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Evaluating Thermal and UV Stability of Some Protective Coatings for Historical Murals in Egypt

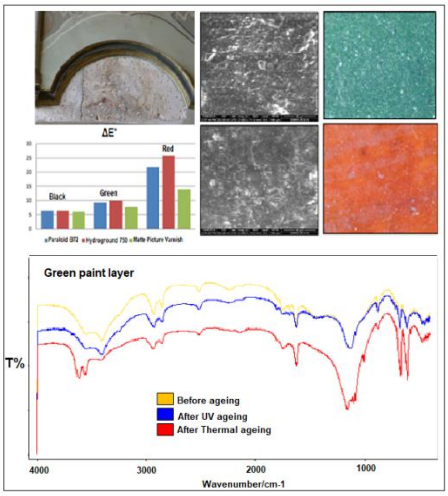
Fatma Refaat[a]*, Hussein Marey Mahmoud[b], Basil El-Sabbagh[c], Atef Brania[b]

a. Ph.D. Student, Department of Conservation, Faculty of Archaeology, Cairo University, Egypt
b. Department of Conservation, Faculty of Archaeology, Cairo University, 12613 Giza, Egypt
c. Housing and Building National Research Centre, Raw Building Materials Technology and Processing Research Institute, Cairo, Egypt.

HIGHLIGHTS

- Evaluating the stability of some acrylic-based polymers (Paraloid B72, Lascaux® Hydroground 750, and Pébéo Satin Matt Picture Varnish).
- Thermal and UV accelerated ageing were performed on laboratory model samples according to literature data .
- The evaluation process was performed using optical microscopy, colorimetry, scanning electron microscopy, and FTIR spectroscopy .
- Among the tested products, the Matt picture varnish (based on acrylic resin and polyethylene wax) showed the best durability against ageing procedures.

GRAPHICAL ABSTRACT



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ABSTRACT

The present study focuses on applying a compatible protective treatment for historical pictorial surfaces in 'El-Harem palace' (the National Military Museum of Egypt). For this purpose, simulated painted samples were prepared according to the original materials. The models were treated with acrylic-based polymeric materials, including Paraloid B72, Lascaux®Hydroground 750, and Pébéo Satin Matt Picture Varnish (a transparent varnish of an acrylic resin and polyethylene wax). The stability of the treated surfaces against accelerated UV and thermal ageing was evaluated via optical microscopy (OM), colour measurements, scanning electron microscopy (SEM), and Fourier-transform infrared spectroscopy (FTIR).

* Corresponding author: fatma_1984p@yahoo.com

The results showed that the behavior of the tested polymers against accelerated ageing tests is comparable, to some extent. Based on its acceptable stability, Pébéo Satin Matt picture varnish was used as a protective coating for the murals. Significantly, the results of this research will help in protecting rare examples of Rococo-style mural paintings in Egypt.

1. Introduction

Rococo is probably derived from two words: 'rocaille' and 'coquille', to describe ornamentations made of small stones and irregular-shape shells. This style is an elegant artistic technique, mainly for interior decorations, which was developed in Europe during the 18th-century.Rococo-style is characterized by soft pastel colours, floral motifs, asymmetrical patterns, and multiple curvy lines [1].The combination of Western influences and the Ottoman art led to the appearance of 'Turkish Rococo Style', which was extensively applied in the second half of the 18th-century [2].During the 19th-centrury in Egypt, Rococo was used extensively to decorate several buildings of Muhammad Ali's family (1805–1952 A.D). In 1826, Mohamed Ali Pasha constructed 'El-Harem palace' to be a residence for his wife. Since 1949, the palace has been operated as a national museum for the military history of Egypt. In 1987, the Supreme Council of Antiquities (SCA), in collaboration with the Egyptian Ministry of Defence, applied an integrated restoration project to the building. In 2011 and after obtaining the necessary permissions from the Administration of the Military Museums of Egypt, the authors of this work started a project to fully uncover an example of the original paintings. The project lasted for three years with the aim of removing undesirable accumulations and recent paint layers. Fig. 1 represents recent renovations on the walls of the National Military Museum, details of uncovered areas and the main deterioration forms.

The uncovered murals showed serious damage caused by the uncontrolled museum's environment. Thus, an intervention for comprehensive restoration was applied, including a protective coating applied to the surface of the murals. Numerous studies have been conducted to characterize paintings materials from historical buildings in Egypt [3-6]. Previous studies were devoted to report the degradation aspects of oil-based paints [7-10], and the durability of surface coatings for their protection [11-14].Protective coatings are usually applied to prevent any damage induced by the surrounding environment. Particularly, these coatings should be resistant to the deleterious effects of ultraviolet radiation. Paraloid B-72 is a stable varnish resin widely used in heritage materials conservation. When dissolved in toluene, it is considered a medium gloss varnish that dries within few minutes. Spraying Paraloid B-72 varnish usually creates a desirable unvarnished appearance of the treated surface [15]. Additionally, Lascaux acrylic dispersions are characterized by their excellent penetration and resistance to ageing. For this, they are applied for conserving multiple heritage materials as consolidants or transparent varnishes. Lascaux dispersions are usually used because of their super protection against UV radiation. For this reason, an aqueous dispersion of Lascaux Hydroground 750 was used to protect historical wall paintings from the Prasakaki house in the Aegean island of Syros [16].It is well known that Matt varnishes are used to avoid reflections and to obtain dull surfaces. In our case, Pébéo Satin Matt Picture Varnish was selected to fulfil this distinctive character. In the present study, the stability of some acrylic-based polymeric materials against artificial accelerated ageing was evaluated. The assessment was designed to determine the optical characteristics, chromatic change, surface morphology, and the molecular behaviour.

2. Materials and methods

2.1. Materials

2.1.1. Polymers

Three acrylic-based polymeric products were selected and subjected to artificial ageing on simulated painted models (see Table 1):

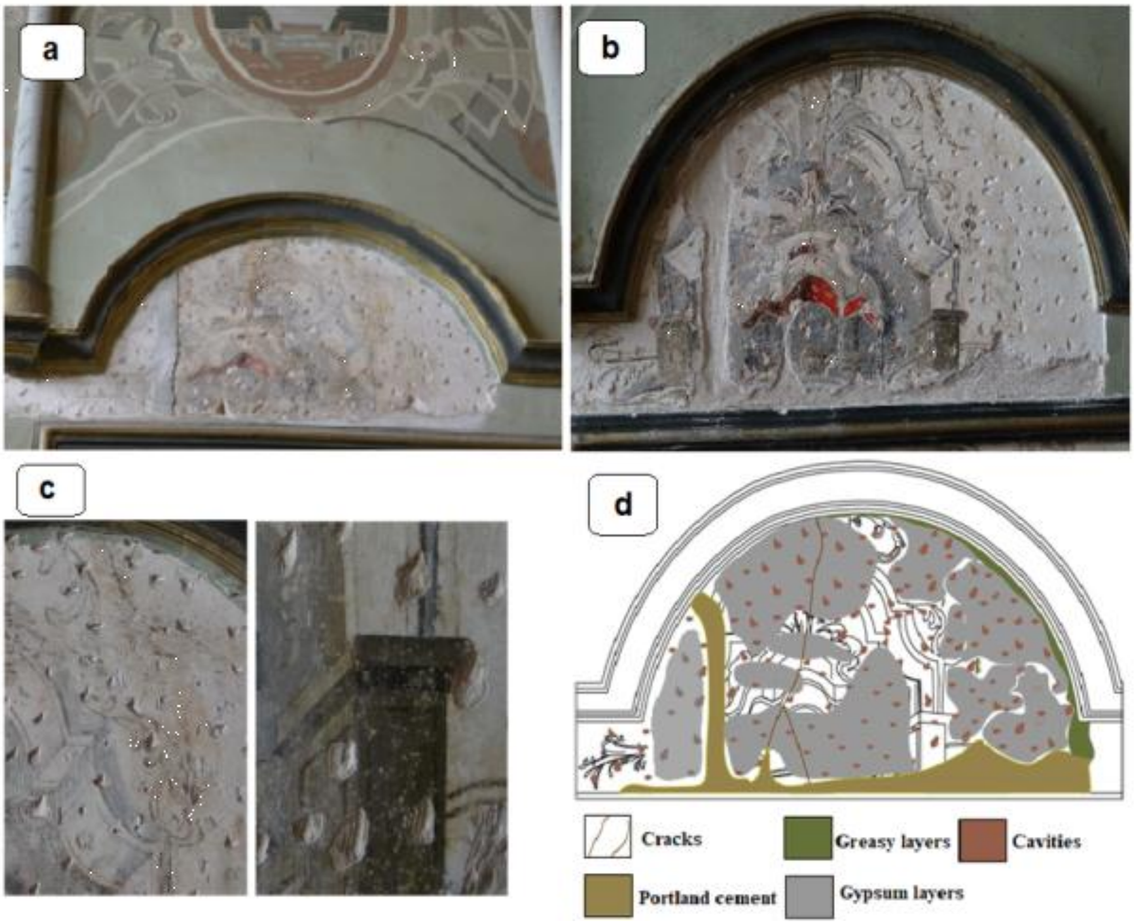

Fig. 1. a) An example of the recent renovations of the National Military Museum 'El Harem palace', uncovered area is also observed, b) A close-up of uncovered area on the walls after preliminary cleaning process, c) Details of the motifs representing the Rococo style, d) Documentation using AutoCAD program reconstruction of the main deterioration aspects affecting the mural.

Table 1.A brief description of the tested polymers

Product	Composition	Concentration/Solvent	Glass transition temperature (Tg)	Manufacturer
Paraloid B-72	100% resin pellets of copolymer of ethyl methacrylate and methyl acrylate	3% dispersed in toluene (weight/volume)	approx. 40 °C	Rohm and Haas Company, USA
Lascaux® Hydroground 750	A pure acrylic resin dispersion	3% dispersed in water(weight/volume)	approx. 25°C	Kremer Pigmente GmbH & Co. KG, Germany
Pébéo Satin Matt Picture Varnish	A transparent ready-to-use varnish based on acrylic resin and polyethylene wax	Ready to use	Undefined value	Pébéo Company, France

- Paraloid B-72, (3% in toluene, weight/volume) (a 100% resin pellets of copolymer of ethyl methacrylate and methyl acrylate, glass transition temperature (Tg) = 40 ^{o}C, this product is commercially produced by Rohm and Haas Company, USA).
- Lascaux® Hydroground 750, (3% in water,weight/volume) (pure acrylic resin dispersion, glass transition temperature (Tg)= 25°C, it is commercially produced by Kremer Pigmente GmbH & Co. KG, Germany).
- Pébéo Satin Matt Picture Varnish-ready to use (a transparent ready-to-use varnish based on acrylic resin and polyethylene wax, commercially produced by Pébéo Company, France).

2.1.2. Laboratory models

Table 2 shows the main elements and minerals identified in the original mural. Based on the analyses which were carried out in our previous study [17], pigments of vermilion (HgS), red ochre (haematite, αFe_2O_3), green earth (di-octahedral mica based on glauconite and celadonite) and carbon black were used in the uncovered murals. Probably, colour tonalities were produced through mixing pigments together (e.g. grey colour, black-reddish colour). Moreover, a binding medium of linseed oil was identified, through FTIR analysis. Accordingly upon these results, laboratory models painted samples were prepared.

2.2. Experiments and instruments

2.2.1. Artificial ageing protocol

Accelerated thermal and photochemical degradation is usually used to determine the stability of polymeric materials. Accordingly, a number of the laboratory models were subjected to an OSRAM L36/37 UV lamp, operated in an effective power of 36 W, for 100 hours (350– 430 nm wavelength, 10 cm distance). Moreover, another group of samples was thermally aged in an oven (Heraeus Series 6000, Germany), at 70°C for one week, then, the temperature was increased into 100°C for two weeks. The thermal ageing protocol was designed following the observations of Blackshaw and Ward [18]. Accroding to Farmakalidis et al. [19], many of the polymers used in conservation field are subjected to temperatures above their Tg values. Further, using a temperature at 100 °C will ensure reaching a notable acceleration of the ageing process.

2.2.2. Optical microscopy

Microscopic observations were helpful to record any change in the appearance of the painted models. The images were obtained using a Veho VMS-004-DELUXE USB digital microscope, with a maximum magnification of 100x.

2.2.3. Colorimetry

Colorimetry is a common method usually used to evaluate the chromatic changes that occurred during the accelerated ageing. The CIE L*a*b*colour values were measured using a Macbeth Colour Eye 3100 laboratory Spectrophotometer unit Module CE 3100 using D65 illuminant and 10° observer. The overall colour difference (ΔE*) (CIELAB, 1976) between the initial measurements and those taken after exposure was calculated using the equation (according to ASTM D2244-16[20]):

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$$

Where:

- ΔL* - the lightness difference: $\Delta L^* = L^*1 - L^*2$
- Δa* and Δb* - chromaticity differences: $\Delta a^* = a^*1 - a^*2$, $\Delta b^* = b^*1 - b^*2$
- L*= refers to Lightness (L= 0 indicates black and L=100 white),
- a*= expresses the hue between red and green (negative a*values indicate green, and the positive ones for red)
- b*= refers to the hue position between yellow and blue (negative b* values are associated with blue, and the positive values for yellow).

2.2.4. Scanning electron microscope

SEM investigation helped in measuring any alterations in the morphological features of the samples due to ageing. For this purpose,

Table 2 Summary of the chemical and mineralogical composition of the original mural (after Refaat et al. [17]).

Sample	Elements*	Minerals**
Plaster	Ca, S, Si, AL, Mg	Gypsum ($CaSO_4 \cdot 2H_2O$), Calcite ($CaCO_3$), Anhydrite ($CaSO_4$) and Quartz (SiO_2)
Preparation layer	Ca, S, Si	Gypsum with traces of quartz
Black pigment	C, Ca, S, Si	Carbon (Graphite) and minerals of the preparation layer
Red pigment	Hg, S, Ca	Vermilion (HgS) and minerals of the preparation layer
Black-reddish pigment	C, Fe, Hg	probably a mixture of carbon black, Hematite and Vermilion
Green pigment	Si, Al, K, Fe, Na	probably green earth (Glauconite)
White pigment	Pb, C, Ca, Si, Al	probably of Hydrocerussite, [$Pb_3(CO_3)2(OH)_2$]

**SEM-EDX was used; ** XRD was used.*
It is worthy to note that FTIR was used on the paint samples and indications on the use of a drying oil (i.e. linseed oil) were reported. Also for the majority of samples, the required amounts for XRD analysis were not available.

Table 3. Summary of the main observations recorded on the laboratory models after ageing.

<table>
<tr><th rowspan="2">Polymer</th><th rowspan="2">Laboratory Model</th><th rowspan="2">Colour change*</th><th colspan="2">Microscopic observations</th><th colspan="2">Morphological aspects</th><th colspan="2">Molecular change</th></tr>
<tr><th>UV ageing</th><th>Thermal ageing</th><th>UV ageing</th><th>Thermal ageing</th><th>UV ageing</th><th>Thermal ageing</th></tr>
<tr><td rowspan="3">Paraloid B-72</td><td>Red</td><td>Unacceptable</td><td>A dense white coat</td><td>White strips</td><td rowspan="3">A slight alteration</td><td rowspan="3">No significant modification</td><td rowspan="3">An increase of the ester carbonyl stretching was noticed at 1750 cm^{-1} due to oxidation</td><td rowspan="3">A little decrease in the absorption intensity of carbonyls</td></tr>
<tr><td>Green</td><td>Unacceptable</td><td>A notable whitening</td><td>A slight whitening</td></tr>
<tr><td>Black</td><td>Acceptable</td><td>Observable lighting</td><td>Darkening occurred</td></tr>
<tr><td rowspan="3">Hydroground 750</td><td>Red</td><td>Unacceptable</td><td colspan="2">Chalky coat</td><td colspan="2" rowspan="3">Homogeneous distribution of the polymer</td><td rowspan="3">A small decrease in the intensity of the ester C=O</td><td rowspan="3">A hydroxyl formation due to oxidative degradation</td></tr>
<tr><td>Green</td><td>Unacceptable</td><td>A deep whitening</td><td>Acceptable whitening</td></tr>
<tr><td>Black</td><td>Acceptable</td><td colspan="2">Acceptable lightening</td></tr>
<tr><td rowspan="3">Pébéo Satin Matt Picture Varnish</td><td>Red</td><td>Unacceptable</td><td colspan="2">Pale red-orange appearance</td><td colspan="2" rowspan="3">A continuous film was formed on the surface</td><td colspan="2" rowspan="3">No critical alterations were reported after the UV/thermal ageing.</td></tr>
<tr><td>Green</td><td>Acceptable</td><td colspan="2">No significant change in appearance</td></tr>
<tr><td>Black</td><td>Acceptable</td><td colspan="2">No significant change in appearance</td></tr>
<tr><td colspan="9">*Values were determined according to ΔE* 1976.</td></tr>
</table>

a Quanta scanning electron microscope FEG 250 (Field Emission Gun, FEI, Netherlands) was used. The instrument was operated using accelerating voltage of 20 kV and magnification of 2000x.

2.2.5. Fourier-transform infrared spectroscopy

The molecular structure of the studied samples was determined using a Jasco 4100 FTIR Spectrometer. Few milligrams of each model sample were removed and mixed with KBr powder to prepare the needed discs for analysis. The transmittance FTIR spectra were collected in the mid-IR region (400–4000 cm^{-1}) with accumulation of 32 scans at a resolution of 4 cm^{-1}. Essential ® FTIR Spectroscopy Software (version 3.1) was applied to measure and handle the recorded spectra.

3. Results and discussion

Table 3 summarizes the main observations recorded on the laboratory models after ageing.

3.1. Microscopic observation

The outer surface of the laboratory models was examined by an optical microscope to observe its appearance before and after ageing. The models painted with vermilion showed a notable aesthetic change (Fig. 2). The control model (untreated) showed horizontal white lines on the painted surface which turned darker after thermal ageing. Concerning the samples treated with Paraloid B72, a significant change in appearance was noticed even before ageing. A chalky white film of the polymer was formed on the surface causing a notable discolouring. Regarding Hydroground 750, the whitening was clear after UV and thermal ageing. Samples treated with Matt picture varnish were less affected than those treated with the other products. In the models painted with green earth (Fig. 3), a slight change in appearance was observed after UV ageing for all samples. After thermal ageing, the samples treated with Matt picture varnish showed the same appearance of the untreated samples. In the models painted with carbon black(Fig. 4), the samples treated with Hydroground 750 and Matt picture varnish showed the most durability against ageing. While the surface of samples treated with Paraloid B72 was changed after treatment and thermal ageing.

3.2. Colorimetric evaluation

The determination of colour properties of the painted models after ageing was used as an indicator for their preservation stability. It is well established that the exposure of polymers to UV radiation leads to serious photo oxidative degradation, which causes yellowing and poor mechanical properties [21]. CIE L*,a*,b* colour parameters were recorded before and after the ageing process. The detection of any discolouring was measured through calculating the ΔE* values. The L* values experienced the largest change in the red coloured samples treated with Hydrogroun 750 (Fig. 5). It was noticed that the values increased suddenly from 35.36 into 43.63. Both Paraloid B72 and the Matt picture varnish showed a less ΔL* change. Concerning the black coloured areas, almost the same values were registered for the three polymers. However for the green coloured areas, Paraloid B72 showed a higher value than the Matt picture varnish. Also for the green painted areas, the values were in an acceptable range of the yellowing effect mainly for Matt picture varnish. Fig. 5 (b,c) shows the Δa* and Δb* values registered upon the treated samples after UV ageing. As for Δa* values, the three polymers showed a high decrease. As for Δb* values, the highest decrease was recorded for Hydroground 750 and Paraloid B72, respectively. As a result, the highest ΔE* values were recorded for the red painted areas treated with Hydroground 750 (Fig. 5d). Samples treated with Paraloid showed the second high ΔE* value. Worthy to note that the high sensitivity of vermilion to light and heat enhanced the recording substantial colour change. While, the lowest colour difference for the three polymers was reported for the black coloured areas.

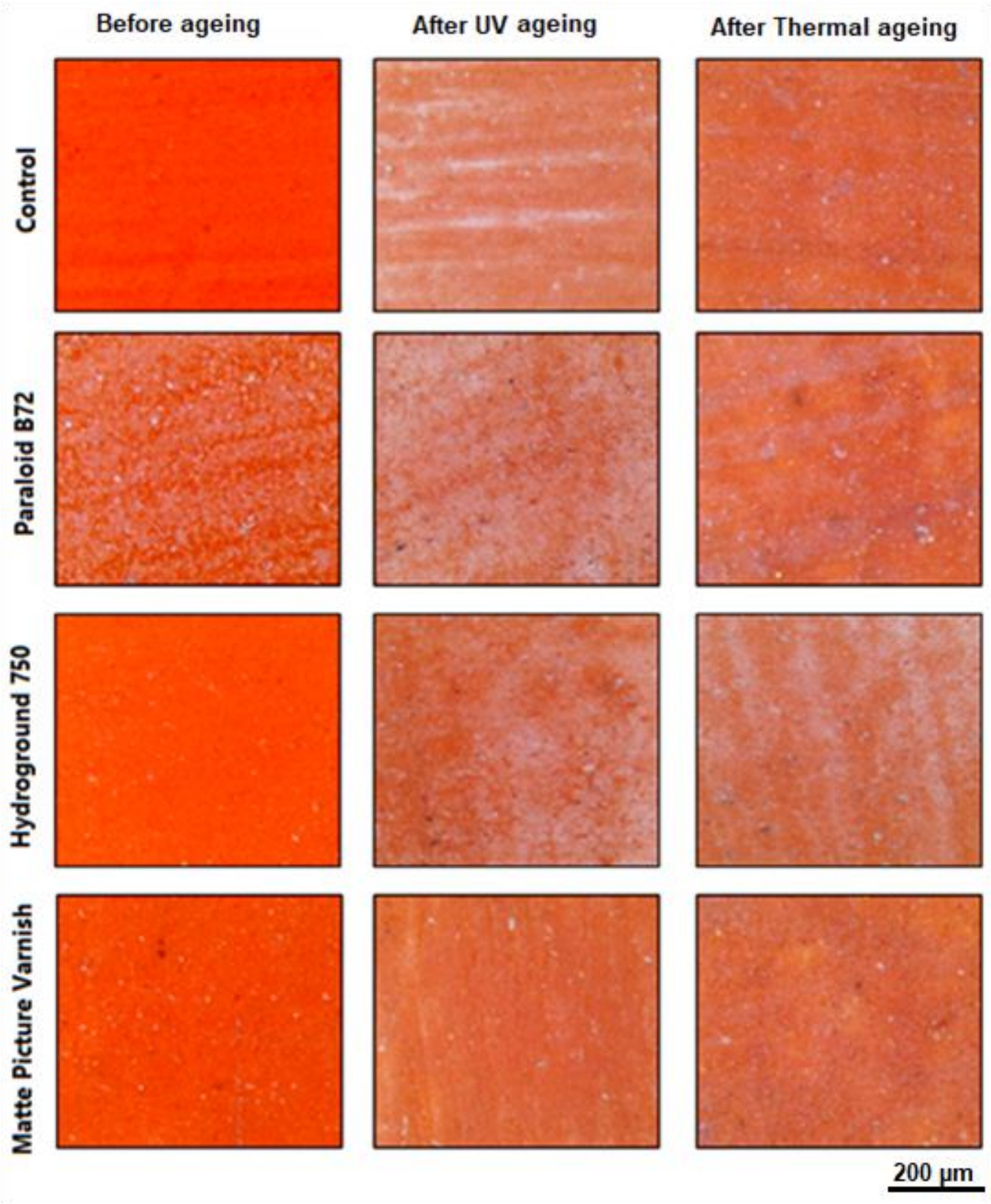

Fig. 2. Microscopic images (at magnification 100 X, scale bar=200µm) of the appearance of red painted models before and after UV/thermal ageing.

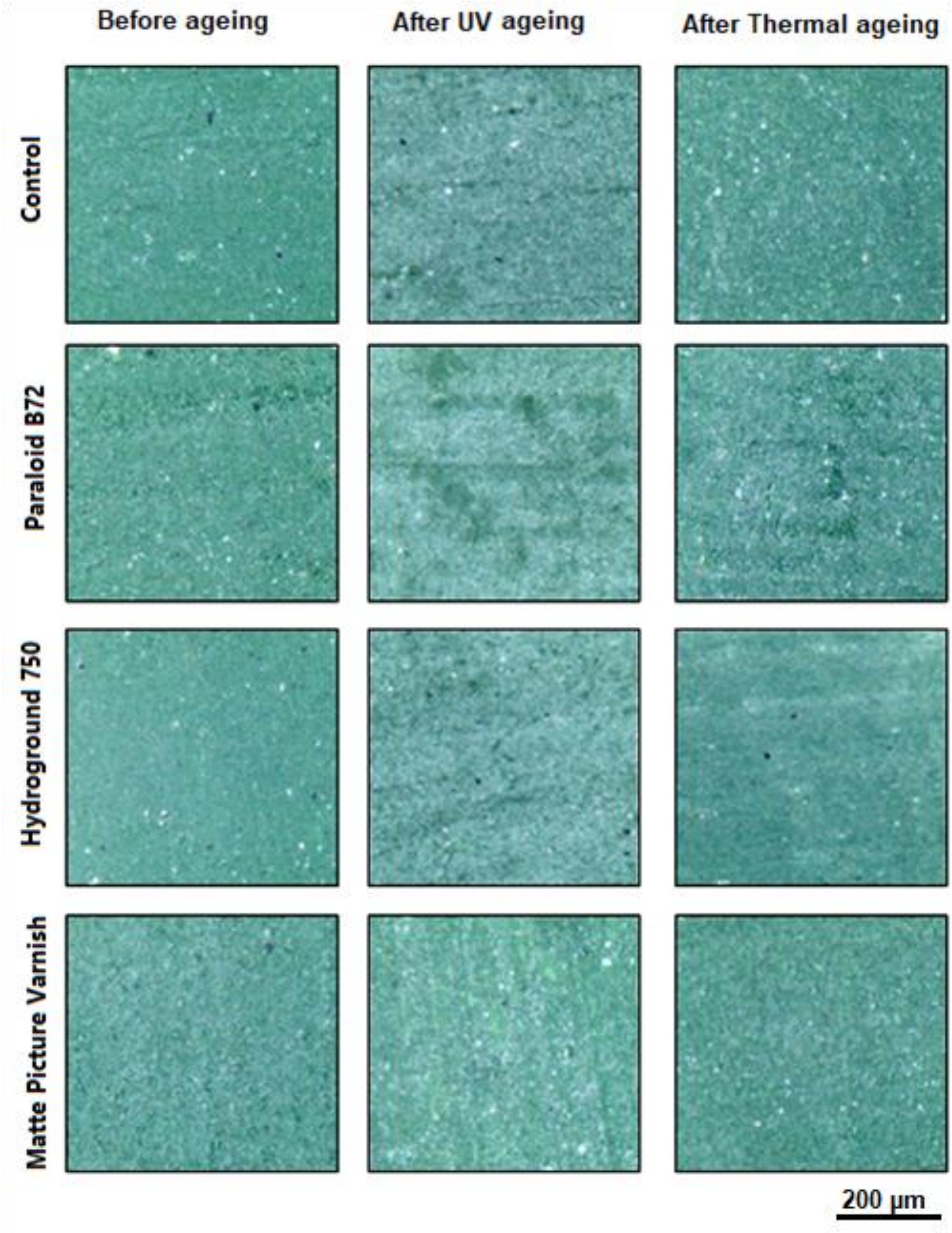

Fig. 3. Microscopic images (at magnification 100 X, scale bar=200µm) of the appearance of green painted models before and after UV/thermal ageing.

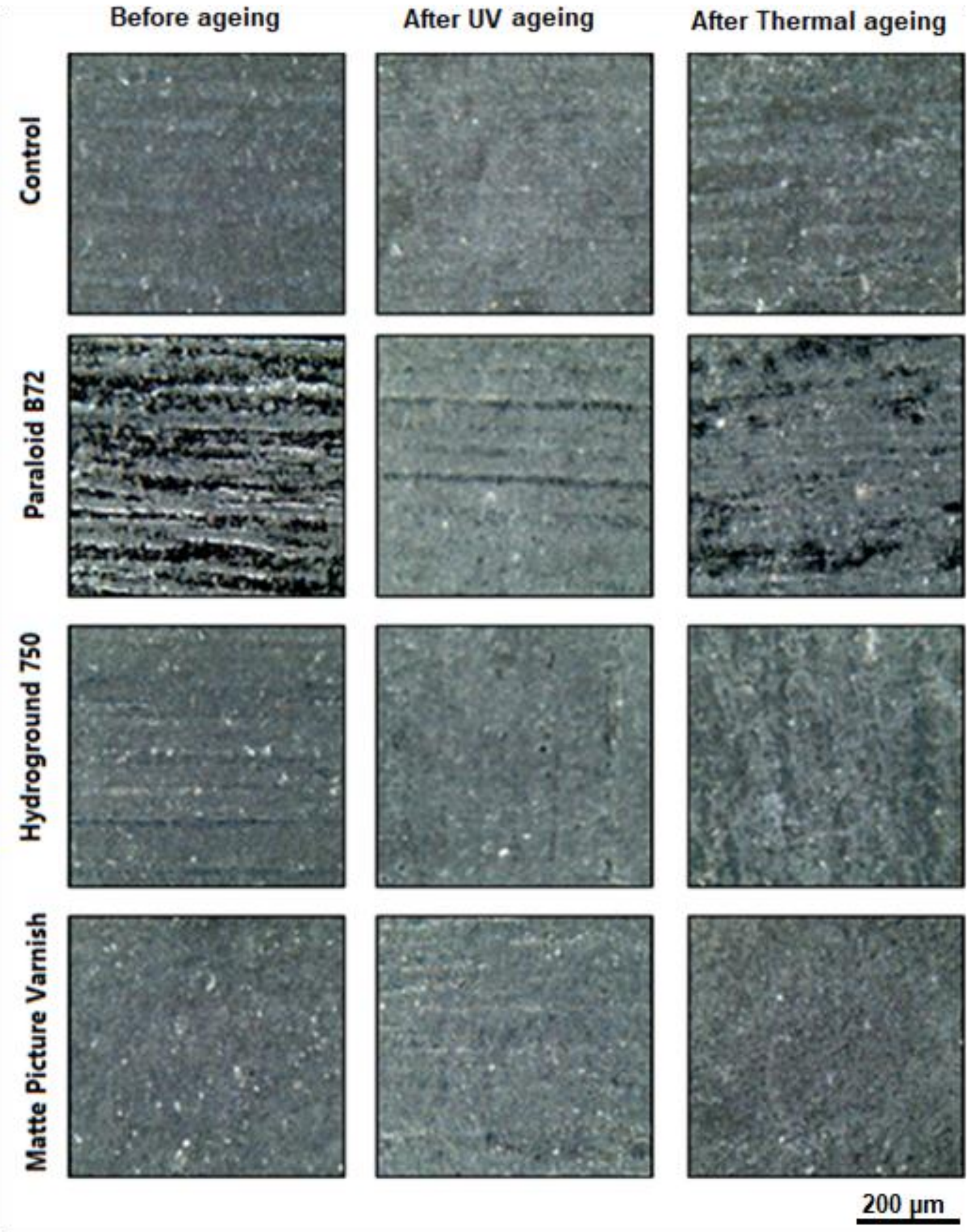

Fig. 4. Microscopic images show (at magnification 100 X, scale bar=200µm) the appearance of black painted models before and after UV/thermal ageing.

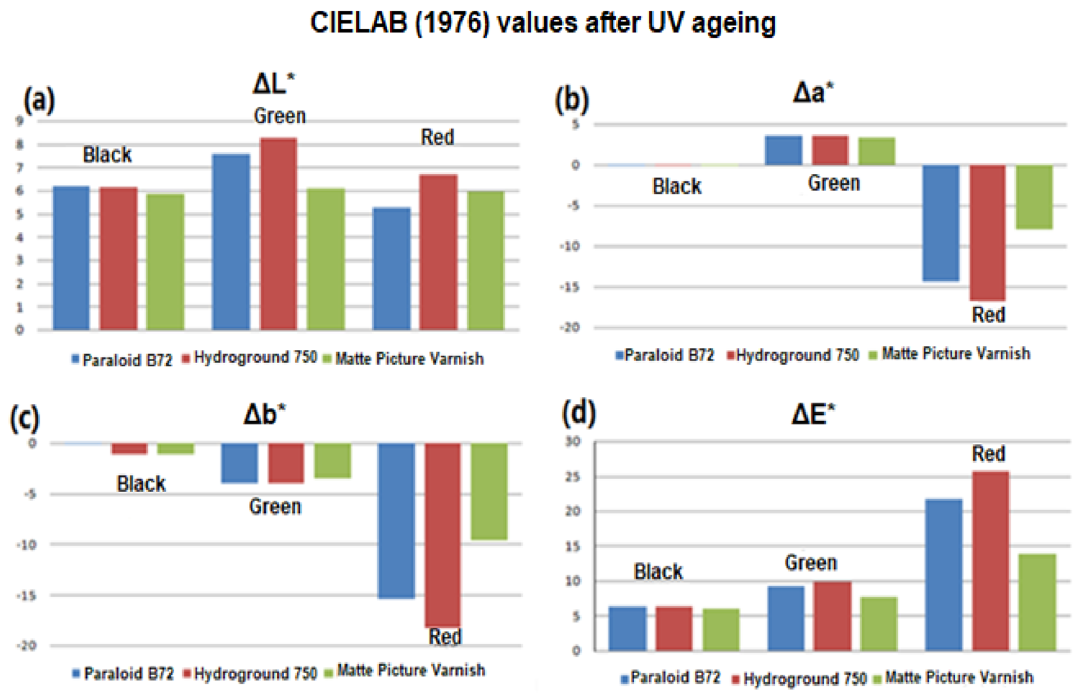

Fig. 5. ΔL*, Δa*,Δb* chromatic values and colour difference (ΔE*) after UV ageing of the laboratory painted models.

3.3. FE-SEM morphological study

FE-SEM investigation was used to determine the alterations of the morphological features of the studied samples. In Figure 6, the FE-SEM micrographs of the control and treated samples are presented. As a result of the UV radiation, all samples painted with red pigment (Fig. 6) showed a slight alteration in the morphological appearance. No significant modification was found due to the effect of thermal ageing, which emphasizes the stability of the three polymers against heat. As for the samples painted with the green pigment (Fig. 7), and treated with the Matt varnish, a homogeneous distribution of the polymer was observed before ageing. However after UV effect, the sample morphology showed a little roughness. FE-SEM micrographs of the samples treated with Paraloid B72 showed no degradation after ageing. In the morphological aspects recorded on Hydroground 750, the polymer has seriously influenced the samples. After ageing, several burrs were formed, due to shrinkage in the polymer material. For the black areas (Fig. 8), both Hydroground 750 and Paraloid B72 gave good morphological results since no alteration occurred. The Matt varnish formed a continuous film on the surface. In a few words, the morphological observations showed that the thermal ageing affected the boundaries of the treated surface due to shrinkage which occurred as a result of the direct effect of heat. In his reference book, Horie [22] stated that heating the thermoplastic polymers in a temperature exceeding their Tg values, will allow them to act as flowing liquids. This will decrease the occurrence of micro-cracks and volume shrinkage after solidification.

3.4. Evaluating changes in the molecular structure

Fig. 9 shows the FTIR spectra recorded on the treated paint models before and after the ageing process. Based on FTIR results, the laboratory models treated with Paraloid B72 showed characteristic peaks as follows: νC–H stretching (at 2981 cm^{-1}), νC=O of ester

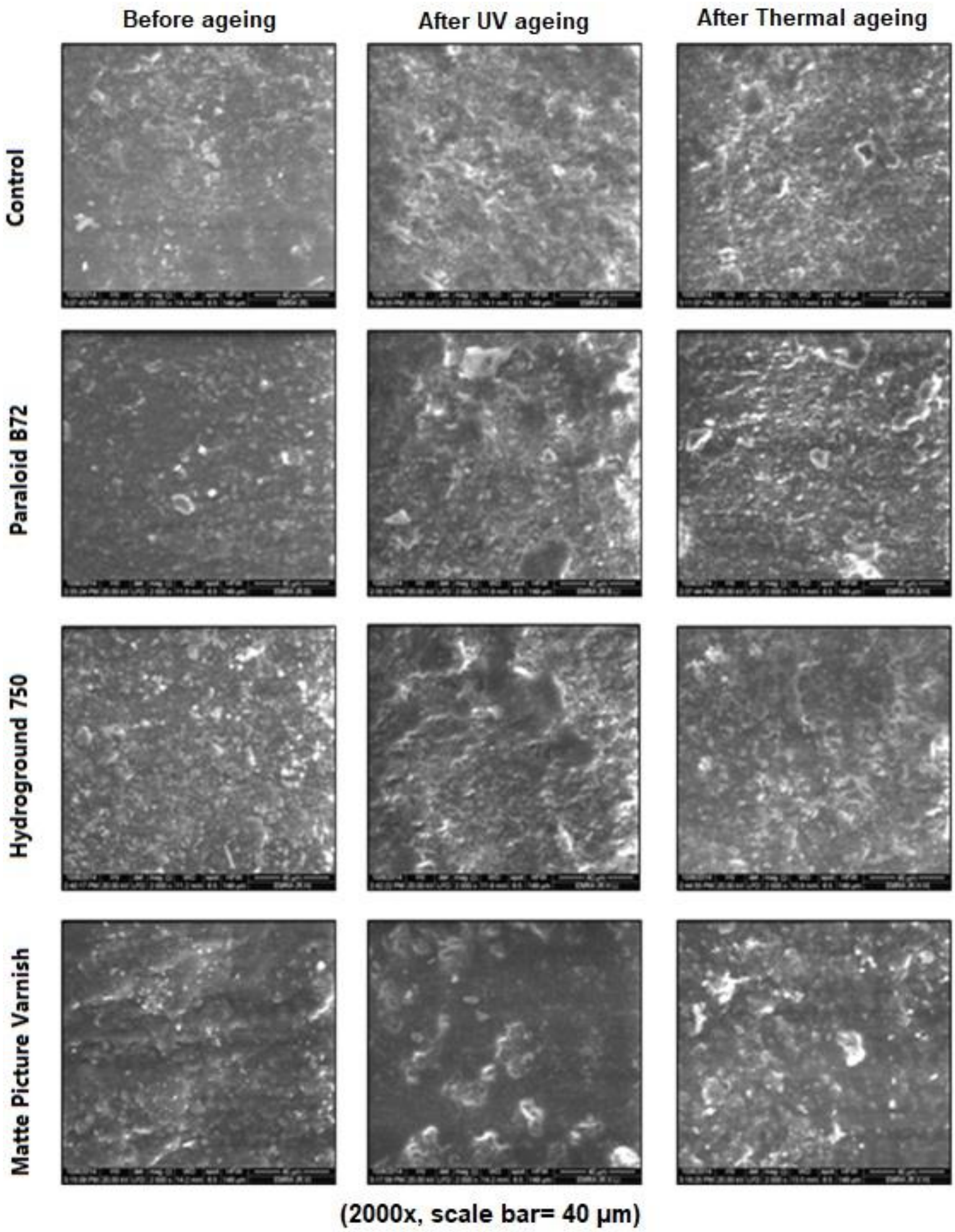

Fig. 6. FE-SEM micrographs (2000x, scale bar = 40μm) of the treated red painted samples before and after UV/thermal ageing.

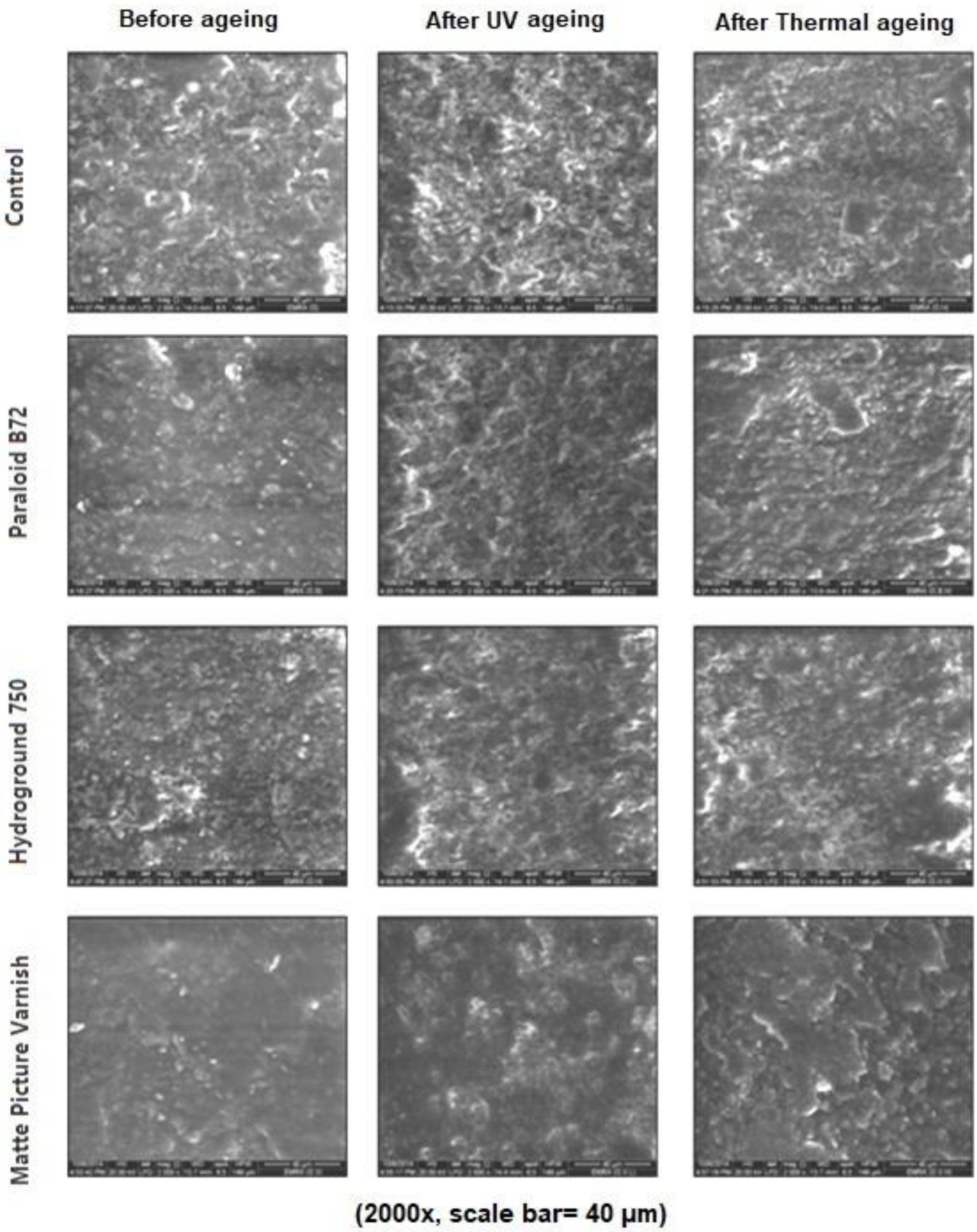

Fig. 7. FE-SEM micrographs (2000x, scale bar = 40µm) of the treated green painted samples before and after UV/thermal ageing

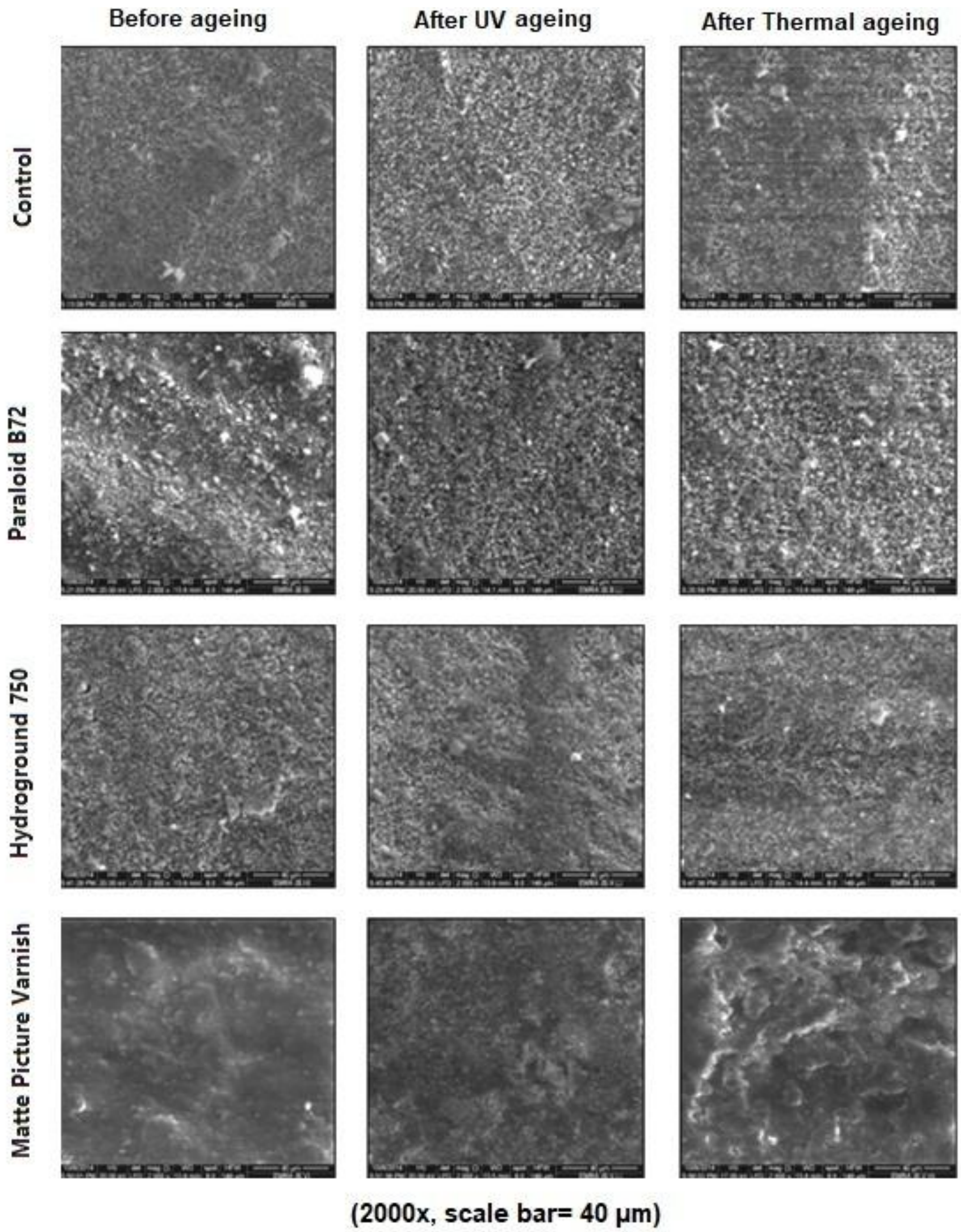

Fig. 8. FE-SEM micrographs (2000x, scale bar = 40µm) of the treated black painted samples before and after UV/thermal ageing.

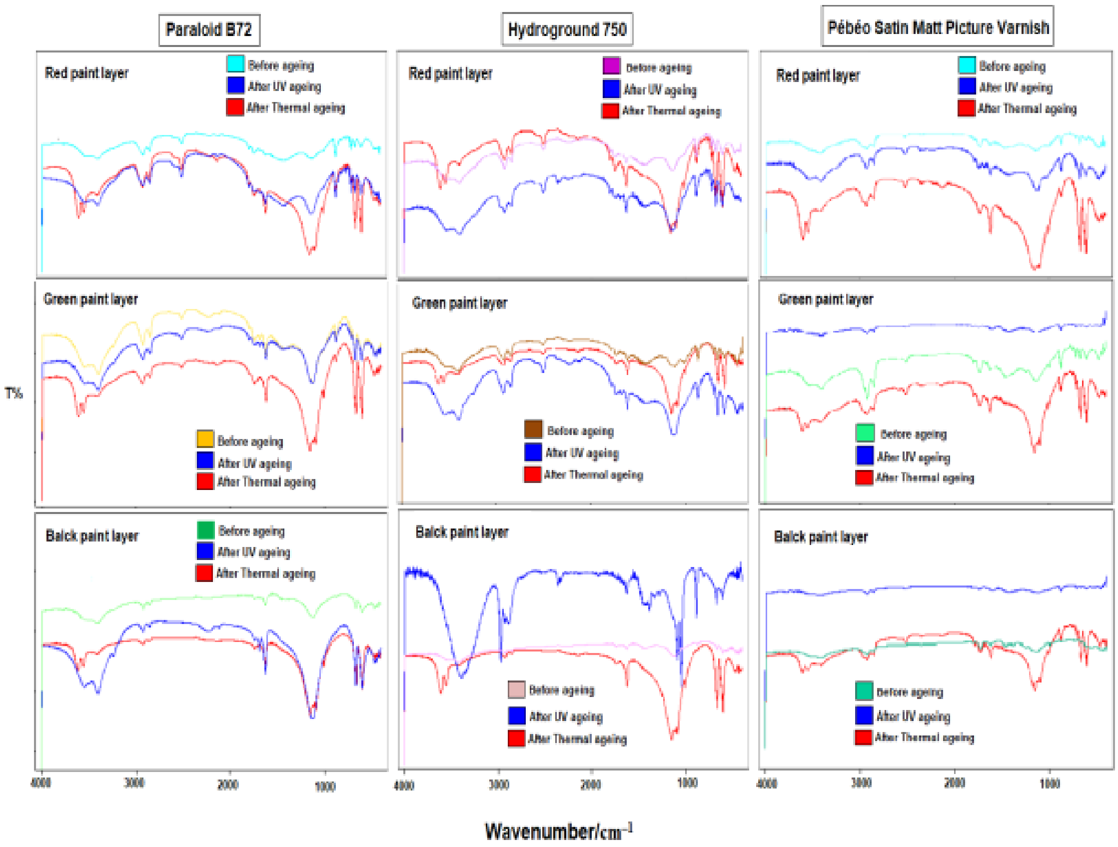

Fig. 9. FTIR spectra recorded on the treated laboratory models, before and after UV/thermal ageing

at 1727 cm^{-1}, C–H bending at 1448 and 1392 cm^{-1}, and ether C–O–C stretching vibration near 1147 cm^{-1}. The infrared spectra of the resins remained almost unchanged throughout the thermal ageing, with a little decrease in the absorption intensity of carbonyl bonds recorded at the final stages of ageing, indicating minor oxidative degradation of the material [23-24]. After UV ageing, the chemical stability has been partially affected. An increase due to oxidation of the ester carbonyl stretching was noticed through the band at 1750 cm^{-1}. According to Hamid and Prichard [25], the primary influence of the oxidative degradation is usually undetectable in the FTIR spectra. Factually, these results are in agreement with the findings of Lazzari and Chiantore [26] on acrylic-based products thermally aged at a temperature of 110 ^{o}C. They stated that for Paraloid B72, restricted decomposition of the esters groups was reported. The thermal ageing increased the stretching vibrations of free and bonded acid OH groups at 3600 cm^{-1}. For Hydroground 750, a group of infrared absorption spectra was registered at: O–H group at 3438 cm^{-1}, C–H stretching at 2958 and 2871 cm^{-1}, ester carbonyl stretching at 1731 cm^{-1}, C–H bending at 1452 and 1398 cm^{-1}, and C–O bond stretching at 1162 cm^{-1}. The UV radiation induced a small decrease in the intensity of the ester groups. The polymer showed a good stability against UV ageing and no considerable change was observed. However, the thermal ageing affected the broad O–H stretching band, which reveals a hydroxyl formation due to an oxidative degradation. For the Matt picture varnish, the infrared absorption spectra showed featured peaks at: C–H stretching at 2960-2850 cm^{-1}, ester carbonyl stretching at 1731 cm^{-1}, δC–H bending at 1471 and 1390 cm^{-1}, and stretching νC–O bonds for esters at the region between 1062-1240 cm^{-1}. Thus, it was concluded that no critical alterations were reported for the polymeric materials after the UV/thermal ageing.

Conclusion

In this contribution, microscopic observations, colour measurements, surface morphology, and the molecular analysis were helpful to evaluate the stability of some acrylic-based polymers for the conservation purpose of 19-th century mural paintings in Egypt. The studied polymers were Paraloid B72, Hydroground 750, and Matt picture varnish. The treated model paint samples were subjected to accelerated UV and thermal ageing. Regardless its stability, Paraloid B72 has affected seriously the appearance of the samples even before the ageing process. On the other hand, the morphological study on the samples showed that no significant alterations occurred. Colour measurements recorded on the samples treated with Hydroground 750 showed the highest ΔE^* values, followed by the samples treated with Paraloid B72. For thermal ageing, the samples showed a good molecular stability mainly for Paraloid B72. In fact, the thermal ageing of polymers usually occurs through an oxidative process or by the direct effect of heat. This was reported for samples treated with Hydroground 750, but it did not affect the material stability. In conclusion, the three polymers showed almost similar stability against UV and thermal ageing, however, Matt picture varnish was the most durable. Thus, it was used as a protective layer for the case study murals .

Acknowledgment

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Conflicts of interest

Authors declare that that they do not have any conflicts of interest.

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