Comparative Study on the Effect of Nano Calcium Hydroxide and Klucel E. as Consolidants Materials for Poly-chromed Wood.

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Abstract

Consolidation process is considered one of the most important step in wood conservation treatment, and there are different materials used as a Consolidant materials such as Nano-restore is a highly compatible with carbonate based materials such as calcareous substrate, plasters, wall paintings and carbonatic stone, Nano-restore also achieving a good results and good re-adhesion of detached pigment flakes without any side effects. Klucel E, furthermore, is one of cellulose ethers (CE) which are widely used as a adhesive materials for wood and paper artefacts. Wherefore this study stablished to evaluate the effect of both on the consolidation of painted wood. different analytical methods such as FTIR and Color meter have been used in the assessment to determine the Chemical alteration which accrue to painting layers, plasters of lime and gypsum and to the wooden base.

Key words

Klucel E, Nano- restore, FTIR, colorimetric measurement

Introduction

Wooden materials are representing a very high percentage from our culture heritage, it used for making different artifacts, and sometimes used as a support for the paint layer (poly-chromed wood/ mono-chromed wood). unfortunately wood is very sensitive to damage according to its hygroscopic nature (coservation of wooden objects) (hager, spchniewind, & unger, 2001) (mecklenburg, tumosa, & erhardt) so that the wooden artifact and it's all layer are very vulnerable to damage when it exposed to different deterioration agent such as (physical, biological and chemical agent)

(Ahmed, Elsheroogy, Al-muheisen, El-oqlah, & villeneuve, 2018) and because of the importance of preserving our cultural heritage, the consolidation process is consider one of the most important process in conservation of wood because it increase the wood strength and its durability {cataldi,deflorian, & pegoretti,2015}{elserogy, Al-muheisen, Villeneuve, & El-pqlah,2019} (elserogy, Al-muheisen, villeneuve, & El-oqlah, 2018) (El Hadidi & Darwish, 2014),wherefore various consolidant materials have been developed and used for the consolidation process of wood and for the paint layer, this consolidant materials were classified into two groups, natural polymers such as (gum Arabic and Funori) and Synthetic polymers such as acrylic resins (Paraloid B 72, primal, Acryloid B72), thermoplastic polymers such as (P(EMA/MA) poly ethyl methacrylate / methyl acrylate, poly vinyl butyral (PVB) and poly vinyl acetate (PVAc) (frances, torroja, folch, & recasens), Thermosetting polymers (as epoxy and polyester) (sideridous, 2016), and Cellulose derivatives as (CMC, Methyl cellulose, poly ethelen glycol and poly (2-Ethyal-2-Oxazoline), (klucel G), and Ethyl hydroxyethyl cellulose (Ethulose), cellulose nitrate, cellulose acetate. (wiki, n.d.)

(pacheco, 2015)Cellulose derivatives are considered one of the most important consolidating materials used in consolidation of cellulosic materials as wood and paper (mohamed & Ali) , (poletto,, zattera,, & santana, 2012) Klucel E , a type of cellulose ethers, (Hydroxy propyl cellulose)is a semi-industrial materials using in consolidation process of the wooden object back 1976 (nastto, et al., 24/4/2015) (feller & wilt, 1990) (5) (horie, 1987). Its particles dissolved in water or different organic solvent such as ethanol or isopropanol, and it has a low viscosity than klucel G (Berry, 2009) so as a result of this it has a good penetration in the wood, and the penetration level of the consolidating material into the object is a most important factor for a good consolidation process. (cataldi, deflorian, & pegoretti, 2015). This material also not make any change in the chemical composition of the wood (sayed, et al.) The ultrasonic mister technique also had been used to consolidate the powdery paint layer. (michalski, dignard, handel, & arnold). However some of this materials improved their success in consolidation process there are some materials failure to consolidate the wood because it caused harmful effect on long term for the conservation aims, an acidic deterioration and it may by not reversible. So researcher now trying to improve the properties of the consolidant materials by mixing different materials with each other such as shellac with colophony or use micro-composites solution consisting of Paraloid with microcrystalline cellulose (El Hadidi & Darwish, 2014) (elserogy, Al-muheisen, villeneuve, & El-oqlah, 2018) (wolbers, mcginn, & duerbeck) (cataldi, deflorian, & pegoretti, 2015) (walsh, et al.) (villanueva, telles, roman, & sales, 2012). But in the recent time nanotechnology improved its success in conservation and restoration of organic materials such as oil painting, wood and paper, inorganic materials such as wall painting, glass and stone (mohmed, shawky, & rashed) (waked) (fernandez, villallaba, fort, & Rabanal, 2017),there are different Nanomaterial's used in the conservation process such as Nano-restore or Nano-lime composed of calcium hydroxide Nano particles, it dissolve in different alcohols as cellulose derivatives, it was used for the conservation of porous limestone, plaster, mortar and wall paintings, in 1960s it was used for the surface consolidation of frescoes but in the early 2000s it was used as a stone consolidant and it began to be widely used (Nano-lime aprtical guide to its use for consolidating weathered limestone, 2017).(D'armada & Hirst , 2012) (oterp, elena charola, grissom, & starinieri, 2017) (frankeova & slizkova, 2012) (Goncalves, 2014) (Vojtechovsky & Bayer, 2016), it has both higher and faster carbonation with a good penetration So at a result of this properties it has a better re-cohesion, faster carbonation and it hasn't make any aesthetic changes like the limewater (oterp, elena charola, grissom, & starinieri, 2017) After the evaporation of the solvent the calcium hydroxide is convert into calcium carbonate by the reaction with carbon dioxide(Ziegenbalg, Brummer, & Pianski, 2010).at high RH(75-95%) the carbonation may by accurse in 9-10 days at room temperature (oterp, elena charola, grissom, & starinieri, 2017) ,Nano-limes also has a good result to detached pigment flakes without causing any further damaged (oterp, elena charola, grissom, & starinieri, 2017). The effect of consolidant Materials should by studied before using directly on the artifacts, there are different analysis technique can use such as microscopic inspection, FTIR, colorimetric measurement, The CIELAB color parameters (L^*, a^*, b^*) were used to detect the color change, The differences in parameters ΔL^*, Δa^*, and Δb^* and the total color differences ΔE^*, where L^* is the lightness, positive (a) is red while negative (a) is green, positive (b) is yellow while negative (b) is blue. (sayed, et al.) (matsuo, et al., 2012),

fourier transform spectroscopy(FTIR) has been used as a simple technique for determine rapid information about the structure of wood and chemical changes taking place in wood due to different treatments methods. (poletto,, zattera,, & santana, 2012) (naumann, peddireddi, kues, & poiie, 18 may 2014) (muller, schopper, vos, kharazipour, & polle, 2014) (traore, kaal, & cortizas, 2016). And it also gives the ratio of lignins, carbohydrates, aromatic and other compounds (naumann, peddireddi, kues, & poiie, 18 may 2014). This technique requires small sample sizes and short analysis time. the spectra were separated into two regions, the OH and CH stretching vibrations in the 3800–2700 cm_1 region and the ‘‘fingerprint’’ region which is assigned to stretching vibrations of different groups of wood components at 1800–800 cm_1 (poletto,, zattera,, & santana, 2012)

Tabel.1 (this table shows functional group of wood, gypsum and lime)

Wavenumber (cm-1)	Compound	References
3800-2700	C-H, O-H stretching	(poletto,, zattera,, & santana, 2012)
3336	O-H stretching	(muller, schopper, vos, kharazipour, & polle, 2014)
3300-3600	O-H stretching	(poni, 2017) (pandey, 1998)(004) (poletto,, zattera,, & santana, 2012)
2800-3000	C-H stretching	(pandey, 1998) (poni, 2017) (Dobrică, Bugheanu, Stănculescu, & Ponta, 2008)
2938-2882	CH- stretch in methyl- and methylene groups	(muller, schopper, vos, kharazipour, & polle, 2014)
1371-1372	C-H deformation in cellulose and hemicellulose	(muller, schopper, vos, kharazipour, & polle, 2014) (naumann, peddireddi, kues, & poiie, 18 may 2014)
1500-200	CH/OH bending	(marchessault)
1730-1740	C=O stretching	(pandey, 1998) (poletto,, zattera,, & santana, 2012) (naumann, peddireddi, kues, & poiie, 18 may 2014) (muller, schopper, vos, kharazipour, & polle, 2014) (poni, 2017)(
1595,1510,1270	C=C/ C-O stretching	(poletto,, zattera,, & santana, 2012)(002) (pandey, 1998)
1800-900	Finger print	(poletto,, zattera,, & santana, 2012) (pandey, 1998)
1318	CH3 wagging	(pandey, 1998)
1163	C-O-C asymmetric vibration	(pandey, 1998)
1140-1080 cm"'	asymmetric SO.'" stretching band	(Derrick , Stulik, & Landry, 1999)
620 cm"'	SO.'" bending band (not shown)	(Derrick , Stulik, & Landry, 1999)
3700-3200 cm	Anti-symmetric and symmetric O-H stretching band	(Derrick , Stulik, & Landry, 1999)
1490-1370 cm·'	CO stretching band	(Derrick , Stulik, & Landry, 1999)

91 0-850 cm"'	O-C-O bending band	(Derrick , Stulik, & Landry, 1999)

Materials and methods

Materials

1-wood:

Pine wood samples were used for this study, its size 3×3×3 cm.

2-consolidants

A: Nano-restore: (calcium hydroxide Nano particles)

B: Klucel E: (Hydroxy propyl cellulose) (HPC)

3-colors

A-limonite

It consists of barium chromate, strontium chromate, or a mixture of lead chromate with lead sulphate. It's a stable yellow with low hiding power.

B-Hematite (fe2o3)

Its redishbrowen color compost of ferric oxide.

C-Goethite

Iron oxy-hydroxide mineral naturally found with iron mineral, manganese and bauxite ores. Goethite is composed of Fe, O and OH. It sometimes contains impurities such as Al2O3, MnO, Cao and SiO2, which may by reach to 5%(morris 1985)

Methods:

1_ preparation of plaster layer

Two plaster layers were prepared, gypsum and lime. In order to prepare the plaster layer at first the glue was dissolved in water 1:50 gm, after that mixed it with the lime and applied it over the wood samples then leave it to dry, and the same steps was applied also to make the gypsum layer.

2_prepration of colors

At the first the colors were grounded to very fine powder after that it was soaked in distilled water for a few days, after this steps the colors were ready to apply in the ground layer.

3-preparation of consolidants

Klucel E dissolved in distilled water at concentration 1%. But the Nano-restore materials was purchase ready to use at concentration 1%.

Results and Discussion

1- Visual Inspection by Digital Microscope:
There are a very slight change in the color of the sample was observed after the artificial aging.

A-wood



Fig.1. Wood Fig.2. wood with klucel E fig.3. wood with nano-restore

B-lime

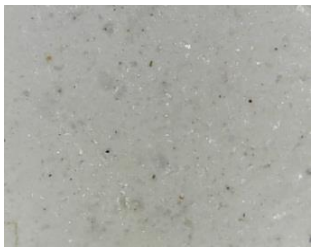
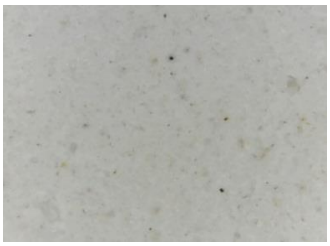




Fig.4Lime Fig.5.Lime with kulcel E Fig.6. Lime with nano-restore

C- Gypsum

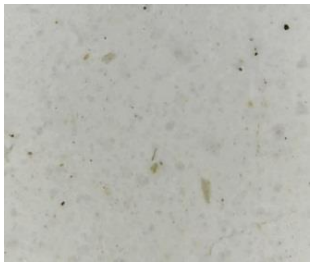
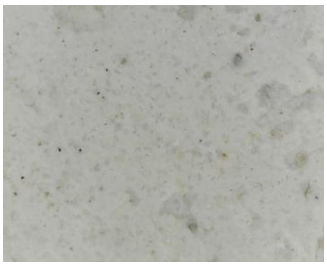




Fig.7.Gypsum Fig.8. gypsum with klucel E Fig.9. gypsum with nano-restore

D-Yellow



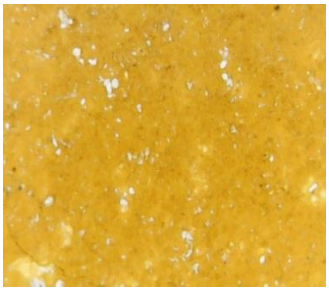


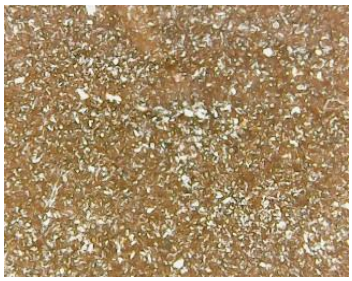
Fig.10.Yellow Fig.11.yellow with klucel E Fig.12.yellow with nano-restore

E-Brown


Fig.13. Brown


Fig.14.brown with klucel E


Fig.15. brown with nano-restore



F- Red

Fig.16.Red

Fig.17.red with klucel E

Fig.18.red with nano-restore

2- Colorimetric Measurement

The monitoring of color changes of all treated samples are given in the table2, all sample give a result below 3.5 (ΔE<4)

Fig.19,20. shows the ΔE value of treated wood sample with kulcel E (ΔE 1.53), and with nano-restore (ΔE 1.88)

Fig.21,22. shows the ΔE value of treated gypsum sample with kulcel E (ΔE 1.51), and with nano-restore (ΔE 1.46)

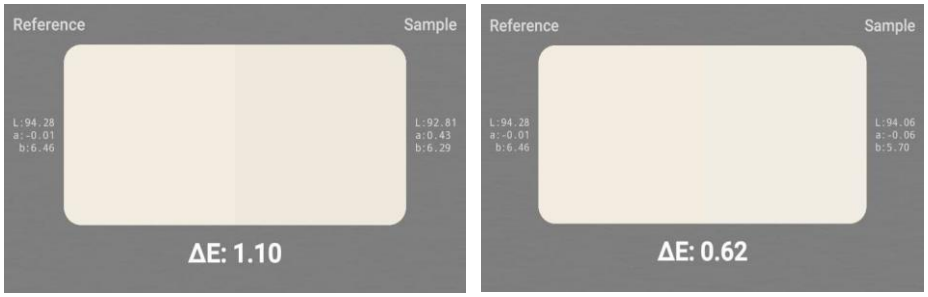


Fig.23,24. shows the ΔE value of treated lime sample with kulcel E (ΔE 1.10), and with nano-restore (ΔE 0.62)

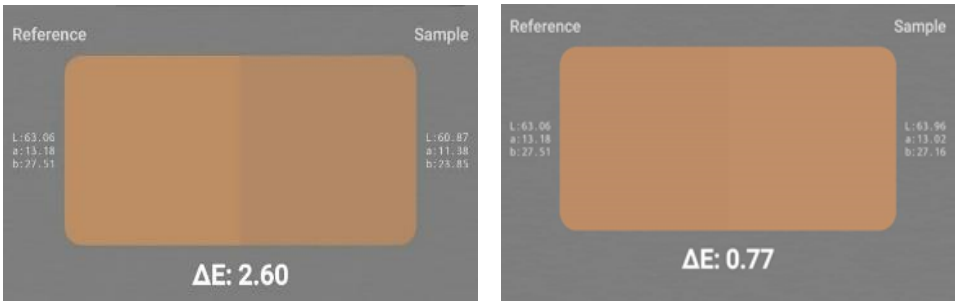


Fig.25,26. shows the ΔE value of treated yellow sample with kulcel E (ΔE 2.60), and with nano-restore (ΔE 0.77)

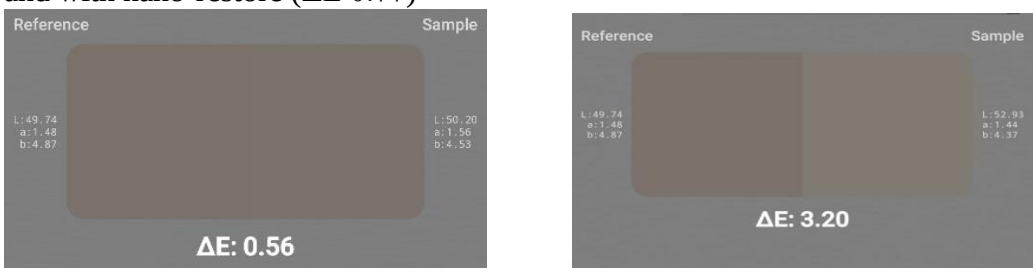


Fig.27,28. shows the ΔE value of treated brown sample with kulcel E (ΔE 0.56), and with nano-restore (ΔE 3.20)

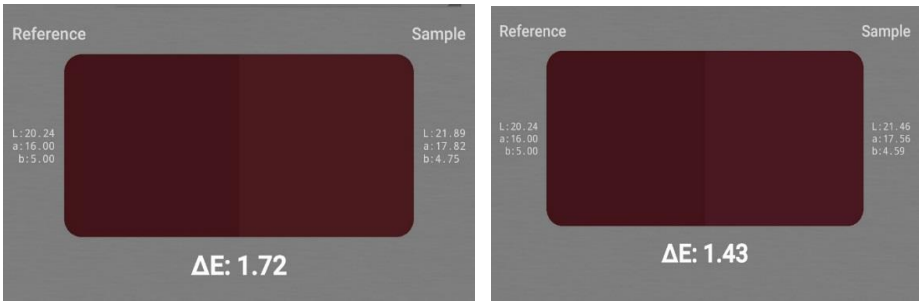


Fig.29,30. shows the ΔE value of treated red sample with kulcel E (ΔE 1.72), and with nano-restore (ΔE 1.43)

Table.2.shows the color metricmeasurment of all samples

Sample	L	A	B	ΔE
W	76.38	6.83	23.47	
WK	76.92	5.52	21.06	1.53
WN	77.02	5.08	20.85	1.88
L	94.28	-0.01	6.46	

LK	92.81	0.43	6.29	1.10
LN	94.06	-0.06	5.70	0.62
G	91.22	0.07	6.68	
GK	92.08	0.51	8.34	1.51
GN	93.57	0.18	6.82	1.46
R	20.24	16.00	5.00	
RK	21.89	17.82	4.75	1.72
RN	21.46	17.56	4.59	1.43
Y	63.06	13.18	27.51	
YK	60.87	11.38	23.85	2.60
YN	63.96	13.02	27.16	0.77
B	49.74	1.48	4.87	
BK	50.20	1.56	4.53	0.56
BN	52.93	1.44	4.37	3.20

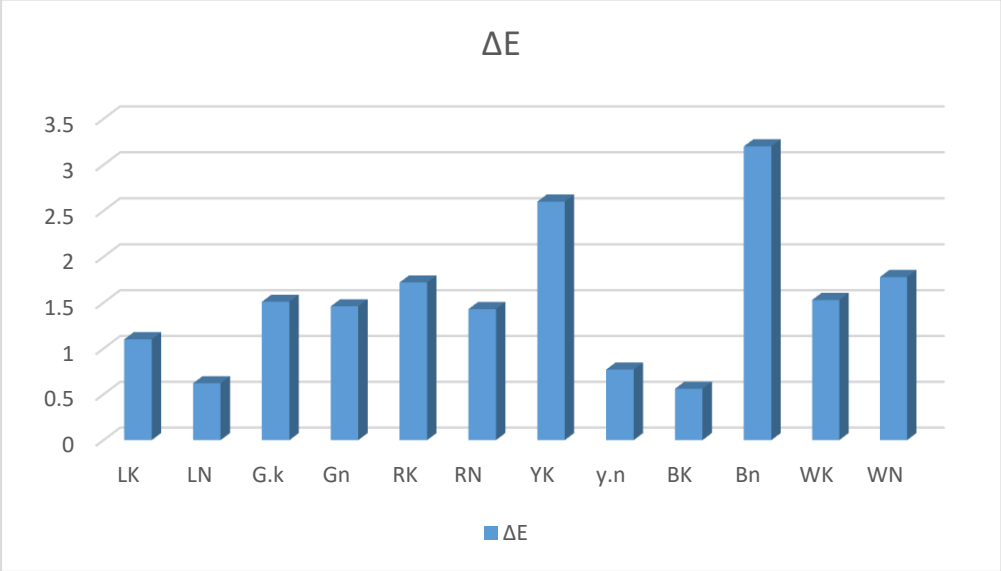


Fig.31 (shows the measured ΔE values for all samples)

3- Fourier transform spectroscopy (FTIR)

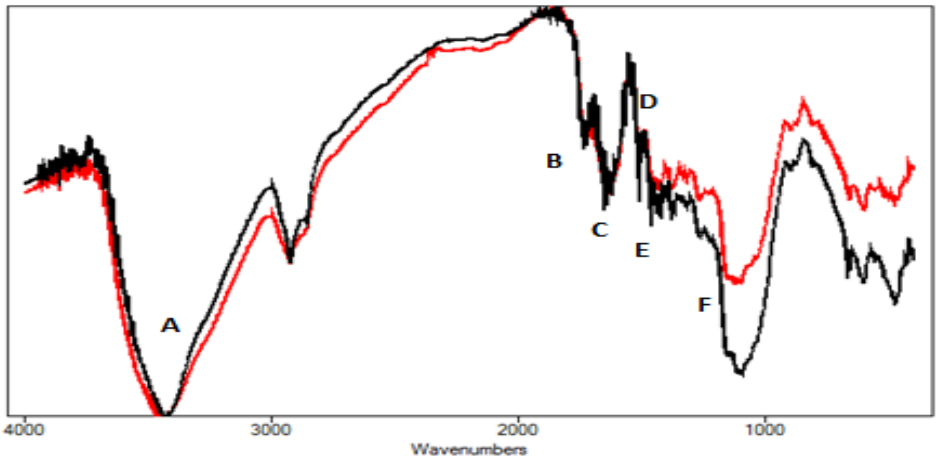


Fig.32.FTIR spectra of treated WOOD wit KULCEL E

Table.3. shows the result of FTIR analysis of treated WOOD wit KULCEL E

SYM	Functional group bands	Sample	Standard	note
A	OH stretching	3300	3345	Cellulose
B	C=O ester	1700	1735	hemicellulose
C	C=0 ketone	**1600**	1642	Cellulose oxidation
D	CH bending	**1440**	1425	Cellulose crystallinity
D1	C=C Aromatic	**1500**	1512	lignin
F	c-o stretching	**1300**	1000-1300	Cellulose polymerization

The treated wood sample with (KE) has a very slight change in OH stretching (3300) and also in Cellulose polymerization area (1300). Hemicellulose (C=O ester) (1700) never has any change. and there is no any change in the Cellulose oxidation area

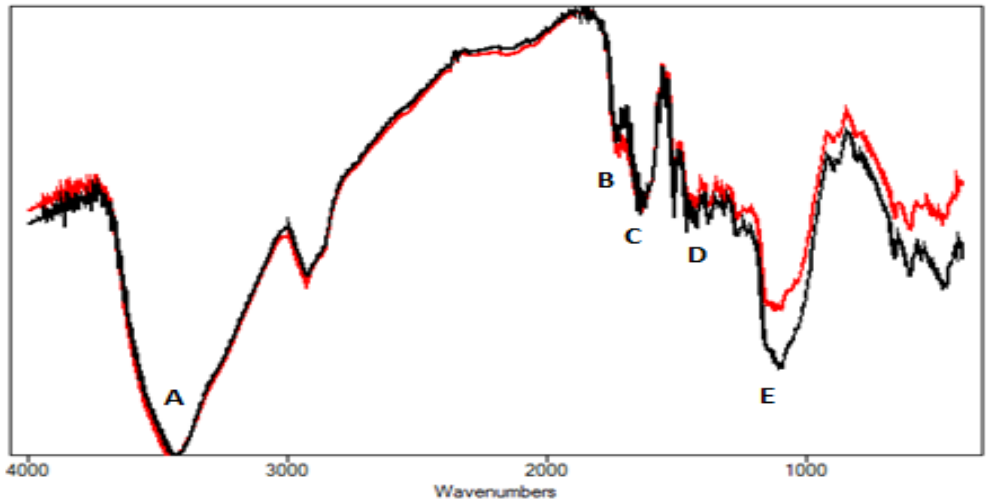


Fig.33.FTIR spectra of treated wood with Nano-restore

Table.4. table shows FTIR analysis of treated wood with Nano-restore

SYM	Functional group bands	Sample	Standard	Note
A	OH stretching	3300	3345	cellulose
B	C=O ester	1700	1735	hemicellulose
C	C=0 ketone	1600	1642	Cellulose oxidation
D	CH bending	1440	1425	Cellulose crystallinity
D1	C=C Aromatic	1500	1512	lignin
F	c-o stretching	1300	1000-1300	Cellulose polymerization

The treated wood sample with Nano-restore has a very slight change in Cellulose polymerization area (1300), and doesn't has any change in OH stretching (3300). hemicellulose (C=O ester) (1700) hasn't any change

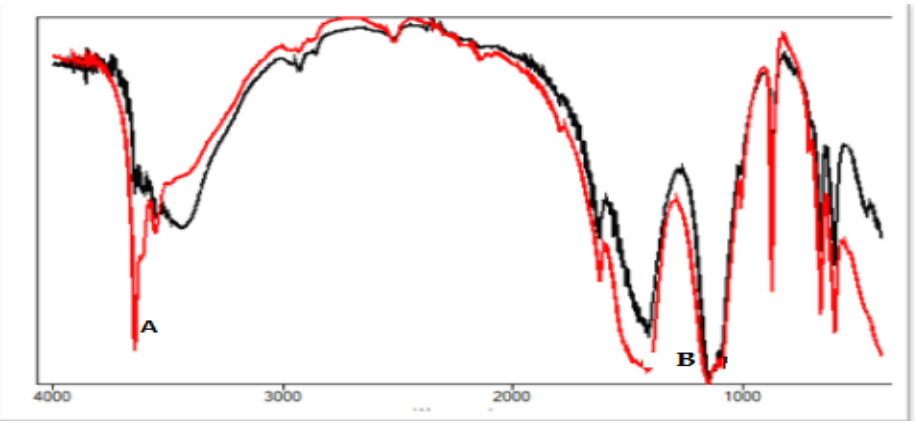


Fig.34.FTIR spectra of treated lime with klucel E

Table.5. Table shows the result of FTIR analysis of treated Lime with klucel E

SYM	Functional group bands	Sample	Standard
A	OH stretching	3300	3345
B	CO3 stretching	1400	1370-1400
C	O-C-O bending	900	850-910

The treated lime with (KE) has a very slight change in CO3 stretching (900). but O-C-O bending never has any change.

The OH stretching appears as a result of using water in lime preparation.

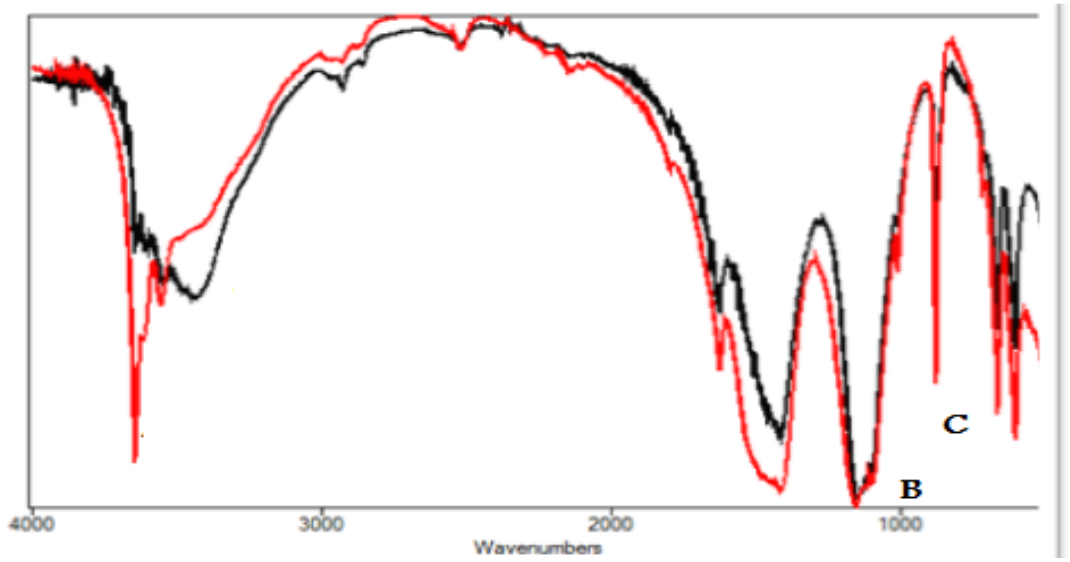


Fig.35.FTIR spectra of treated lime with Nano-restore

Table. 6.Table shows the result of FTIR analysis of treated lime with Nano-restore

SYM	Functional group bands	Sample	Standard
A	OH stretching	3300	3345
B	CO3 stretching	1400	1370-1400
C	O-C-O bending	900	850-910

The treated lime with Nano-restore has a very slight change in CO3 stretching (900) because the chemical composition of Nano-restore like the lime composition.

O-C-O bending never has any change.

The OH stretching appears as a result of using water in lime preparation.

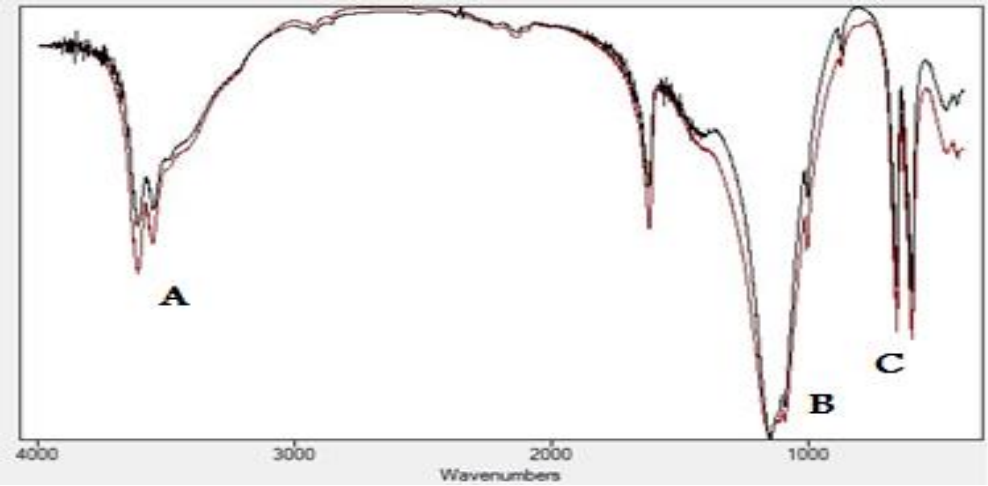


Fig.36.FTIR spectra of treated gypsum KULCEL E

Table.7. Table shows the result of FTIR analysis of treated gypsum KULCEL E

SYM	**Functional group bands**	Sample	Standard
A	OH stretching	3600	3200-3700
B	SO stretching	**1100**	1080-1140
C	SO bending	**800**	850-910

The treated gypsum with (KE) has no any change in OH stretching and SO bending, but it has a very slight change in SO stretching

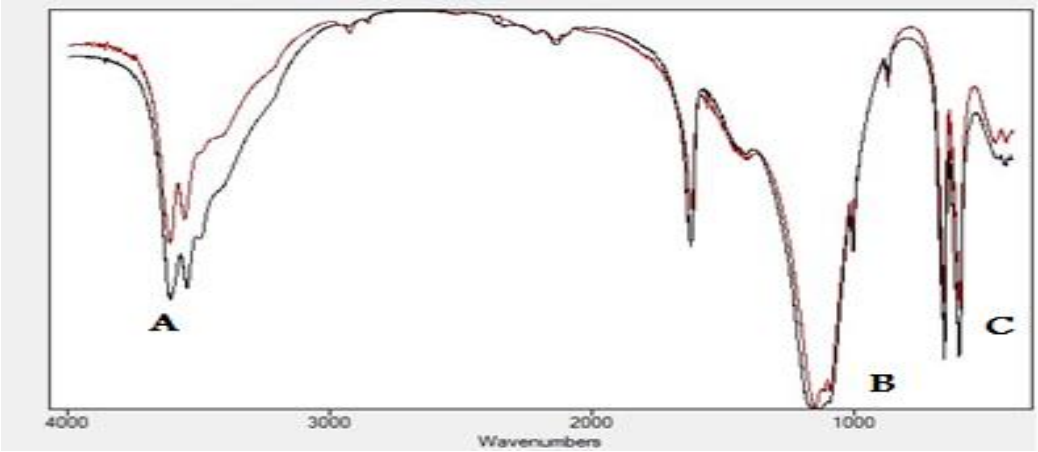


Fig.37.FTIR spectra of spectra of treated gypsum Nano-restore

Table.8. Table shows the result of FTIR analysis of treated gypsum with Nano-resto

SYM	**Functional group bands**	Sample	Standard
A	OH stretching	3600	3200-3700
B	SO stretching	**1100**	1080-1140

C	SO bending	**800**	850-910

The treated gypsum with Nano-restore has no change in SO stretching and SO bending, but it has a very slight change in OH stretching because the gypsum is unstable in water.

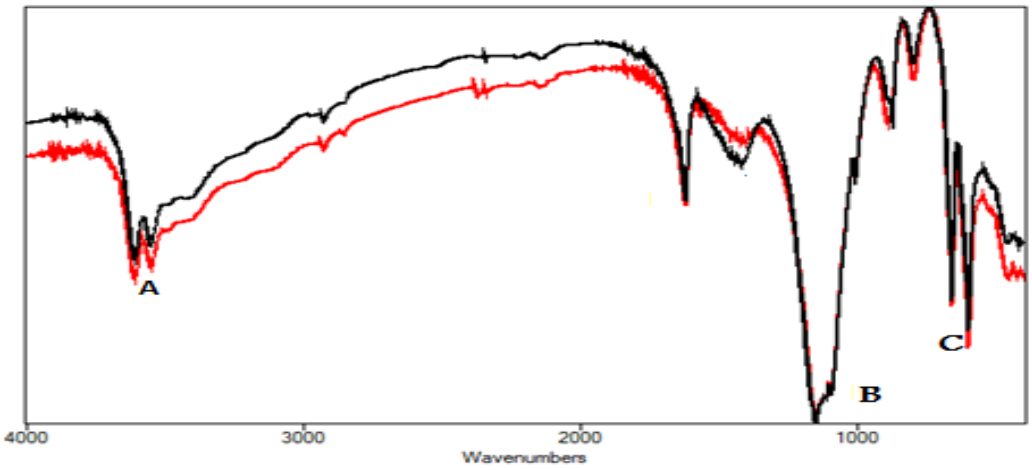


Fig.38.FTIR spectra of treated brown color with klucel E

Table.9. Table shows the result of FTIR analysis of treated brown color with klucel E

SYM	Functional group bands	Sample	Standard
A	OH stretching	3600	3200-3700
B	SO stretching	1100	1080-1140
C	SO bending	800	850-910

The FTIR analysis of treated brown color with (KE) has a very simple result because the composition color appears after 400 cm_1, and the most clear peaks are the gypsum peak because it used as a ground layer.

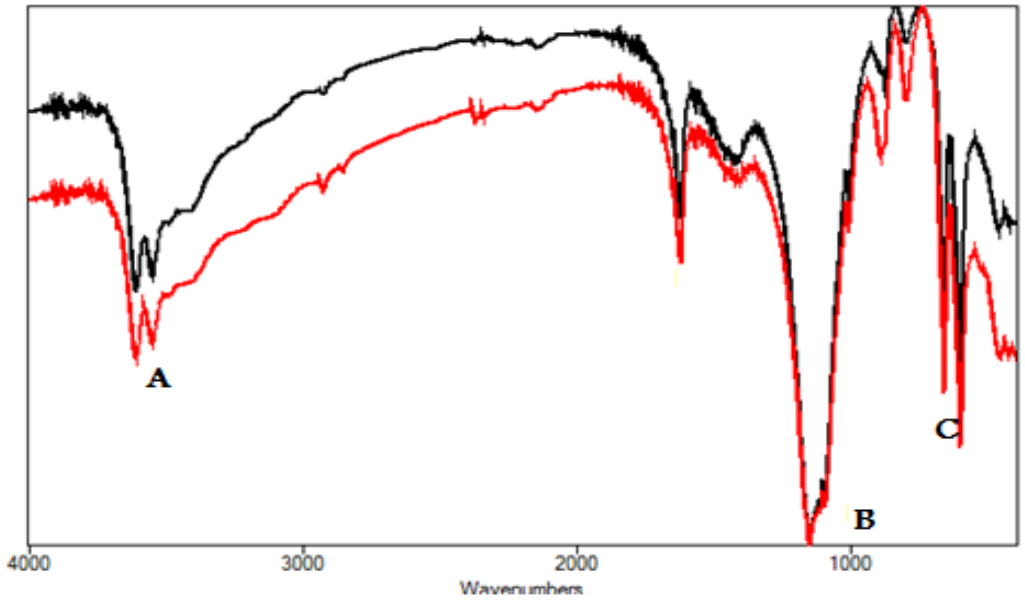


Fig.39.FTIR spectra of treated brown color with Nano-restore

Table.10. Table shows the result of FTIR analysis of treated brown color with Nano-restore

SYM	Functional group bands	Sample	Standard
A	OH stretching	3600	3200-3700
B	SO stretching	**1100**	1080-1140
C	SO bending	**800**	850-910

The FTIR analysis of treated brown color with Nano-restore has a very simple result because the composition color appears after 400 cm_1, and the most clear peaks are the gypsum peak, it has no change in SO stretching, SO bending and has only change in OH stretching**.**

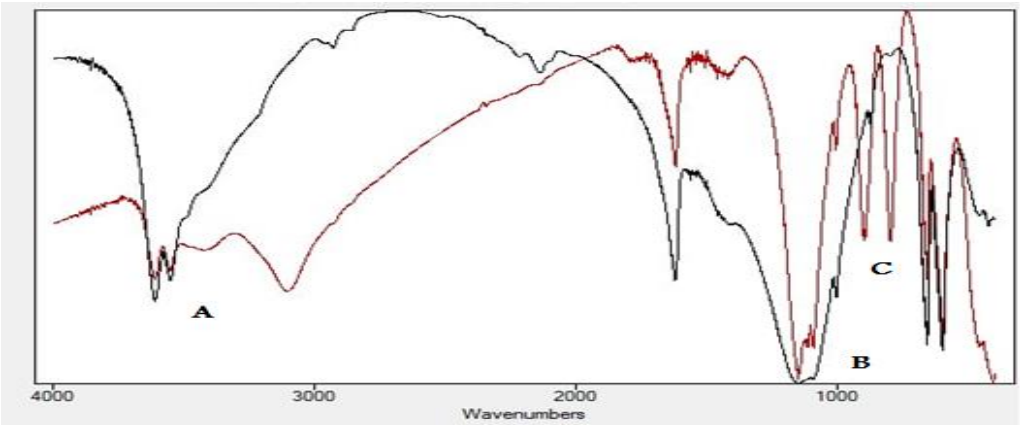


Fig.40.FTIR spectra of treated yellow color with klucel E

Table.11. Table shows the result of FTIR analysis of treated yellow color with klucel E

SYM	Functional group bands	Sample	Standard
A	OH stretching	3600	3200-3700
B	SO stretching	1100	1080-1140
C	SO bending	800	850-910

the FTIR analysis of treated yellow color with (KE)has a very simple result because the composition color appears after 400 cm_1, and the most clear peaks are the gypsum peak, it has change in SO stretching, OH stretching and has no change in SO bending**.**

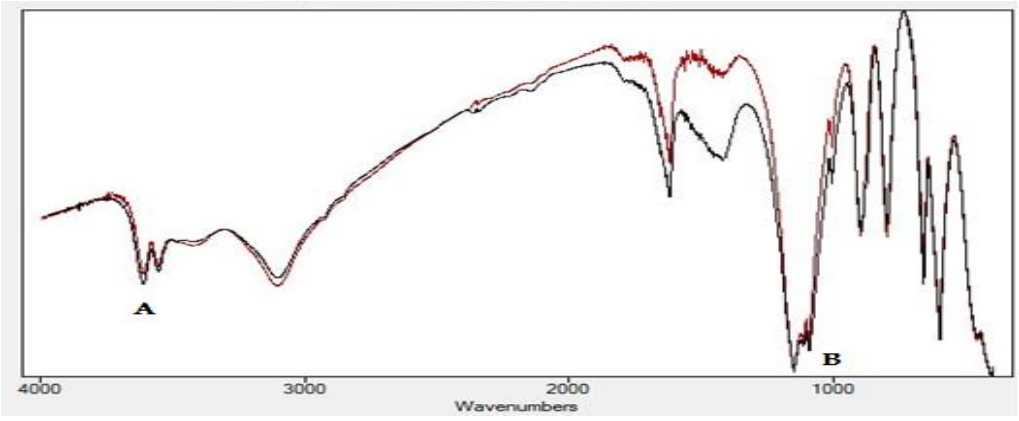


Fig.41.FTIR spectra of treated yellow color with Nano-restore

Table.12. Table shows the result of FTIR analysis of treated yellow color with Nano-restore

SYM	Functional group bands	Sample	Standard
A	OH stretching	3600	3200-3700
B	SO stretching	1100	1080-1140
C	SO bending	800	850-910

The FTIR analysis of treated yellow color with Nano-restore has a very simple result because the composition color appears after 400 cm_1, and the most clear peaks are the gypsum peak, and it hasn't any change.

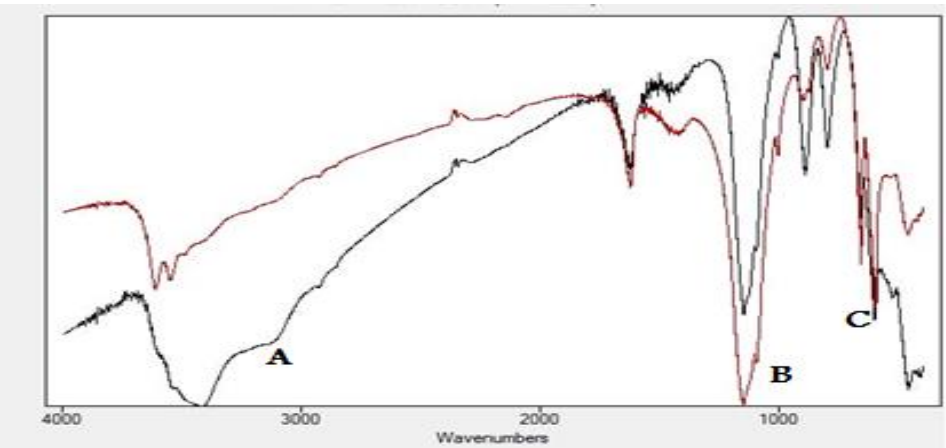


Fig.42.FTIR spectra of treated red color with klucel E

Table.13. Table shows the result of FTIR analysis of treated red color with klucel E

SYM	Functional group bands	Sample	Standard
A	OH stretching	3600	3200-3700
B	SO stretching	1100	1080-1140
C	SO bending	800	850-910

the FTIR analysis of treated red color with (KE)has a very simple result because the composition color appears after 400 cm_1, and the most clear peaks are the gypsum peak, and it has very slight change in SO stretching, OH stretching and has no change in SO bending**.**

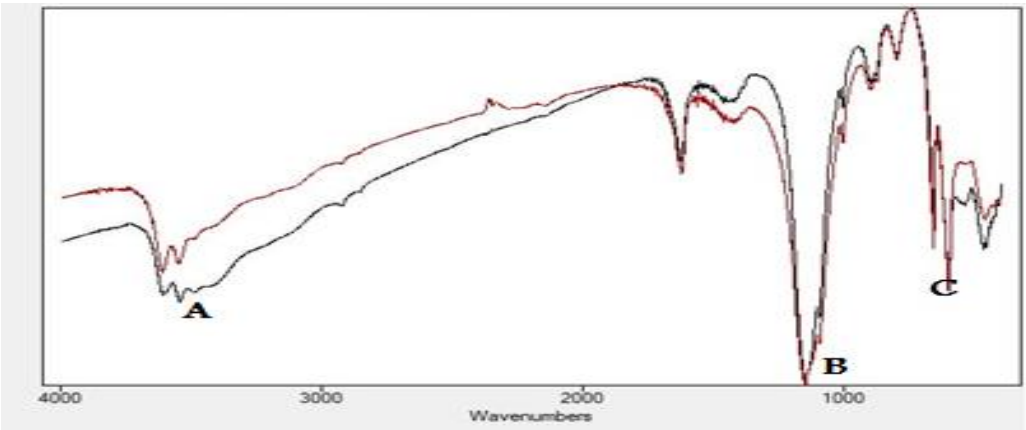


Fig.43.FTIR spectra of treated red color with Nano-restore

Table.14. Table shows the result of FTIR analysis of treated red color with Nano-restore

SYM	Functional group bands	Sample	Standard
A	OH stretching	3600	3200-3700
B	SO stretching	**1100**	1080-1140
C	SO bending	**800**	850-910

The FTIR analysis of treated red color with Nano-restore has a very simple result because the composition color appears after 400 cm_1, and the most clear peaks are the gypsum peak, and it has very slight change in OH stretching and has no change in SO bending and SO stretching.

Conclusion

The two consolidant materials, klucel E and Nano-restore didn't effect on the visual appearance and chemical composition of the wood, ground layer and colors and also didn't caused any color change.

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دراسة مقارنة حول تأثير هيدروكسيد الكالسيوم النانوي وكلوسيل إي كمواد داعمة للخشب متعدد الكروم

تعتبر عملية التدعيم من أهم الخطوات في معالجة الحفاظ على الخشب ، وهناك مواد مختلفة تستخدم كمواد مجمعة مثل Nano–restore وهو متوافق للغاية مع المواد القائمة على الكربونات مثل الركيزة الجيرية واللصقات واللوحات الجدارية والحجر الكربوني ، كما يحقق Nano–restore نتائج جيدة وإعادة التصاق جيدة لرقائق الصباغ المنفصلة دون أي آثار جانبية. علاوة على ذلك ، فإن Klucel E هو أحد إيثرات السليلوز (CE) التي تستخدم على نطاق واسع كمواد لاصقة للقطع الأثرية الخشبية والورقية. لذلك أنشأت هذه الدراسة لتقييم تأثير كليهما على توحيد الخشب المطلي. تم استخدام طرق تحليلية مختلفة مثل FTIR ومقياس اللون في التقييم لتحديد التغيير الكيميائي الذي يعود إلى طبقات الطلاء والجص من الجير والجبس والقاعدة الخشبية. تمثل المواد الخشبية نسبة عالية جدا من تراثنا الثقافي ، وتستخدم لصنع القطع الأثرية المختلفة ، وتستخدم أحيانا كدعم لطبقة الطلاء (خشب متعدد الكروم / خشب أحادي الكروم).

لم تؤثر المادتان المدمجتان ، Klucel E و Nano–restore ، على المظهر المرئي والتركيب الكيميائي للخشب والطبقة الأرضية والألوان ولم تتسبب أيضا في أي تغيير في اللون.

Key words

Klucel E, Nano- restore, FTIR, colorimetric measurement