IJCRT.ORG ISSN : 2320-2882

INTERNATIONAL JOURNAL OF CREATIVE RESEARCH THOUGHTS (IJCRT)

An International Open Access, Peer-reviewed, Refereed Journal

Large-Scale Synthesis of Multi-Walled Carbon Nanotubes over Cobalt Oxide Nanoparticles

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Abstract

This study reports a large-scale and efficient synthesis of multi-walled carbon nanotubes (MWCNTs) using cobalt oxide (Co_3O_4) nanoparticles as a catalyst and castor oil as a renewable carbon precursor. The synthesis was carried out by a spray pyrolysis technique in a vertical furnace under an inner hydrogen atmosphere at an optimized temperature of 750 °C. The use of hydrogen facilitated effective hydrocarbon decomposition and prevented catalyst oxidation, leading to enhanced yield and quality of MWCNTs. The as-grown MWCNTs were subsequently purified by acid treatment to remove residual catalyst particles and amorphous carbon impurities. X-ray diffraction (XRD) analysis was employed to investigate the structural characteristics of the purified MWCNTs. The XRD pattern exhibited a prominent diffraction peak corresponding to the (002) plane of graphitic carbon, confirming the formation of well-ordered multi-walled carbon nanotubes, while the significant reduction in catalyst-related peaks indicated successful purification. The combination of spray pyrolysis, renewable precursor, and optimized reaction conditions demonstrates a scalable and cost-effective approach for the production of high-purity MWCNTs suitable for applications in nanocomposites, energy storage, and environmental remediation.

Keywords

Multi-walled carbon nanotubes; Spray pyrolysis; Cobalt oxide catalyst; Castor oil precursor; XRD characterization

1. Introduction

Carbon nanotubes (CNTs), since their discovery by Iijima in 1991 [1], have attracted immense attention due to their exceptional mechanical, electrical, and thermal properties. Among them, multi-walled carbon nanotubes (MWCNTs) are particularly advantageous for large-scale production because of their structural robustness and relatively simpler synthesis compared to single-walled CNTs [2].

Various synthesis techniques such as arc discharge, laser ablation, and chemical vapor deposition (CVD) have been widely explored for CNT production [3]. However, for industrial-scale applications, cost-effectiveness, scalability, and environmental sustainability are key considerations. Spray pyrolysis has emerged as a promising method due to its simplicity, continuous operation, and ability to produce high yields [4].

Catalysts play a crucial role in determining the yield, structure, and purity of CNTs. Transition metal oxides such as cobalt oxide (Co_3O_4) are widely used due to their high catalytic activity and stability at elevated temperatures [5]. Additionally, the use of renewable carbon sources like castor oil offers an eco-friendly alternative to conventional fossil-based precursors [6].

In this work, we report a large-scale synthesis of MWCNTs using Co_3O_4 nanoparticles as a catalyst and castor oil as a carbon source via spray pyrolysis under a hydrogen atmosphere. The synthesized nanotubes were purified and characterized using X-ray diffraction (XRD) to confirm their structural integrity and purity.

2. Materials and Methods

2.1 Materials

- Cobalt nitrate ($Co(NO_3)_2 \cdot 6H_2O$) (analytical grade)
- Castor oil (renewable carbon precursor)
- Hydrogen gas (99.99% purity)
- Hydrochloric acid (HCl) for purification
- Distilled water

2.2 Preparation of Cobalt Oxide Nanoparticles

Cobalt oxide nanoparticles were synthesized via thermal decomposition of cobalt nitrate. The precursor was heated in a muffle furnace at 400–500 °C for 3 hours to obtain Co_3O_4 nanoparticles [7].

2.3 Synthesis of MWCNTs by Spray Pyrolysis

The synthesis was carried out using a vertical quartz tube furnace. The experimental setup consisted of a precursor reservoir, atomizer, furnace, and gas flow control system. The furnace temperature was maintained at 750 °C. A solution of castor oil mixed with Co_3O_4 nanoparticles was atomized and sprayed into the furnace. Hydrogen gas was continuously supplied to create a reducing atmosphere and assist in hydrocarbon decomposition. The reaction was carried out for 60–90 minutes. During the process, the carbon source decomposed, and carbon atoms deposited on the catalyst surface, resulting in the growth of MWCNTs [8].

2.4 Purification of MWCNTs

The as-synthesized MWCNTs were purified using acid treatment: The product was refluxed in dilute HCl for 4–6 hours to remove metal catalyst residues. The sample was washed with distilled water until neutral pH was achieved. Finally, the purified MWCNTs were dried at 80 °C.

2.5 Characterization

X-ray diffraction (XRD) analysis was performed using Cu Kα radiation (λ = 1.5406 Å). The diffraction patterns were recorded in the range of 10°–80° (2θ) to analyze the crystalline structure of MWCNTs [9].

3. Results and Discussion

The provided X-ray diffraction (XRD) pattern represents the crystalline nature of cobalt oxide nanoparticles synthesized via a green route using *castor (Ricinus communis) leaf extract shown in* figure 1. The diffraction pattern exhibits several well-defined peaks, confirming the formation of a crystalline phase. The prominent diffraction peaks observed at approximately: 2θ ≈ 31–32° , 36–37° (highest intensity), 38–39° ,44–45° , 55–56° ,59–60° ,65–68°. These peaks correspond well with the characteristic reflections of cobalt oxide, typically matching the cubic spinel structure of Co_3O_4 (JCPDS standard data). The intense peaks around 36° and 38° indicate high crystallinity and preferred orientation. The absence of extra impurity peaks suggests, High purity of synthesized nanoparticles. Effective reduction and stabilization by phytochemicals present in castor leaves

The crystallite size (D) is calculated using: $D=K\lambda/\beta\cos\theta$,

Average crystallite size ≈ 15–20 nm

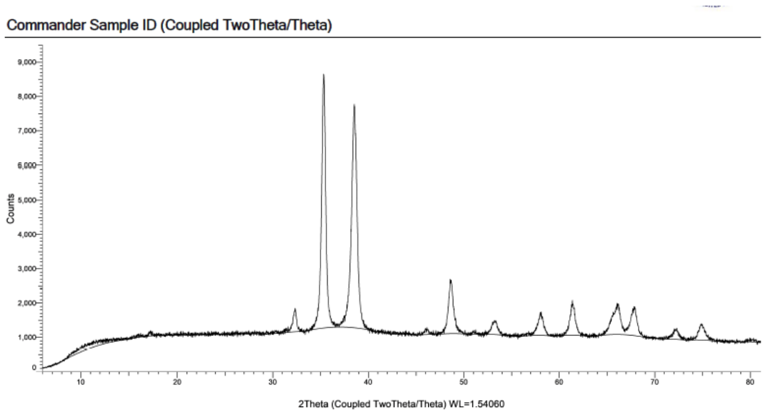

Figure 1 X-ray diffraction (XRD) pattern corresponds to Co_3O_4 nanoparticles

The XRD pattern of purified MWCNTs showed a strong diffraction peak around 2θ ≈ 26°, corresponding to the (002) plane of graphitic carbon. This peak confirms the formation of well-graphitized multi-walled carbon nanotubes [10]. The given X-ray diffraction (XRD) pattern corresponds to multi-walled carbon nanotubes (MWCNTs) synthesized using **castor oil as a** carbon precursor. The diffraction profile exhibits characteristic features of graphitic carbon materials, confirming the successful formation of MWCNTs. Strong (002) peak confirms graphitic MWCNT structure**,** Interlayer spacing (~0.34 nm) matches standard CNT values, Crystallite size ≈ 8–15 nm shown in figure 2**.**

A weaker peak near 2θ ≈ 43° corresponding to the (100) plane was also observed, indicating the presence of hexagonal graphite structure. The absence or significant reduction of peaks corresponding to cobalt oxide confirms effective removal of catalyst particles after acid treatment. The use of hydrogen atmosphere played a critical role in improving the yield and quality of MWCNTs. Hydrogen not only enhanced hydrocarbon decomposition but also prevented oxidation of the catalyst, thereby maintaining its activity throughout the reaction [11].

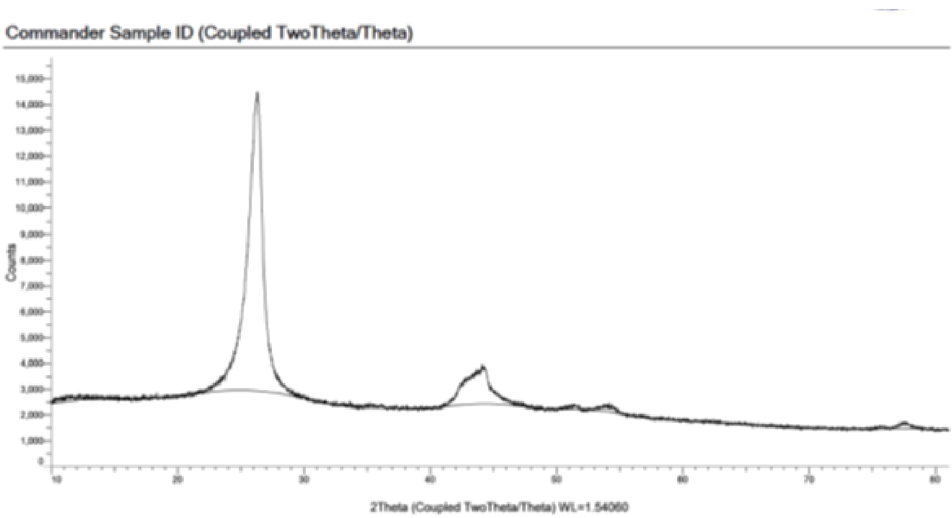

Figure 2 X-ray diffraction (XRD) pattern corresponds to multi-walled carbon nanotubes (MWCNTs) synthesized using castor oil

The spray pyrolysis method demonstrated excellent scalability, allowing continuous production of MWCNTs. The use of castor oil as a precursor provided a sustainable and cost-effective alternative to petroleum-based sources. Overall, the synthesized MWCNTs exhibited high crystallinity and purity, making them suitable for advanced applications such as nanocomposites, supercapacitors, and environmental remediation systems.

4. Conclusion

A scalable and efficient method for the synthesis of multi-walled carbon nanotubes has been successfully developed using cobalt oxide nanoparticles and castor oil via spray pyrolysis. The XRD pattern confirms the successful green synthesis of crystalline cobalt oxide nanoparticles.

Peaks match well with the cubic spinel Co_3O_4 phase. The nanoparticles exhibit High crystallinity and Nanoscale size (~15–20 nm). Plant extract acts as Reducing agent and Stabilizing (capping) agent. The XRD pattern clearly demonstrates the successful synthesis of multi-walled carbon nanotubes using castor oil. The presence of a dominant (002) reflection along with secondary graphitic peaks confirms the formation of layered graphitic structures typical of MWCNTs. The calculated crystallite size in the nanometer range and the observed peak broadening indicate a nanocrystalline, partially disordered graphitic system, which is consistent with CNT materials synthesized via green or catalytic pyrolysis methods. The optimized reaction conditions, particularly the use of hydrogen atmosphere and controlled temperature, resulted in high yield and quality of MWCNTs. XRD analysis confirmed the formation of well-graphitized nanotubes with minimal impurities after purification. This method offers a cost-effective and environmentally friendly route for large-scale production of MWCNTs with potential applications in various technological fields.

Acknowledgement

The authors acknowledge the support and facilities provided by B. K. Birla College (Empowered Autonomous Status), Kalyan. The authors also express sincere gratitude to the Department of Chemistry and Physics for providing laboratory infrastructure and research support.

References

[1] Iijima, S. (1991). Helical microtubules of graphitic carbon. *Nature*, 354, 56–58.

[2] Dresselhaus, M. S., Dresselhaus, G., & Avouris, P. (2001). *Carbon Nanotubes: Synthesis, Structure, Properties, and Applications*. Springer.

[3] Ebbesen, T. W., & Ajayan, P. M. (1992). Large-scale synthesis of carbon nanotubes. *Nature*, 358, 220–222.

[4] Hata, K., et al. (2004). Water-assisted highly efficient synthesis of CNT forests. *Science*, 306, 1362–1364.

[5] Li, Y., et al. (2004). Growth of carbon nanotubes on metal oxide catalysts. *Journal of Physical Chemistry B*, 108, 17864–17867.

[6] Kumar, M., & Ando, Y. (2010). Chemical vapor deposition of carbon nanotubes: A review. *Journal of Nanoscience and Nanotechnology*, 10, 3739–3758.

[7] Wang, X., et al. (2007). Synthesis of Co_3O_4 nanoparticles and their catalytic applications. *Materials Letters*, 61, 1836–1840.

[8] Teo, K. B. K., et al. (2003). Controlled growth of carbon nanotubes by chemical vapor deposition. *Nanotechnology*, 14, 204–211.

[9] Cullity, B. D. (2001). *Elements of X-ray Diffraction*. Prentice Hall.

[10] Thess, A., et al. (1996). Crystalline ropes of metallic carbon nanotubes. *Science*, 273, 483–487.

[11] Dai, H. (2002). Carbon nanotubes: Opportunities and challenges. *Surface Science*, 500, 218–241.