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CHARACTERIZATION OF THE BEST CONSOLIDATION MATERIAL FOR BLACK RESIN FOR THE LATE PERIOD COFFIN

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ABSTRACT

Many archaeological wooden artefacts covered with a layer of black resin were found in a bad condition, and it was urgent to choose the best consolidation material. Black resin had a special case on conservation because it contains natural resin in composition, and it turns into small fragments and separated from the artefacts. Color meters were used to assess the rate of chromatic change by studying the change reflectance of the sample. The present study focuses on evaluating the efficiency of many consolidation materials used for consolidating the black resin samples, e.g. Nano Paraloid B-72, Paraloid B72, Klucel G (Klucel G, hydroxyl propyl cellulose) and Klucel E (Klucel G, hydroxyl propyl cellulose). The optical properties of black resin were studied using Opti-Match and gloss meter. Nano Paraloid B-72 in xylene gave the best result for consolidating the black resin layer.

KEYWORDS: Black resin, Wood, Gloss, Reflection, Nano Paraloid B-72

1. INTRODUCTION

Wood, as a natural composite material, was used mainly in exterior construction and building applications (Teacă and Bodîrlău, 2018; Afifi, et al., 2019). Wooden artefacts undergo complex alteration and degradation during ageing (Zoja, et al., 2017; Zidan, et al., 2016). The conservation of black resin is very complicated because it contains natural resin and bitumen. Consequently, we had to make a test to choose the best consolidation material.

Over the last decade, polymer-nanoparticle composites attracted great interest in the field of cultural heritage conservation due to their unique multifunctional properties, resulting from the high surface area and chemical activity of the nanoparticles dispersed in the polymers (Helmi, and Hefni, 2016). Poly ethyl methacrylate/methyl acrylate (70:30) was used to improve the properties of Nano composites to be used in the consolidation and protection of black resin layer (Al-Dosari, et al., 2017). Paraloid B72 is one of the most common consolidates used in the field of conservation and restoration. It is an ethyl-methacrylate copolymer (Ewais, 2014).

Luster is classified as a surface property. It refers to specular reflection where incidence and reflection angles are the same. Based on the definition of gloss, the highest amount of reflected light is ascribed to the most lustrous surface. A perfect specular surface is able to reflect all light in accordance with the law of reflection, while opaque surfaces are recognized by their minimal gloss (Ajeli and Ahmad, 2014).

2. MATERIALS AND METHODS

2.1 Materials

The wood of the coffin was identified as sycamore species. The authors prepared new samples of sycamore wood (3×3 cm²).

Nano Paraloid B-72 and paraloid B72 were dissolved in xylene and Klucel G (hydroxyl propyl cellulose), and Klucel E (hydroxyl propyl cellulose) was dissolved in ethanol.

2.2 Preparation of Poly EMA-co-MA [Nano-Paraloid (B-72)]

Poly (EMA-co-MA) with co-monomer (70: 30) was synthesized by a continuous process using an emulsion system containing 5% of monomer mixture concentration and 23*10⁻³ gm sodium dodecyl sulphate (SDS) emulsifier. Co-monomers (EMA and MA), 0.027 gm Potassium persulphate initiator (PPS) and distilled water were put in a 250ml three-necked

round bottom flask, equipped with a reflux condenser, two additional funnels and a rounded anchor like stirrer, in a temperature control water bath.

The stirrer was adjusted at ~350 rpm during the whole process. The continuous process involves the addition of the monomer drop wisely to the emulsifier solution.

In a typical procedure, the experiment was carried out, as follows: in a 250-ml three-necked round-bottomed flask, equipped with reflux condenser, the emulsifier dissolved in 30 ml distilled water was overnight mechanically stirred at room temperature. Then, the weighed initiator was dissolved in 15 ml water, and the first portion of this solution (30%) was added to the reactor and left in a water bath at 65 oC (decomposition temperature of the initiator) in the presence of a pure nitrogen gas. The monomers concentration and the second portion of the initiator (60%) were added to the aqueous phase drop wisely over one hour. The third portion of the initiator (ca 10%) was added, and the reaction content was left for two other hours to complete the polymerization.

2.3 Materials Used for the Preparation of Nano Paraloid

The monomers Ethyl methacrylate (EMA; Across company, New Jersey, USA) and Methacrylate (MA; Sigma company, Germany) were purified via filtration through an alumina column (R) and silica gel column, respectively. The purified monomers were stored in a dark container, refrigerated, and used within one month of purification.

Sodium dodecyl sulfate (SDS) - purchased from across comp., New Jersey (USA) - was used as received without further purification. Potassium persulfate (PPS; BDH laboratory Supplies Poole, BH15 1TD, England) was used as water-soluble initiator.

3. TREATMENTS AND ACCELERATED AGING

A total of (12) samples were utilized. One sample was the standard. Three samples were used as the aging reference. The remaining 8 samples were treated by a brush 1% and 3% of Nano paraloid B72 in xylene, paraloid B72 in xylene, Klucel G 1% and 3% in ethanol and Klucel E 1% and 3% in ethanol. Accelerated aging was applied to the samples at 35oC and 30% humidity for 120h. This procedure was carried out in aging ovens at the National Research Centre, Dokki- Egypt.

Table 1. sample numbers

Test	Sample number	Definition
Reference sample	ST	Standard sample
Light aging	STL	Standard light aging sample
Thermal aging	STT	Standard thermal aging sample
Aged reference sample	STTL	Standard thermal and light aging sample
NanoParaloid B-72 in xylene (1%)	NBX1	
NanoParaloid B-72 in xylene (3%)	NBX2	
Paraloid B-72 in xylene (1%)	B1	
Paraloid B-72 in xylene (3%)	B2	
Klucel G in ethanol (1%)	KG1	
Klucel G in ethanol (3%)	KG2	
Klucel E in ethanol (1%)	KE1	
Klucel E in ethanol (3%)	KE2	

4. ANALYSIS TECHNIQUES

Reflection Change Measurements: The brightness of the samples was measured with an Optimatch 3100®SDL spectrophotometer. All samples were measured in a visible region (Ali and Mohamed, 2015) with a wavelength range of 400-700 μm .

Gloss measurement: Mini Gloss 101Nn (www.aliexpress.com) was used with ISO 7668 standard (sheen instruments.com 2018). It was easy to use and the measuring geometry was 60 Degrees.

All measurements were made before and after treatment and compared to that of the aged control and standard samples.

5. RESULTS AND DISCUSSION

5.1 Visual inspection

Visual observation was employed to detect the visual changes of the treated experimental sample (Rasha et al 2017; Moustafa et al., 2017). The samples treated with Nano paraloid B72 in xylene 1% and 3% give good result by naked eye than other samples.

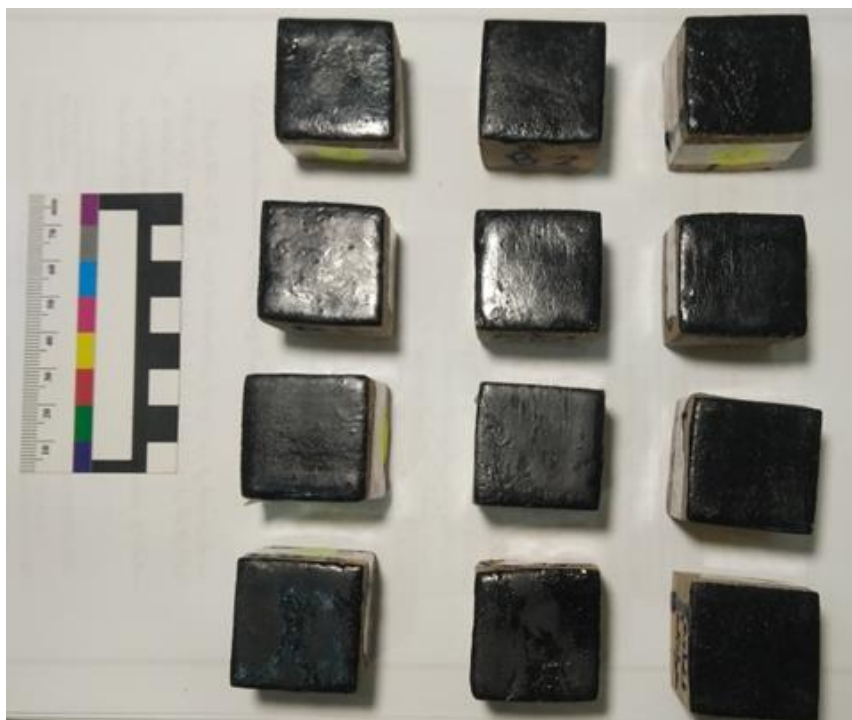


Figure 1. The treated samples after accelerated aging compared to aged control sample.

5.2 Color measurements

Black resin had a special case in evaluating the best materials for conservation because it was black. Thus, only the reflection could be employed in such

evaluation. All sample measured in visible wavelength 500 μm , and device results are given in percentage. All the samples were conducted according to the CIE lab system (Osman et al., 2011) ISO105-Jo1:1989.

Table 2 gives the reflection measured for the standard samples after 240 hours of exposure in thermal and light aging.

Table 2. Values of reflection for the standard samples before and after aging

Sample	Reflection %
ST	13.47
STT	14.7
STL	14.83
STTL	13.86

Table 3 gives the degree of reflection for the samples treated with different consolidation materials before and after aging after 240 hours of exposure.

Table 3. Values of reflection for the treated samples (A) before and (B) after aging

Sample number	A	B
NBX1	14.89	14.89
NBX2	15.6	15.60
B1	15.49	15.49
B2	15.31	15.31
KE1	15.1	15.10
KE2	15.54	15.54
KG1	15.69	15.69
KG2	15.04	15.04



Figure 2. Values of reflection on the visible spectrum; (ST) standard sample; (STT) thermal aging; (STL) light aging; (STTL) after light and thermal aging

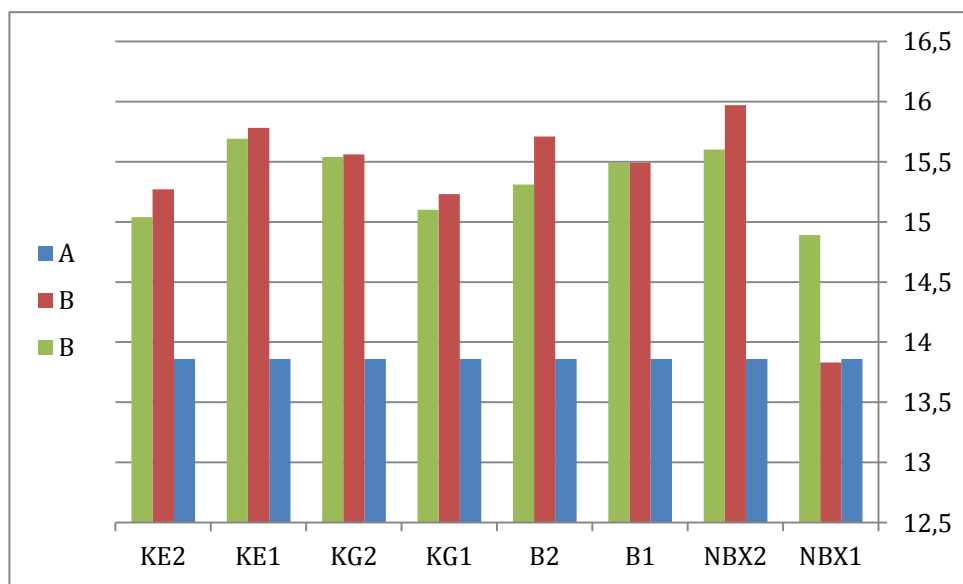


Figure 3. Values of reflection degree; (A) standard; (b) after the application of the consolidation materials; (c) after aging in 500 μm

The samples treated with NanoParaloid B-72 in xylene 1% showed the least change in the reflection degree (14.89) from the standard samples (Fig. 3). Klucel G in ethanol 3% showed that the degree of reflection was (15.54) and Paraloid B-72 in xylene 3% showed that the degree of reflection was (15.1).

5.3 Luster measurement

The degree of gloss was measured because the black resin was shiny.

The results of measuring the degree of luster for the standard samples and thermal aging and light aging samples, shown in Table 4 indicate that the standard sample gives the highest degree of gloss 38.65, the standard sample of light aging gives 35,

the standard sample of thermal and light aging gives 34.4, and the standard sample of thermal aging 23.95.

Table 4. Gloss results using the Gloss meter for the standard samples after light and thermal limitations

Sample number	Gloss
ST	38.65
STL	35
STT	23.95
STTL	34.4

Fig.4 shows that the difference between the degree of gloss and thermal and light aging.

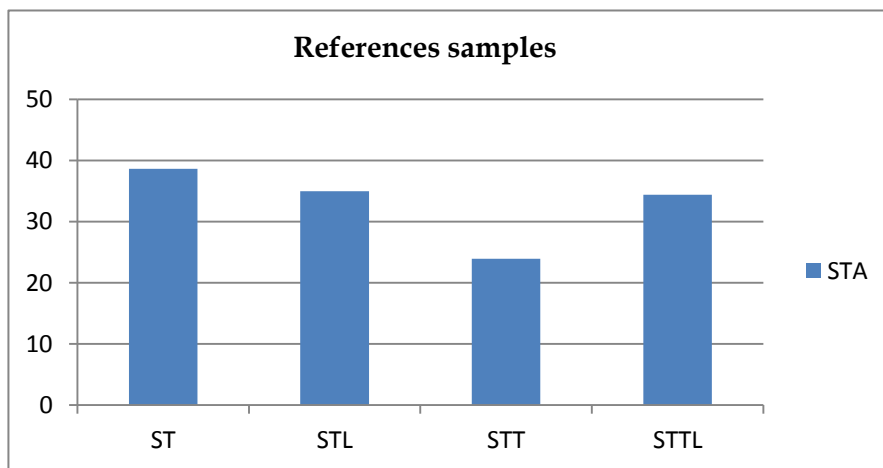


Figure 4. Comparing the values of change in the brightness of the standard samples and the light and thermal aging samples.

5.4 The degree of gloss

The degrees of gloss of the standard samples, light aging samples (STL), thermal aging (STT), and light and thermal aging samples (STTL) were 38.65, 35, 23.95, and 34.4, respectively. The measurement scale of Gloss by Units are the so called Gloss Units (GU).

Gloss was measured for all samples after 240 hours of exposure, and Table 5 shows the difference of gloss measurement for the standard sample (A) and treated sample by different consolidation materials (before B, after C); whenever it is high this implies increase of gloss of the sample.

Table 5. Gloss results using the Gloss meter for the experimental samples (A) standard sample, (B) before light and thermal aging, (C) after light and thermal aging and the application of the consolidation materials

Sample number	A	B	C
NBX1	34.4	26.8	21.55
NBX2	34.4	25.6	19.15
B1	34.4	14	17.1
B2	34.4	12.1	9.2
KG1	34.4	18.8	10.3
KG2	34.4	12.6	17.95
KE1	34.4	12.3	12.3
KE2	34.4	10.1	9.5

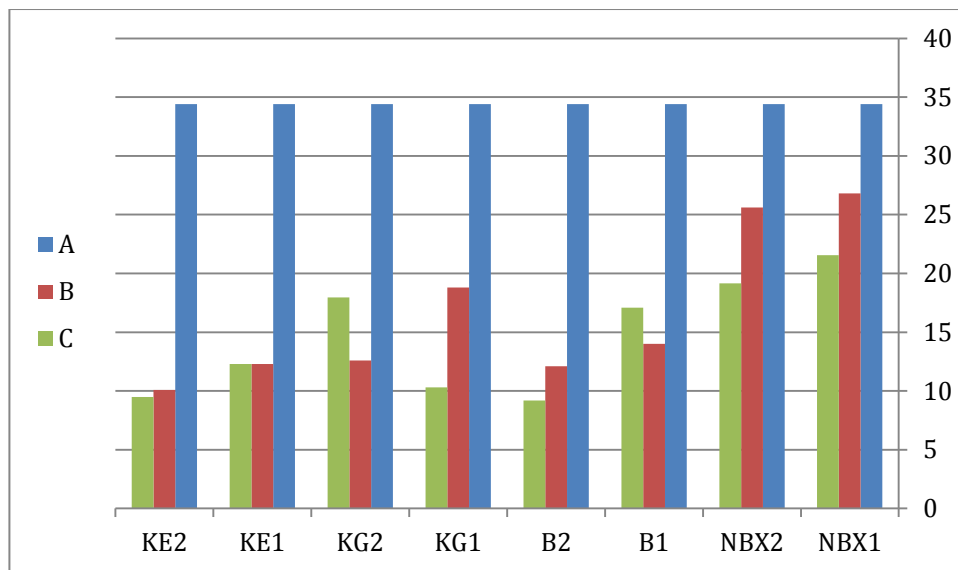


Figure 6. Comparison of the values of change in the degree of gloss of the samples: standard samples (A), after the application of experimental consolidation materials (B) and after aging (C)

The degrees of gloss of the experimental samples after thermal and light aging and the application of experimental consolidation materials were taken.

The results illustrate (figure 6).that the samples treated by Nano-paraloid B72 in xylene 1% (21.55), paraloid B72 in xylene 1% (17.1), and Klucel G in alcohol 1% (10.3) faded and were not visible to the naked eye. Paraloid B72 in xylene 1% showed the best result in gloss measurement.

The values of the change in the degree of gloss of the experimental samples treated with different materials varied. The higher the number, the higher the

brightness, and the lower the number. The sample was fading as a result of the consolidation material

6. CONCLUSION

The study has shown that the samples treated with Nano paraloid B72 in xylene 3% gave the best result in the gloss meter. From measuring the degree of gloss using Mini Gloss device and the degree of reflection using Opti-Match device for different consolidation materials and after aging, the treated samples showed that the samples treated with Nano paraloid B72 in xylene gave the best results for consolidating the black resin layer.

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