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# Study of Gamma Irradiation on FTIR and XRD of MWCNTs-Clay-$TiO_2$ Nanocomposite

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**Abstract**

Structural and chemical analysis of gamma-irradiated green-synthesized $TiO_2$–MWCNTs–Bentonite nanocomposite obtained by Melia azedarach leaf extract have been studied. Dose dependent changes in bonding configuration, crystallinity and functional groups were studied along with the impact of doses by gamma (25, 50, 75 and 100 kGy) in FTIR and XRD analyses. XRD data results showed that moderate irradiation doses (25–50 kGy) enhanced the formation of the $TiO_2$ phase and interfacial adhesion among $TiO_2$, MWCNTs, and clay matrices. These modifications produced an increase in lattice perfection and surface hydroxylation, which were essential for photocatalysis and adsorption. Higher doses (75–100 kGy), on the contrary, resulted in an incomplete amorphization and defect formation, with a significant bond scission comprising of both bond breaking and oxidation. The dual role of gamma irradiation, in increasing structure at mild doses and decreasing at higher levels indicates its application as a tunable tool for nanocomposite design. Technically, the tunable surface chemistry and crystallinity of irradiation as applied could serve as a promising way to adjust photocatalytic activity of the hybrid nanomaterials for environmental purification and photochemistry-responsive practices.

***Keywords:*** Gamma irradiation; Nanocomposite; MWCNTs; Titanium dioxide

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## 1. Introduction

The rapid development of nanotechnology has facilitated the preparation and utilization of multifunctional nanocomposites with improved physicochemical properties and catalytic activities applied in environmental and biomedical fields [1,2]. Natural clay minerals and of all the random hybrid systems, one system from titanium dioxide ($TiO_2$) based with multi-walled carbon nanotubes (MWCNTS), have received tremendous interests owing to their unique synergetic structural characteristics together high surface activity and photocatalytic efficiency [3-4]. The introduction of such repeat units could improve charge separation, adsorption capacity and mechanical properties, that's to say evolving the potential applications including water cleaning, pollutant degradation as well as antibacterial fields. Indeed, some recent works have demonstrated that the structure and the physical properties of nanocomposites may be affected by external physical treatments (e.g., ionizing radiation) [5]. Strong and green process of high dose gamma irradiation is known to be effective in morphology, crystal structure, surface chemistry modification for nanomaterial's [6,7]. It is known that chemical bonding and bond formation in a solid lattice can be broken, link the two bonds to form defects and atomic rearrangement between positions. These effects can again alter crystallite size, lattice strain and surface functional groups leading to material generation with optical, catalytic properties [8]. Use of green synthesis combined with radiation modification is also under development in environmentally-benign alternative to conventional chemical processes. Various plant extracts have been utilized as nontoxic capping and reducing agents for example flavonoids, terpenoids phenolics compounds to manage the synthesis of metal oxide NPs without involving hazardous tools [9,10]. In the reported methods, derived products of M. azedarach has been derivatized that exhibit template like strong bio-reduction and stabilization effects on $TiO_2$ NPs [11], thus green and nontoxic for biological applications [11,12]. This green strategy when combined to gamma irradiation, can produce new physicochemistry interactions between the organic bioactive species vs. inorganic phases and improve catalytic-sorptive properties. Notwithstanding this progress, an important research need endures. Previous studies were mainly either on radiation-induced effects in inorganic or polymeric nanocomposite or on green synthesis without irradiation [13,14]. Some studies have focused on the individual components of $TiO_2$–MWCNT–clay nanocomposites and their long-term environmental remediation capacity. The strong photocatalytic activity and chemical resistance properties of titanium dioxide ($TiO_2$) is well known, having been extensively applied in the degradation process of pollutants under UV exposure as shown by Baltac et al. (2025) [2] and Alsmadi et al. (2025) [15]. In bentonite clays is available in layered silicate structure and high cation exchange capacity enhances the adsorption potential and also serve as a mechanical support as reported well by Gharbi (2022) [16] and Hossain (2022) [17]. MWCNTs hybridization with $TiO_2$ and clay reported the best performance for dye degradation, heavy metals sorption and antimicrobial activity exploiting enhanced surface area, porosity and variety of functional groups (Gupta, 2023; Elqahtani, 2025) [14,18]. Meanwhile, gamma irradiation has been developed to be an effective post-synthesis modification method. Additionally, Fu (2020) [19] elaborated on the action of ionizing radiation in reassembling the nanostructures and altering surface functionalities, therefore enhancing the conductivity and durability. Green methods of synthesis employing plant extracts have also garnered interest for their environmentally and biologically benign nature. Extracts from Melia azedarach, with its wealth in flavonoids and phenolic compounds, have been known effectively reduce and stabilize $TiO_2$ nanoparticles to obtain well-dispersed nanostructures of high photocatalytic activity (Karthika, 2017; Jebril, 2020) [11,20]. Despite these progresses, the majority of the work reported so far have been confined to either γ-irradiation of inorganic or polymer based systems, or green synthesis leading to metal oxides but without post irradiation. The synergy of the gamma irradiation on plant extract–mediated synthesized bio-functionalized $TiO_2$–MWCNTs–clay nanocomposites is under-researched to date as reported by Kamil (2014) [21]. However, no systematic studies are available with respect to the long-term combined effect of gamma irradiation on biofunctionalized plant extract synthesized $TiO_2$–MWCNT–Bentonite nanocomposites. The comprehension of the interactions between the clay matrix, CNTs, metal oxide network and phytochemical entities upon gamma-radiation is also essential for sustainable nanomaterial design.

Accordingly, we are aiming in the current study to examine, for the first time, structural and compositional changes induced in a green-synthesized $TiO_2$–MWCNT–Bentonite nanocomposite by gamma irradiation using Melia azedarach leaf extract. In particular, irradiation-induced changes in bonding coordination, crystallinity and surface hydroxylation is analyzed with FTIR and XRD methods. Furthermore, this work aims at understanding the relationship between gamma dose and structural disorder to demonstrate how controlled irradiation could be employed as an applied methodology for tailoring the physicochemical properties of hybrid nanocomposites. The outcomes are expected to provide valuable insights into the rational design of radiation-responsive, eco-friendly nanomaterials for advanced photocatalytic and environmental remediation applications.

## 2. Materials and Methods

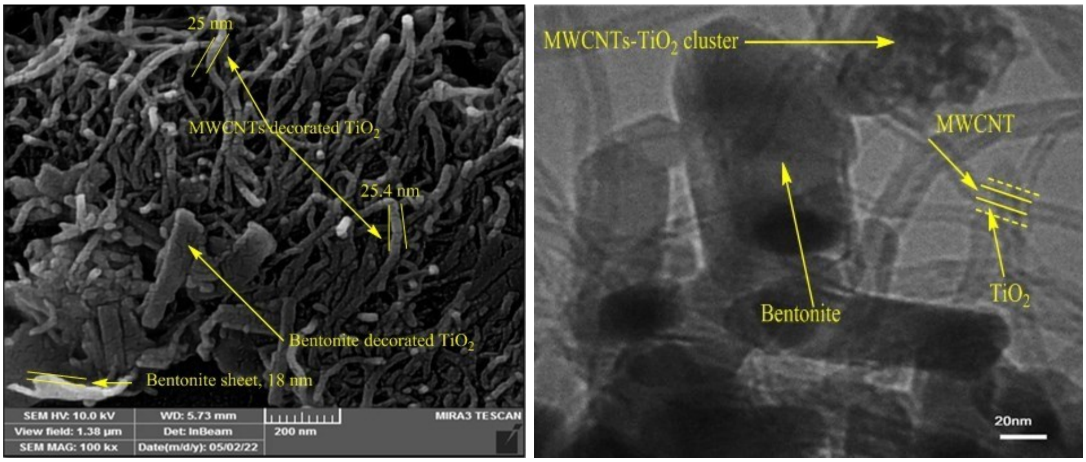
15 g of bentonite clay were soaked in deionized water for 24 hours and then mixed with 0.5 g of MWCNTs in 100 ml of deionized water. The mixture was thereafter sonicated using an ultrasonic probe (750 W, Sonics, USA) for 30 minutes to afford a homogenized solution. 5 ml of titanium isopropoxide dissolved in ethanol was added dropwise under stirring to the above mixture. The mixture was stirred for 15 minutes and then 100 ml of *Melia azedarach* leaf extract was added (25 g of *Melia azedarach* leaves in 100 ml deionized water). Thereafter, the mixture was heated to 60°C under stirring for 2 hrs. The resulting clay nanocomposite was collected by centrifugation at 4000 rpm, washed, and dried for 24 hours at 60°C. The dried composite was calcined at 300°C for 6 hours to get the required compound.

To investigate the effects of high-energy radiation, the synthesis nanocomposite sample were subjected to gamma irradiation using a 60 Co source at a dose rate of approximately 30 Gy/h. Four total doses were applied: 25, 50, 75, and 100 kGy. Irradiation was performed at Physics Department, College of Science, University of Baghdad, at room temperature under ambient atmospheric conditions. After exposure to radiation, samples were stored in tightly closed containers before spectral analysis [2].

## 3. Results and Discussion

The SEM micrograph (Fig. 1a) clearly shows a homogenously entangled, three-dimensional network of MWCNTs fitted into the clay-$TiO_2$ matrix. The nanotubes seem continuous and entangled to construct the conductive framework which encapsulates $TiO_2$ nanoparticles and bentonite sheets. Bright contrast originating from well dispersed $TiO_2$ particles located on the surface of both MWCNTs and clay platelets is evident, showing good interfacial adhesion between the organic and inorganic components. An average diameter of the MWCNTs was estimated as 25 nm, and its slightly rough surface provides a beneficial anchoring place for $TiO_2$ nanoparticles. Furthermore, thin (thickness lower 20nm) bentonite sheet material could be observed well-dispersed in the nanotube network, indicating that incorporation of the clay phase is efficient. This interpenetrated structure is indicative of the successful hybridization of these three components and the establishment of a percolation network that not only promotes charge transport but also increases mechanical stability. Such structural integration is particularly advantageous for photocatalytic applications, as it maximizes the interfacial contact area and increases the number of reactive sites available for adsorption and redox reactions. The homogeneous distribution of $TiO_2$ onto the MWCNT-bentonite support is crucial for effective light utilization, as well as charge separation, which are very important factors in enhancing catalytic and environmental functionality [21,22].

The transmission electron microscopy (TEM) image (Fig. 2b) further indicates that nanopores in the two devices are of similar size. It provides comprehensive information of the internal microstructure of the green synthesized $TiO_2$–MWCNTs–Bentonite nanocomposite prior to irradiation. The micrographs were captured at 20 nm scale, and it is clear that the well dispersed nanoscale structure of MWCNTs, clay platelets, $TiO_2$ nanoparticles in conjunction with organic phase (obtained from extract of Melia azedarach) is extremely inhomogeneous. The MWCNTs present as a network of long tubular paths housing numerous concentric tubes, within the three-dimensional porous structure high surface area and potential electronic conductive territory of the composites. The (comparatively) uniformly scattered non-clear indefinite dark shapes of the $TiO_2$ nanoparticles are present on the outer surface of nanotubes and can also be observed inside the clay matrix in some cases. This close contact of $TiO_2$ with MWCNTs is an indication of good adhesion at the interface for electron transfer across the interface and photoactivity. The clay takes the form of soft, disordered lamellae with fuzzy edges, so it can be known as being capable to "flexibly" bond on the carbon scaffold. The less contrasted regions surrounding these inorganic entities are an evidence of the Melia azedarach bio-organics acting as natural stabilizing and capping agents, which eventually suppresses the agglomeration and ensures homogenous dispersion [18]. Finally, the observing TEM images further confirmed that a multi-phase hybrid system was well fabricated and each phase has its own special contributions to structure or function. MWCNTs constituted the conductive scaffold, $TiO_2$ NPs acted as active catalytic sites, clay layers were for adsorption and support, while organic matrix was responsible of cohesion and stability. Since the analyzed samples will not have been irradiated, such a morphology can be considered as the reference baseline for comparison with structural or chemical alterations induced by γ-irradiation.



(a) (b)

**Fig. (1) (a) SEM image and (b) TEM image of the synthesized MWCNTs-clay-$TiO_2$-*Melia azedarach L.* nanocomposite**

An energy-dispersive X-ray spectroscopy (EDS) analysis as shown in Fig. (3) and table (1) aimed to study the elemental composition of a nanocomposite prepared using multi-walled carbon nanotubes (MWCNTs), natural clay, and titanium dioxide ($TiO_2$) and surface functionalization with Melia azedarach plant extract. The EDS spectrum shows the intensity of emitted X-rays (in the vertical axis) from the sample at different energies photon energies and gives valuable information about which elements are present in that material The experimental elemental analysis confirms successful synthesis of a complex nanocomposite containing MWCNTs, clay, titanium dioxide ($TiO_2$) and bioactive components synthesized from Melia azedarach. The peaks of the spectrum are shown to exhibit characteristics in energy corresponding to those associated with characteristic X-ray lines of important elements such as Ti (Kα at 4.5 keV), Si, Al, Mg, Fe, Ca and O arising from inorganic/organic phases. The main peak of Ti is ascribed to the existence of few $TiO_2$ nanoparticles which are absolutely necessary for photocatalytic as well as electronic applications. The strong Si Kα fluorescent line indicates a large amount of admixing from the silicate-like clays if their stoichiometric composition were taken into consideration Al, Mg and Fe witnesses regarding the mineralogical complexity of the matrix. and life's origins Guojian Sampling of complex molecular reaction pathways and their effects on has recently been proposed that the trace elements are weakly occurring in lower intensity depositions might be the natural precursors or residues of synthesis for multifunctions in behavior of the composite. The strong oxygen peak indicates the oxide nature of the matrix and carbon signal proves that MWCNTs and plant derived organic material has been embedded. Overall, these spectral data indicate a homogeneous blended hybrid nanostructure with variable elemental composition indicating suitability for environmental remediation, catalysis and tailored material engineering applications [17,23].

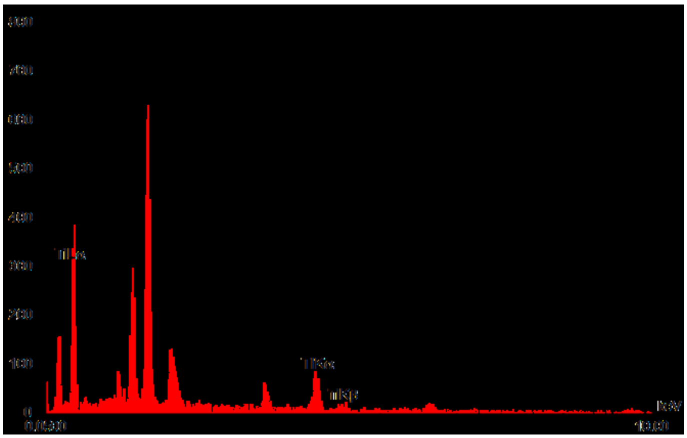


**Fig. (2) EDX spectrum of the synthesized MWCNTs-clay-$TiO_2$-*Melia azedarach L.* nanocomposite**

**Table (1) Elemental signatures and X-ray line intensities from EDX analysis of the synthesized nanocomposite**

| Element | Line | Int | keV | Prob% |
|---|---|---|---|---|
| C | Ka1 | 20.8 | 0.29 | 100 |
| Ca | Ka1 | 6.7 | 3.69 | 100 |
| O | Ka1 | 36.5 | 0.53 | 100 |
| Fe | Ka1 | 3.9 | 6.4 | 100 |
| Mg | Ka1 | 8.6 | 1.26 | 100 |
| Al | Ka1 | 17.1 | 1.5 | 100 |
| Si | Ka1 | 88 | 1.75 | 100 |
| Nb | La1 | 15.4 | 2.17 | 100 |
| Ag | La1 | 1.1 | 2.99 | 100 |
| Ti | Ka1 | 14.2 | 4.51 | 100 |
| Ba | La1 | 1 | 4.46 | 100 |
| Bi | Ma1 | 1.4 | 2.42 | 100 |
| Ir | La1 | 0.8 | 9.14 | 20 |

The FTIR spectra of the bentonite-MWCNTs-$TiO_2$ nanocomposite shown in Fig. (3) before and after gamma irradiation doses of 25, 50, 75, and 100 kGy as showing fig4. In the non-irradiated sample, there was a strong absorption band seen at ~1030 $cm^{-1}$ is due to the Si–O–Si stretching vibrations of bentonite. The broad band centered at ~3430 $cm^{-1}$ is due to O–H stretching vibrations of surface hydroxyl groups and adsorbed water molecules. The absorption at ~1630 $cm^{-1}$ is attributed to the bending mode of molecular water, whereas the bands at ~500–800 $cm^{-1}$ are due to Ti–O–Ti lattice vibrations of $TiO_2$. At a dose of 25 kGy, the reduction in transmittance intensity of Si–O– Si and O–H bands is minimum indicating the partially breaking up of H-bonded network as well as possible dehydroxylation. At the higher doses (50–75 kGy) larger differences were observed, and especially broadening of the O–H. stretching band and significant reduction in the intensity of the Si–O– Si peak. This demonstrates radiation- Induced chemical disordering in the bentonite layers and incomplete disorganization of the silicate lattice. Notably, the low frequency (~500–800 $cm^{-1}$) Ti–O–Ti vibration band was reduced at 100 kGy, possibly due to the latticewarping and local crystalline-amorphous amorphization in TiO2 phase caused by high-energy gamma irradiation. The observed shifts in modified nanocomposite (bentonite-MWCNTs) may associate with the bond breaking and restructuring within the matrix of the hybrid nanocomposite, FTIR data confirm that gamma irradiation plays a vital role in destabilization of structures and altering chemical bonds of bentonite-MWCNTs-$TiO_2$. The magnitude of those changes increases with radiation, indicating the overall effect of high energy photons on inorganic and organic components Characteristics of composite [24,25].

The X-ray diffraction (XRD) patterns of the MWCNTs-clay-$TiO_2$-*Melia azedarach* nanocomposite before and after gamma irradiation at doses of 25, 50, 75, and 100 kGy as shown in Fig. (4). The patterns display characteristic reflections of graphitic carbon, in particular the (002) plane at 26.7°, and anatase $TiO_2$,superimposed on a broad diffraction background due to the clay matrix as well as remaining organic moieties from the plant extract. The effect of gamma irradiation caused an increment of the peak broadening, a decrease of intensities, and small 2θ shifts with dose increase higher than 50 kGy. These variations suggest that there is a gradual decrease in the coherent crystallite sizes and increase in lattice strain due to irradiation-induced defects, generation of oxygen vacancies and distortion of the lattice (MWCNT as well $TiO_2$ sites) Scherrer size calculations based on the main reflections were around 208–560 nm with a value close to ~332 nm for (002) peak from graphitic phase. The microstrain estimated by Williamson–Hall procedure lies between 0.0022 and 0.0069and could increase with increase in the peak broadening. The anatase reflections are present at all doses of irradiation, so the phase is preserved and less intense graphitic peaks could indicate that stacking of MWCNTs is interrupted and even walls crosslinked. Lack of major basal peaks in clay is indicative of the widespread exfoliation/intercalation, which may be promoted by synergism between radiation and nanocomposite heterogeneity. Such structural changes should have implications in the composite functional properties due to an increase in active site density for moderate doses and loss of performance at too-high exposures [2,19,26].

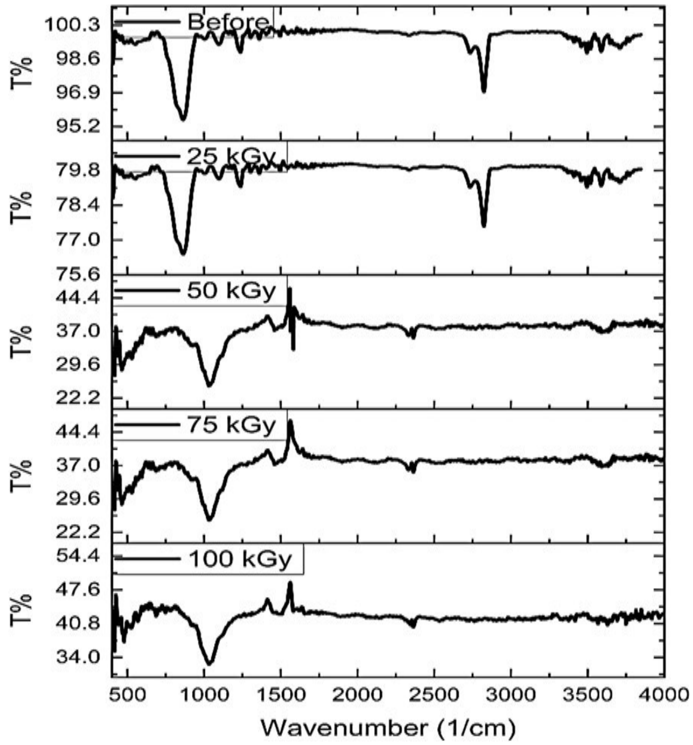


**Fig. (3) FTIR spectra of the MWCNTs-clay-$TiO_2$ nanocomposites before and after gamma irradiation at different dose (25, 50, 75, and 100 kGy )**

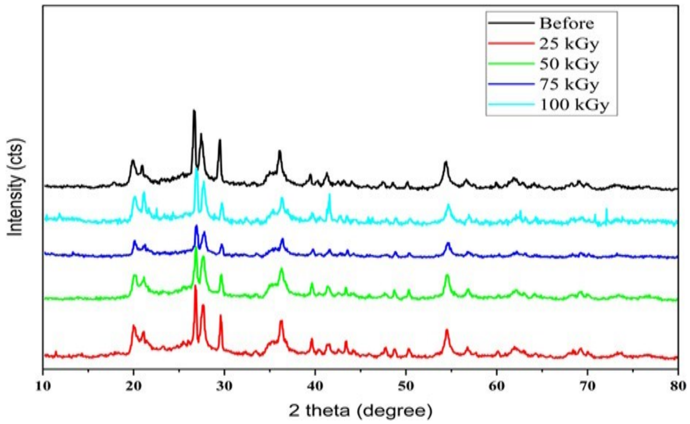


**Fig. (4) XRD patterns of the MWCNTs-clay-$TiO_2$ nanocomposites before and after gamma irradiation at different dose (25, 50, 75, and 100 kGy)**

## 4. Conclusion

The results clearly indicate the dose-dependent influence of gamma irradiation on structural and molecular characteristics of MWCNT–clay–$TiO_2$ nanocomposites. Besides the increase of $TiO_2$ crystallinity and small changes in both clay and NT layers, excepted by functional groups, monomorphism took place, higher radiation doses (75–100 kGy) would lead to a larger degree of defects. These effects reflect the "dualism" of gamma irradiation as a preparation procedure: one the one hand, actin-structures become more easily observed or even (semi-)conserved; on the other hand, they suffer from the dosage. These findings have practical significance in the optimization of properties of nanocomposites which are pursued and optimized for environmental remediation, construction of advanced materials and similar kinds of multifunctional systems. The approach can be extended to other bio-functionalized nanomaterials in the further studies in order to develop deployment strategies for practical application.

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