

It determines the percentage of elements of Carbon , Hydrogen and Nitrogen in archaeological wood and blood wood.So C,H,N analysis is important to compare between structure of archaeological wood and a new wood which used in extracting vegetable dye. This analysis was made in (Micro Analytical Center, Cairo University) as shown in next tables no.(1,2)

Table (1) shows the results of C,H,N analysis of archaeological wood

Sample	C%	H%	N%
Ceder wood	37.25	5.22	1.60

Table (2) shows the results of C,H,N analysis of Blood wood

Sample	C%	H%	N%
Bloodwood	44.93	6.02	0.51

According to obtained results it is found that the ratio of Carbon and Hydrogen in the Blood wood is higher than archaeological wood because it is fresh and has no damage like archaeological wood. The nitrogen components in archaeological cedar wood were higher than blood wood as a result of insect infection and changes was occurring in archaeological wood as a result of insects activities but nitrogen ratio was low in fresh blood wood because heamatoxylin dye consistsof $C_{16}H_{14}O_6$as shown in figure (10).

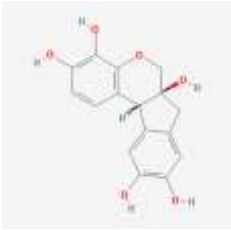


fig.(10) Shows 2D Structure of $C_{16}H_{14}O_6$

https://pubchem.ncbi.nlm.nih.gov/conpound/Heamatoxylin

3-2-3. XRD

XRD analysis which shown in Figure (11) illustrates that archaeological wood consists of cellulose ($C_6H_5O_{10}$) card No. 00-050-2241, and quartz (SiO_2) card No. 00-002-0285.XRD of Blood wood fig.(12) consists of Iron Phosphate Hydrate ($FePO_4.2H_2O$)Card number 03-0452 as one of the Iron components in the blood wood, Quartz SiO_2 Card number 27-0605 as a kind of silicate components ,Calcite $CaCO_3$ Card number 5-0586. Calcite may be from Soil feeding . Finally traces of Cellulose ($C_6H_5O_{10)n}$. This vegetable dye can give its colour to several materials like fibers and it has insecticide propertys(Tucker,S.1997).

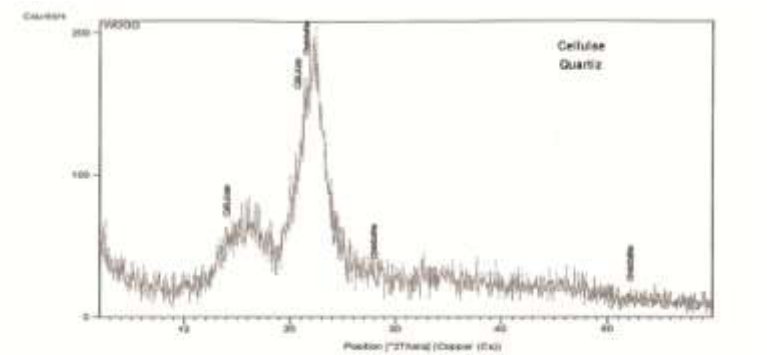


Figure (11) shows XRD chart of archaeological wood

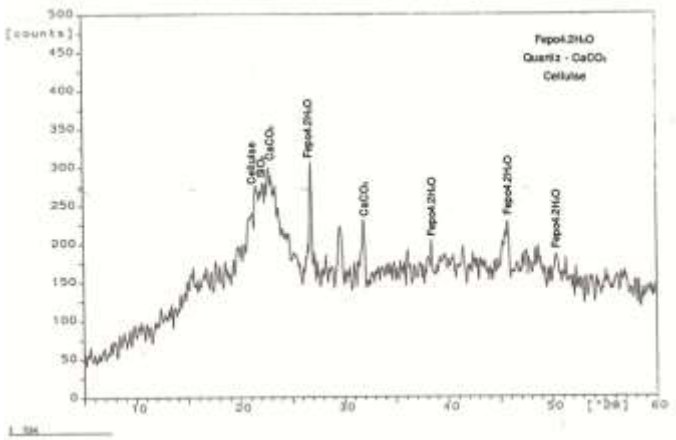


Figure (12) shows XRD chart of blood wood

3-2-4.EDX

EDX analysis of archaeological wood analysis shown in Figure (13) consists of (C, O, Ca, K,) elements as the main components of the wood. The percentage of calcium is high as a remains pigments used on surface, (S, Cl) are elements of chemical deterioration on the archaeological wood.

. EDX analysis of blood wood shown in Figure (14) illustrates that It consists of (Fe, Mn, Ca, Si, Al, Ti, C, O, Mg) elements are the main components of vegetable dye in Blood wood.

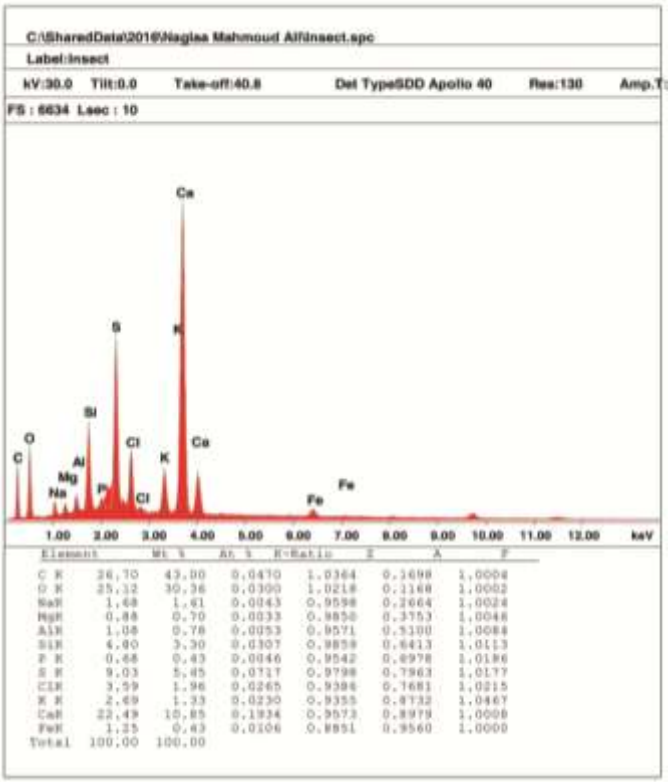


Figure (13) shows EDX chart of archaeological wood

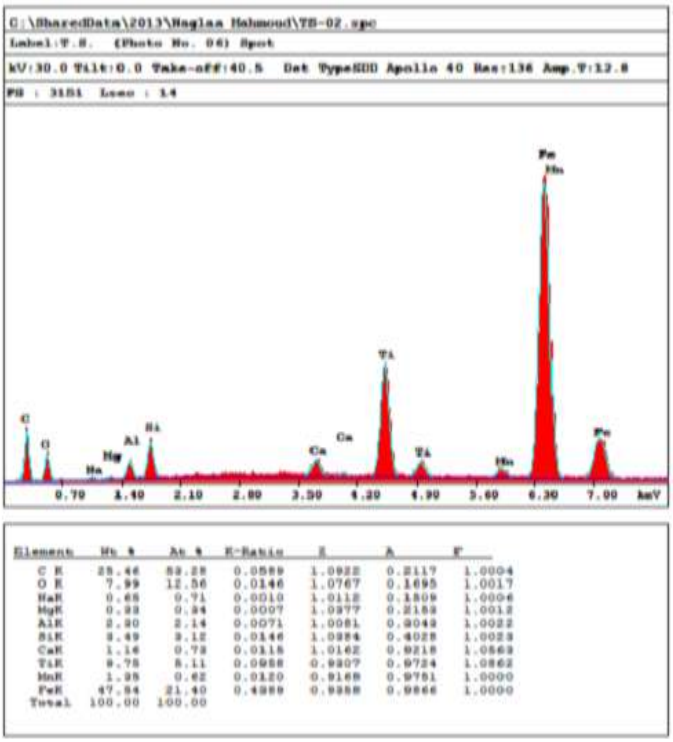


Figure (14) shows EDX chart of blood wood

3-2-5. FTIR

FTIR analysis of Archaeological wood sample, Blood wood sample, and two double form of completing mixture (A,B),(Ah,Bh) show in fig.(15) illustrates that all samples participate in more than one functional groups, but two samples (B), (Bh)which consisted of (Beva) 371 10%, medical cotton, metal oxides) different in the pigment materials as a results heterogeneity in after thermal ageing appear in shrinking of sample after thermal ageing. where there are homogeneity and stability in mixture (A), (Ah) which consists of (Beva 371 10%, medical cotton, vegetable dye) as shown in fig. (16).

Peak find

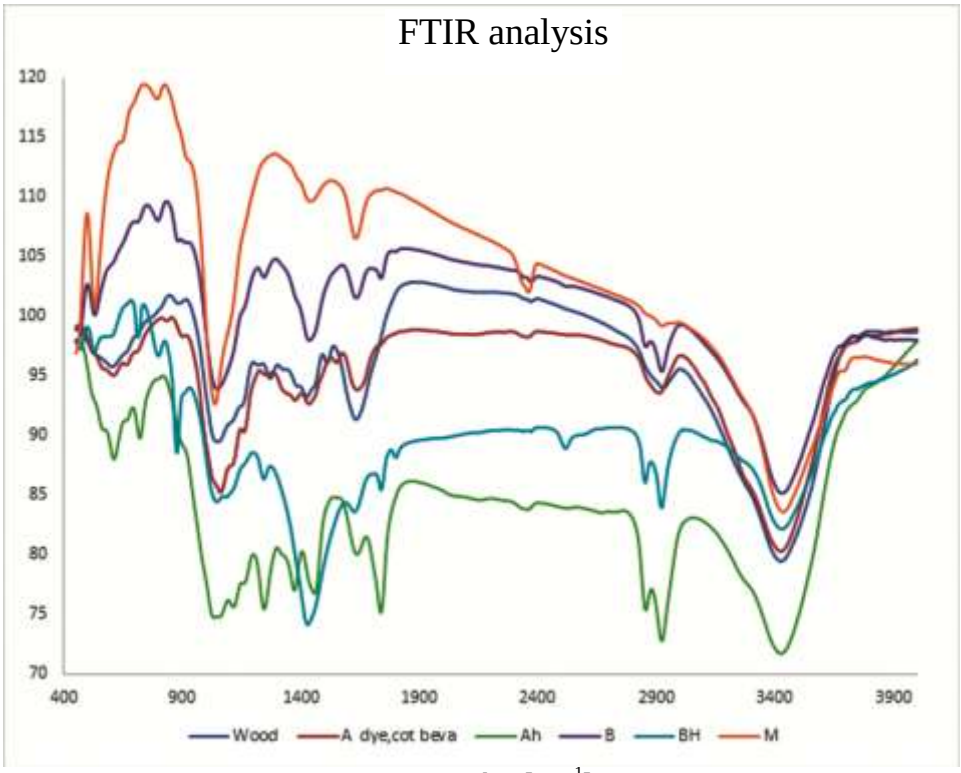


Figure (15) shows FTIR chart of - archaeological wood,-A completing mixture with vegetable dye ,AH the same mixture after thermal ageing - B completing mixture colored with mineral oxides

,-BH the same mixture after thermal ageing –M mineral oxids with cotton filler only.

Fig. (16) Shows completing mixtures after and before artificial ageing

A= mixture of (Bebva 371+cotton+vegatabl dye) befor artificial ageing
AH= A mixture after artificial ageing
B= mixture of (Bebva 371+cotton+metal oxids) befor artificial ageing
Bh=B mixture after artificial ageing

3-2-6. Light meter

archaeological wood (Arch. Face) = archaeological wood surface, (Arch. Back) = archaeological wood back are measuring with light meter and obtained the following results which recorded in table no. (3)

Table no.(3) shows light meter measuring for archaeological wood and experimental samples.

Sample	L*	A*	b*	ΔE	K / S
A*	42.99	13.42	23.40	-	2.6515
AH*	42.02	13.13	25.59	2.41	2.4908
B*	40.03	9.69	18.47	-	2.6389
BH*	32.57	8.69	16.15	7.88	4.441
Arch. Face	51.23	10.48	27.48	-	1.3642
Arch. Back	45.39	9.42	25.06	6.41	2.0448

From the previous table (B), (Bh) which consisted of (Beva 371 10%, medical cotton, and metal oxides) after artificial ageing shows high changing in color degree, but mixture (A), (Ah) which consists of (Beva 371 10%, medical cotton, and vegetable dye) have good stability in color degree after artificial ageing. So the last mixture is the most suitable mixture for completing insects holes in Sabil Al-Kazlar as shown in fig. (17, 18).

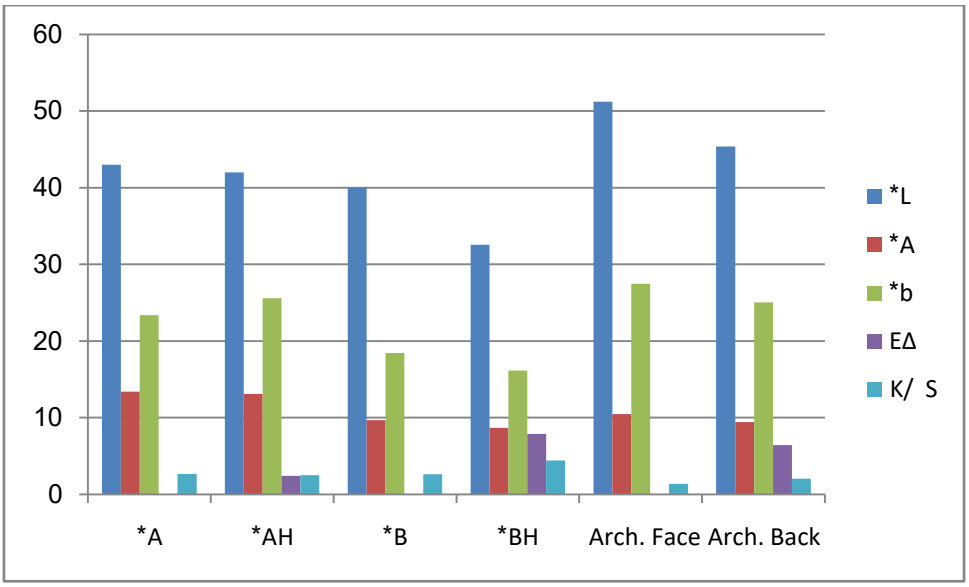


Figure (17) shows results of light meter

L*= color lightness. $\Delta E=\sqrt{(\Delta L*)^2+(\Delta a*)^2+\Delta b*)^2}$

a*= reddish /green. K /S= The colour strength.

b*= yellowish /bluish.

Figure (18) shows insect holes on archaeological wood surface before and after restoration

Conclusions

archaeological wood from sabil Al-Kazlar demonstrate that the wood consists of Lebanese cedar where Blood wood is the common name of Haemtoxylum camechianum L. tree heart wood in this tree is dark brown color because the Hematoxylin dye concentrates in it.

The Scanning Electron Microscope (SEM) examination shows the insect holes damage on the surface of archaeological wood, while SEM micrographs show the surface of heart wood where is the vegetable dye.
According to(C, H, N) analysis, the researcher find that the ratio of Carbon and Hydrogen in the Blood wood higher than archaeological wood because it is fresh and has no damage such as archaeological wood,while the nitrogen component in archaeological cedar wood were higher than Blood wood as a result of insects infection which caused some changes for archaeological wood .
XRD analysis illustrates that archaeological wood sample consists of cellulose and quartz. while Blood wood consists of Iron Phosphate Hydrate, Silicon oxide and cellulose.

EDX analysis of archaeological wood sample analysis shows that archaeological wood consists of (C, O, Ca, K) elements as the main components of the wood, and (S, Cl) are elements of deterioration , and the (Si, Al, Fe, Mg, Na) elements represent the dust on the archaeological wood. EDX analysis of blood wood illustrates that it consists of (Fe, Mn, Ca, Si, Al, Ti, C, O, Mg)) which conceder the main components of vegetable dye in Blood wood.

FTIR analysis of Archaeological wood sample, Blood wood sample, and two double form of completing mixture (A,B),(Ah,Bh) illustrates that all mixture participates in more than one functional groups, but two mixture (B, Bh) consisted of (Beva 371 10%, medical cotton, and metal oxides) were different in the pigment materials (mineral oxides) so there are heterogeneity in naked eye examination FTIR chart after thermal ageing such as shrinking of sample and change of color degree after thermal ageing as a result of changing in some groups. where there are homogeneity and stability in mixture (A), (Ah) which consists of (Bava 371 10%, medical cotton, and vegetable dye).

From Light meter analysis the researcher can observe that (Bh) which consisted of (Beva 371 10%, medical cotton, and metal oxides) after thermal ageing appear has high changing in color degree, but mixture (Ah) which consists of (Beva 371 10%, medical cotton, and vegetable dye) have good stability in color degree after thermal ageing. So the last mixture is the most suitable mixture for completing insects holes in Sabil Al-Kazlar.

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