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Synthesis and Characterisation of Carbon Nanofibers Using Castor Oil as a Carbon Source over a Nickel Oxide Catalyst

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Abstract

Carbon nanofibers (CNFs) are one-dimensional carbon nanostructures that have attracted significant attention due to their exceptional mechanical strength, electrical conductivity, chemical stability, and wide-ranging applications in catalysis, energy storage, sensors, and environmental remediation. The synthesis of CNFs using renewable and sustainable carbon sources has become a major research focus in recent years. Castor oil, a non-edible and eco-friendly vegetable oil, serves as an excellent carbon precursor owing to its high carbon content and thermal stability. This review article presents a comprehensive overview of the green synthesis of Nickel-based catalysts using castor leaves and microwave-assisted methods, followed by the synthesis of metal-catalyzed carbon nanofibers (MCNFs) using castor oil through the catalytic chemical vapor deposition (CCVD) method in a hydrogen atmosphere at 750 °C. The purification and functionalization of CNFs via acid treatment are also discussed. Structural and morphological characterization techniques such as Fourier Transform Infrared Spectroscopy (FT-IR), X-ray Diffraction (XRD), and Scanning Electron Microscopy (SEM) used for analyzing both CNFs and Nickel oxide catalysts are critically reviewed. The review highlights the sustainability, efficiency, and potential applications of castor oil-derived CNFs synthesized using green catalysts.

Keywords: Carbon Nanofibers; Castor Oil; Green Synthesis; Nickel Catalyst; CCVD Method

1. Introduction

Carbon nanofibers (CNFs) belong to the family of nanostructured carbon materials and possess unique properties such as high surface area, excellent electrical conductivity, chemical resistance, and superior mechanical strength. These properties make CNFs promising materials for applications in fuel cells, supercapacitors, lithium-ion batteries, catalyst supports, sensors, and biomedical devices [1,2]. Conventionally, CNFs are synthesized using fossil fuel-based hydrocarbons such as acetylene, methane, and benzene, which pose environmental and sustainability concerns. In recent years, there has been a paradigm shift toward the use of renewable carbon sources, particularly vegetable oils, for CNF synthesis [3]. Castor oil, derived from Ricinus communis, is a non-edible oil rich in ricinoleic acid and offers advantages such as low cost, renewability, and high carbon yield [4]. Catalysts play a crucial role in determining the yield, morphology, and quality of CNFs. Transition metals such as iron, Nickel, and nickel are commonly used, with Nickel-based catalysts showing superior catalytic activity and stability for CNF growth [5]. Furthermore, green synthesis approaches for catalyst preparation using plant extracts have gained attention due to their eco-friendly nature and reduced chemical usage [6]. This review focuses on the synthesis of Nickel oxide catalysts using castor leaf extract and microwave assistance, followed by the synthesis of MCNFs using castor oil via the CCVD method. The purification, functionalization, and characterization of CNFs are also discussed in detail.

2. Materials and Methods

2.1 Synthesis of Nickel Oxide Catalyst.

The green synthesis of Nickel oxide catalyst involves the use of castor (Ricinus communis) leaf extract as a natural reducing and stabilizing agent. Fresh castor leaves are thoroughly washed, dried, and boiled in distilled water to obtain an aqueous extract. A Nickel salt solution (such as Nickel nitrate) is mixed with the leaf extract under continuous stirring.

Microwave irradiation is applied to the mixture to accelerate nucleation and particle formation. The microwave-assisted process ensures uniform heating, reduced reaction time, and controlled particle size. The obtained precipitate is filtered, dried, and calcined at elevated temperatures to yield Nickel oxide (NiO) nanoparticles [6,7].

2.2 Synthesis of Metal-Catalysed Carbon Nanofibers (MCNFs):

The synthesis of MCNFs is carried out using the catalytic chemical vapor deposition (CCVD) technique. The green-synthesized Nickel oxide catalyst is placed in a quartz boat and inserted into a tubular furnace. The furnace temperature is raised to 750 °C under a continuous flow of hydrogen gas to reduce the Nickel oxide to metallic Nickel.

Castor oil is introduced into the reaction chamber as a carbon source using a controlled vaporization system. Under high temperature and hydrogen atmosphere, castor oil decomposes on the catalyst surface, leading to the growth of carbon nanofibers. After completion of the reaction, the system is cooled to room temperature under inert conditions [8].

2.3 Purification and Functionalization of Carbon Nanofibers:

The as-grown CNFs contain residual metal catalyst particles and amorphous carbon. Purification is achieved by treating the CNFs with concentrated acids such as nitric acid (HNO_3) or a mixture of sulfuric acid (H_2SO_4) and nitric acid.

Acid treatment removes metallic impurities and introduces oxygen-containing functional groups (–COOH, –OH) on the CNF surface, enhancing their dispersibility and chemical reactivity. The purified CNFs are repeatedly washed with distilled water until neutral pH and then dried [9].

3. Results and Discussion

3.1 FT-IR Analysis:

Figure 1 the FT-IR spectrum of NiO nanoparticles synthesized via green route using castor leaf extract reveals the presence of various functional groups responsible for reduction, stabilization, and capping of nanoparticles [6]. A broad and intense absorption band observed around 3200–3500 cm^{-1} corresponds to O–H stretching vibrations of hydroxyl groups present in alcohols and phenolic compounds. These biomolecules from the plant extract play a crucial role as reducing as well as stabilizing agents. The peaks appearing near 2920–2850 cm^{-1} are attributed to C–H stretching vibrations of aliphatic hydrocarbons, indicating the presence of organic constituents such as proteins and lipids from the leaf extract. A prominent band around 1600–1650 cm^{-1} is assigned to C=O stretching (amide I) or C=C stretching, suggesting the presence of proteins or polyphenols. These compounds are responsible for binding and capping the nanoparticle surface. The absorption band observed near 1380–1450 cm^{-1} corresponds to C–N stretching vibrations of amines or O–H bending, indicating involvement of biomolecules like amino acids and flavonoids in nanoparticle synthesis. A peak around 1000–1100 cm^{-1} is attributed to C–O stretching vibrations of alcohols, ethers, or esters, confirming the presence of plant-derived phytochemicals. Most importantly, the characteristic strong absorption band in the region 400–600 cm^{-1} is assigned to Ni–O stretching vibrations, confirming the formation of nickel oxide nanoparticles.

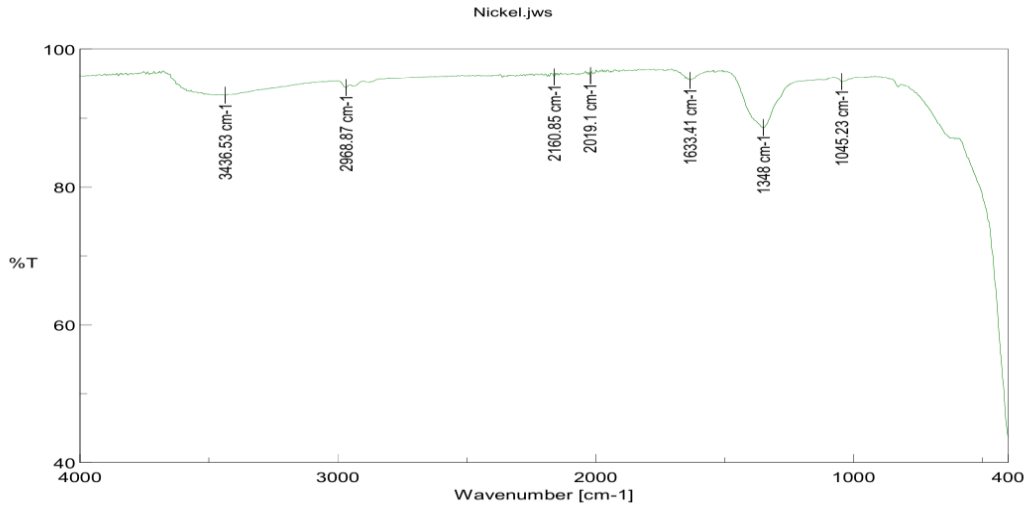

Figure1. The FT-IR spectrum of NiO nanoparticles synthesized using castor leaf extract

Figure 2 the FT-IR spectrum of CNTs synthesized using castor oil in the presence of NiO nanoparticles as catalyst provides evidence for the formation of carbon nanotubes along with residual surface functional groups [9].

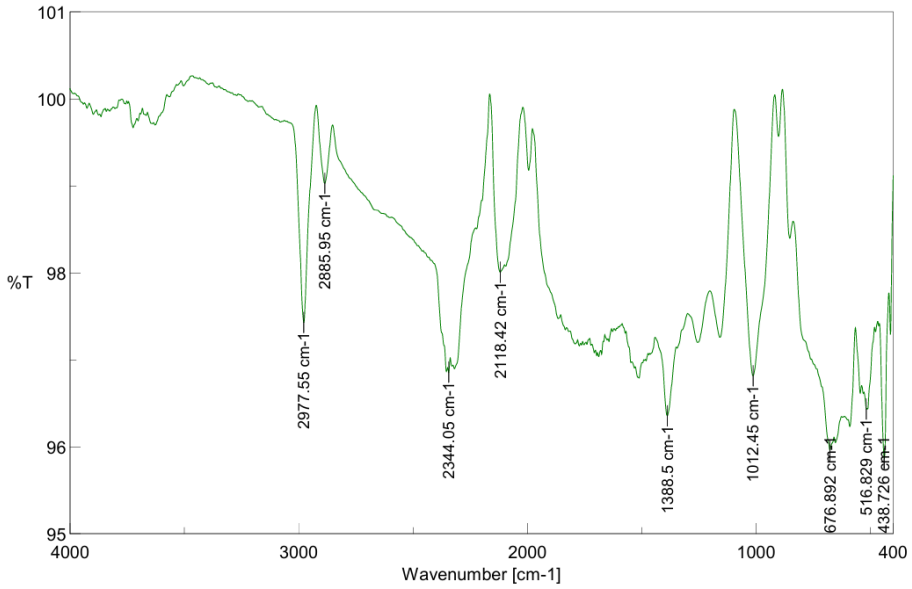

Figure 2 The FT-IR spectrum of CNTs synthesized using castor oil in the presence of NiO nanoparticles

A broad absorption band observed around 3200–3500 cm^{-1} corresponds to O–H stretching vibrations, indicating the presence of hydroxyl groups. These may arise due to adsorbed moisture or slight oxidation of CNT surfaces during or after synthesis.

The peaks appearing near 2850–2920 cm^{-1} are attributed to C–H stretching vibrations of aliphatic $–CH_2$ and $–CH_3$ groups. These bands suggest the presence of residual hydrocarbon fragments derived from the decomposition of castor oil precursor. A band around 1600–1650 cm^{-1} is assigned to C=C stretching vibrations, which is a characteristic feature of the graphitic structure of CNTs. This peak confirms the formation of sp^2 hybridized carbon networks. The absorption peak near 1380–1450 cm^{-1} corresponds to C–H bending vibrations, further supporting the presence of hydrocarbon moieties attached to CNT surfaces. A weak band in the region 1000–1200 cm^{-1} is attributed to C–O stretching vibrations, indicating the presence of oxygen-containing functional groups such as alcohols, ethers, or esters. These groups may form due to partial oxidation or residual plant oil components. Additionally, a low-intensity band observed around 500–600 cm^{-1} may be assigned to Ni–O vibrations, indicating trace amounts of catalyst (NiO nanoparticles) remaining attached to the CNT surface.

3.2 XRD Analysis:

Figure 3 the X-ray diffraction (XRD) pattern of nickel oxide (NiO) nanoparticles synthesized via *Ricinus communis* (castor) leaf extract confirms the crystalline nature and phase purity of the obtained material [7].

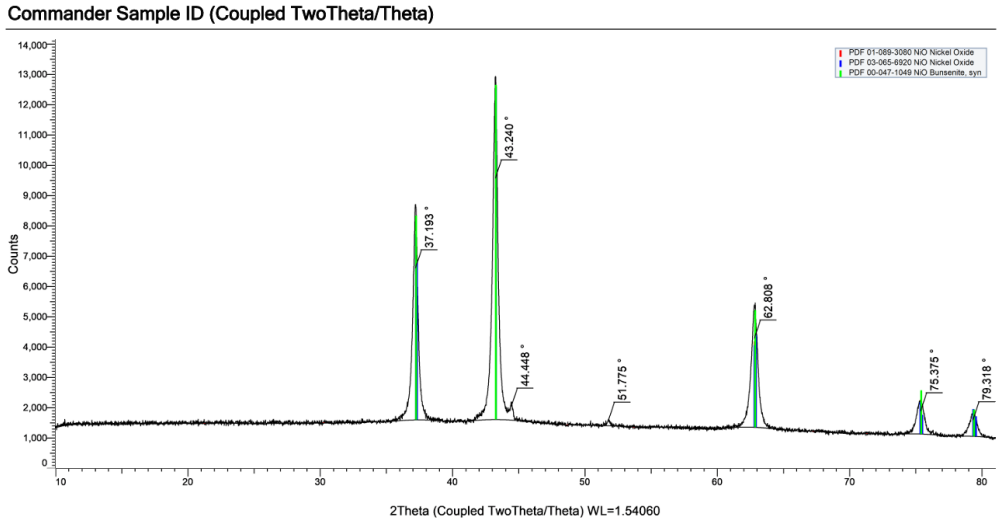

Figure 3 XRD patterns of the Nickel oxide catalyst using Castor leaves

The diffraction peaks observed at approximately 2θ = 37.1°, 43.2°, 62.8°, 75.3°, and 79.3° correspond to the characteristic planes of NiO. These peaks are indexed to the (111), (200), (220), (311), and (222) planes, respectively. The pattern matches well with standard JCPDS data (e.g., PDF No. 47-1049), confirming the formation of face-centered cubic (fcc) NiO structure. The presence of sharp and intense diffraction peaks indicates that the synthesized NiO nanoparticles are highly crystalline in nature. The absence of broad humps suggests minimal amorphous content. No extra impurity peaks corresponding to nickel hydroxide or other nickel-based phases are significantly observed, indicating high phase purity of NiO nanoparticles. Minor low-intensity peaks, if present, may be due to residual organic matter from the plant extract. The crystallite size (D) can be estimated using the Debye–Scherrer equation:

$$D = \frac{0.9\lambda}{\beta \cos\theta}$$

The calculated crystallite size typically lies in the range of **10–30 nm**, confirming the nanoscale nature of the synthesized NiO particles. A minor peak around **51.77°** may be attributed to slight lattice distortion or trace intermediate phases, but its low intensity suggests it does not significantly affect the overall phase purity.

Figure 4 the XRD pattern of the synthesized carbon nanotubes (CNTs) shows characteristic features confirming the formation of graphitic carbon structures along with residual catalyst signals. A prominent and intense diffraction peak at around 2θ ≈ 25–26° is observed, which corresponds to the (002) plane of graphitic carbon. This peak is a signature of multi-walled carbon nanotubes (MWCNTs) and indicates the presence of well-ordered graphitic layers. The sharpness and high intensity of this peak suggest a relatively good degree of graphitization of the CNTs formed from castor oil.

A broad and low-intensity peak around 2θ ≈ 42–44° is also visible, which is indexed to the (100)/(101) planes of hexagonal graphite. This peak further supports the formation of graphitic carbon, though its broad nature indicates partial disorder or defects within the CNT structure [5, 8]. The broadening of peaks in the XRD pattern indicates that the CNTs possess nanocrystalline or turbostratic structure, which is typical for CNTs synthesized via catalytic chemical vapor deposition (CVD) using natural precursors like castor oil. Additionally, weak and less intense peaks observed at higher angles (around 51–55° and 75–78°) may be attributed to residual NiO or metallic Ni catalyst particles. These peaks confirm that NiO nanoparticles acted as an effective catalyst for CNT growth, although small amounts of catalyst residue may remain embedded within the CNT matrix. The relatively high background and peak broadening suggest the presence of amorphous carbon along with crystalline CNTs, which is common in green synthesis routes.

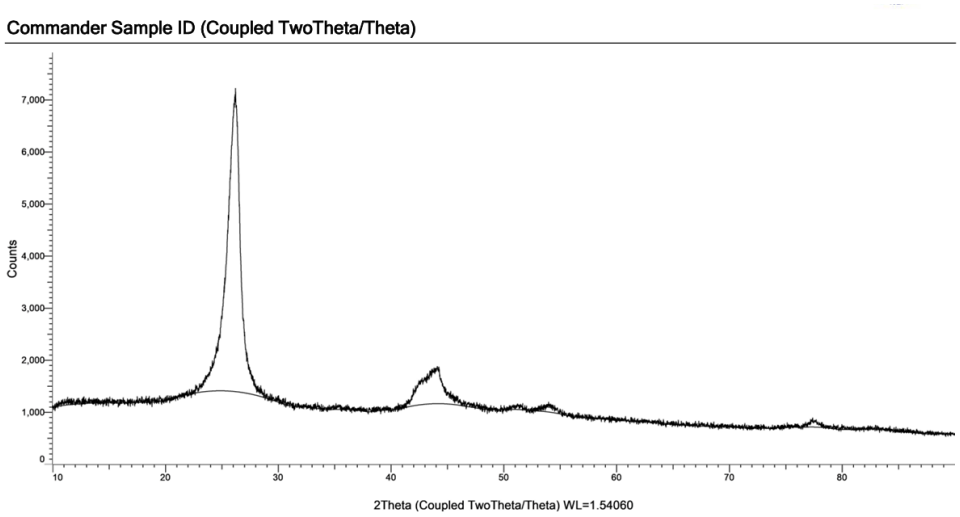

Figure 4 XRD pattern of the CNTs obtained using the castor oil over NiO NPs

4. Conclusion:

This work highlights an environmentally friendly and sustainable approach for the synthesis of carbon nanofibers using castor oil as a renewable carbon source over a green-synthesised nickel oxide catalyst. The use of castor leaf extract and microwave-assisted methods for catalyst preparation significantly reduces chemical usage and energy consumption. The CCVD synthesis at 750 °C in a hydrogen atmosphere produces high-quality CNFs with well-defined morphology. Acid purification effectively removes catalyst residues and introduces functional groups, enhancing CNF applicability. Characterisation techniques such as FT-IR and XRD confirm the structural integrity and quality of both the catalyst and CNFs. Overall, this approach offers a promising pathway for large-scale, sustainable production of carbon nanofibers for advanced technological applications.

The FT-IR analysis confirms that phytochemicals present in castor leaf extract such as phenols, flavonoids, proteins, and carbohydrates are actively involved in the reduction of nickel ions and stabilization of NiO nanoparticles. The presence of a distinct Ni–O band validates the successful formation of NiO nanoparticles. The FT-IR spectrum confirms the successful formation of carbon nanotubes with characteristic graphitic C=C bonds. The presence of minor functional groups such as –OH and C–O indicates slight surface oxidation, which can enhance dispersibility. Residual NiO peaks suggest that catalyst particles may still be present on CNT surfaces, which is common in catalytic growth processes.

The XRD analysis confirms the successful synthesis of pure, crystalline NiO nanoparticles with a face-centered cubic structure using an eco-friendly green synthesis method. The nanoscale crystallite size and absence of impurities demonstrate the effectiveness of *Ricinus communis* leaf extract in producing high-quality NiO nanoparticles suitable for catalytic and electronic applications. The XRD analysis confirms the successful synthesis of multi-walled carbon nanotubes (MWCNTs) using castor oil over NiO nanoparticles. The presence of the strong (002) peak indicates good graphitic structure, while minor peaks corresponding to catalyst residues and broad features suggest a combination of crystalline and amorphous carbon phases.

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