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PRESERVATION OF HISTORICAL GLASS PLATE NEGATIVES USING E-BEAM TECHNOLOGY

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Abstract.

Glass-plate photographic negatives constitute valuable cultural heritage materials that preserve historical, scientific, and documentary information. However, their long-term preservation is frequently challenged by microbiological contamination and material degradation. In this study, the effects of electron beam irradiation on historical glass-plate photographic negatives were investigated to evaluate its potential as a decontamination technology for cultural heritage applications. Samples were irradiated with doses ranging from 2 to 25 kGy under both ambient (25 °C) and cryogenic (-70 °C) conditions. The effects of irradiation were assessed by Fourier transform infrared spectroscopy (FTIR), visual inspection, digital image analysis, and microbiological evaluation. FTIR results showed no detectable chemical modifications in the glass matrix following irradiation. Visual analyses indicated that radiation-induced optical changes increased with dose but were significantly reduced when irradiation was performed under cryogenic conditions. Microbiological analyses identified fungal contamination in non-irradiated samples, whereas no fungal growth was observed after irradiation, demonstrating the effectiveness of the treatment for biological decontamination. Although minor visual alterations were detected in the digitized images, the photographic information remained preserved. The results demonstrate that electron beam irradiation is a promising, residue-free technology for the decontamination of historical glass plate photographic negatives, particularly when combined with temperature-control strategies to minimize radiation-induced optical effects.

Key Words: Electron beam irradiation; Cultural heritage; Glass-plate photographic negatives; Biodeterioration; Decontamination; Conservation science.

1. Introduction

The preservation of cultural heritage collections remains a major challenge due to the wide variety of materials involved and their susceptibility to physical, chemical, and biological degradation. Among these materials, glass-plate photographic negatives represent valuable documentary and historical records. However, their long-term preservation is often compromised by the fragility of the glass support and by biological contamination, including fungi, bacteria, and insects. These agents can affect both the physical stability of the substrate and the quality of the photographic image, emphasizing the need for effective decontamination procedures compatible with conservation requirements [1].

Several methods have been employed for the disinfection and decontamination of cultural heritage materials, including chemical treatments [2], anoxic atmospheres [3], freezing [4], and ultraviolet irradiation [5]. Although these techniques have demonstrated effectiveness in specific applications, they may present limitations such as incomplete microbial inactivation, long processing times, residue generation, or potential adverse effects on the treated materials.

Consequently, the development of alternative decontamination technologies that combine high biocidal efficiency with minimal impact on heritage objects remains an active field of research.

Ionizing radiation has been increasingly investigated as a decontamination tool for cultural heritage materials. Its advantages include high effectiveness against a broad range of microorganisms, the absence of chemical residues, and the ability to treat packaged or complex objects. In particular, radiation-based technologies offer precise control of processing parameters, such as absorbed dose and dose rate, enabling treatment protocols to be adapted to different materials and conservation requirements [6], [7], [8]. These characteristics have contributed to the growing interest in radiation processing for the preservation of cultural heritage collections [3].

Despite its potential benefits, the application of ionizing radiation to glass-based materials requires careful evaluation. Previous studies have reported radiation-induced effects in glass, including color center formation, darkening, and modifications in optical and structural properties, depending on the glass composition and irradiation conditions. Therefore, the identification of irradiation parameters that ensure effective decontamination while minimizing material alterations is essential for the safe implementation of this technology in conservation practices [9].

In this context, the present work evaluates the effects of electron beam irradiation on glass-plate photographic negatives. Particular attention is given to strategies aimed at reducing radiation-induced changes, including temperature control during irradiation and post-irradiation recovery treatments. The results contribute to a better understanding of the interaction between ionizing radiation and historical glass photographic materials and provide relevant information for the development of safe radiation-based decontamination protocols for cultural heritage applications.

2. Materials and Methods

2.1 Materials

For the destructive analyses and the development of the proposed method, commercial samples of ordinary glass (VC), historical glass-plate photographic negatives from the Cida Rivelli (CR) private collection, and photographic negatives from the Museum of Zoology (MZ) collection were used. The dimensions and thicknesses of the samples used are presented in Table 1.

TABLE 1. DIMENSIONS AND THICKNESSES OF THE GLASS SAMPLES AND PHOTOGRAPHIC NEGATIVES USED IN THIS STUDY

Sample	Image	Dimension (cm)	thickness (mm)
VC		3 x 1	2
CR		9 x 6,5	2
MZ		9 x 6,5	2

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2.2 Electron beam irradiation

Electron beam irradiation was carried out at the Radiation Technology Center of the Nuclear and Energy Research Insitute (IPEN, São Paulo, Brazil) using a JOB 188 electron accelerator (Radiation Dynamics Inc., USA). The accelerator operates at a maximum energy of 1.5 MeV, with a beam current of up to 25 mA and an effective scanning width of 120 cm.

Samples were irradiated at absorbed doses of 2, 6, 10, and 25 kGy, using a dose rate of 2.3 kGy s^{-1}. To investigate the influence of temperature during irradiation, the samples were placed inside tempered-glass containers and irradiated under two different conditions: (i) ambient temperature (25 °C) and (ii) cryogenic conditions (-70 °C). A schematic view of the irradiation setup is shown in Fig. 1.



FIG. 1 – *Irradiation facility and experimental arrangement used during electron beam processing*.

2.2 Characterization techniques

2.3.1 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier transform infrared (FTIR) spectra were acquired using a Cary 630 FTIR spectrometer (Agilent Technologies, USA) equipped with an attenuated total reflectance (ATR) accessory. Analyses were performed at the Microarchaeology Laboratory of the Museum of Archaeology and Ethnology, University of São Paulo (USP).
Spectra were collected in the 4000–400 cm^{-1} range with a spectral resolution of 2 cm^{-1} and 128 accumulated scans per measurement. Data acquisition and processing were performed using Agilent MicroLab software.

2.3.2 Fungal identification

Fungal contamination analyses were conducted at the Laboratory of Toxigenic Fungi and Mycotoxins, Institute of Biomedical Sciences, University of São Paulo (USP).

Microorganisms were sampled using sterile cotton swabs moistened with sterile distilled water and rubbed across the entire surface of each photographic negative. The collected material was inoculated onto 90 × 15 mm sterile Petri dishes containing Dichloran Glycerol Agar (DG18;

Neogen®, USA) supplemented with 0.01% chloramphenicol, following established methodologies reported in the literature [10], [11], [12].

DG18 medium was selected due to its suitability for the isolation and enumeration of xerophilic fungi commonly associated with low-moisture substrates, such as photographic materials [13]. After inoculation, plates were incubated for 15 days in a biochemical oxygen demand (BOD) incubator at 25 °C and 47% relative humidity.

Following incubation, fungal growth was evaluated by determining the number of colony-forming units (CFU) per sampled area. Fungal identification was performed through macroscopic and microscopic morphological characterization according to standard taxonomic procedures [14]. Macroscopic observations included colony morphology, texture, growth rate, and pigmentation. Microscopic analyses were performed by mounting fungal structures in lactophenol cotton blue and examining them under an optical microscope at 400× magnification.

2.3.3 Image Digitization and Digital Analysis

The photographic negatives were digitized to document and evaluate possible visual alterations induced by the irradiation treatments. Image acquisition was performed using a Nikon D5100 digital single-lens reflex (DSLR) camera equipped with an 18–55 mm lens. The camera was mounted on a tripod and positioned above a uniform light table to ensure reproducible illumination conditions during image capture (Fig. 2).
The acquired images were subsequently processed using the open-source software GIMP (version 2.10.36), where the negative images were digitally converted into positive images to facilitate qualitative visual assessment and comparison.



FIG. 2 – *Experimental setup employed for image acquisition of the glass-plate photographic negatives, consisting of a DSLR camera, tripod, and illuminated light table.*

3. Results and Discussion

3.1 FTIR Analyses

FTIR spectroscopy was employed to investigate possible chemical modifications induced by electron beam irradiation in both commercial glass (VC) and historical glass-plate photographic negatives. The spectra obtained for the analyzed samples are presented in Fig. 3.

The spectra exhibited the characteristic absorption bands of silicate-based glasses, predominantly associated with Si–O–Si stretching and bending vibrations that constitute the vitreous network. Comparison between non-irradiated and irradiated samples revealed no significant changes in band position, intensity, or profile over the investigated dose range. These results indicate that electron beam irradiation up to 25 kGy did not promote detectable chemical modifications in the glass structure.

The absence of significant spectral variations suggests that the irradiation conditions employed in this study preserved the chemical integrity of the glass matrix. Similar findings have been reported for silicate materials exposed to sterilization and decontamination doses, where radiation-induced effects are predominantly associated with optical rather than chemical alterations.

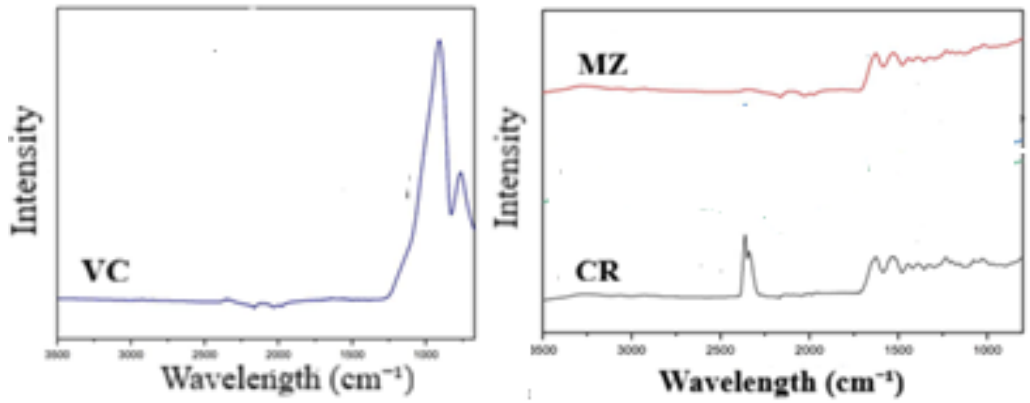

FIG. 3 – *FTIR spectra of commercial glass (VC) and historical glass-plate photographic negative samples before and after electron beam irradiation*.

3.2 Visual Assessment of Irradiated Glass Samples

The visual appearance of glass samples irradiated under different temperature conditions is shown in tabele 2. Progressive optical changes were observed with increasing absorbed dose, particularly in samples irradiated at room temperature (25 °C). These changes were characterized by increased darkening and reduced transparency of the glass substrate.

In contrast, samples irradiated under cryogenic conditions (-70 °C) exhibited significantly less pronounced visual alterations. The results indicate that temperature plays an important role in the development of radiation-induced optical defects in glass.

The reduced darkening observed at low temperatures may be attributed to the suppression of defect formation and stabilization processes within the glass network. Previous studies have demonstrated that radiation-induced color centers, which are responsible for glass darkening, are influenced by atomic mobility and defect recombination mechanisms. Lower temperatures reduce structural mobility, limiting the formation and stabilization of these defects and consequently mitigating optical degradation [15].

These findings suggest that sample cooling during irradiation represents an effective strategy for minimizing undesirable visual changes while maintaining the decontamination efficiency of the radiation process.

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TABLE 2 - COMMERCIAL GLASS SAMPLES IRRADIATED AT 25 °C AND -70 °C WITH ABSORBED DOSES OF 2, 6, 10, 15, AND 25 kGy

Dose kGy	25°C	-70°C
2		
6		
10		
15		
25		

3.3 Evaluation of Fungal Contamination

Microbiological analyses revealed fungal contamination in the non-irradiated photographic negatives. Based on macroscopic and microscopic characterization, the predominant fungal genera identified were *Aspergillus*, *Cladosporium*, and *Penicillium*. Additionally, nine isolates were classified as non-sporulating fungi (NSF) because they failed to produce reproductive structures under the cultivation conditions employed. Following electron beam irradiation, no fungal growth was detected in any of the treated samples. The complete absence of colony formation demonstrates the effectiveness of electron beam processing for the elimination of fungal contamination present on the surface of the photographic negatives in Fig.4.

The observed biocidal effect is consistent with the well-established action of ionizing radiation on microbial cells, which involves damage to nucleic acids and other essential cellular components, ultimately preventing cell replication and survival. Considering the sensitivity of historical photographic materials to conventional chemical treatments, the use of electron beam

irradiation represents a promising alternative for biological decontamination without direct chemical intervention.

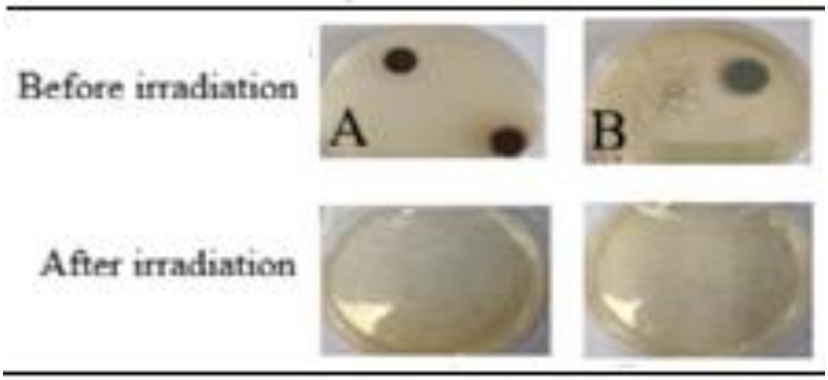

Fig. 4 – *Photographic negative samples from the Cida Rivelli (CR) collection (A) and the Museum of Zoology (MZ) collection (B) before and after irradiation at 6 kGy*.

3.4 Digital Image Evaluation

To facilitate the assessment of potential irradiation-induced alterations, the digitized negatives were converted into positive images using GIMP software. Representative images before and after irradiation are presented in Fig. 5.

Visual comparison revealed subtle modifications in image appearance after treatment, including slight reductions in tonal contrast, localized lightening, and minor losses of image definition. These effects may be associated with radiation-induced optical changes in the glass substrate and/or alterations in the photographic emulsion layer.

Differences in the response of the two historical collections were also observed. The MZ samples exhibited more pronounced visual changes than the CR samples after exposure to the same irradiation dose. This behavior may be related to the conservation condition of the photographic negatives prior to treatment. Historical degradation processes, including silver mirroring, silver oxidation, emulsion deterioration, and previous environmental exposure, may increase the susceptibility of photographic materials to irradiation-induced effects.

Despite the observed visual modifications, the overall image content remained preserved, indicating that electron beam irradiation at 6 kGy can achieve effective fungal decontamination while maintaining the documentary information contained in the photographic negatives.



Fig. 5 – *Digitized positive images of CR and MZ photographic negatives before and after electron beam irradiation at 6 kGy.*

5. Conclusion

This study evaluated the effects of electron beam irradiation on historical glass-plate photographic negatives with the objective of assessing its suitability as a decontamination method for cultural heritage materials. FTIR analyses demonstrated that irradiation at doses up to 25 kGy did not induce detectable chemical modifications in the glass matrix, indicating preservation of the material's chemical structure. Visual assessments revealed that radiation-induced optical changes were dependent on irradiation conditions, with sample cooling at -70 °C significantly reducing glass darkening and other undesirable visual effects.

Microbiological analyses confirmed the presence of fungal contamination in non-irradiated negatives, including genera commonly associated with the biodeterioration of archival materials. Electron beam irradiation at 6 kGy successfully eliminated fungal growth, demonstrating the effectiveness of the process for biological decontamination.

Although minor visual alterations were observed in the digitized images after irradiation, the photographic information remained preserved, suggesting that electron beam processing can provide an effective balance between microbial inactivation and conservation of historical records. Furthermore, the implementation of cooling during irradiation proved to be a promising strategy for mitigating radiation-induced optical changes in glass-based photographic materials.

Overall, the results demonstrate that electron beam irradiation is a viable and environmentally friendly alternative for the decontamination of historical glass-plate photographic negatives. The methodology shows significant potential for application in cultural heritage preservation, particularly when combined with temperature-control strategies aimed at minimizing alterations to sensitive materials. Future studies should focus on quantitative optical characterization and long-term aging assessments to further validate the conservation safety of the proposed treatment.

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Conflicts of interest

The authors declare that they have no conflicts of interest

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