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Synthesis and Characterisation of Carbon Nanofibers from Ricinus communis L. Oil by Chemical Vapour Deposition

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ABSTRACT: Carbon nanofibers (CNFs) are a fascinating class of carbon nanomaterials with unique properties and a wide range of applications. CNFs obtained using plant-based oil (*Ricinus communis* L.) as a sustainable carbon precursor via the Oil Vapor chemical vapor deposition (OV-CVD) method. Iron-based catalysts were placed in a quartz substrate, and the OV-CVD process was carried out at 800°C under a flow of Hydrogen atmosphere. The synthesized CNFs were characterized using Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD) and Thermogravimetric Analysis (TGA). Morphological analysis revealed the formation of entangled fibrous structures with average diameters in the range of 50–70nm and lengths up to several micrometers. Structural analysis confirmed the graphitic nature of the CNFs with varying degrees of defects depending on the synthesis conditions. The high surface area and thermal stability of the synthesized CNFs suggest their potential for Biomedical applications. This work demonstrates a promising pathway for the sustainable production of valuable carbon nanomaterials from readily available biomass resources.

1. INTRODUCTION

Carbon nanomaterials (CNMs) such as carbon nanotubes (CNTs), graphene, and carbon nanofibers (CNFs) have garnered significant attention due to their exceptional mechanical, electrical, thermal, and chemical properties [1, 2]. Among these, carbon nanofibers, characterized by their high aspect ratio, excellent conductivity, and tunable surface properties, find widespread applications in fields ranging from composite materials, catalyst supports, sensors, to energy storage devices [3, 4].

Traditional methods for CNF synthesis, primarily chemical vapor deposition (CVD), involve the use of hydrocarbon gases (e.g., methane, ethylene, acetylene) as carbon precursors [5]. While effective, these precursors are often derived from fossil fuels, contributing to environmental concerns and resource depletion. The growing global emphasis on sustainability and green chemistry necessitates the exploration of renewable and environmentally friendly alternatives for nanomaterial production [6]. Biomass-derived materials, including plant-based oils, offer a compelling sustainable source of carbon due to their abundance, renewability, and relatively low cost. Utilizing such precursors aligns with the principles of a circular economy and reduces the carbon footprint associated with nanomaterial synthesis.

Previous research has explored the use of various biomass sources for carbon material synthesis, including agricultural waste, lignin, and different plant extracts [7-8]. However, the direct synthesis of high-quality carbon nanofibers from plant-based oils via CVD remains an area with significant potential. Plant oils, being rich in carbon and hydrogen, can serve as excellent precursors for carbon material formation, undergoing pyrolysis and decomposition at elevated temperatures to release carbon species required for CNF growth [9-10].

The CVD method is a versatile and scalable technique for synthesizing CNMs, offering precise control over reaction parameters such as temperature, gas flow rates, and catalyst composition, which directly influence the morphology and properties of the

resulting carbon structures [11-12]. The process typically involves the decomposition of a carbon-containing gas over a transition metal catalyst (e.g., Fe, Co, Ni) at high temperatures.

This paper aims to investigate the synthesis of carbon nanofibers from a readily available plant-based oil (e.g., Castor oil) using the CVD method. The primary objectives are to establish optimal CVD conditions for the growth of economical CNFs from plant-based oil over a cobalt oxide catalyst. The findings of this study will contribute to the development of greener and more sustainable pathways for the production of advanced carbon nanomaterials, paving the way for their broader adoption in various industrial and technological applications.

2. MATERIALS AND METHODS

2.1 Materials

The plant-based oil chosen for this study was refined castor oil, purchased from a local supplier. Cobalt (II) nitrate hexahydrate 99.9% purity, Sigma-Aldrich) was used as the catalyst precursor for synthesizing cobalt oxide nanoparticles. High-purity Hydrogen (99.999%) gas was used to maintain an inert atmosphere during the reaction. Ethanol (99.9% purity) and deionised water were used for cleaning.

2.2 CVD Setup

The CVD synthesis was carried out in a horizontal quartz tube furnace (inner diameter 100 mm, length 1 meter) equipped with a resistive heating element. The furnace was connected to a gas delivery system comprising mass flow controllers for precise regulation of Hydrogen gas. A peristaltic pump was used to introduce the liquid plant-based oil into a vaporization unit heated to approximately 200,°C before entering the main reaction zone. The system also included a vacuum pump for purging and a collection system for the synthesized carbon materials. A schematic diagram of the CVD setup is shown in Figure 1.

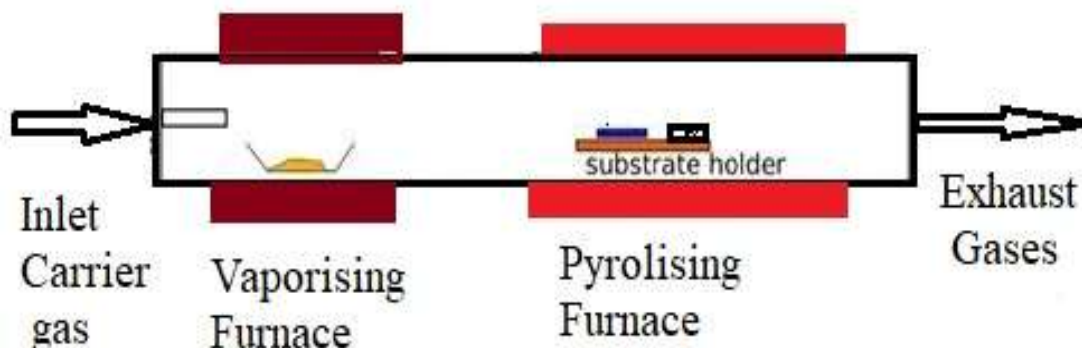


Figure 1 A schematic diagram of the CVD setup for synthesis of CNFs using Castor oil

2.3 Synthesis Procedure

2.3.1 Catalyst Preparation

Weigh 10 g of fresh leaves, grind to a paste and transfer into a 250 mL beaker containing 100 mL distilled water. Heat the mixture at 70–80°C for 30 min. Cool and filter through filter paper. Store the filtrate at 4°C for further use. Dissolve 2.91 g of cobalt nitrate hexahydrate in 100 mL distilled water to prepare a 0.1 M solution. Stir continuously until complete dissolution. Take 50 mL of 0.1 M cobalt nitrate solution in a 250 mL beaker. Add 25 mL of castor leaf extract slowly under continuous stirring. Adjust pH to about 8–9 using dilute NaOH solution. Transfer the reaction mixture into a microwave-safe vessel. Subject the mixture to microwave irradiation at 700 W for 5–10 min with intermittent stirring every 1–2 min. The solution color changes from pink to dark brown or black, indicating the formation of cobalt oxide nanoparticles. Cool the reaction mixture to room temperature and Centrifuge. Wash the precipitate with distilled water followed by ethanol. Dry the obtained material at 80°C overnight. Calcine at 400–500°C for 2 hours in a muffle furnace. The final black powder obtained is crystalline cobalt oxide (Co₃O₄) nanoparticles.

2.3.2 Carbon Nanofibers Growth

The cobalt oxide powder catalyst was placed on a quartz boat inside the horizontal quartz tube. The tube was sealed, and the system was purged with high-purity Hydrogen gas for 30,minutes at a flow rate of 200,scm to remove any residual air. Subsequently, the furnace temperature was raised to the desired reaction temperature (800,°C at a heating rate of 10,°C/min) under a flow. Upon reaching the target temperature, hydrogen gas (50,scm) was introduced for 15,minutes to reduce the Cobalt oxide nanoparticles to active cobalt metal nanoparticles, which catalyze CNF growth. After reduction, the flow of plant-based oil was initiated at a rate of 0.20mL/min into the preheated vaporization unit. The hydrogen flow was maintained at 100,scm as a carrier gas. The reaction was allowed to proceed for 60minutes. After the desired reaction time, the flow of plant-based oil and hydrogen was stopped, and the furnace was allowed to cool down to room temperature under a continuous flow of Hydrogen gas. The synthesised carbon materials were then collected.

3. Results and Discussion

3.1 Morphological Characterization

SEM images (Figure 2a,) clearly show the formation of a dense network of entangled carbon nanofibers on the silicon substrate. The fibers appear relatively uniform in diameter and exhibit typical fibrous morphology. The high density of CNFs suggests efficient carbonization of the plant-based oil precursor under the chosen CVD conditions. The average diameter of the CNFs, estimated from multiple SEM images, ranged from approximately 50nm to 60nm. The lengths of the fibers extended to several micrometres.

Figure 2b - placeholders for TEM images provided more detailed insights into the internal structure and morphology of the individual CNFs. The TEM images confirmed the fibrous nature, showing hollow or partially hollow core structures for some nanofibers, characteristic of vapor-grown carbon nanofibers. The diameters measured from TEM images were consistent with those from SEM, typically ranging from 50 nm to 60nm. The walls of the CNFs appeared graphitic, composed of stacked carbon layers.

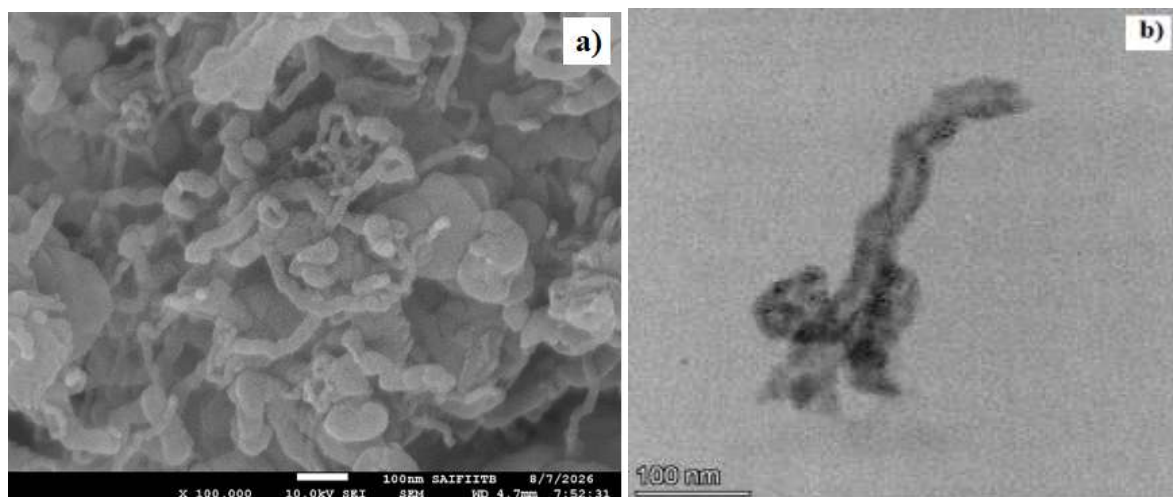


Figure 2 a) Scanning Electron Microscopy and b) Transmission Electron Microscopy images of the CNFs obtained by CVD method using Castor Oil

Figure 3 The X-ray diffraction (XRD) pattern of the carbon nanofibers (CNFs) synthesized by the chemical vapour deposition (CVD) method using castor oil as the carbon precursor is shown in the figure. The analysis was carried out using Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$). The XRD pattern exhibits a prominent diffraction peak at approximately $2\theta = 26.2\text{--}26.5^\circ$, which can be indexed to the (002) plane of graphitic carbon. This reflection arises from the periodic stacking of graphitic layers and confirms the formation of a carbonaceous material with a graphitic structure. Using Bragg's law,

$$n\lambda = 2d \sin \theta$$

the interlayer spacing corresponding to the (002) reflection is approximately 3.4 Å, which is characteristic of graphitic/turbostratic carbon.

A second relatively broad diffraction feature is observed around $2\theta = 43\text{--}45^\circ$, which can be assigned to the (100)/(101) planes of graphitic carbon. This reflection is associated with the in-plane ordering of carbon atoms within the graphitic layers. The broad nature of these reflections indicates that the CNFs possess limited crystallite size and some structural disorder, which is commonly observed in CVD-derived carbon nanofibers. A weak feature at higher diffraction angle, around 77° , may be associated with higher-order graphitic reflections. The absence of several sharp crystalline peaks indicates that the product is predominantly carbonaceous rather than a highly crystalline material.

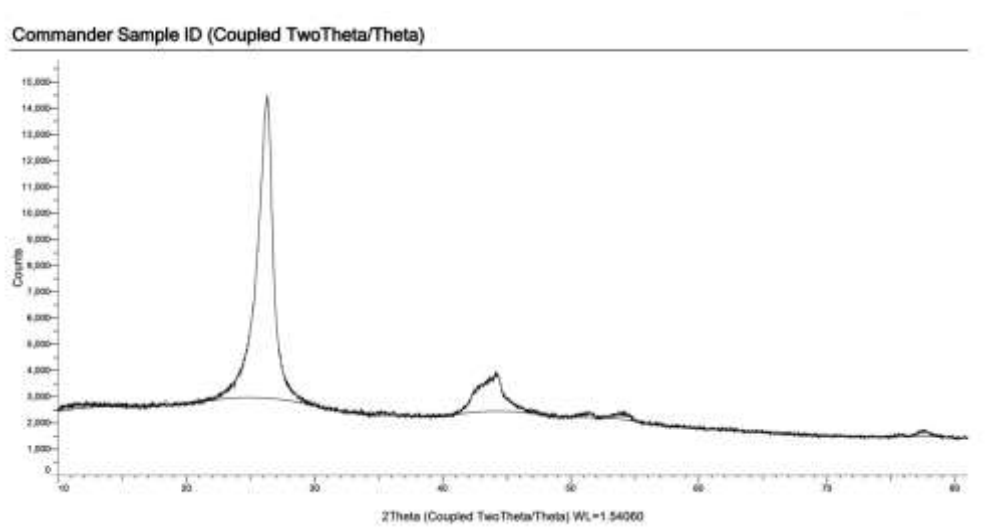


Figure 3 XRD image of the CNFs obtained by CVD method using Castor Oil

Figure 4 the TGA and DTA curves of the carbon nanofibers (CNFs) synthesised by the chemical vapour deposition (CVD) method using castor oil as the carbon precursor provide information about their thermal stability and decomposition behaviour. The TGA curve shows that the CNFs exhibit very little weight loss at lower temperatures, indicating the absence of significant moisture and volatile organic impurities. An initial weight change of approximately 0.12% at 24.4°C is observed, which may be attributed to adsorbed moisture or surface-bound volatile species. As the temperature increases, the sample remains relatively stable up to approximately $500\text{--}550^\circ\text{C}$. A pronounced weight-loss region begins around $550\text{--}600^\circ\text{C}$, followed by a steep decrease between approximately 600 and 700°C . The major degradation occurs near 621°C , as indicated by the DTA curve. The total weight loss is approximately 88.5%, leaving about 11.5% residual mass at $\sim 798^\circ\text{C}$. This residual mass may correspond to metal catalyst residues, metal oxides, and/or inorganic ash remaining from the CVD synthesis.

The DTA curve displays a prominent thermal event with a maximum at approximately **621°C** , corresponding to the major oxidation/decomposition of the carbonaceous material. The pronounced thermal event indicates that the synthesized CNFs possess appreciable thermal stability before undergoing rapid oxidation.

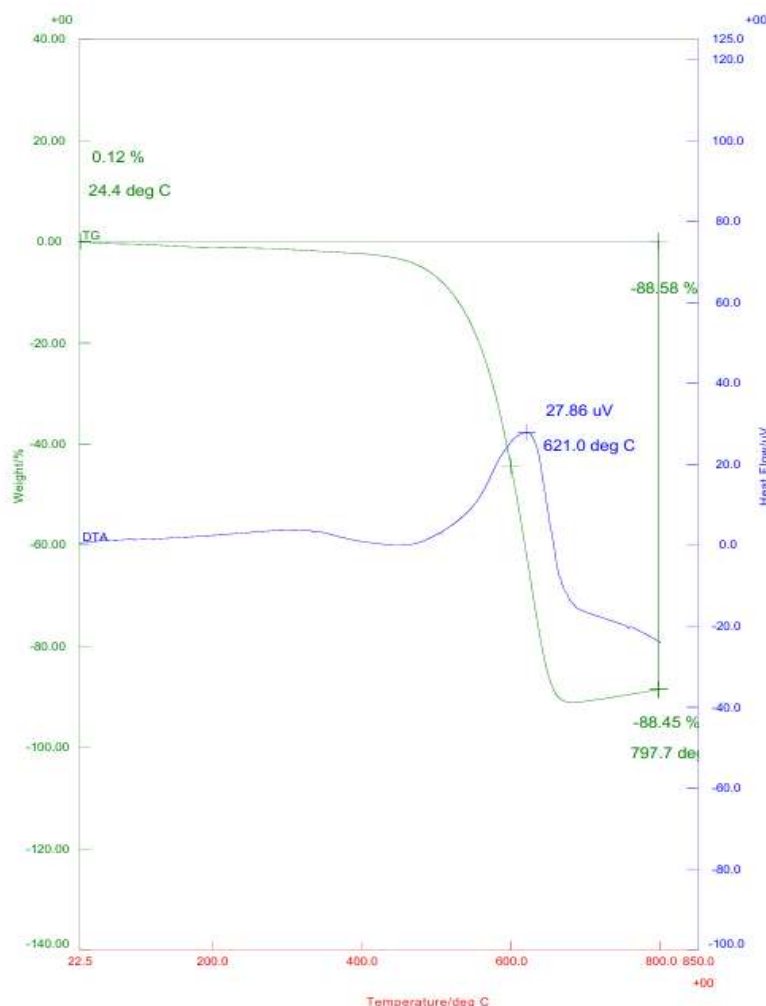


Figure 4. TGA and DTA curves of carbon nanofibers synthesized by the CVD method using castor oil as a carbon precursor

3.2 Effect of Synthesis Parameters

While this paper primarily focuses on demonstrating the feasibility of CNF synthesis from plant-based oil, preliminary experiments indicated that synthesis parameters significantly influence the properties of the resulting CNFs. For instance:

- **Reaction Temperature:** Higher temperatures (e.g., 750–800,°C) generally promote higher graphitization and larger diameters, potentially leading to lower graphitisation, but excessively high temperatures can lead to amorphous carbon formation or catalyst deactivation. Lower temperatures (600–650,°C) tend to produce thinner, more defective fibers.
- **Oil Flow Rate:** An optimal flow rate of the plant-based oil precursor is crucial. Too low a flow rate can lead to insufficient carbon supply, resulting in sparse CNF growth or thin, discontinuous fibers. Too high a flow rate can lead to excessive amorphous carbon deposition on the catalyst and substrate, hindering CNF growth and leading to lower purity.
- **Catalyst Concentration/Particle Size:** The concentration and size of the catalyst nanoparticles play a critical role in controlling CNF diameter and density. Smaller catalyst particles generally yield thinner nanofibers. Further systematic studies are required to comprehensively optimize each of these parameters to tailor the CNF properties for specific applications.

4. CONCLUSION

In this study, we successfully demonstrated the synthesis of carbon nanofibers using plant-based oil as a sustainable and renewable carbon precursor via the Chemical Vapor Deposition (CVD) method. The synthesized CNFs exhibited typical fibrous

morphology with diameters in the range of 50–60nm, as confirmed by SEM and TEM analyses. Structural characterization by XRD and Raman spectroscopy indicated the formation of graphitic carbon with varying degrees of defects, which is characteristic of vapor-grown CNFs. The high thermal stability make these sustainable CNFs attractive for various applications. This work highlights a promising and environmentally benign pathway for the production of advanced carbon nanomaterials from readily available biomass resources, reducing reliance on fossil fuel-derived precursors. The use of plant-based oils not only promotes sustainability but also contributes to the valorization of agricultural products. Future research will focus on further optimising the CVD parameters to control the morphology and structural properties of the CNFs, exploring different plant-based oils, and investigating their performance in specific applications such as electrocatalysis, energy storage, and functional composites.

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