

Effect of Gamma Irradiation on Structural and Morphological Properties of MWCNTs–Ag Nanocomposites Synthesized Using *Melia azedarach L.* Extract

Wrood K. Abood

Department of Physics, College of Education, Al-Iraqia University, Baghdad, IRAQ
Email: wrood.k.abood@aliraqia.edu.iq

In this paper, we studied the influence of 60 kGy gamma-ray irradiation on greenly prepared MWCNT–Ag nanocomposite using *Melia azedarach* leaf extract. The structural characterization of the studied materials (XRD, SEM-EDX TEM, FTIR and EDX) found that the crystallinity is increased with the improvement in AgNP dispersion as-well-as selective surface modifications following irradiation. The whole structure was also composed of the MWCNT skeleton, suggesting good stability of the composites. The results (presented here) constitute a very significant refinement for the post-synthesis processing of nanocomposites, in pharmaceutical and catalytic area, by gamma irradiation.

Keywords: Gamma irradiation; Nanocomposites; Green synthesis; MWCNTs

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1. Introduction

Multi-walled carbon nanotubes (MWCNTs) are well-known for their excellent electrical conductivity, mechanical strength and thermal stability, which have rendered them promising materials for electronics, catalysis and biomedical engineering [1,2]. MWCNTs have been used in hybridized with silver nanoparticles (AgNPs) they form an improved hybrid nano. These CNT AgNPs hybrids demonstrate not only enhanced capacity charge transport and greater catalytic activity but also the antimicrobial prop [3]. Most of the existing synthetic methods are combined with toxic reducing agents and harsh environment, leading to environmental pollution and potential safety risk.

In comparison, green synthesis of nanoparticles through plant extract has opened a new era of biosynthesis with environment-friendliness and One such promising route to capitalize this natural material is by employing plant extracts [4]. *Melia azedarach L.* is a phytochemical rich plant in which flavonoids, terpenoids, and alkaloids found was reducing $Ag(I)$ ions and are also responsible in acts as phyto-reducer for stabilizing nanocomposites [5,6]. Gamma irradiation is a classical physical process applied for nanomaterials modification. It triggers changes in crystallography, the creation of atomic defects and surface functional groups, and thus can affect physicochemical properties [7,8]. Gamma irradiation has been reported to cause considerable change in the FTIR spectra and structures of carbon-based nanomaterials [9, 10]. Nevertheless, the simultaneous influence of green synthesis and gamma

irradiation for hybrid nanocomposites is poorly understood [11]. Accordingly, in this paper we report investigations on the structural and chemical changes generated as a result of gamma irradiation of MWCNT–Ag nanocomposites prepared from *Melia azedarach* extract. The results should allow a better understanding of the influence of irradiation to improve lubricant performance of nanocomposites.

2. Materials and Methods

A bentonite-MWCNT-Ag nanocomposite was synthesized by a dispersion of 15 g of bentonite and 0.5 g of MWCNTs in deionized water with sonication. Silver nitrate (2 g) was added and subsequently reduced using 50 ml of *Melia azedarach* leaf extract. The mixture was stirred at 60 °C for 2 h, then centrifuged, washed and dried. Precipitated composite was treated by Co-60 gamma ray 60 kGy (30 Gy/h), in closed containers have been done at University of Baghdad, to keep sample stability. The 60 kGy dose was also chosen on the basis of previous work in which good structural modification of carbon-based nanomaterials has been shown to occur at this level without damaging the physical integrity. This dose is found to be adequate for achieving energy deposition substantial enough to create both crystallographic and surface modifications but not compromising the integrity of the composite's structure. Dose-response evaluate the potential dose relationship, a wider range of IC group doses is planned for further study.

3. Results and Discussion

Energy Dispersive X-ray Spectroscopy (EDX) also showed the presence of carbon, oxygen, silver, and silicon as main components of the MWCNT–Ag nanocomposite material. The strong Ag peak at 3.000 keV is indicative of an effective decoration of the nanoparticles, while signal from aluminum, magnesium and silicon are to be ascribed to the bentonite matrix. Some small peaks from calcium, barium and gold were also detected (the latter likely from that put on the SEM). The corresponding spectrum (Figure 1) contains well-separated peaks that confirm the structural integrity and compositional homogeneity of this nanocomposite Film. Bentonite was used as a supporting and dispersing reagent. Because of its sheet-like silicate structure, it provides multiple sinks for high-level anchoring of MWCNTs and AgNPs, while the indication of presence in the EDX spectrum (Si, Al and Mg) asserts that it was incorporated. Although it does not directly contribute to the reduction, it improves the mechanical stability and radiation tolerance of composite.

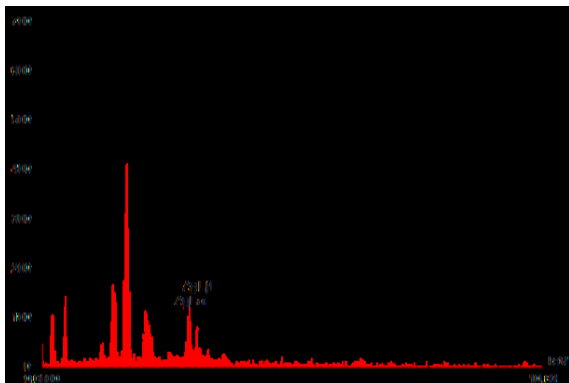


Fig. (1) EDX spectrum of MWCNTs-Ag-*Melia azedarach L.* nanocomposite

The surface morphology of the resulting MWCNT–Ag nanocomposite was revealed by Scanning Electron Microscopy (SEM). Figure 2 shows quite dense entangled network of multi-walled carbon nanotubes decorated with silver particles as observed from the micrograph. The surface reveals agglomerates irregularities/filamentous textures which confirms the nanoparticles anchoring as well as some clusters forming throughout the composites matrix. The observed roughness and porosity give an indication of increased available surface area which could improve catalytic and antimicrobial activity of the material. The difference between the carbonaceous matrix and metallic nanoparticles is distinct, indicating a heterogeneity of the nanocomposite. The morphology confirms the success of the green synthesis route and its suitability for further functionalization through gamma

irradiation. Approach and support the material's suitability for further functionalization via gamma irradiation. Figure (3) shows the TEM image of the MWCNT–Ag nanocomposite synthesized using *Melia azedarach L.* extract prior to gamma

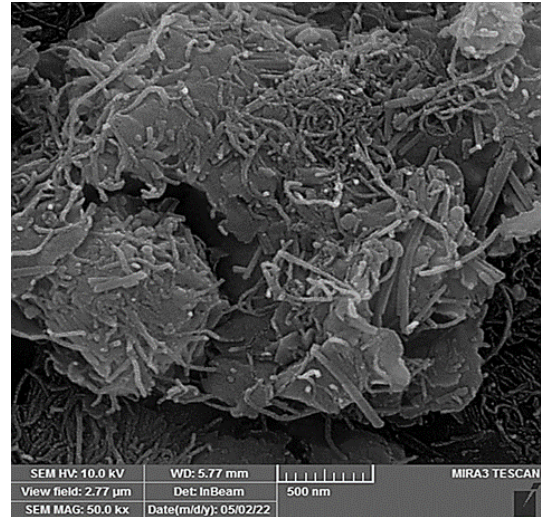


Fig. (2) SEM image of the synthesized MWCNTs-Ag-*Melia azedarach L.* nanocomposite

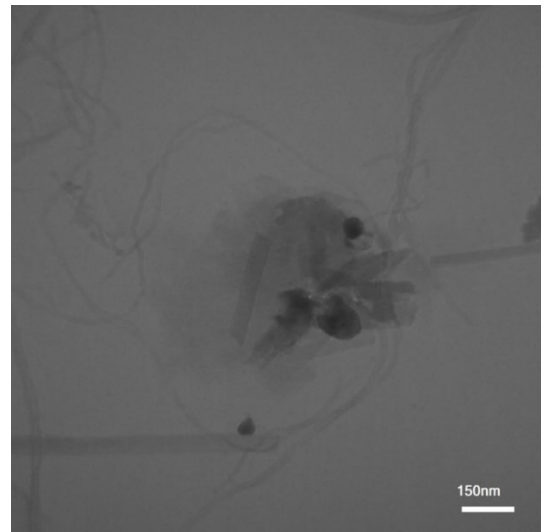


Fig. (3) TEM image of the synthesized MWCNTs-Ag-*Melia azedarach L.* nanocomposite

Figure (4a) shows the XRD pattern of the MWCNT–Ag nanocomposite before gamma irradiation, revealing sharp peaks indicative of a crystalline structure. The most intense peak at $2\theta \approx 26.74^\circ$ attributed to (002) plane of graphitic carbon, verifying the integrity of nanotube is retained. Peaks at $2\theta = \sim 32.31^\circ, 38.25^\circ, 44.37^\circ$ and 64.57° correspond to (111), (200) and (220) planes of the FCC structure of silver in AgNPs-loaded VAM spine suggesting successful loading of AgNPs [45]. The observed peak broadening indicates nanosized

crystallites and some degree of lattice imperfection. The XRD of the MWCNT–Ag nanocomposite after gamma irradiation at 60 kGy is shown in Fig. (4b). The major diffraction peaks on MWCNTs and FCC silver can be still observed, indicating the maintenance of a structure.

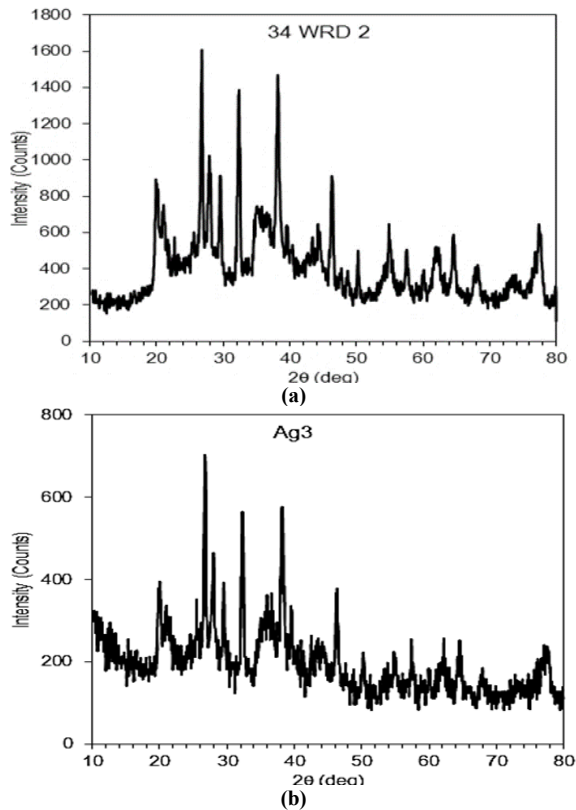


Fig. (4) XRD patterns of MWCNT–Ag nanocomposite synthesized using *Melia azedarach L.* extract (a) before and (b) after gamma irradiation at 60 kGy

To quantitatively assess the effect of gamma irradiation on crystallinity, the crystallite size of silver nanoparticles was calculated using the Scherrer equation

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (1)$$

where D is the crystallite size, $K=0.9$ is the shape factor, $\lambda=0.15406$ nm is the X-ray wavelength, β is the full-width at half maximum (FWHM) of the diffraction peak, and θ is the Bragg angle

Based on the (111) peak at $2\theta \approx 38.25^\circ$, the FWHM before irradiation was approximately 0.42° , and after irradiation it decreased to 0.31° . The calculated crystallite sizes were

$$D = \frac{0.9 \times 0.15406}{0.00733 \times \cos(19.125^\circ)} \approx 19.2 \text{ nm}$$

before irradiation, and

$$D = \frac{0.9 \times 0.15406}{0.00541 \times \cos(19.125^\circ)} \approx 26.0 \text{ nm}$$

after irradiation.

This increase in crystallite size confirms grain growth and enhanced crystallinity due to gamma-

induced atomic rearrangement and possible annealing effects.

The FTIR spectrum before irradiation (Fig. 5a) displayed characteristic bands for O–H, C=O, C=C, N–H, C–N, and Ag–O groups, confirming the presence of phytochemical residues and successful nanoparticle formation. Exposure to 60 kGy (Fig. 5b), evident spectral changes were observed such as the decreased and shifted from red to blue of O–H band, and the enhanced C=O peak intensity, suggesting surface oxidation as well as depressed hydrogen bonding. It can be seen that the Ag–O peak at 918 cm^{-1} did not change, signifying the preservation of the nanoparticle. These changes convey tailored surface properties but not the structure of composite

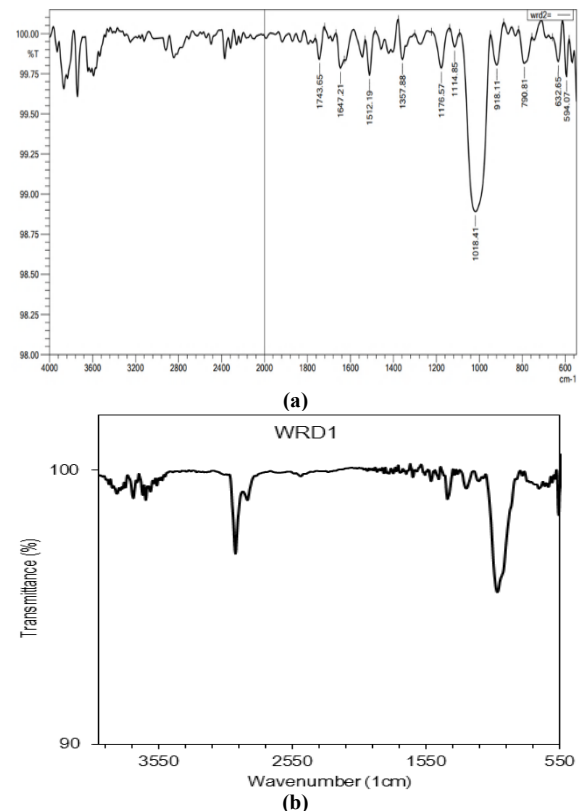


Fig. (5) FTIR as analyzed for MWCNT–Ag nanocomposite using *Melia azedarach* extract (a) and (b) after gamma irradiation

For an easier analysis of the modifications on the surface, the raw O–H stretching band (3420 cm^{-1}) was additionally deconvoluted both before and after irradiation. Thus FWHM decreased from 142 cm^{-1} to 108 cm^{-1} indicating decrease of hydrogen bonding and partial clearing of the phytochemical moieties. The C=O band (1630 cm^{-1}) was also stronger in intensity and red-shifted, indicating the surface oxidation. These spectral changes are suggestive of radiolysis-induced cleavage of the organics and preferential reactivity at the surface

functional groups [14]. In most cases the major interaction of γ -irradiation with nanocomposites is dominated by Compton scattering which produces secondary high-energy electrons that can lead to bond scission, targeted activation of defects and local heating. Such actions may cause recrystallization of metal NPs and desorption of organic capping. The increase in AgNP crystallite size and changes in FTIR spectra observed in this study is considered as being due to radiolytic scission of phytochemicals and defect-induced structural rearrangement.

4. Conclusions

Significant enhancement in crystallinity and nanoparticle dispersion as well as surface chemistry was found before 60 kGy gamma-irradiation versus after it. These enhancements should be helpful to further enhance catalytic performance and antimicrobial activity of this nanocomposite, just like those with AgNP-decoration carbon materials reported in literatures. The findings validate the feasibility of gamma irradiation as a post-synthesis parameter to tune nanocomposites for biomedical and environmental applications.

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