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Radiation-induced copper oxide formation in a clinical gel composite: a novel approach for dosimetry

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Abstract: This research is intended to investigate radiation-induced changes in a clinical gel composite with copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$). This study aimed to investigate the mechanisms was to understand the mechanisms of interaction between radiation and the material, within the proposal to develop accessible dosimetric materials that can efficiently monitor radiation doses. The samples were previously studied by ultraviolet-visible (UV-Vis) spectroscopy, demonstrating their potential for dosimetric use. For this work, the material was irradiated with gamma doses of 30 and 100 kGy and analyzed by nuclear magnetic resonance (NMR) spectroscopy and Raman spectroscopy. It was observed that the color of the samples changes from blue to red with increasing radiation dose, suggesting the formation of copper oxides. NMR analysis revealed changes in longitudinal (T1) and transverse (T2) relaxation times, indicating interactions between the gel matrix and copper sulfate. Raman spectroscopy confirmed the formation of new peaks associated with the presence of copper oxides. pH measurements were also performed to corroborate the results.

Keywords: dosimetry, gamma irradiation process, clinical gel, $CuSO_4 \cdot 5H_2O$.

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Formação de óxido de cobre induzida por radiação em um compósito de gel clínico: uma nova abordagem para dosimetria

Resumo Este trabalho se baseou na investigação das mudanças radioinduzidas em um compósito de gel clínico com sulfato de cobre pentahidratado ($CuSO_4 \cdot 5H_2O$). O objetivo foi compreender os mecanismos de interação da radiação com o material, dentro da proposta de desenvolver materiais dosimétricos acessíveis, que possam monitorar doses de radiação de maneira eficiente. As amostras foram estudadas previamente por espectroscopia no ultravioleta-visível (UV-Vis), demonstrando seu potencial para uso dosimétrico. Para este trabalho, o material foi irradiado com doses gama de 30 e 100 kGy e analisado por espectroscopia por ressonância magnética nuclear (RMN) e espectroscopia Raman. Observou-se que a coloração das amostras muda de azul para vermelho com o aumento da dose de radiação, sugerindo a formação de óxidos de cobre. A análise por RMN revelou mudanças nos tempos de relaxação longitudinal (T1) e transversal (T2), indicando interações entre a matriz do gel e o sulfato de cobre. A espectroscopia Raman confirmou a formação de novos picos, associados à presença de óxidos de cobre. Medidas de pH também foram realizadas para corroborar os resultados.

Palavras-chave: dosimetria, processo de irradiação gama, gel clínico, $CuSO_4 \cdot 5H_2O$.

1. INTRODUCTION

Radiation dosimetry is a fundamental discipline in various scientific, industrial, and medical applications, including the sterilization of medical products, food preservation, and radiotherapy [1,2]. In these applications, precise control of the radiation dose is essential to ensure both the effectiveness of the process and the safety of the materials and individuals involved. In this context, the development of accessible dosimetric materials capable of efficiently monitoring radiation doses becomes strategic. A promising approach for this purpose is the use of materials that undergo measurable observable changes, such as color alterations, in response to exposure to ionizing radiation [3,4].

The clinical gel composite with copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) stands out for its ability to gradually change its color from blue to shades of red or brown as the radiation dose increases. Preliminary studies have shown that this color transition is proportional to the absorbed radiation dose, suggesting great potential for applications in visual dosimetry of high doses, with an upper detection limit of 100 kGy [4]. The samples were reevaluated one month after irradiation and it was noted that the color changed over time, but without returning to the original color. This suggests its safe use in the first hours after irradiation. However, the chemical mechanisms responsible for these changes are not yet fully understood, limiting the optimization and practical application of this material in environments where dose control is essential.

A detailed understanding of the chemical mechanisms involved requires the use of analytical techniques that allow the exploration of molecular and structural interactions induced by ionizing radiation. In previous work, the samples were analyzed by ultraviolet-visible spectroscopy (UV-Vis), used to study the optical absorption of the material before and after radiation exposure [4]. This technique allowed correlating the intensity and shift of the absorption peaks with the observed color changes, providing a quantitative and qualitative analysis of the optical changes occurring in the irradiated composite. In the specific case of $CuSO_4 \cdot 5H_2O$, the absorption peak around 470 nm is related to the blue color, while shifts or changes in intensity in this spectrum may indicate chemical transformations, such as the partial reduction of copper (from Cu^{2+} to Cu^{+}) or the formation of copper oxides [4]. In another study, samples were evaluated using magnetic resonance imaging (MRI). Since copper sulfate is paramagnetic, differences in gray tones in MRI of samples with different proportions of this charge were tested. Irradiated and non-irradiated samples were analyzed and it was possible to demonstrate that the samples undergo changes due to the gamma irradiation process [5]. To assist in elucidating the mechanisms involved, two techniques were used in the present study: Raman spectroscopy and nuclear magnetic resonance (NMR) spectroscopy.

NMR was used to analyze samples of pure clinical gel, clinical gel with copper sulfate, and clinical gel with irradiated copper sulfate, focusing on the behavior of the water signal (4.7 δ). Relaxometry data were proposed to help understand changes in the interaction between the gel matrix and copper sulfate due to irradiation. Raman spectroscopy is widely used to investigate structural and chemical changes in irradiated materials [6]. This technique provides information on molecular vibrations, chemical bonds, and changes in the crystal lattice. In the case of $CuSO_4 \cdot 5H_2O$, Raman analysis can reveal possible changes in the coordination of copper ions or interaction with water molecules, which are fundamental to the characteristic blue color of the material.

The combined use of these techniques aimed at a comprehensive analysis of the chemical and structural phenomena involved, providing support for the development and practical application of the material. Furthermore, understanding the mechanisms involved may pave the way for the creation of new dosimetric materials based on similar principles but with improved performance in terms of sensitivity, precision, and stability.

2. MATERIALS AND METHODS

The clinical gel samples with added $CuSO_4 \cdot 5H_2O$ were obtained by manually mixing analytical grade $CuSO_4 \cdot 5H_2O$ (100 mg) in 100 ml of colorless clinical gel from the manufacturer Mercur®. The clinical gel is a hydrophilic polymer gel obtained by hydrating polyacrylic acid (PAA), which forms carboxyl polymers. The gel formulation employed in this study complises deionized water (dH_2O), polyacrylic acid, EDTA tetrasodium (EDTA 4Na), 1,3-dihydroxymethyl-5,5-dimethylhydantoin, triethylamine, and 1,2-propylene glycol. The quality of the gel was guaranteed by the manufacturer through a declaration of absence of impurities contained in the Chemical Product Safety Information Sheet, published by mercur.zendesk.com, in accordance with the Brazilian Standard - NBR 14725-4:2014 [7-9]. Samples of PPA gel and those with added copper sulfate were separated for irradiation in 10ml vials, as shown in Figure 1.

Figure 1: Samples of PPA gel/$CuSO_4 \cdot 5H_2O$ (bluish) and PPA gel (colorless) and separated in a vial for the irradiation process.

Source : authors' archive.

In Figure 1, two samples can be observed: a colorless one, which is the PPA gel, and a bluish one, which is the PPA gel/$CuSO_4 \cdot 5H_2O$. The presence of copper sulfate gives the sample its bluish color. Samples of PPA gel/$CuSO_4 \cdot 5H_2O$ were used for gamma irradiation with doses of 30 and 100 kGy at a constant dose rate. The irradiation process was conducted in the multipurpose irradiator of the Gamma Irradiation Laboratory (LIG) at the Center for the Development of Nuclear Technology (CDTN), which is equipped with a Cobalt-60 source. A month was waited before starting the characterizations to ensure that the radiation-induced changes had stabilized.

NMR analyses were conducted at the High-Resolution Magnetic Resonance Laboratory of the Federal University of Minas Gerais (UFMG) – LAREMAR using a Bruker Avance Neo 600 MHz spectrometer (model: Avance Neo 600 MHz) with a 4.7 T magnet, 5mm PROBE TXI probe, at a controlled temperature of 25 °C (SmartCooler BCU I system). Samples of PPA gel, PPA/$CuSO_4 \cdot 5H_2O$ (non-irradiated), and PPA gel/$CuSO_4 \cdot 5H_2O$ irradiated with 30 and 100 kGy were prepared with D_2O solvent. Relaxometry data were obtained considering the water signal, i.e., through the ^1H-NMR spectrum. The parameters obtained by relaxometry were the longitudinal (T1) and transverse (T2) relaxation times. These were used to correlate with the physicochemical characteristics of the samples, looking for changes in the gel matrix caused by the addition of copper sulfate. Subsequently, changes in the PPA gel/$CuSO_4 \cdot 5H_2O$ samples due to the irradiation process were studied.

Raman spectroscopy analyses were conducted in partnership with the Laboratory of Characterization and Processing of Nanomaterials (LCPNano) at UFMG, using a Witec Alpha300RA micro-Raman spectrometer: The equipment operates in spectral imaging capacity with a spatial resolution of about 1 micron, containing three laser lines (633 nm, 532 nm, and 457 nm), three diffraction gratings (600 lines/mm, 1800 lines/mm, and 2400 lines/mm), an EMCCD detector, and an atomic force microscopy (AFM) system that allows for topographic imaging. The excitation configuration is "up-right" (top illumination). For this work, the 457 nm laser line, i.e., blue laser with ten accumulations, grating G1: 600 g/mm, BLZ=500nm, central wavelength at 529.514 nm, and spectral center at 3000.0 rel. 1/cm, was used. A Zeiss EC Epiplan-Neofluar 100x / 0.9 objective was used. Non-irradiated PPA gel/$CuSO_4 \cdot 5H_2O$, $CuSO_4 \cdot 5H_2O$, PPA gel, and PPA gel/$CuSO_4 \cdot 5H_2O$ irradiated with 100 kGy were analyzed.

Complementary pH data of samples were conducted using a digital pH meter M/Micronal type B474.default style.

3. RESULTS AND DISCUSSIONS

For this work, the samples were irradiated with doses of 30 and 100 kGy, and in accordance with previously obtained results [4], a color change was observed in the samples. The non-irradiated samples had a bluish-green color due to the addition of copper sulfate to the clinical gel. However, with irradiation, the samples changed color, presenting a red hue. Since the gel is colorless, the color change is attributed to copper sulfate through interaction mechanisms with radiation, leading to the formation of copper oxide, which has a reddish color.

The proposed use of this material is for dosimetric purposes, seeking a linear relationship between the color change and the absorbed dose. Therefore, the objective was to make evident the transformation of copper sulfate into copper oxide, proposing a reaction that would explain the color change. For this, changes in the PPA gel matrix were analyzed through NMR using longitudinal (T1) and transverse (T2) relaxation data calculated based on the water signal (4.7 δ). Initially, non-irradiated PPA gel and PPA gel/$CuSO_4 \cdot 5H_2O$ samples were analyzed by NMR to study the changes introduced by copper sulfate in the matrix. Subsequently, PPA gel/$CuSO_4 \cdot 5H_2O$ samples irradiated with 30 and 100 kGy were analyzed.

Thus, the study of T1 and T2 relaxation data of the samples becomes relevant. This way, it was possible to obtain information about the effect of the presence of copper sulfate in the matrix and also the irradiation process of the composite. In Table 1, we can see the obtained results.

Table 1 : Relaxometry data of the samples analyzed by NMR

Relaxation times (4,688 ppm peak)	**T1 (s)**	**SD**	**T2 (s)**	**SD**
PPA gel non-irradiated	6.90	2.72	50.40	3.42
PPA gel/ $CuSO_4 \cdot 5H_2O$ non-irradiated	8040.42	7.03	16.14	1.27
PPA gel/ $CuSO_4 \cdot 5H_2O$ irradiated (30 kGy)	1001,22	1.12	12.78	3,03
PPA gel/ $CuSO_4 \cdot 5H_2O$ irradiated (100 kGy)	1468,14	1.34	11,52	4,34

With the data presented in Table 1, it was possible to make inferences that are described in detail in the following topics.

1.1 Effects of copper sulfate in PPA gel before irradiation

Copper sulfate is a paramagnetic agent due to the presence of the copper ion (Cu^{2+}), which has unpaired electrons [10]. This ion can affect the relaxation times of hydrogen protons in the clinical gel matrix as proposed:

- T1 (longitudinal relaxation) – The local magnetic field generated by Cu^{2+} ions provide fluctuations that increase the efficiency of spin-lattice interaction, accelerating the return to equilibrium configuration. This increases T1, as the spin-lattice interaction is favored compared to PPA gel.
- T2 (transverse relaxation) – The same paramagnetic effect intensifies spin-spin interactions, causing a greater loss of coherence between nuclear spins. This significantly reduces T2.

1.2 Effect of irradiation on PPA gel with copper sulfate

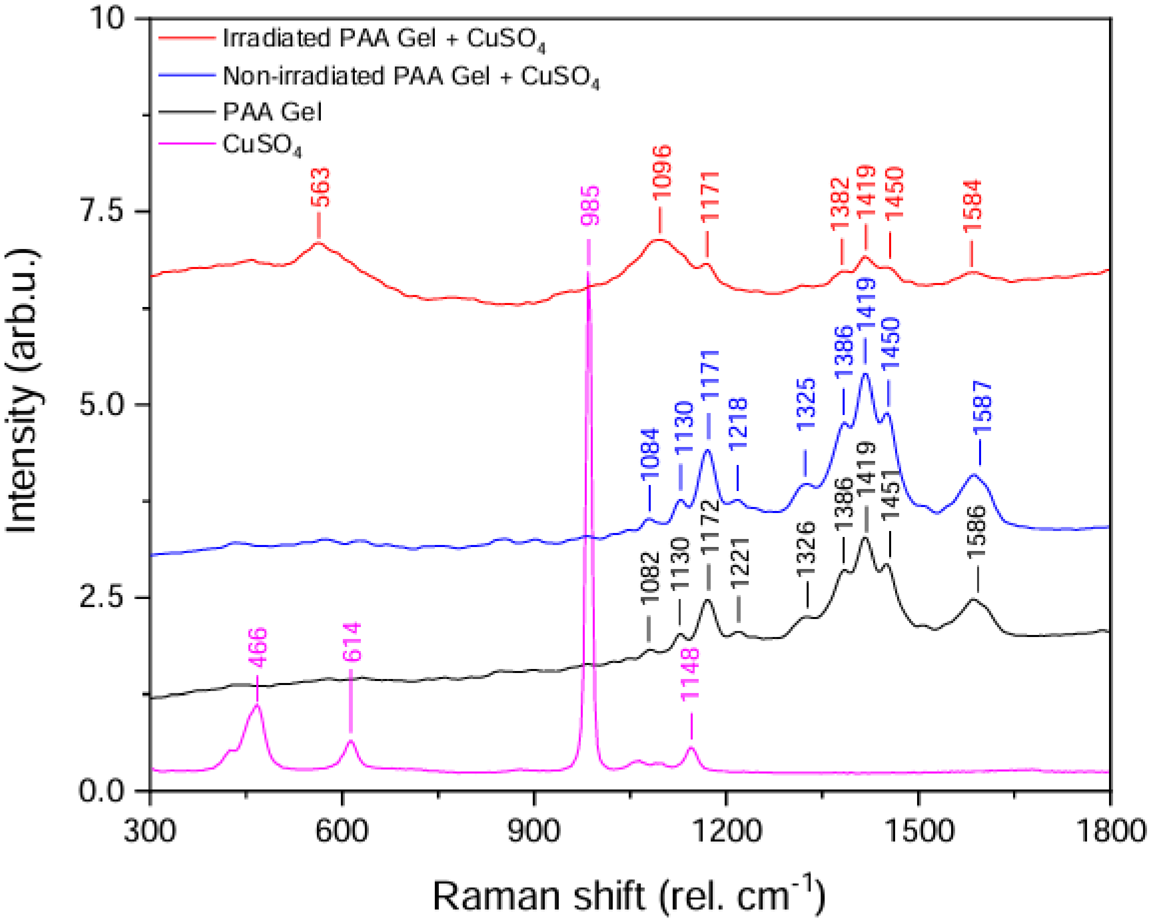
When adding copper sulfate to the matrix and subjecting it to high-dose irradiation, the relaxation dynamics change significantly due to the presence of Cu^{2+}. When the samples are irradiated, the following is observed:

- Decrease in T1 – Radiation can reduce the paramagnetic effects of Cu^{2+} by modifying the chemical environment or causing partial precipitation of copper-containing compounds. This generates more intense local magnetic fluctuations, accelerating longitudinal relaxation (T1).
- Small alteration in T2 – Copper ions continue to dominate spin-spin interactions. Despite the chemical changes induced by radiation, the impact on transverse relaxation (T2) is small, as copper ions still play a dominant role.

1.3 Results obtained by Raman spectroscopy and pH analysis of the samples

In Figure 2, the Raman spectra of $CuSO_4{\cdot}5H_2O$, PPA gel, non-irradiated PPA gel/$CuSO_4{\cdot}5H_2O$, and PPA gel/$CuSO_4{\cdot}5H_2O$ irradiated with 100 kGy can be seen. Analyzing the Figure 2, the Raman spectrum corresponding to the peak line reveals the characteristic bands of the $CuSO_4{\cdot}5H_2O$ in the range of 300rel.cm^{-1} to 1800cm^{-1}. The internal vibrations modes of sulfate ions are observed at 466rel.cm^{-1}, 614rel.cm^{-1}, 984rel.cm^{-1} and 1146rel.cm^{-1} corresponding to the ν_2 [SO_4^{2-}], ν_4 [SO_4^{2-}], ν_1 [SO_4^{2-}] and ν_3 [SO_4^{2-}], respectively [11,12].

Figure 2: Raman spectra of $CuSO_4 \cdot 5H_2O$, non-irradiated PPA gel, non-irradiated PPA gel/$CuSO_4 \cdot 5H_2O$, and PPA gel/$CuSO_4 \cdot 5H_2O$ irradiated with 100 kGy.



Source : authors' archive.

Notably, when comparing the pure PAA gel (black line, Figure 2), with the PAA gel mixed with $CuSO_4 \cdot 5H_2O$ (blue line, Figure 2), the vibrational modes corresponding to PAA remain unchanged. The minor variations observed are within the standard deviation.

However, when the $CuSO_4 \cdot 5H_2O$ is dissolved in the PAA gel, the vibrational modes of [SO_4^{2-}] associated with the coordination of Cu^{2+} disappear, indicating a change in the sulfate ion's interaction within the polymeric matrix.

The vibrational modes of PAA gel can be identified as follows: 1082 rel. cm^{-1} for corresponds to the $\varrho(CH_2)$ mode [7], 1130 rel. cm^{-1} is likely associated with the triply degenerate ν_3 mode of the SO_4^{2-} group with a shift [11,12], 1326 rel. cm^{-1} is attributed to the

ν_s (CCO) stretching mode [7], 1386 rel. cm^{-1} is assigned to a strong band from the Cu^{2+}EDTA complex, which shows a shift from the original 1400 rel. cm^{-1}[13], and 1451rel.cm^{-1} corresponds to the δ (CH_2) bending mode [7]. The vibrational mode of the -CH_2CO-OH group in EDTA typically exhibits a characteristic frequency around 1200 rel. cm^{-1}, which can be related to the band at 1221 rel. cm^{-1} [13]. The peak at 1171 rel. cm^{-1} can be attributed to vibrational modes associated with the symmetry of the C-CO-C bridge, with a significant contribution from in-plane bending vibration of OH, which is consistent with the presence of carboxylate (–COO–) or hydroxyl (–OH) groups in compounds such as polyacrylic acid or 1,3-dihydroxymethyl-5,5-dimethylhydantoin [2].

The peak at 1419 rel. cm^{-1} can be attributed to the carboxylate (COO^-) stretching vibration, which typically appears due to the deprotonation of carboxyl (COOH) groups. In this context, this peak suggests the presence of deprotonated carboxylate groups, likely originating from polyacrylic acid, which contains carboxyl functional groups that can undergo pH-dependent deprotonation. The Raman peak at 1586 cm^{-1} in the PAA gel may be attributed to the C=O stretching vibration of carbonyl groups, similar to what has been observed in flavonols, suggesting contributions from carboxylate (COO^-) groups in polyacrylic acid [14].

The Raman spectrum of the irradiated matrix (Figure 2, red line) exhibited broadened features in several regions, with few well-defined peaks. The peak at 563 rel. cm^{-1} may correspond to the copper chelation coordination with carboxylate groups, and it appears after irradiation [9,15]. A peak at 1096 rel. cm^{-1} can be identified as a combination of the 1082 rel. cm^{-1} (ϱ(CH_2) mode) and 1130 rel. cm^{-1} (ν_3 [SO_4^{2-}]) peaks. The peak at 1171rel. cm^{-1} can be associated with the C-CO-C bridge, with a significant contribution from in-plane bending vibration of OH. The peak at 1382 rel. cm^{-1} corresponds to a strong band from the Cu^{2+}-EDTA complex. The peak at 1419 rel. cm^{-1} can be attributed to the carboxylate (COO^-) stretching vibration, while the peak at 1450 rel. cm^{-1} is attributed to the δ (CH_2) bending mode. Finaly, the peak at 1586 cm^{-1} is the C=O stretching vibration of carbonyl groups [7,11, 15].

1.4 Proposed interpretation of the data obtained by NMR and Raman spectroscopy

A radiation-induced reaction consistent with the experimental data can be proposed considering the following factors.

- Radiolysis of water: The PPA gel matrix contains a large amount of water, and ionizing radiation induces radiolysis, generating highly reactive species such as hydroxyl radicals (OH·), hydrated electrons (e^{-aq}), and hydrogen ions (H^+).
- Reduction of Cu^{2+}: Hydrated electrons and hydroxyl radicals can reduce Cu^{2+} to Cu^+, promoting the formation of copper oxide.
- Decomposition of sulfate (SO_4^{2-}): Radiation can induce the degradation of the sulfate ion, releasing sulfur trioxide (SO_3), which can react with water to form sulfuric acid (H_2SO_4).

On the other hand, in relation to the composition of the gel itself, it is possible to consider PAA gel is a weak polyelectrolyte, and its conformation and mobility are strongly influenced by its degree of ionization. In an alkaline medium, a higher concentration of COO^- and OH^- groups is present, leading to competition for interactions with cations, such as Cu^{2+} or other metals [7-9]. When $CuSO_4 \cdot 5H_2O$ is added to the PAA gel matrix, it dissociates, releasing Cu^{2+} and SO_4^{2-} ions into the gel. Copper (Cu^{2+}) in the PAA gel can form coordination complexes through interactions with the carboxylate groups of polyacrylic acid (PAA). In aqueous solution, Cu^{2+} exhibits a strong affinity for oxygen-containing ligands, particularly carboxylate (COO^-) and hydroxyl (OH^-) groups. The sulfate ions might also interact via ionic interactions. The complexation process is influenced by factors such as pH, polymer ionization degree, solvent environment, and temperature [9]. In this configuration, the gamma irradiation induced method can be used to synthesize Cu_2O particles [16,17].

The proposed reaction involves the formation of copper oxide, resulting in a color change in the composite. This transformation is detailed in previous studies, where irradiated samples changed color from blue to yellow, orange, and red [4]. This result is consistent with the literature, as according to Pozharov et al. (2016), Cu_2O particles exhibit color variations depending on size and extension conditions, and can display shades ranging from yellow to dark red due to their semiconductor optical properties [18].

According to the proposed reaction, the additional formation of SO_3, an acidic oxide, is expected to lower the pH of the samples due to the subsequent formation of H_2SO_4. Therefore, pH measurements of the samples were carried out, and the results can be seen in Table 3.

Table 3 : pH values of the analyzed samples.

Samples	**PPA gel non-irradiated**	**PPA gel irradiated (100 kGy)**	**PPA gel/ $CuSO_4 \cdot 5H_2O$ non-irradiated**	**PPA gel/ $CuSO_4 \cdot 5H_2O$ irradiated (100 kGy)**
pH values	7.06	6.67	6.13	5.6

In Table 3, it can be observed that the clinical gel sample has a neutral pH. The addition of $CuSO_4 \cdot 5H_2O$ to the PPA gel slightly alters its pH. However, the pH of the PPA gel/$CuSO_4 \cdot 5H_2O$ sample changes more significantly after irradiation. The decrease in pH for the irradiated composite is consistent with the proposed radiation-induced formation of SO_3 in the sample.

The studied material can be compared with other dosimeters that also use color changes, through similar mechanisms, to monitor radiation doses. For example, gel-based dosimeters with silver nanoparticles (AgNPs), which change color from yellow to brown when exposed to radiation. The material prepared with porcine gelatin, isopropanol and

AgNPs can change color due to the surface plasmon resonance (SPR) phenomenon, monitored by UV-VIS molecular absorption spectrophotometry [19]. In comparison with the material proposed in this work, both materials have advantages in terms of sensitivity and ease of use. However, the PPA gel composite with $CuSO_4 \cdot 5H_2O$ stands out for its ability to monitor extremely high doses, up to 100 kGy, while silver AgNPs dosimeters are more suitable for lower doses. The choice of material depends on the specific needs of each application, with both being promising for visual dosimetry in different contexts.

4. CONCLUSIONS

The results of this study demonstrate that the PPA gel composite with copper sulfate is a promising material for visual dosimetry of high radiation doses. The color change observed in the irradiated samples can be used as a parameter for evaluating the absorbed dose, allowing easy visualization and monitoring of radiation doses. NMR and Raman analyses provided important information for understanding the chemical mechanisms involved, confirming the formation of copper oxides as a result of the interaction between radiation and the material. Additionally, pH measurements corroborated the proposed radiation-induced reaction. This work paves the way for the development of new dosimetric materials based on similar principles, with potential applications in various scientific, industrial, and medical fields.

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CONFLICT OF INTEREST

All authors declare that they have no conflicts of interest.

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