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GAMMA IRRADIATION-INDUCED MODIFICATIONS IN THE STRUCTURAL AND DIELECTRIC PROPERTIES OF GO/PVA/AgNW NANOCOMPOSITES

MODIFIKACIJA STRUKTURNIH IN DIELEKTRIČNIH LASTNOSTI NANOKOMPOZITOV GO/PVA/AgNW S POMOČJO OBSEVANJA Z GAMA ŽARKI

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Solution casting was used to create GO/PVA/AgNW nanocomposites, which were then exposed to gamma irradiation at dosages of (8, 25 and 50) kGy. XRD, SEM, XPS, and dielectric spectroscopy were used to examine the nanocomposites' structural, morphological, and dielectric characteristics. XRD analysis confirmed the successful synthesis of the nanocomposites and indicated that no significant oxidation occurred due to γ-irradiation. Interestingly, the sample exposed to 25 kGy radiation showed the lowest degree of crystallinity. With no discernible morphological changes across all irradiation doses, SEM images revealed a homogeneous dispersion of GO inside the polymer matrix and a random distribution of AgNWs throughout the composite. XPS results demonstrated that silver retained its metallic state, while gamma irradiation enhanced its surface contribution. Dielectric measurements revealed that the sample irradiated at 8 kGy exhibited the highest dielectric permittivity and electrical conductivity. This response is fundamentally linked to the increased freedom of charge carriers, a consequence of polymer chain scission processes and the simultaneous introduction of oxygenated functional moieties. These structural modifications facilitate easier charge transport pathways within the composite matrix.

Key words: gamma irradiation, nanocomposite, graphene oxide, silver nanowire

Avtorji v članku opisujejo raziskavo vpliva obsevanja z gama žarki na strukturne in dielektrične lastnosti nanokompozitov vrste GO/PVA/AgNW. Nanokompozite so pripravili z nalivanjem raztopine na podlago. Raztopina je bila predhodno ultrazvočno obdelana iz mešanice posameznih komponent; i.e.: grafenovega oksida (GO), polivinil alkohola (PVA) in srebrnih nanožičk (AgNW). Nato so nastali tanek film nanokompozita obsevali z različnimi dozami gama žarkov (8, 25 in 50) kGy. Za oceno vpliva obsevanja na strukturne in dielektrične lastnosti sintetiziranih kompozitov so uporabili rentgensko difrakcijsko spektroskopijo (XRD), vrstični elektronski mikroskop (SEM), rentgensko fotoelektronsko spektroskopijo (XPS) in dielektrično spektroskopijo (DS). XRD analize so potrdile uspešno sintezo nanokompozitov in potrdile, da ni prišlo do pomembne oksidacije zaradi obsevanja z gama žarki. Zanimiva ugotovitev avtorjev je bila, da je pri dozi obsevanja 25 kGy prišlo do najmanjše stopnje kristaliničnosti. Pri vseh odmerkih obsevanja so SEM posnetki pokazli, da ni prišlo do opaznih morfoloških sprememb ter homogeno disperzijo GO znotraj polimerne matrice z naključno porazdelvijo AgNW po celotnem kompozitu. Rezultati XPS analiz so pokazali, da je srebro ostalo v kovinskem stanju, medtem ko je gama sevanje povečalo njegov prispevek na površini. Dielektrične meritve so pokazale, da so vzorci obsevani z 8 kGy imeli najvišjo permitivnost in električno prevodnost. Ta odziv je v osnovi povezan s povečano svobodo nosilcev naboja, kar je posledica procesov cepitve polimernih verig in hkratnega uvajanja oksigeniranih funkcionalnih skupin. Te strukturne spremembe omogočajo lažje poti za prenos nabitih delcev znotraj kompozitne matrike.

Ključne besede: obsevanje z gama žarki, nanokompozit, grafenov oksid, srebrne nanožičke

1 INTRODUCTION

Over the past decade, GO/PVA-based hybrid systems have garnered significant attention within the realm of nanocomposite research, primarily owing to their remarkable synergy of chemical versatility and superior physicochemical performance metrics. This unique combination positions them at the forefront of advanced functional material development. The high density of oxygen-containing functional groups in GO, along with its excellent dispersibility in water, facilitates its incorporation into polymer matrices such as PVA, enabling the straightforward fabrication of stable and homogeneous composite films.[1] These nanocomposites have found diverse applications across several advanced technological fields, including humidity and gas sensors,[2,3] supercapacitor electrodes,[4] and radiation detection systems.[5] The tunability of their properties through parameters

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such as filler content[6] and the reduction degree of GO[7] allows for versatile design and functionalization tailored to specific applications.

Beyond tailoring their initial compositional architecture, the intrinsic structural configuration and multifunctional attributes of GO/PVA composites can be profoundly modulated through strategic post-fabrication interventions. Such treatments enable deliberate manipulation of microstructural features and interfacial phenomena, thereby tuning the overall material behavior.[8] Methods such as thermal annealing, electron beam exposure, and γ irradiation have been extensively studied for inducing physical and chemical changes within the polymer matrix or at the filler-matrix interface.[9–11] Another effective approach involves introducing metallic nanostructures into the GO/PVA matrix, which can lead to the formation of metal–semiconductor interfaces and significantly impact the composite's electrical, optical, and thermal behavior.[12] Among the various metallic additives, silver has garnered considerable interest due to its well-known antimicrobial activity, excellent electrical conductivity, and ability to enhance thermal and mechanical stability.[13] Cobos et al. demonstrated that GO/PVA composites functionalized with silver nanoparticles exhibit promising properties for wound healing applications.[14] Furthermore, these engineered nanocomposites exhibit substantially improved light-transmission capabilities alongside reinforced mechanical robustness, attributes that collectively render them highly promising contenders for integration into next-generation flexible optoelectronic platforms. For instance, the optical transmittance of PVA/GO-Ag nanocomposites crosslinked with glutaraldehyde has been reported to increase by up to 51 %.[15]

Another important direction in materials science is the use of γ irradiation for post-synthesis modification of polymers and nanocomposites.[16] Gamma rays, being high-energy electromagnetic radiation, can induce significant chemical transformations, including chain scission, cross-linking, and the reduction of oxygen functionalities, particularly in systems containing GO and PVA.[17,18] These changes can dramatically affect the optical, mechanical, and dielectric properties of the composites. Previous studies revealed that γ irradiation influences strain distribution in GO/AgNWs-based materials,[19] increases hardness at specific doses,[20] and enhances conductivity in optimized irradiation regimes.[21] A. A. Basfar et al. reported that the CNB–PVA polymer nanocomposite exhibited 100 % crosslinking when irradiated with a γ-ray dose of 50 kGy.[22] In the case of the PVA/PANI polymer compound, γ-irradiation was found to increase the optical band gap. However, for GO/PVA nanocomposites, the band gap decreased with increasing γ-radiation dose. This reduction is attributed to the formation of new energy levels within the forbidden band, which lowers the energy required for electronic transitions.[23] Similarly, γ-irradiated PVA–Ag systems demonstrated a significant band gap reduction of up to 4 eV.[24] Other studies further confirmed that the band gap of PVA–Ag nanocomposites decreases from 4.57 eV to 3.72 eV upon exposure to a 50 kGy γ-ray dose.[25]

In this investigation, emphasis is placed on both the fabrication and gamma irradiation-driven modulation of GO/PVA nanocomposites embedded with one-dimensional silver nanowires AgNWs, which serve to augment electron conduction pathways and enhance mechanical stability. These hybrid materials were synthesized employing a solution casting strategy, followed by exposure to gamma doses of (8, 25 and 50) kGy. A comprehensive suite of analytical techniques – including XRD, TEM, SEM, XPS, and dielectric spectroscopy – was employed to systematically elucidate the effects of irradiation on their structural, morphological, and electrical attributes. Through this study, we seek to unravel the interplay between gamma radiation dosage and the resultant physical properties of GO/PVA/AgNW composites, thereby furnishing critical insights pertinent to their prospective deployment in electronic, sensor, and biomedical device applications.

2 MATERIALS AND METHODS

2.1 Nanocomposite fabrication

GO was synthesized employing a refined version of the Hummers' protocol, wherein graphite underwent oxidative treatment with potassium permanganate in concentrated sulfuric acid under controlled low-temperature conditions. This was sequentially followed by gradual dilution, oxidative stabilization via hydrogen peroxide, and extensive acid washing to eliminate residual impurities. The final GO product was subsequently air-dried at ambient temperature.[19] AgNWs were fabricated through an adapted polyol synthesis route. In this method, ethylene glycol functioned dually as the solvent and reducing medium, while polyvinylpyrrolidone (PVP) served as a steric stabilizer to guide nanowire formation. A meticulous introduction of copper bromide and sodium chloride was critical in steering anisotropic growth. A mixture of silver nitrate and additional PVP solutions was incrementally introduced dropwise at 160 °C, promoting the elongated nanostructure formation. The resulting AgNWs were harvested via acetone-induced precipitation followed by centrifugation and ultimately re-dispersed in ethanol for storage and subsequent use.[19]

GO/PVA/AgNW nanocomposites were fabricated using a solution casting method. GO was exfoliated by sonication in water and mixed with a heated aqueous solution of PVA. AgNWs were added to the mixture under stirring and then sonicated further to ensure homogeneity. To create homogeneous thin films, the resultant mixture was poured into petri dishes and allowed to dry at room temperature. The composites contained 3 *w*/% GO and 1 *w*/% AgNWs relative to PVA. To examine radiation-induced changes in the films' structural and

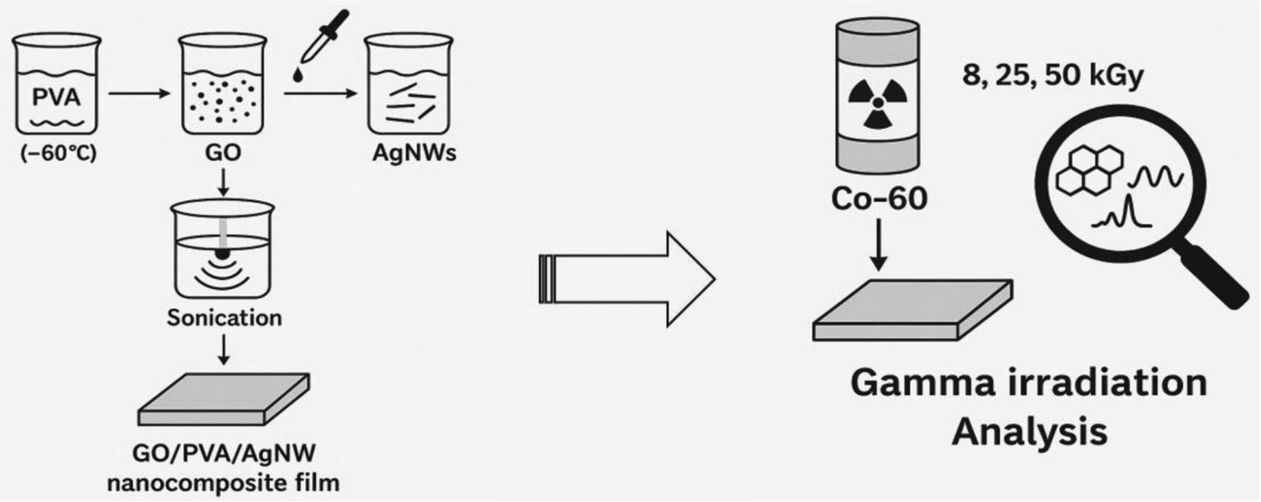

Figure 1: Conceptual schematic depicting the sequential fabrication process of GO/PVA/AgNW nanocomposites, followed by their controlled exposure to varying doses of gamma irradiation

physicochemical characteristics, they were subjected to gamma irradiation at different dosages (8, 25 and 50) kGy after drying (**Figure 1**).

2.2 Characterization

The structural characteristics of the fabricated nanocomposite samples were investigated employing a Rigaku MiniFlex 600 X-ray diffractometer, utilizing Cu Kα radiation with a wavelength of 0.15406 nm. Morphological features were characterized through scanning electron microscopy (SEM) on a FIB-SEM ZEISS Crossbeam 550 system, which was also equipped with an energy dispersive X-ray spectroscopy (EDS) detector for elemental analysis. Additional microstructural insights were obtained via transmission electron microscopy (TEM) using a JEM-1400 (JEOL, Japan) operating at an accelerating voltage range of 80–120 kV. Surface chemical composition analyses were conducted by X-ray photoelectron spectroscopy (XPS) on a Versa Probe 3 instrument outfitted with a monochromatic Al Kα source. Both survey and high-resolution spectra were collected for the principal elements (C 1s, O 1s, and Ag 3d), with charge referencing performed against the C 1s peak positioned at 284.7 eV. Prior to the analysis, selected specimens underwent argon ion (Ar^+) sputtering to effectively eliminate surface contaminants.

Gamma irradiation of the nanocomposite films was executed using a ^{60}Co source, delivering photons with an average energy of 1.25 MeV.

3 RESULTS AND DISCUSSION

3.1 XRD analysis

Diffraction patterns for non-irradiated and irradiated samples are shown in **Figure 2**. PVA is characterized by a large diffraction peak at $2\theta = 19.88°$ (JCPDS 36-1451). Furthermore, in accordance with JCPDS card no. 04-0783, distinct peaks at $2\theta = 38.49°$, 45.21°, 64.82°, and 81.78° correspond to the (111), (200), (220), and (311) crystal planes of AgNWs. Notably, the XRD patterns do not clearly show the typical GO peaks at $2\theta = 10.97°$ and 42° (given to the (001) and (100) planes, JCPDS card #75-2078), suggesting that GO was evenly distributed in the PVA matrix without generating. However, in pattern 2c, a weak diffraction peak at $2\theta = 10.97°$ can be seen, which may suggest localized aggregation of GO sheets in certain regions of the composite. Despite irradiation under atmospheric conditions, no diffraction peaks corresponding to silver oxide (AgO) are detected, indicating that significant oxidation of Ag did not occur. Additionally, the sample exposed to 25 kGy radiation shows the largest whole width at half maximum in its XRD pattern, indicating that γ irradiation reduced crystallinity.

3.2 Microscopy

SEM images of the GO/PVA/AgNWs nanocomposites are shown in **Figure 3**. AgNWs are not oriented in a

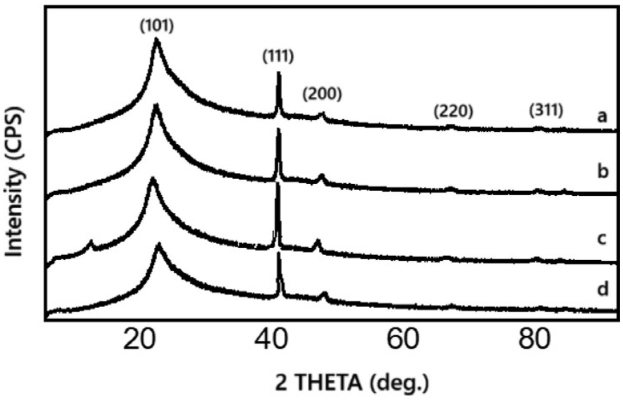

Figure 2: XRD diffraction patterns of GO/PVA/AgNWs nanocomposites: a) non-irradiated sample, b) irradiated at 8 kGy, c) irradiated at 25 kGy, d) irradiated at 50 kGy

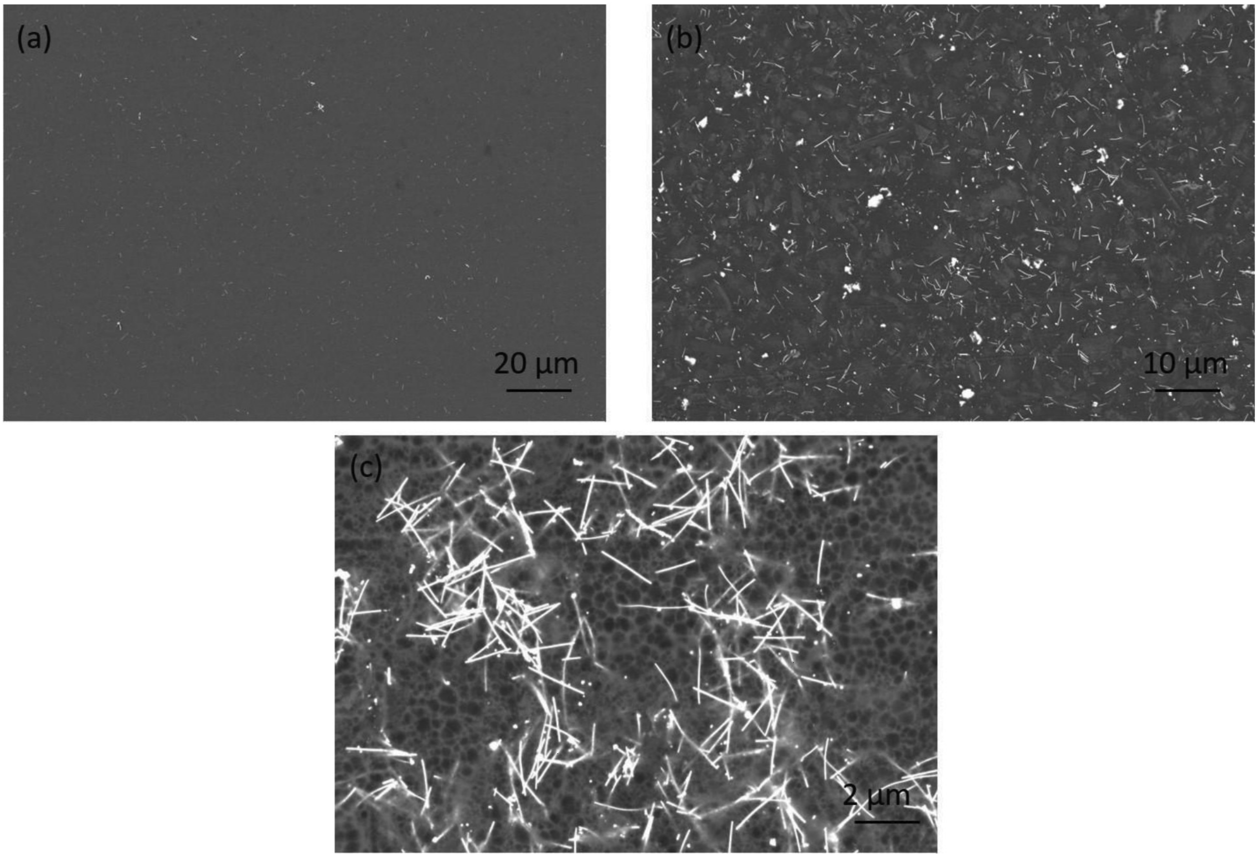

Figure 3: SEM images of GO/PVA/AgNW nanocomposites: a) upper surface, b) bottom surface, c) bottom surface enlarged

single orientation inside the polymer matrix, consistent with previous observations. The top and bottom surfaces of the composite film are shown in **Figures 3a** and **3b**, respectively. Comparative analysis of these images suggests that during film formation, a portion of the filler material – due to gravitational effects – accumulated at the lower layers of the composite. In addition, larger particles that did not form into wire-like structures during synthesis were also observed to settle at the bottom surface. **Figure 3c** shows a higher-magnification image of the bottom surface, revealing that some nanowires are in contact with each other within the matrix. AgNWs positioned perpendicular to the imaging plane are responsible for the bright spots shown in the picture. Notably, GO sheets are not visibly detected in the SEM images, likely due to their nanoscale dispersion within the PVA matrix. Additionally, the composite surface shows porosity, which might be caused by interactions of the electron beam with the polymer during SEM imaging. Furthermore, the SEM pictures of the irradiated samples show no discernible morphological alterations, indicating that γ radiation did not modify the nanocomposites' overall morphology.

3.3 XPS analysis

High-resolution XPS analyses were performed to investigate the chemical state of silver within the

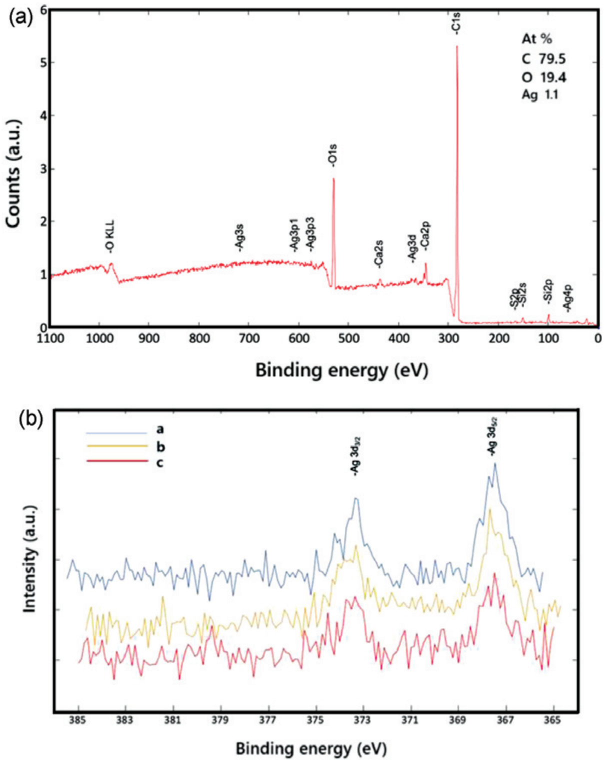

Figure 4: High-resolution XPS spectra depicting the Ag 3d signals in GO/PVA/AgNW nanocomposites irradiated at different gamma doses: a) 50 kGy, b) 8 kGy, c) 0 kGy (unirradiated reference)

GO/PVA/AgNW nanocomposite films. These measurements were carried out on the samples subjected to different gamma irradiation doses (0, 8, and 50) kGy, illustrated with spectra a, b, c in **Figure 4**. The chemical composition of the nanocomposite was determined to include approximately C = 79 $\varphi/\%$, O = 19 $\varphi/\%$, and Ag = 1 $\varphi/\%$.

The Ag 3d spectrum revealed two distinct peaks attributed to spin–orbit splitting: Ag $3d_{5/2}$ and Ag $3d_{3/2}$, located at binding energies of ≈368 eV and ≈374 eV, respectively. These values align well with the reference data for metallic silver (Ag^0).[22] The consistent spin–orbit splitting of around 6.0 eV between the two peaks confirms the presence of unoxidized, elemental silver in the composite. The symmetric and narrow shapes of the Ag 3d peaks further indicate that silver exists primarily in its metallic state. Moreover, the area under the Ag 3d peaks increased with rising the γ irradiation dose, suggesting that γ radiation may enhance silver exposure and/or promote its reduction on the composite's surface. This effect was most pronounced in the sample irradiated at 50 kGy (**Figure 6a**), where the intensity of the Ag $3d_{5/2}$ peak was significantly higher compared to the non-irradiated sample.

3.4 Dielectric spectroscopy

Figure 5 illustrates the logarithmic variation of dielectric permittivity with frequency for GO/PVA/AgNW nanocomposites exposed to different gamma irradiation doses (8, 25 and 50) kGy over a temperature span of 293–383 K. Subfigures 5A, 5B, 5C, and 5D correspond to the frequency-dependent dielectric behavior of the non-irradiated, 8 kGy-, 25 kGy-, and 50 kGy-irradiated samples, respectively. The plots clearly reveal that dielectric permittivity rises as temperature increases. Within the examined frequency interval of approximately 1.2×10^2 to 10^6 Hz, the real component of dielectric permittivity (ε') displays a consistent decline with growing frequency. This characteristic frequency dependence underscores the relaxation dynamics inherent to these nanocomposites, reflecting contributions from both dipolar and interfacial (Maxwell–Wagner) polarization processes. At lower frequencies, elevated dielectric permittivity is mainly attributed to efficient dipole orientation, facilitated by the slowly varying external field that provides ample time for dipolar alignment. Conversely, as frequency escalates, dipoles struggle to reorient rapidly enough, resulting in a progressive reduction in ε'. At sufficiently high frequencies, ε' becomes largely insensitive to further frequency changes, since the swift oscillations of the electric field surpass the dipolar relaxation capabilities, thereby diminishing their overall impact on the dielectric behavior. Furthermore, the increase in dielectric permittivity with rising temperature is associated with the enhanced segmental mobility of the polymer chains, which facilitates the orientation of dipoles and increases overall polarization.[23,24]

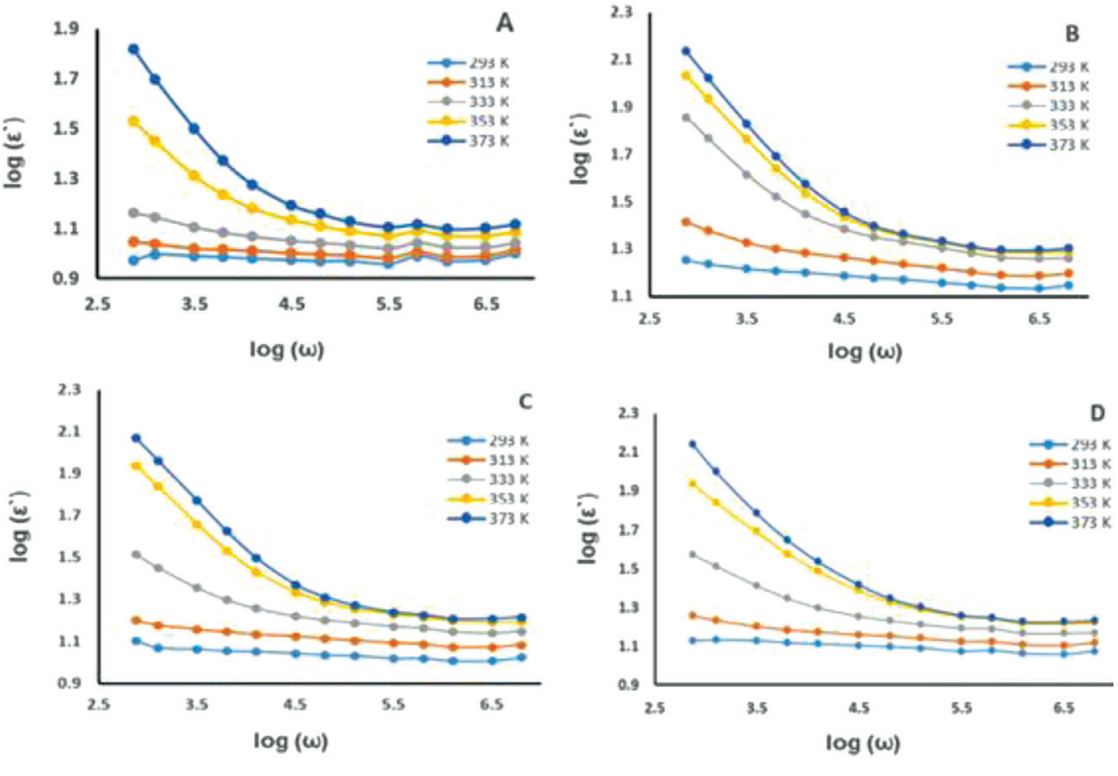

Figure 5: Logarithmic plots of dielectric permittivity (ε') as a function of frequency for GO/PVA/AgNW nanocomposites evaluated across a temperature range of 293–373 K. Panels show (A) the non-irradiated specimen and the specimens irradiated at (B) 8 kGy, (C) 25 kGy, and (D) 50 kGy.

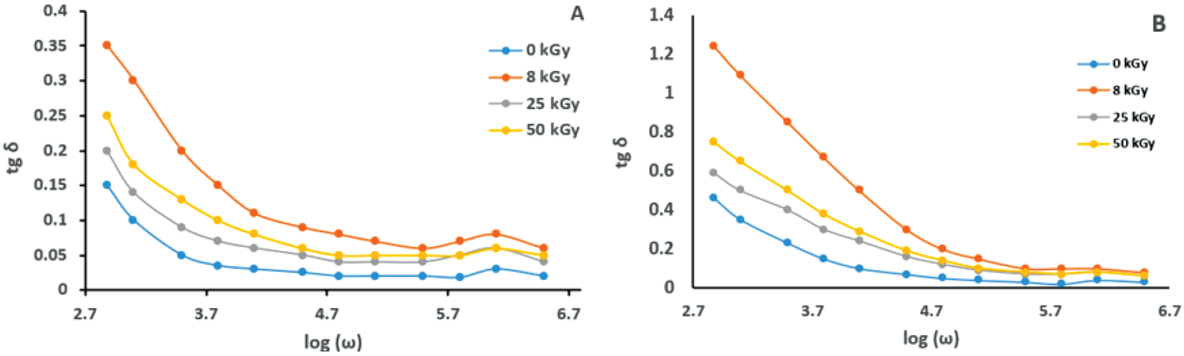

Figure 6: Variation in the dielectric loss tangent (tan δ) with frequency for GO/PVA/AgNW nanocomposites exposed to different gamma irradiation doses, recorded at (A) 313 K and (B) 333 K

The impact of gamma irradiation on the dielectric behavior of the nanocomposites is clearly reflected in the experimental data. Notably, the specimen exposed to 8 kGy (**Figure 5B**) demonstrates the highest dielectric permittivity among all the samples examined. At ambient temperature, for instance, the dielectric permittivity of the unirradiated composite registers at 9.33, whereas irradiation at 8 kGy elevates this value markedly to 17.78. This pronounced enhancement is primarily linked to the generation of radical species induced by gamma exposure, which fosters the incorporation of oxygen-rich functional groups and promotes partial cleavage of polymer chains. These structural transformations collectively facilitate increased dipolar mobility and augment the overall polarity of the material, culminating in a higher dielectric permittivity. In contrast, samples subjected to higher irradiation doses of 25 kGy and 50 kGy (**Figures 5C** and **5D**) exhibit a noticeable decline in dielectric permittivity. This reduction is predominantly ascribed to the onset of radiation-induced crosslinking processes within the polymer matrix, which constrain the rotational freedom of dipoles and hinder their effective alignment under an external electric field. Furthermore, it is well recognized that gamma irradiation can partially reduce graphene oxide (GO),[25] diminishing the concentration of oxygenated functional moieties on its surface. This decrease in polar functional groups directly translates into a lowered composite polarity, thereby contributing to the observed drop in dielectric permittivity.

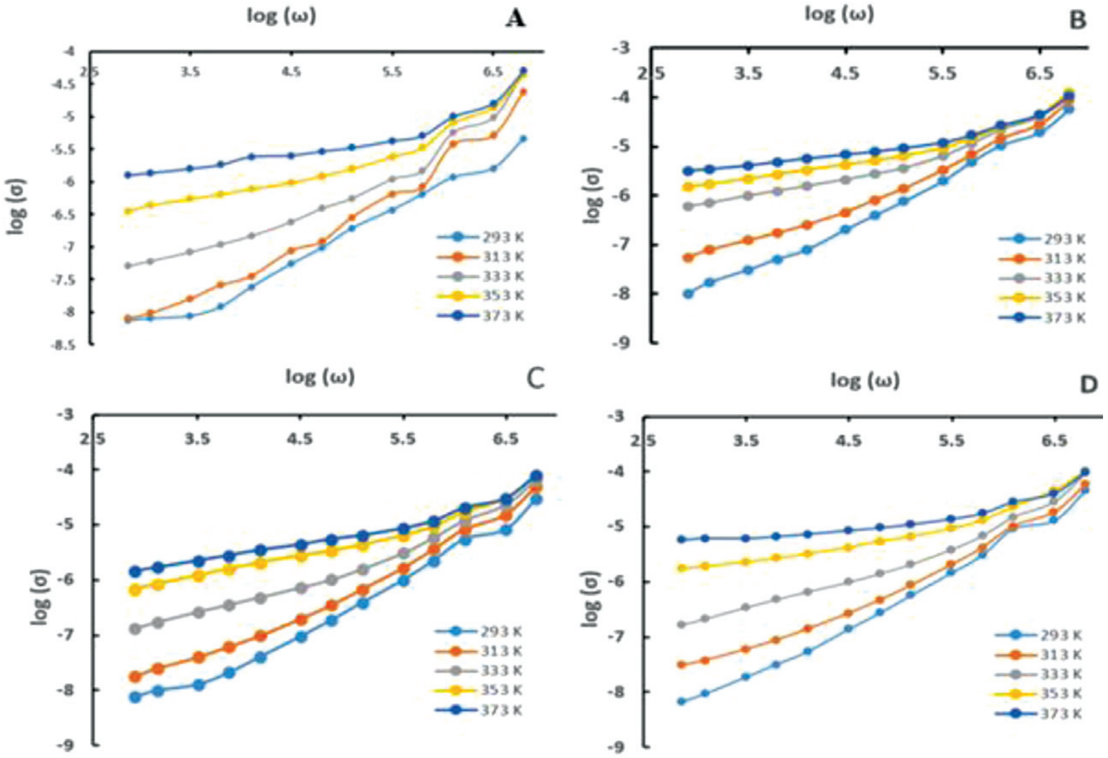

Figure 7: Electrical conductivity (σ) logarithmic frequency dependence for GO/PVA/AgNW nanocomposites subjected to varying dosages of gamma irradiation: (A) samples not exposed to radiation, (B) exposed to 8 kGy, (C) exposed to 25 kGy, (D) exposed to 50 kGy

Figure 6 depicts how the dielectric loss tangent (tan δ) varies with frequency at two representative temperatures, 313 K and 333 K, for the GO/PVA/AgNW nanocomposites. The plots clearly indicate that dielectric losses rise with increasing temperature. This trend is primarily attributed to the greater segmental mobility of the polymer chains at elevated temperatures, which enhances the ability of dipolar entities to realign in response to the alternating electric field. As a result, thermal activation facilitates more pronounced dipolar rotations, thereby amplifying dielectric losses. The highest tan δ values were observed in samples subjected to 8 kGy of gamma irradiation, a phenomenon that can be linked to the formation of additional polar functional groups and a higher density of irradiation-induced structural defects. These defects likely act as localized sites for energy dissipation, contributing to the elevated dielectric loss. In contrast, at higher irradiation doses, tan δ exhibits a noticeable decline. This reduction is likely due to the onset of radiation-induced crosslinking along with partial scission of polymer chains, structural changes that collectively impede dipolar mobility and reorientation, ultimately leading to diminished dielectric losses.

Figure 7 shows the frequency-dependent conductivity (σ) behavior of GO/PVA/AgNW nanocomposites at various gamma irradiation doses. It is clear that electrical conductivity rises as temperature rises, a feature shared by nanocomposites based on polymers. This temperature dependence arises from enhanced thermal energy that facilitates charge carrier mobility within the polymer matrix and increases the number of available hopping or transit sites.[26] The nanocomposite exposed to 8 kGy radiation showed the greatest conductivity among the samples under study. This enhancement is attributed to gamma irradiation-induced bond scission, which generates free electrons and ions, thereby increasing the number of mobile charge carriers. Additionally, gamma irradiation introduces defects into the system, which act as localized trap sites. These traps enable charge transport via thermally activated hopping mechanisms. However, at higher doses (as seen in **Figures 7C** and **7D**), conductivity decreases. This decrease results from the polymer matrix's excessive crosslinking, which prevents charge carriers from moving freely. Moreover, the increased radiation dose also leads to a higher concentration of defects, which, beyond a certain threshold, act predominantly as deep traps, capturing charge carriers and limiting their mobility.[27]

4 CONCLUSIONS

Solution casting was used to effectively create GO/PVA/AgNW nanocomposites, which were then subjected to gamma irradiation at dosages of (8, 25 and 50) kGy. Comprehensive characterization was performed using XRD, TEM, SEM, XPS, and dielectric spectroscopy. XRD analysis confirmed the effective formation of the nanocomposites and indicated that gamma irradiation did not induce significant oxidation. Moreover, analysis of FWHM revealed the lowest degree of crystallinity in the sample irradiated at 25 kGy. Within the PVA matrix, SEM images revealed a uniform distribution of GO and a random, chaotic distribution of AgNWs. No substantial morphological changes were observed with increasing irradiation dose. XPS results indicated that silver nanoparticles retained their metallic state, while gamma irradiation enhanced their surface exposure. Dielectric measurements revealed a marked increase in dielectric permittivity in the sample irradiated at 8 kGy. The creation of oxygen-containing functional groups, which raise the system's polarity, as well as the improved mobility and rotational flexibility of dipoles caused by bond breakage are responsible for this improvement. The same sample also exhibited the highest electrical conductivity, which is ascribed to improved charge carrier mobility facilitated by the breaking of chemical bonds. However, at higher doses, an increase in structural defects and trap density led to a reduction in conductivity due to hindered charge transport.

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Competing Interests

The authors declare that they have no competing interests.

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