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Gamma radiation cured polyester resin for restoration of cultural heritage wood

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ABSTRACT

In the process of restoring and conserving cultural heritage artifacts and documents, a wide range of coatings based on resins and adhesives are used, including natural products and various types of synthetic polymers. The aim of this study was to examine the effectiveness of polyester-based resins with polymeric monomers cured by gamma radiation without a catalyst. These resins are typically used to impregnate wooden artifacts that have been compromised by insect damage and cannot be consolidated using other methods. Gamma radiation has been effectively used to treat cultural heritage objects, such as rare books, sacred or museum images, and canvas paintings. It is used for insect control, disinfection of harmful microorganisms, such as fungi and bacteria, and for creating new materials for restoration. For this study, formulations of polyester resin, methyl methacrylate monomer (MMA), and styrene (SM) were prepared with different concentrations and combinations. In the absence of catalysts, these formulations were exposed to gamma radiation at a dose of 50 kGy and a rate of 1 kGy/h. After curing, they were analyzed using gel fraction, thermogravimetry (TGA), differential scanning calorimetry (DSC), infrared (FTIR), scanning electron microscopy (SEM) and Optical microscopy (MO). The results show that polyester-based resins were successfully cured by gamma radiation without catalysts or chemicals. This process provides a form of long-lasting protection against fungi, bacteria, and insects, as well as protection against moisture exchange with the environment. These findings demonstrate the effectiveness of the proposed methodology in the conservation and preservation of wooden artworks, being capable of providing support, stability, and quality to the object.

1. Introduction

Cultural heritage is understood as an asset that must be protected owing to its value and representativeness for a given society (FUNARI, 2000). This includes material intentionally crafted by humans which, with the passing of time, has become historical documentation of an era. In the case of works of art, it may also be the sole representative of an artistic expression. Materials that carry information about the trajectory of humankind, their habits, beliefs, fears, ways of interaction with others and with nature (Canani, 2005)- (Francioni, 2014)- (Cruz, 2012), as well as their subsistence, social organization, and trade routes (MacKova et al., 2019), are marked for preservation. Conserving such materials and artifacts allows us to view and understand the present through the prism of the past and even make inferences about the future (De Fátima Bojanoski et al., 2017). The preservation, conservation, and restoration of cultural assets constitute an interdisciplinary activity situated at the intersection of many related domains (Marušić et al., 2022)- (Rizzo et al., 2002), such as cultural heritage management, conservation science, archaeology, and many others.

In the last century, the use of gamma rays, originating from Cobalt-60, has emerged as an alternative for the application in the consolidation and restoration of various types of cultural heritage materials. When compared to traditional methods, such as the use of catalysts for curing polyester-based resins, the use of gamma rays offers a range of benefits. These include ease of application, immediate success, absence of chemical residues, and elimination of the need for quarantine. Gamma rays also afford the possibility of treating materials in their original packaging and definitively eliminating insects and fungi since radiation acts at all stages of their life cycle. However, it does not prevent recontamination if stored in an unhygienic environment (Cortella, 2019)- (Moise et al., 2019). Following irradiation, the absorbed dose in the artifact is cumulative. Disinfection and pest control of cultural

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heritage artifacts have recently met with success through the appliation of gamma radiation with little, or no, apparent alterations (side effects) in the irradiated material. Furthermore, ionizing radiation can be used to consolidate porous materials in combination with the use of resin (Cortella et al., 2020)- (Nagai et al., 2021)- (Appoloni et al., 2022).

It was reported by Tran in 2017 that styrene unsaturated polyester resin cured by gamma radiation could be used for the consolidation of 18th century polychrome wooden sculpture (IAEA, 2017)- (Cortella and Commission, 2022). The process involved consolidating the sculpture with solvent-free resin. Following impregnation of the resin, the sculpture underwent the gamma radiation process using a cobalt-60 source to promote the polymerization of the resin and enable the recovery of the sacred sculpture. Consolidation of the damaged parts allowed for the restoration of the degraded areas and, thus, less susceptible to climatic variations.

Initial investigations of radiation-induced polymerization were driven by the quest for knowledge, particularly the interface between polymers and radiation chemistry, as well as the possibility of developing processes that could compete with traditional polymer production techniques. Recent activities have focused on studying radiation-induced processes in complex chemical mixtures, or environments, using gamma radiation sources and available analytical methods (Moise et al., 2017)- (Lungu et al., 2023)- (Barsbay and Güven, 2020).- (Ashfaq et al., 2020)- (Gueven, 2004). While reports on the use of polyester resins for consolidation can be found in the literature, these studies neither characterize the process nor advance new formulations. To address this gap in our knowledge, we herein report novel formulations feasible for wood consolidation, replacing chemical catalysts with gamma radiation for resin curing.

2. Materials and methods

Unsaturated, isophthalic and low-reactivity polyester resin (LP 8847®), methyl methacrylate monomer (MMA) and styrene monomer (SM) were supplied by Reichhold. Formulations and prepared as follows: Resin 1: Polyester LP 8847®, Resin 2: MMA, Resin 3: 50 % Polyester LP 8847® with 50 % MMA, Resin 4: 33 % Polyester LP 8847® with 67 % SM, the Batch AD dosimeter, type 3042 range of 1–30, and the Batch NE dosimeter, type 4034, range of 5–50, and wood samples of pau-marfim (*Balfourodendron riedelianum*) and cedar (*Cedrela spp.*)

The formulations were placed in containers, homogenized, and left to rest for 24 h to prevent the formation of bubbles. After this period, they were homogenized again and sealed. Following this procedure, they were placed in the Multipurpose Cobalt-60 Irradiator at the Institute of Energetic and Nuclear Research (IPEN) and exposed to gamma radiation with a dose of 50 kGy and a dose rate of 1 kGy/h. The dose rate ensures slow curing in order to maintain the integrity of an impregnated piece.

Batch AD dosimeters, type 3042, and Batch NE dosimeters, type 4034, both made of PMMA (polymethyl methacrylate), were used to calculate the absorbed dose of the samples. Prior to the curing procedure, a preliminary measurement of the distance between the source and the sample was performed to ensure that the received energy would be sufficient to achieve a dose rate of 1 kGy/h. For this determination, the Batch AD dosimeter was used. However, once the appropriate distance was defined, the samples were positioned for curing, together with the dosimeters in direct contact with them. After 50 h of irradiation, the dosimeters were removed and the transmittance/absorbance was measured using a spectrophotometer at specific wavelengths, comparing the results with the calibration curve that is periodically maintained for reading this type of dosimeter. The distance between the source and the sample determines the irradiation time required for curing to occur gradually, which is an essential condition in wood treatment in order to avoid the formation of cracks. It is noteworthy, however, that this distance is relative, being directly associated with the intensity of the radioactive source. In the Cobalt-60 Multipurpose Irradiator, on the date the irradiation was performed, the calculated distance between the radioactive source and the sample to achieve a dose rate of 1 kGy/h was 111 cm.

3. Characterization

a) To characterize the gel fraction, resin samples cured were placed on a nylon mesh (mesh 400) as support. Each piece of mesh was cut into an envelope shape and weighed. Following this, the resin sample was placed inside, and the combined weight of the envelope and resin was recorded. These samples were prepared in triplicate and subjected to extraction in a Soxhlet apparatus with xylene solvent at 120 °C for 12 h. After extraction, the samples were dried in an oven at 60 °C until a constant mass was achieved. The envelopes containing the samples were then weighed, and the mass of the mesh was subtracted. The remaining insoluble portion was considered the irreversible mass of the sample. The gel fraction was calculated as

$$GF\ (\%) = (mf/mi)\ x\ 100, \quad \text{(Eq 1)}$$

Where GF is the gel fraction, mf is the dry mass of the resin after extraction and drying, and mi is the initial mass of the resin before extraction.

b) Fourier Transformation of Infrared Spectroscopy (ATR-FTIR) analysis was conducted using a MIRacle Single Reflection Horizontal ATR Accessory with a ZnSe Crystal Plate Pike® installed on a Nicolet® 6700 FT-IR spectrometer equipped with a cooled MCT detector with N_2 liquid. This infrared spectroscopy technique was employed to characterize the functional groups present in the monomeric units of the sample through the vibration frequency of the atoms.

c) Thermogravimetric analysis (TGA) was employed to monitor the sample's mass variation as a function of temperature or time under a controlled atmosphere. Alumina crucibles were used for the experiments. Heating was conducted at a rate of 10 °C per minute, reaching an equilibrated temperature of 25 °C. The atmosphere used was N_2 with a flow rate of 100 ml per minute, and the maximum temperature achieved was 600 °C. The equipment utilized was the TA Instruments SDT Q6000.

d) Differential Scanning Calorimetry (DSC), was used to determine the polymer transition temperatures, such as melting crystallization and glass transition, enabling the evaluation of energetic, physical, and/or chemical phenomena occurring during sample heating (Bonfim et al., 2014; Andrade et al., 2008). These analyses were conducted simultaneously with the TGA, under the same conditions, using the TA Instruments SDT Q6000 model, with the first derivative being analyzed.

e) Scanning Electron Microscopy (SEM) was used to analyze the formation of pores and cracks on the resin's surface. The analyses were carried out with a Jeol field emission scanning electron microscope (FE-SEM) (JSM-6701 F), operating at 3 kV.

f) Optical microscopy was employed to examine the presence of macropores in the samples, both before and after their impregnation with resin. The resin was introduced into the macropores of the wood, which had been treated using gamma radiation. The analyses were conducted using a Zeiss Primotech optical microscope.

4. Results and discussion

4.1. Gel fraction analysis

Polyester resins are known for their thermosetting properties, as described in the literature (Greene and Greene, 2021). The curing process is typically achieved using catalysts, and the curing of these resins is controlled according to the specific requirements of each application (KRISHNAN et al., 2008)- (Varma et al., 2000)- (Crawford et al., 2002).

Table 1
Samples of resins of cured through gamma radiation and the gel fraction in percentage after extraction.

Formulations	Samples 100 % cure	Gel fraction %
100 % MMA		3.0
100 % LP 8847®		96.5
50 % LP 8847® with 50 % MMA		86.0
33 % LP 8847® with 67 % SM		95.0

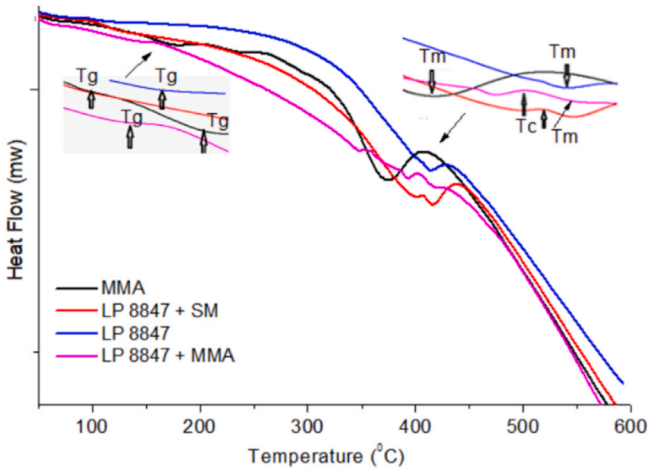

Fig. 1. DSC curves of gamma radiation-cured resins.

In this study, however, catalysts were replaced by the gamma radiation process, yielding significant results, with a total curing of 100 % without the need for such catalysts.

Table 1 presents the results of total curing and the percentages of curing for all resins with Polyester LP 8847® in the absence of catalysts. The resin obtained from MMA monomer showed a reversible process with a gel fraction of only 3 %, thus failing to fall into the category of thermosetting resins. Gel fraction analysis was performed in triplicate and indicated the insoluble portion, the value of which could be attributed to the polymeric fraction of curing by polymer modification. The contribution of 33 % styrene in the formulation is associated with the resin's reversibility. Although the measured difference is only 1.5 %, this is considered an environmentally relevant factor. Regarding the control sample composed solely of styrene, it is known that pure styrene is not curable by radiation, as it does not form the three-dimensional networks required for curing. Styrene becomes viable when blended with the LP 8847® polyester resin, contributing to a reduction in the overall irreversibility of the polymer matrix. These results show that gamma radiation curing is effective, clean and safe, thus respecting the principles of Green Chemistry.

4.2. Differential scanning calorimetry (DSC) analysis

The thermal events in Fig. 1 can be observed through endothermic peaks, indicated by arrows in the auxiliary figure of the graph. These peaks are associated with the glass transition for all resins, ranging from 120 to 177 °C and the melting temperature ranging from 371 to 414 °C. The methyl methacrylate resin presents characteristics of a reversible resin showing a defined melting point around 371 °C and decomposition

Table 2
Values of T_g, T_m and ΔHf of gamma radiation-cured resins.

Samples	T_g (°C)	T_m (°C)	ΔHf (J/g^{-1})
LP 8847 ®	139	414	13
50 % LP 8847® with 50 % MMA	120	394	319
33 % LP 8847® with 67 % SM	125	408	21
MMA	177	371	133

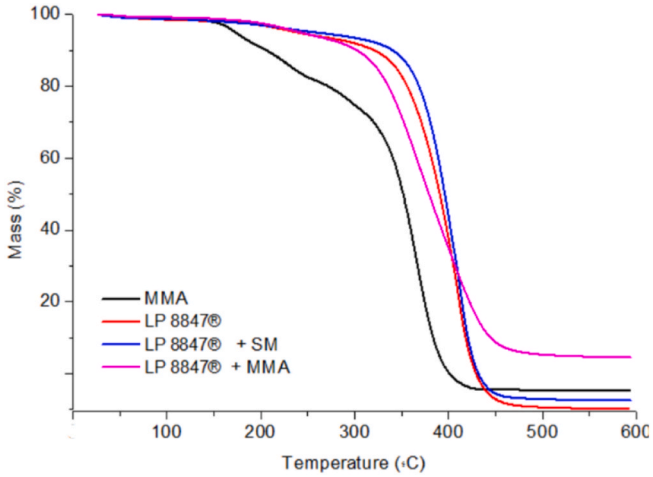

Fig. 2. – TGA curves of the gamma radiation-cured resins.

in the region of 420 °C. The addition of MMA and SM to LP 8847® resins causes a reduction in the glass transition (T_g) and fusion temperature (T_m) values of the resins compared to LP 8847® without additives, as shown in Table 2. We can see that polyester resin mixed with methyl methacrylate and cured by gamma radiation in the absence of catalyst presents a value of ΔHf = 319 J/g^{-1}, which is significant relative to the values of other resins indicated in Table 2.

It is assumed that the crystals which formed, as observed under microscopy (SEM) (see Fig. 5B), were responsible for the increase in ΔHf. Crystals are characteristics that add benefits to polymeric materials. In the literature, it is reported (Yeon et al., 2018)-(Martínez-Barrera et al., 2008) that the addition of MMA monomer to LP 8847® resin through the chemical curing process offers benefits superior to those provided by the addition of styrene, resulting in improvements in the general properties of the polymer, depending on the specific application. These results observed by DSC corroborate with the gel fraction (Table 1), indicating that the presence of MMA in LP8847 favors significant properties, making the new resin with crystallinity and improved reversibility. No observation of crystalline characteristics obtained by the traditional catalyst curing process was found in the literature. However, crystalline polymers have been very attractive for obtaining new materials for the industrial area, for example for automotive parts It should be noted that formation of crystallites occurs in polyester resin with MMA cured by gamma rays. However, the presence of crystals is not significant for wood preservation use.

4.3. Thermogravimetric analysis (TGA) analysis

Cured polyester resins, regardless of added monomer composition, experienced spontaneous loss of mass near the temperature of 218 °C, as observed in Fig. 2. This is a characteristic of all vinyl copolymers which are known to degrade into monomeric units at high temperatures. Thermogravimetric curves display only two mass loss events under a nitrogen atmosphere for all polyester resins and three events for the MMA resin, as well as lower thermal stability compared to other resins containing polyester.

Similar characteristics of mass loss for polyester resins were observed

Table 3
– Onset temperatures (T_{onset}) of mass loss for gamma radiation-cured resins.

Resin	$1°T_{onset}$	$2°T_{onset}$	$3°T_{onset}$
MMA	157	252	326
LP 8847®	217	316	–
50 % LP 8847® with 50 % MMA	217	279	–
33 % LP 8847® with 67 % SM	217	327	–

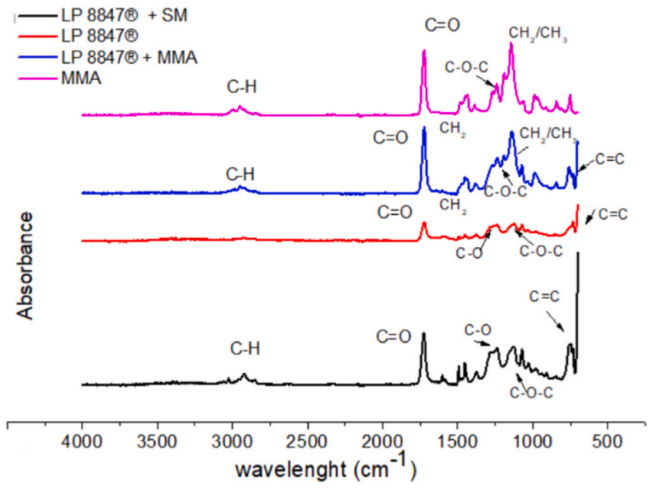

Fig. 3. – FTIR spectra of gamma radiation-cured resin samples.

in the literature in a study conducted through the chemical curing process (Sanchez et al., 2000). Table 3 shows the temperatures at which mass losses occur in the gamma radiation-cured resins, indicating the onset of degradation, also known as the onset temperature (T_{onset}).

Compared to pure LP 8847 resin, the resin composed of 33 % LP 8847 with 67 % SM showed an increase of 11 °C in thermal stability. On the other hand, the resin with 50 % of LP 8847® with 50 % MMA shows a reduction of 38 °C in thermal stability compared to pure LP 8847® resin. It is noteworthy that the presence of MMA decreases the thermal stability of LP 8847® resin, whereas the presence of styrene increases it. This difference in behavior is related to curing density and phase segregation in the different samples.

4.4. Fourier Transform Infrared Spectroscopy (FTIR) analysis

When monomers such as methyl methacrylate (MMA) and styrene (SM) are exposed to gamma radiation, ionization or molecular excitation occurs, resulting in the formation of highly reactive free radicals. These radicals initiate polymerization reactions or chemical modification of the polymer matrix. For MMA (CH_2=C(CH_3)$COOCH_3$), radicals such as •CH_2–C(CH_3) ($COOCH_3$) are formed. In the case of SM (CH_2=CH–Ph), species such as •CH_2–CH(Ph) are generated. These radicals propagate chain reactions, promoting the formation of new polymeric structures. After radical generation, the MMA and SM monomers can integrate into the polyester matrix through different mechanisms, among them curing. The monomers react with the double bonds present in the unsaturated polyester chains, forming covalent bridges between distinct polymer chains. This results in more resistant and rigid three-dimensional networks.

Fourier Transform Infrared Spectroscopy (FTIR) is an essential tool for monitoring polymer curing and observing structural changes in the monomers within the final material. Fig. 3 shows the **(**FTIR) absorption bands of gamma radiation-cured resin samples. The C-H groups are identified in the ranges of 2950 cm- (FUNARI, 2000) to 3000 cm- (FUNARI, 2000), followed by the presence of carbonyl in the region of 1730 cm- (FUNARI, 2000). A characteristic band of MMA is observed in the region of 1450 cm- (FUNARI, 2000), which is shifted to 1440 cm- (FUNARI, 2000) in the presence of polyester. For the groups in the range of 1143 cm- (FUNARI, 2000) of MMA, a shift of the band to 1132 cm- (FUNARI, 2000) is noted in the presence of polyester, which is accompanied by a slight reduction in intensity. Previous studies (Hamulić et al., 2020) report the presence of these groups with similar functions in resins cured by chemical catalysts. The bands shown in Fig. 3 indicate that styrene increased the intensity of the bands present in LP 8847® resin. The same phenomenon occurs in the bands with higher intensity

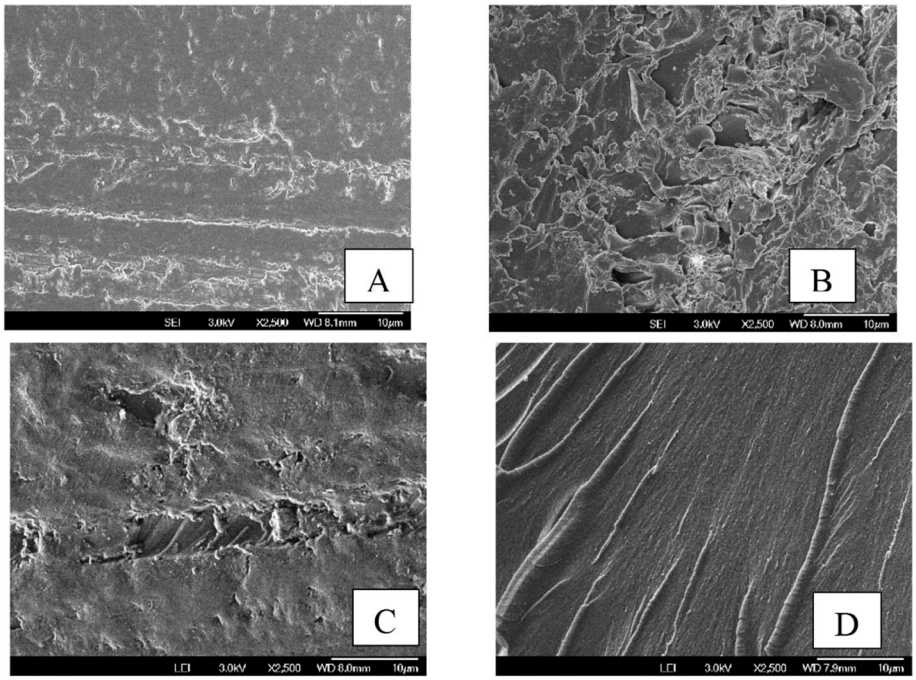

Fig. 4. – SEM images of LP 8847®: (A) cross section; (B) fracture; 33 % LP 8847® with 67 % SM (C) cross section; (D) fracture; both magnified at 2,500×, cured by gamma radiation.

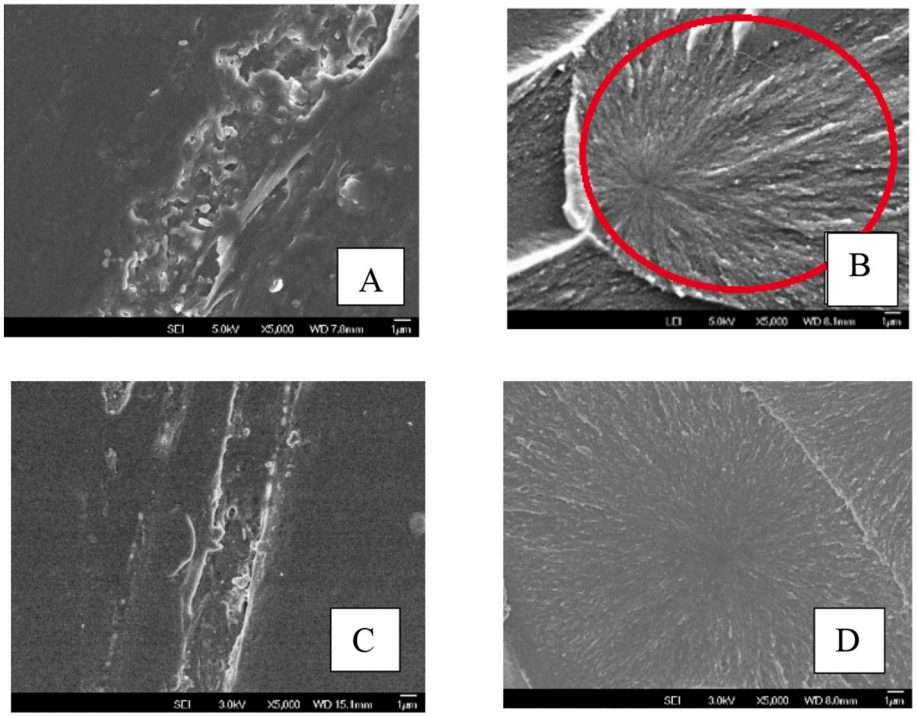

Fig. 5. – SEM image of LP 8847® with MMA: (A) cross section; (B) fracture; MMA (C) cross section; (D) fracture; both magnified at 5,000×, cured by gamma radiation.

with the presence of MMA in LP 8847® resin. These results are consistent with the results obtained from the gel fraction, indicating the occurrence of curing by gamma radiation. According to Barbosa (2007), it is possible to identify specific areas of vibrational infrared wherein each type of bond presents a distinct absorption range (El-Shamy et al., 2017)- (Tretinnikov and Ohta, 2002)- (Jeong et al., 2020).

4.5. Scanning electron microscopy (SEM) analysis

The morphologies of samples were observed in both cross sections and fractures, at Scanning Electron Microscopy (SEM). However, the images resulting from the fractures exhibit homogeneous characteristics, appearing similar in all samples, considering the degree of image magnification. Nevertheless, all samples present gaps and roughness, both in the cut and in the fractured area. It is hypothesized that differences in the pattern of roughness and gaps between LP 8847® and 33 % LP 8847® with 67 % SM is related to the concentration of styrene. During the curing process by a chemical catalyst, some studies in the literature report an increase in the probability of microgel formation and, hence, overlapping tangles when high concentrations of styrene are present in the polyester resin during curing. Expansions caused by styrene are also observed (Yang and Lee, 1988)- (Mun and Choi, 2008)- (Zhao et al., 2018). For LP 8847® resins with MMA, sharp regions related to the formation of crystals during the curing of the sample by gamma radiation were observed in the fractures of samples, indicated by a cycle in Fig. 5b. The formation of crystals, which does not occur when the polyester resin is cured with a catalyst, is an important characteristic for the development of new polymeric materials. Because is characteristically intrinsic to crystalline structures that make them attractive for the development of new materials The formation of these crystals is consistent with the value observed in ΔHf in Figs. 1 and 5B. Figs. 4 and 5 show images generated by the four different resin compositions.

Table 4
Presents comparisons between the techniques used to characterize resins cured by gamma radiation.

Analytical techniques	Formulations			
	LP 8847®	LP 8847®+SM	LP 8847®+MMA	MMA
Gel fraction	96.5 %	95,0 %	86.0 %	3,0 %
DSC	ΔHf (J/g^{-1}) = 13	ΔHf (J/g^{-1}) = 21	ΔHf (J/g^{-1}) = 319	ΔHf (J/g^{-1}) = 133
TGA	$2°T_{onset}$ = 316 °C	$2°T_{onset}$ = 327 °C	$2°T_{onset}$ = 279 °C	$2°T_{onset}$ = 252 °C
FTIR	main peaks C=O; C-O-C; CO; CH/CH	main peaks C=O; C-O-C; CO; CH/CH; CH band emergence in the 2800 region	main peaks C=O; C-O-C; CO; CH/CH, CH band emergence in the 2800 region	main peaks C=O; C-O-C; CO; CH/CH, CH band emergence in the 2800 region
SEM	The transversal morphology of the sample presents irregular or amorphous regions, smooth surfaces and the fracture presents gaps and roughness	The transversal morphology of the sample presents irregular regions and gaps, while the fracture presents a smooth surface and lines indicating sublayers.	The transversal morphology of the sample presents roughness and a smooth surface, while the fracture presents cleavage planes and regular arrangements, characteristic of a crystalline structure.	The transversal morphology of the sample presents roughness and gaps, while the fracture surface appears smooth with regular lines.

4.6. Comparisons between the techniques used to characterize resins cured by gamma radiation

In Table 4, comparisons are presented between the techniques used

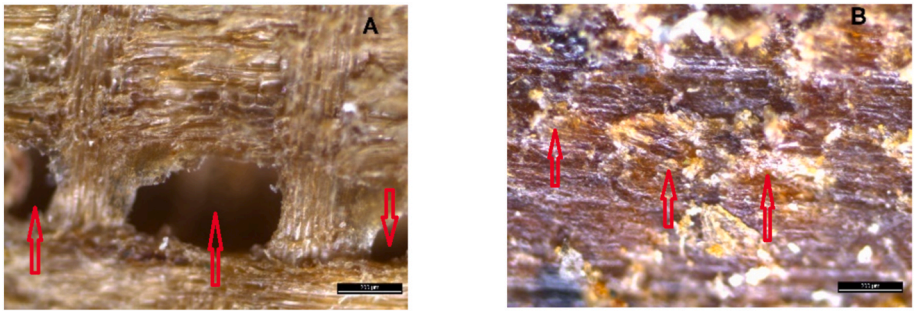

Fig. 6. Optical Microscopy of Cedar (*Cedrela* spp., *P. Browne*) Sample – Surface: (A) Before irradiation, without resin; (B) With resin and after irradiation.

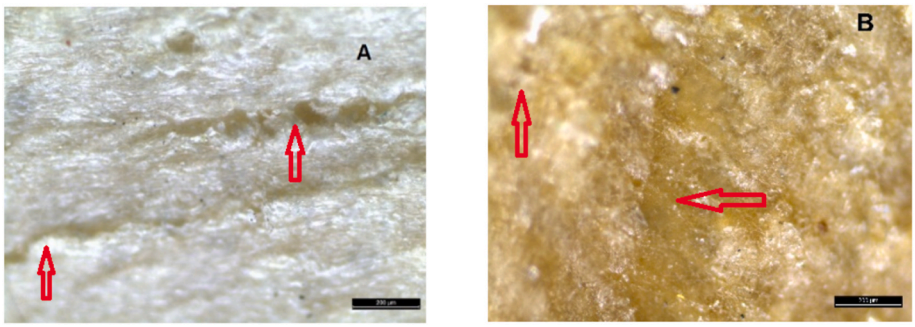

Fig. 7. Optical Microscopy of Pau-Marfim Sample – Surface: (a) Before irradiation without resin; (b) With resin and irradiated.

for the characterization of gamma radiation-cured resins. The four resins analyzed show potential for application in wood impregnation. Although the results indicate that the resins containing MMA exhibit distinct characteristics—such as lower gel fraction and higher ΔHf value (J/g^{-1}), indicative of crystallinity—the morphology observed by SEM is consistent with the formation of crystalline phases. However, crystallinity was not the focus of this research, since it is not considered a relevant factor for the performance of the resin in wood. For the impregnation process, it is necessary to use a sufficient amount of resin so that the wood remains completely submerged throughout the procedure. After completing the physicochemical characterizations, the LP 8847® formulation with styrene was the one that remained available in greater quantity. Thus, cedar and ivory wood samples were selected, as these species are traditionally used by artisans in wood carving. Additionally, the LP 8847® resin with SM exhibited advantageous properties, particularly its higher thermal stability compared to the other formulations, as well as it was lower cost. It is noteworthy that the acquisition cost of MMA is approximately five times higher than that of LP 8847® with SM.

4.7. Optical microscopy (MO) analysis

In Optical Microscopy Figs. 6 and 7 the samples of wood were examined both before and after the impregnation with resin, curing of 33 % polyester LP 8847® and 67 % styrene. Optical microscopy revealed the presence of macropores in the canafistula and ivorywood samples prior to impregnation indicated with arrows in the image. The impregnation with polyester/styrene resin resulted in the filling of macropores, as evidenced by the color change in the ivorywood. This change indicates a higher concentration of resin in the macropores indicated with arrows in the image. The formation of a homogeneous polymer matrix, confirmed by this optical microscopy technique, demonstrates the efficient penetration of the resin into the wood samples in figure. These results, along with the observation that the macropores were filled with resin, suggest that impregnation with radiocurable resins could be a viable technique for the preliminary treatment of sacred imagery pieces with degraded structure, providing protection against insect damage and consolidating areas damaged by such actions.

5. Conclusion

This study demonstrated that polyester, SM, and MMA based resins cured by gamma radiation show potential for application in the impregnation and restoration of wooden artifacts damaged by insect attack. The ability to control curing through the absorbed dose in kGy represents a significant advantage over the traditional catalyst-based method. The results indicate that the use of gamma radiation in resin curing constitutes an innovative and clean strategy, aligned with the principles of Green Chemistry, opening new perspectives for the conservation of structurally fragile wooden cultural heritage materials.

CRediT authorship contribution statement

Maria Aparecida M.G. Pereira: Writing – original draft, Visualization, Validation, Resources, Project administration, Methodology, Investigation, Formal analysis. **Maria José Alves. Oliveira:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization, Maria José Alves Oliveira, Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Larissa Otubo:** Writing – review & editing, Supervision, Methodology. **Leni M.P.R. Lima:** Writing – review & editing, Visualization, Supervision. **Pablo Antonio Salvador Vasquez:** Writing – original draft, Visualization, Validation, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

No data was used for the research described in the article.

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