2026 International Nuclear Atlantic Conference – INAC 2026
Escola de Guerra Naval, Rio de Janeiro, RJ, August 24 - 28, 2026

Gamma Irradiation Effects on Indigo Pigment Dispersed in Paraloid® B-72 and Xylene: Implications for Cultural Heritage Conservation

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1. Introduction

Cultural heritage comprises tangible and intangible assets of historical, artistic, and symbolic significance, whose preservation is essential for maintaining collective memory (1). Among these assets, works of visual art produced on different media stand out, frequently susceptible to degradation caused by fungi, bacteria, and insects. Conventional disinfestation methods may present limitations, such as residual toxicity, microbial resistance, and physicochemical alterations in materials, while alternatives such as fumigation, anoxia, and heat treatments have high costs and application times (2- 3). In this context, gamma radiation has stood out since the 2000s as a safe and effective technology for the decontamination of cultural collections without significantly compromising their original characteristics (4-5-6). Considering that each material presents different levels of sensitivity to ionizing radiation and that, for the disinfestation of microorganisms, such as fungi, doses between 6 and 10 kGy are generally employed, this work aimed to investigate the effects of gamma radiation on indigo pigment solubilized in Paraloid® and xylene. This system is widely used both in easel paintings and in processes. restoration and chromatic recomposition of pictorial works.

2. Methodology

Indigo pigment was dispersed in a 1:1 ratio in Paraloid B-72, a material used for reintegration in restorations due to its reversibility. Paraloid B-72 is a copolymer of ethyl methacrylate and methyl methacrylate, soluble in aromatic hydrocarbons, with xylene used as the pigment dispersion medium for application onto the fabric screen. After preparing the screen, the samples were divided into two parts. In Part 1, the samples were irradiated at doses of 5 and 15 kGy. In Part 2, a layer of Winsor & Newton High Gloss Varnish spray varnish and a layer of Winsor & Newton Matt Varnish, both used in reversible restorations, were applied, and subsequently subjected to the same irradiation doses. After irradiation, the samples were characterized by Fourier transform infrared spectroscopy (FTIR) in ATR (diamond crystal single reflection) mode, using an Agilent Technologies spectrometer (Cary model, DTGS detector). Analyses were conducted with a spectral resolution of 4 cm^{-1} and 128 cumulative scans per spectrum.

3. Results and Discussion

Figure 1 shows the FTIR spectra of indigo pigment samples irradiated with xylene at 0 kGy, 5 kGy, and 15 kGy, recorded in the range of 400 to 4000 cm^{-1}. Fig. 2A and 2B highlight different spectral regions, aiming to facilitate the observation of the changes verified in the spectra.

As highlighted (in Fig. 2A), the decrease in intensity of the bands at 652, 677, and 720 cm^{-1} is noted, already observed at the dose of 5 kGy, while the intensity of the shoulder at 869 cm^{-1} decreases at 15 kGy. These bands correspond to out-of-plane C–H aromatic deformations [7], present both in the molecular structure of the indigo pigment and in xylene compounds. This behavior suggests that the observed changes are related to the rupture of the molecular structure of these compounds or to the loss of conjugation of the aromatic ring. There is no evidence of the formation of oxidized compounds in the recorded FTIR spectra. The bands at 2852 and 2920 cm^{-1} are attributed to aliphatic C–H symmetric stretching vibrations (Fig. 2B), probably related to the aliphatic compound Paraloid B-72. The decrease in the intensity of these bands suggests alterations in the molecular structure of the compound, such as rearrangement or the formation of cross-links between polymer chains, reducing the number of CH_2/CH_3 groups and, consequently, the intensity of the vibrational modes associated with them. No significant changes were observed in the other spectral regions. The analyzed samples consist of a mixture of indigo pigment (2-(3-hydroxy-1H-indol-2-yl)indol-3-one), Paraloid B-72, and xylene. It is important to consider that Paraloid B-72 is a thermoplastic acrylic resin, while xylene corresponds to a mixture of the isomers o-dimethylbenzene, m-dimethylbenzene, and p-dimethylbenzene. Another relevant aspect is that commercial pigments, such as indigo, often contain small concentrations of inorganic additives or extenders used to adjust color properties according to the manufacturer.

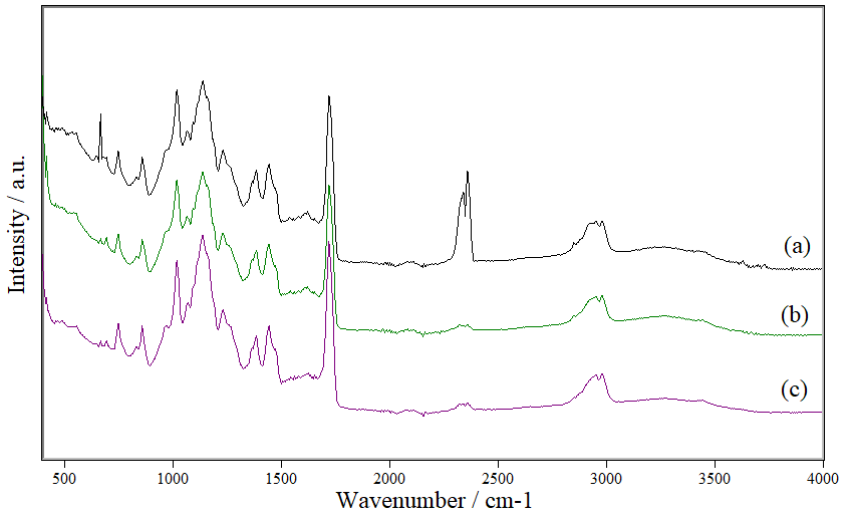

Figure 1. Indigo / xylene pigment (a) 0 kGy; (b) 5 kGy; (c) 15 kGy

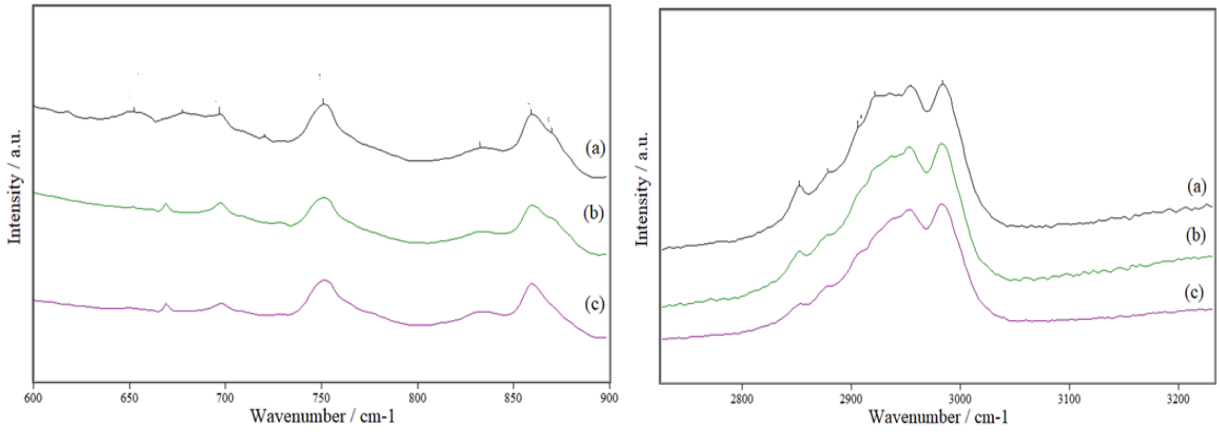

Figure 2. Indigo/xylene pigment, (a) 0 kGy; (b) 5 kGy; (c) 15 kGy.

In Fig. 3A, the FTIR spectra of the indigo pigment samples with xylene and with varnishes applied to the surface are observed. The samples were prepared under the same conditions described previously, with the addition of two layers of varnish on the surface, and irradiated at doses of 5 and 15 kGy. Figs. 4A and 4B highlight different spectral regions in which changes were observed in the materials exposed to radiation.

In Fig. 4A, a subtle increase in the relative intensity of the band at 1070 cm^{-1} is noted for the dose of 5 kGy, related to C–O stretching vibrations [7]. In addition, a decrease in the bands at 2853 and 2920 cm^{-1}, attributed to aliphatic C–H stretching [8], is observed after exposure to the dose of 15 kGy (Fig. 4B), which may be related to the formation of chemical bonds in organic compounds. The mode at 728 cm^{-1} is related to C–C stretching vibrations and rocking bending of $–CH_2$ [9 - 10], while the band at 697 cm^{-1} corresponds to aromatic ring deformation modes [1]. The decrease in the relative intensity of these bands may be associated with the loss of aromatic ring conjugation or the disruption of the molecular structure. Bands below 470 cm^{-1} are generally associated with silicates, commonly used as extenders in synthetic pigments. In this sense, the appearance of the band at 463 cm^{-1} in the material exposed to 15 kGy is interpreted as most likely related to sample heterogeneities, possibly resulting from a higher concentration of silicates at the point analyzed by FTIR-ATR.

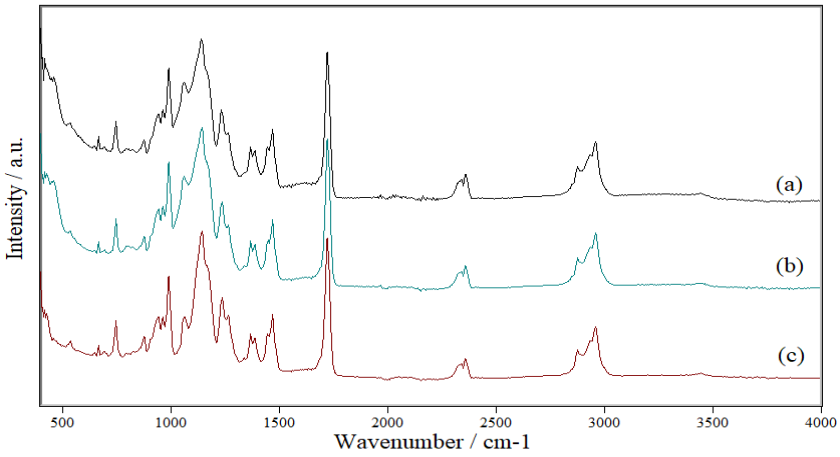

Figure 3. Indigo pigment / xylene / varnishes (a) 0 kGy; (b) 5 kGy; (c) 15 kGy.

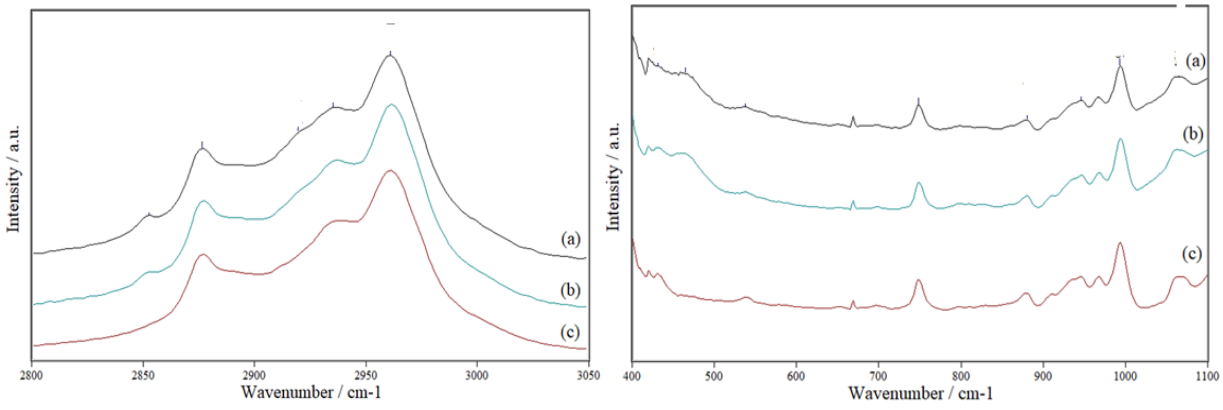

Figure 4. Indigo pigment / xylene / varnishes (a) 0 kGy; (b) 5 kGy; (c) 15 kGy.

4. Conclusions

In the varnished samples, additional spectral variations were detected, mainly at 15 kGy, although no evidence of significant oxidation products was identified. Overall, the results indicate that the materials preserved their main structural characteristics after exposure to doses of gamma radiation commonly used

for the decontamination of cultural heritage. These findings reinforce the potential applicability of gamma irradiation as a safe and effective method for the conservation and decontamination of easel paintings and restoration materials, while also highlighting the importance of assessing the radiation sensitivity of each restoration system individually.

Acknowledgements

The authors are grateful to the Project Development and Implementation of Cultural Heritage Preservation Using Ionizing Radiation Technology (Nº. F22082) of the International Atomic Energy Agency (IAEA), and the research group led by Dr. Ximena Villagran from the Microarchaeology Laboratory of the Museum of Archaeology and Ethnology at the University of São Paulo.

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