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Synthesis of Silica-doped multi-walled carbon nanotubes (Silica-MWCNTs) using a spray pyrolysis-assisted chemical vapor deposition (CVD) technique

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Abstract

Silica-doped multi-walled carbon nanotubes (Silica-MWCNTs) have attracted us due to their favorable structural, mechanical, and electrical characteristics, which make them attractive options for energy storage and nanoelectronic applications. In this work, we used tetraethoxysilane (TEOS) as the Silica precursor to create Silica-MWCNTs utilizing an in-situ Chemical Vapor Deposition (CVD) approach. By introducing TEOS into a ferrocene-toluene solution during the CVD process, Silica atoms were successfully incorporated into the carbon nanotube lattice, leading to noticeable modifications in the structural and electronic characteristics of the material. The synthesized Silica-MWCNTs were systematically characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), Raman spectroscopy, and atomic force microscopy (AFM). Silica was successfully integrated and verified by XRD analysis, as shown by peak broadening and lattice aberrations. While AFM studies showed surface roughness and mechanical reinforcement as a result of Silica incorporation, SEM images showed nanotubes. Increased D-band intensity and 2D-band broadening were seen in Raman spectroscopy, suggesting defect-induced changes and changed electronic characteristics. The controlled doping of Silica into MWCNTs, which affects their conductivity, mechanical robustness, and optical characteristics, is confirmed by these structural and spectroscopic results. The experimental results show that the in-situ CVD technique provides a scalable and controllable way to create Silica-MWCNTs with specific mechanical and electrical characteristics, which makes them suitable for use in energy storage systems, optoelectronics, and nanoelectronics.

Keywords: TEOS, chemical vapor deposition (CVD), CNTs, Silica-MWCNTs, structural characterization, energy storage.

Introduction

Carbon nanotubes (CNTs) have shown their usefulness as a versatile nanomaterial due to their good mechanical strength, electrical conductivity, and structural stability, making them a good candidate for applications in nanoelectronics, energy storage, and advanced composite materials (Syduzzaman et al. 2025; Fenta and Mebratie 2024). Among various CNT modifications, Silica-doped multi-walled carbon nanotubes (Silica-MWCNTs) have gained a significant attention because of their advantages conferred by Silica incorporation, such as improved electronic properties, mechanical resilience, and tunable structural characteristics. Silica-MWCNTs are good candidate for electronic devices, optoelectronic systems, and high-performance energy storage solutions (Campos-Delgado et al. 2010; Arjmandi, Sasanpour, and Rashidian 2012). Silica integrated into the CNT lattice through various synthesis techniques, including plasma-enhanced CVD, sol-gel methods, and laser ablation. However, these methods often face challenges such as inhomogeneous doping, poor structural integrity, and scalability limitations (Ben Ishai and Patolsky 2009). To address these challenges, we have employed an in-situ Chemical Vapor Deposition (CVD) approach, utilizing tetraethoxysilane (TEOS) as the Si precursor. By using TEOS into a ferrocene-toluene solution during the CVD process, Silica atoms are successfully incorporated into the CNT framework, leading to controlled structural and electronic modifications (Jeong et al. 2003; Sharif Zein and Boccaccini 2008). To confirm the material synthesis, we have done some characterization of our synthesized Silica-MWCNTs by using X-ray diffraction (XRD), scanning electron microscopy (SEM), Raman spectroscopy, and atomic force microscopy (AFM) to analyze their structural, morphological, and spectroscopic properties. XRD studies shows about lattice distortions and peak broadening, confirm the successful incorporation of Silica into the CNT matrix. SEM imaging shown the presence of well-aligned nanotubes with their clear morphology, while AFM measurements indicated surface roughness and mechanical reinforcement due to Silica incorporation (Zhao et al. 2021; Zheng et al. 2009). Raman spectroscopy shown an improved D-band intensity and increase of the 2D-band, suggesting defect-induced modifications that effect electronic conductivity and optical behavior (Yoo et al. 2012; Kopanski et al. 2011). The findings of this study make in-situ CVD synthesis method as a scalable and controllable way to synthesize Silica-MWCNTs with tailored properties. Obtained results suggest the possible use of Silica-MWCNTs for applications in nanoelectronics, optoelectronics, and energy storage systems (Brachetti-Sibaja et al. 2021;

Chen, Long, and Cui 2022).

Experimental Section

Reagents and Chemicals

Ferrocene (catalyst) was procured from Otto Chemie, while toluene (carbon source) was obtained from Molychem India. TEOS (Silica precursor) was purchased from Alfa Aesar. All used reagents were based on their high-grade purity.

Synthesis of Silica-Doped Multi-Walled Carbon Nanotubes (Silica-MWCNTs)

Silica-MWCNTs were synthesized via a spray pyrolysis-assisted Chemical Vapor Deposition To synthesize Silica-MWCNTs, spray pyrolysis-assisted Chemical Vapor Deposition (CVD) method, which we have used in our previous works to synthesize multiwalled carbon nanotubes (Wang et al. 2006; Gonzalez et al. 2022). First, we have prepared a homogeneous precursor solution by adding 1.5 g of ferrocene which act as catalyst, into 40 mL of toluene which serves as carbon source, and 10 mL of tetraethoxysilane (TEOS, silica precursor) at room temperature. The solution mixture was magnetically stirred for 15 minutes to ensure homogeneity. Prior to these we have already turned ON the CVD Furnace. The CVD setup consists of a horizontal quartz tube (120 cm length, 3 cm diameter) mounted within a programmable quartz tube furnace. The furnace was heated to 830°C at a ramp rate of 13.8°C/min. Prior to the injection of the precursor solution, argon gas was introduced into the quartz tube furnace at a flow rate of 200 standard cubic centimeters per minute (SCCM), when the furnace heated to 200°C, to establish an inert atmosphere. The precursor solution was loaded into a syringe pump and atomized into fine droplets using a spray nozzle. When the desired growth temperature reached to 830 °C, we started to inject the precursor solution. The precursor solution was injected into the reactor at a controlled rate of 0.83 mL/min, with argon serving as the carrier gas. Inside the reaction chamber the precursor thermally decomposed as into iron (Fe) nanoparticles, acting as catalytic nucleation sites for CNT growth and Toluene pyrolyzed to release carbon atoms, which dissolved into Fe nanoparticles and precipitated as graphitic layers to form MWCNTs. TEOS decomposed to form silica (SiO₂) species, adsorbed onto the CNT surface to form a doped architecture. The growth duration was kept 60 minutes to ensure sufficient growth time to yield well-aligned silica-doped MWCNTs on the quartz tube's inner walls. After completion, the furnace was cooled to room temperature under argon flow. The deposited silica-doped MWCNTs were carefully scraped from the quartz tube reactor walls

using a clean spatula, in the form of a black, fibrous powder. The collected material exhibited Silica-doped MWCNTs. (Kolodziejczyk et al. 2016; Li and Yang 2023). Previous studies have demonstrated that Silica incorporation into the CNT lattice significantly alters the structural, electronic, and mechanical properties of CNTs, enhancing their performance for nanoelectronic, optoelectronic, and energy storage applications (Mélinon et al. 2007; Peng et al. 2008). The in-situ CVD-based doping approach presented in this work ensures scalability and reproducibility, making it a viable technique for the controlled synthesis of Silica-MWCNTs with tailored properties. All steps were conducted in the Nanomaterials Laboratory Department of Physics, Institute of Science, Banaras Hindu University, Varanasi under the supervision of Prof. Anchal Srivastava, A schematic representation of the synthesis process is shown in Figure 1, illustrates the reaction pathway from precursor preparation to Silica doped nanotube formation.

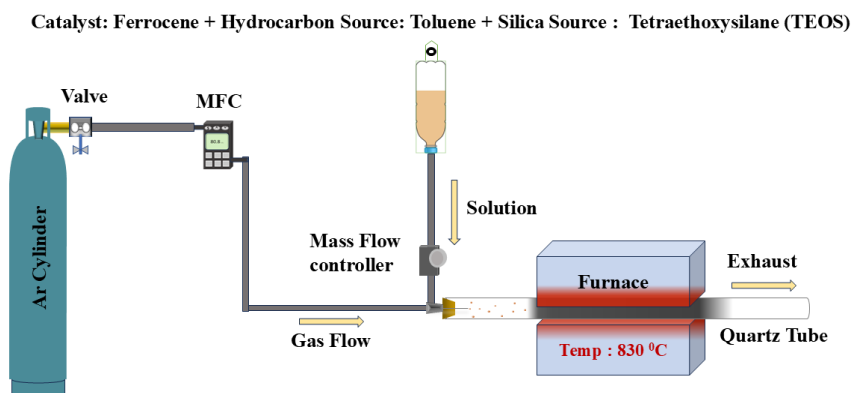


Figure 1: Schematic diagram of the synthesis of Silica-doped multi-walled carbon nanotubes.

Results and Discussion

The structural and morphological characteristics of the synthesized Silica-doped multi-walled carbon nanotubes (Silica-MWCNTs) were comprehensively analyzed using a combination of advanced techniques, including X-ray diffraction (XRD) (Malvern Panalytical, UK) using $\text{Cu K}\alpha$ radiation, scanning electron microscopy (SEM) (ZEISS, Germany), atomic force microscopy (AFM) (Park XE7, South Korea), and Raman spectroscopy (WITec alpha300 RAS, Germany). These characterization methods provided crucial insights into the crystallographic structure, phase composition, surface morphology, and defect density of the doped nanotubes.

X-ray Powder Diffraction (XRD) Analysis

The crystallographic structure and phase composition of the synthesized silica-incorporated multi-walled carbon nanotubes (Silica-MWCNTs) were analyzed using X-ray powder diffraction (XRD) on a Malvern Panalytical system (UK) with Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$). The XRD pattern revealed well-defined diffraction peaks characteristic of hexagonal graphitic carbon (MWCNTs), indexed to the (002), (100), (101), and (103) crystallographic planes, consistent with the reference JCPDS card No. 75-1621. The prominent (002) reflection at $\sim 26^\circ$ corresponds to the interlayer stacking of graphitic carbon, while the in-plane (100) and (101) reflections confirm the structural integrity of the MWCNT framework. A low-intensity peak observed at $2\theta \approx 43^\circ$ was assigned to the (110) plane of metallic iron (JCPDS 88-2324), attributed to residual catalyst particles from the synthesis process (Pandey et al. 2019; Sun et al. 2024). The incorporation of silica into the MWCNT matrix was evidenced by distinct diffraction peaks corresponding to the α -quartz phase of SiO $_2$ (JCPDS 89-8951), specifically the (100), (101), (110), (102), (111), (003), and (103) reflections. The presence of these peaks indicates partial crystallization of silica within the composite, with peak broadening suggesting lattice strain and structural distortion due to heteroatom integration into the carbon lattice. Additional secondary peaks, indicative of silica-carbon interfacial phases, further corroborate successful doping, likely arising from covalent interactions between silica and the MWCNT surface (Pandey et al. 2022). Such structural modifications are known to alter charge distribution and enhance mechanical stability in hybrid nanotube systems, aligning with reported effects of silica doping on the electronic and mechanical properties of CNTs (Qian et al. 2010). Figure 2: XRD pattern of as synthesized Silica-doped multi-walled carbon nanotubes confirms the coexistence of graphitic carbon, crystalline α -quartz, and residual Fe within the composite, demonstrating the efficacy of the synthesis route in achieving a structurally modified Silica-MWCNT hybrid material.

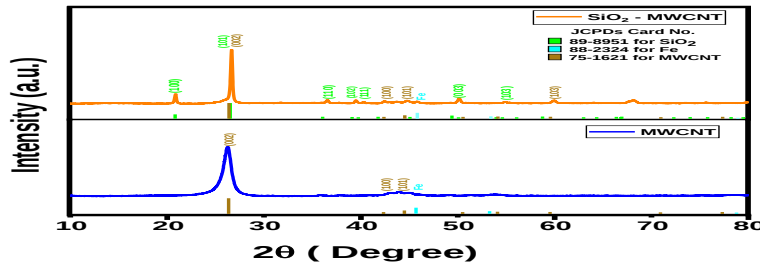


Figure 2: XRD pattern of as synthesized Silica-doped multi-walled carbon nanotubes

Scanning Electron Microscopy (SEM) Analysis

The morphological characteristics and alignment of the Silica-MWCNTs were investigated using scanning electron microscopy (SEM) (ZEISS, Germany). The SEM images revealed a uniform distribution of nanotubes with high aspect ratios and well-defined tubular structures. The presence of Silica doping was inferred from the surface roughness and structural modifications observed in the CNT walls, which appeared slightly thicker compared to undoped MWCNTs (Rahman et al. 2022). Figure 3: SEM images of the synthesized Silica-doped multi-walled carbon nanotubes showed that the nanotubes maintained a good degree of vertical alignment, which is critical for enhancing their electrical and thermal properties in potential applications such as nanoelectronics and energy storage devices (Al-Zu'bi, Anguilano, and Fan).

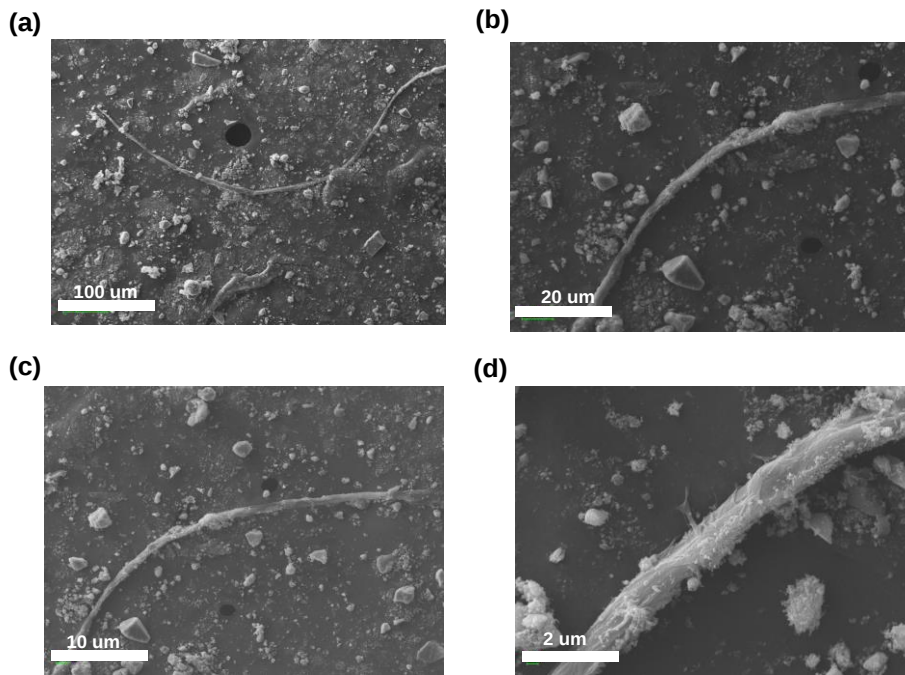


Figure 3: SEM images of the synthesized Silica-doped multi-walled carbon nanotubes

Atomic Force Microscopy (AFM) Analysis

To further examine the surface morphology and topographical features of the Silica-MWCNTs, atomic force microscopy (AFM) (Park XE7, South Korea) was performed. The AFM images provided high-resolution nanoscale details, revealing variations in tube diameter and surface roughness due to Silica incorporation (Marteeny 2008). Figure 4: AFM

topography of the Silica-doped multi-walled carbon nanotubes shows an increased tube diameter compared to pristine CNTs, suggesting successful doping and possible Silica deposition on the nanotube walls (Nguyen et al. 2019). Moreover, the mechanical properties, including surface adhesion and stiffness, were also affected by Silica doping, confirming structural modifications at the atomic level (Lu 2006).

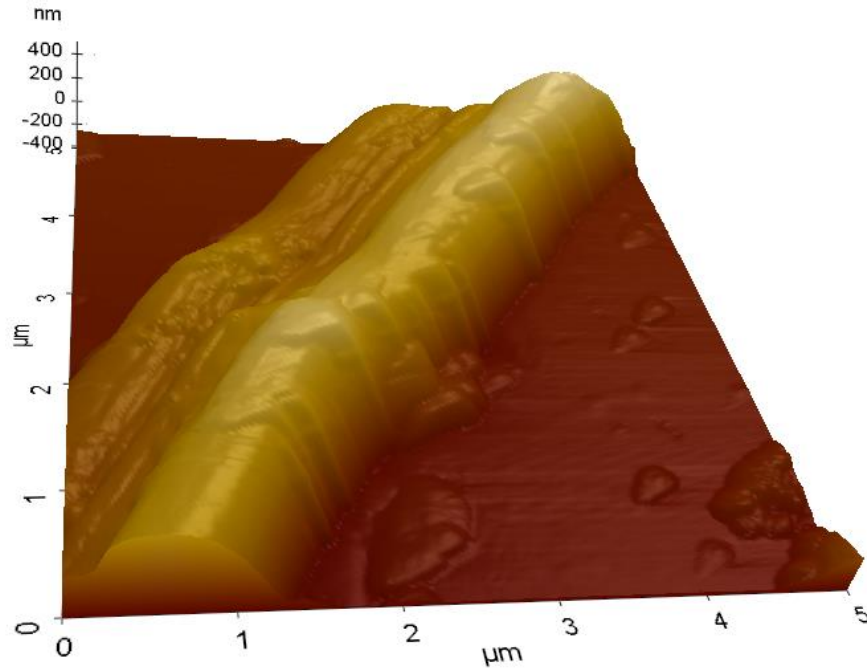


Figure 4: AFM topography of the Silica-doped multi-walled carbon nanotubes

Raman Spectroscopy Analysis

Raman spectroscopy was conducted using a WITec alpha300 RAS (Germany) Raman microscope to evaluate the vibrational properties and structural integrity of the synthesized Silica-MWCNTs. The Raman spectra exhibited prominent D-band ($\sim 1350 \text{ cm}^{-1}$), G-band ($\sim 1582 \text{ cm}^{-1}$), and 2D-band (2692 cm^{-1}), which are characteristic of carbon nanotube structures (Wu et al. 2023). The D-band, associated with defects and disorder in the carbon lattice, showed intensity variation in Silica-doped samples, indicating the introduction of structural defects due to Silica incorporation (Prete et al. 2024). The G-band, representing the graphitic nature of CNTs, remained prominent, confirming that the nanotubes retained their sp^2 -hybridized carbon network despite Silica doping. The presence of Silica in the MWCNTs compared to pristine MWCNTs

support the presence of doping-induced defects (Quach et al. 2021). The Raman spectra exhibited prominent primary Raman peak at are typically observed at 464 cm^{-1} . This peak is attributed to the symmetric stretching of the Si-O-Si bonds. Due to the bending modes of the Si-O-Si unit we see a peak at 202 cm^{-1} and depending on the structure we get a low frequency peak around 124 cm^{-1} (ZIEMANN 2004). Furthermore, the 2D-band, indicative of the number of graphene layers and degree of stacking order, exhibited noticeable broadening, which suggests alterations in the electronic structure of the nanotubes due to Silica incorporation (Roy and Das 2025). Figure 5: Raman spectra of the as synthesized Silica-doped multi-walled carbon nanotubes shows the Silica doping introduced localized states in the CNT lattice, thereby modifying its electronic and optical properties (Evanoff et al. 2012).

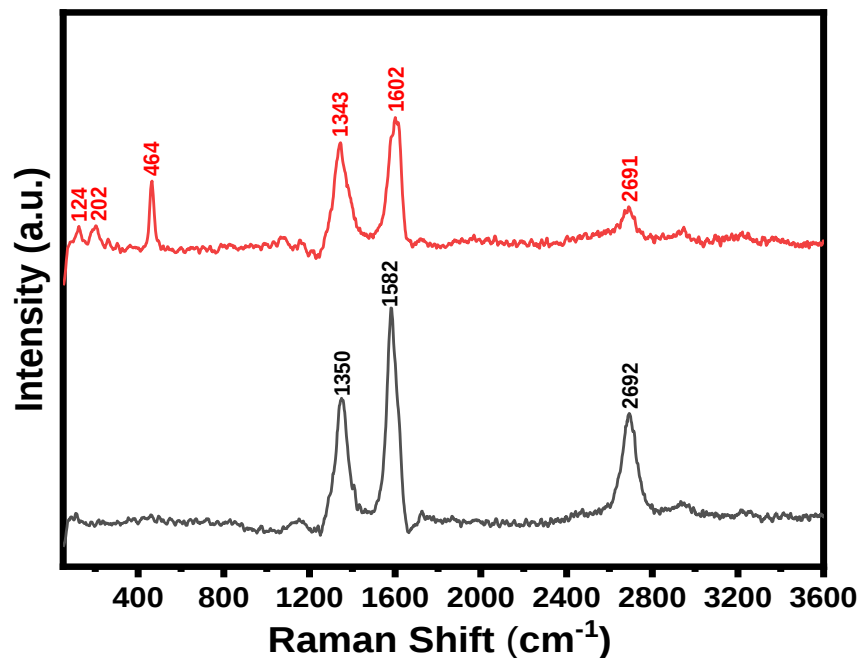


Figure 5: Raman spectra of the as synthesized Silica-doped multi-walled carbon nanotubes

Conclusion

The detailed structural and morphological characterization confirmed the successful incorporation of Silica into the MWCNTs. XRD analysis demonstrated lattice distortions due to doping, SEM revealed uniform morphology and alignment, AFM confirmed changes in tube diameter and mechanical properties, and Raman spectroscopy highlighted doping-induced defects and electronic modifications. These findings suggest that

Silica-doped MWCNTs exhibit structural and electronic properties suitable for applications in nanoelectronics, optoelectronics, and energy storage devices.

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