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Synthesis and structural characterization of chitosan-based green nanocomposites reinforced with multi-walled carbon nanotubes

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Abstract

The growing need for sustainable and eco-friendly materials has driven extensive research on green polymer nanocomposites. Among various biopolymers, chitosan (CS) has emerged as a promising candidate due to its biodegradability, renewability, and film-forming ability. When reinforced with multi-walled carbon nanotubes (MWCNTs), chitosan-based nanocomposites exhibit enhanced structural and electrical potential, making them suitable for environmental and sensing applications. This study focuses on the synthesis and structural characterization of CS/MWCNT nanocomposites to explore their potential for sustainable conductive material-related applications. In the present work, green polymer nanocomposites reinforced with multi-walled carbon nanotubes synthesized from three different carbon sources were prepared. Qualitative dispersion behavior of CNTs in the chitosan matrix was evaluated through visual observation during the preparation process. Structural analysis based on X-ray diffraction (XRD) confirmed the successful incorporation of carbon nanotubes into the chitosan matrix and revealed a reduction in chitosan crystallinity due to physical interactions and polymer chain wrapping. These findings demonstrate that MWCNT-reinforced chitosan biopolymer nanocomposites offer promising potential for applications in sustainable conductive materials and green electronic systems.

Keywords: chitosan, carbon nanotubes, dispersion, green nanocomposites, X-ray diffraction
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1. Introduction

In the rapidly advancing field of materials science, polymer nanocomposites have attracted significant research interest due to their ability to exhibit enhanced mechanical, electrical, and functional properties. The ease of processing and tunable structure of polymer matrices allow the incorporation of nanoscale fillers to achieve superior performance compared to conventional composites. In particular, conductive polymer composites formed by the addition of carbon nanotubes (CNTs) to insulating polymers are considered a promising approach for applications in sensor technologies, electronics, and energy systems [1, 2]. In CNT-based nanocomposites, electrical conductivity is governed by tunneling and percolation mechanisms, which strongly depend on the morphology and surface characteristics of the CNTs, as well as the dielectric properties of the polymer matrix [3–4]. In this context, the selection of chitosan as the polymer matrix is of particular importance. As a naturally derived, biocompatible, and film-forming polymer, chitosan offers advantages in terms of environmental sustainability and functional applications [5]. Although many studies have focused on the CNT content, surface functionalization, and concentration, the influence of the carbon source used for CNT synthesis on the electrical properties of the resulting nanocomposites has not been sufficiently investigated. Therefore, the aim of this work is to fabricate chitosan-based nanocomposites reinforced with CNTs synthesized from three different carbon sources and to comparatively discuss their potential electrical conduction behavior based on structural considerations. This study provides new insights into the role of the carbon source in the formation of percolative conductive networks and contributes to the design of environmentally sustainable conductive materials. This paper is organized as follows. In Section 2, the materials used and the experimental procedures for the synthesis of multi-walled carbon nanotubes and the preparation of CS/MWCNT nanocomposites are described. Section 3 presents and discusses the structural characterization results obtained by X-ray diffraction analysis. Finally, the main conclusions of the study are summarized in Section 4.

2. Materials and methods

2.1. Materials

The chitosan used in this study was of commercial origin, and information regarding the manufacturer was not available. The material was used as Received, without any additional chemical treatment. Multi-walled carbon nanotubes (MWCNTs) were synthesized from three different carbon sources (cyclohexane, xylene, and n-heptane) using an aerosol-assisted chemical vapor deposition (CVD) method. A 2 wt.%

acetic acid (CH_3COOH) solution was employed to dissolve chitosan. For the initial dispersion of the MWCNTs, a 1 wt.% solution of the non-ionic surfactant Tween 20 was used. All chemical reagents were used without further purification.

2.2. Synthesis of MWCNTs

Multi-walled carbon nanotubes (MWCNTs) were synthesized from three different carbon sources—xylene, cyclohexane, and n-heptane—using an aerosol-assisted chemical vapor deposition (CVD) method. All syntheses were carried out at a temperature of 950 °C under an Ar/H_2 carrier gas atmosphere. Ferrocene was employed as the catalyst, with a concentration of 20 mg mL^{-1} in the precursor solution. The precursor–ferrocene mixture was converted into an aerosol via ultrasonic dispersion at 800 kHz and subsequently introduced into the reaction zone by the carrier gases. A movable furnace traveling at a speed of 20 mm min^{-1} along a quartz tube enabled the deposition of carbon nanotubes on the inner wall of the tube. After completion of the synthesis, the nanotubes were thermally purified under an argon atmosphere and maintained in an inert environment until the system cooled down. According to the carbon source used, the samples were denoted as Xyl-MWCNTs, CyHex-MWCNTs, and nHep-MWCNTs. A more detailed description of the synthesis methodology has been reported in our previous work [6].

2.3. Preparation of CS/MWCNT Nanocomposites

A total of 400 mg of chitosan was dissolved in 20 mL of a 2 wt.% acetic acid solution under magnetic stirring for 12 h at 25 °C. To ensure better dispersion of the nanotubes within the polymer matrix, the MWCNTs were initially dispersed in a solution of the non-ionic surfactant Tween 20. The CNTs were ultrasonically dispersed in the Tween 20 solution for 50 min using an ultrasonic bath. Subsequently, the homogeneous CNT dispersion was gradually added to the chitosan solution, and the mixture was further processed using magnetic stirring combined with ultrasonic treatment. The resulting mixture was poured into Petri dishes and dried in a low-airflow oven at 30 °C for 72 h, yielding CS/MWCNT nanocomposite films.

3. Results and discussion

3.1. Dispersion behavior of CNTs in the chitosan matrix

The effective distribution of carbon nanotubes within the chitosan matrix plays a crucial role in determining the structural and functional properties of the nanocomposites. Therefore, the dispersion behavior of CNTs was qualitatively evaluated based on visual observations during the nanocomposite preparation process. Figure

1 shows the CNT dispersion in a Tween 20 solution after 50 min of ultrasonic bath treatment, as well as the chitosan/CNT system after the gradual addition of CNTs into the chitosan solution followed by 12 h of magnetic stirring (Figure 1b). After ultrasonic treatment in the presence of the non-ionic surfactant Tween 20, the CNT dispersions exhibited a uniform dark appearance without visible agglomerates, indicating effective debundling of the nanotubes in the liquid medium. Following the gradual incorporation of the CNT dispersion into the chitosan solution and prolonged stirring, visually homogeneous chitosan/CNT mixtures and continuous nanocomposite films were obtained. Although these visual observations do not provide quantitative information on dispersion at the nanoscale, they suggest that the applied dispersion strategy facilitates the distribution of carbon nanotubes within the chitosan matrix. These findings are consistent with the XRD analysis presented in the following section, indirectly confirming the physical wrapping of polymer chains around the carbon nanotubes and weak intermolecular interactions within the polymer matrix.

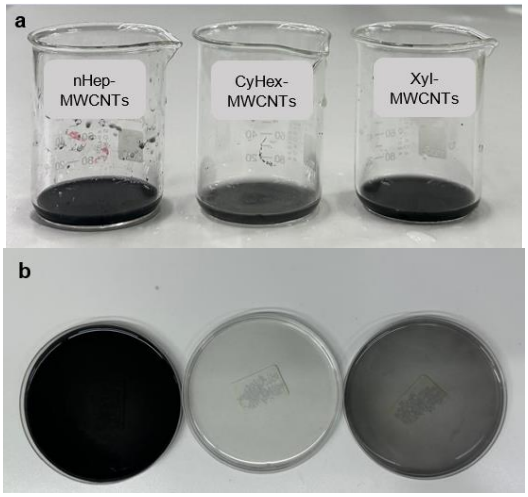

Figure 1. Visual observation of dispersion during nanocomposite preparation: (a) CNT dispersion in Tween 20 solution after 50 min of ultrasonic bath treatment, showing the absence of visible agglomerates; (b) chitosan/CNT system after 12 h of magnetic stirring, resulting in a visually homogeneous mixture and continuous nanocomposite film. These observations provide qualitative evidence supporting effective dispersion of CNTs prior to film formation.

3.2. X-ray Diffraction (XRD) Analysis

Figure 2 presents the XRD patterns of pristine chitosan (CS) and CS/Xyl-MWCNTs, CS/CyHex-MWCNTs, and CS/nHep-MWCNTs nanocomposites prepared by incorporating carbon nanotubes previously characterized in our earlier work [6] into the

chitosan matrix. As observed from the diffraction patterns, pristine chitosan exhibits a sharp diffraction peak in the range of 2θ ≈ 19–21°, which is characteristic of its semi-crystalline structure. In addition, weak diffraction features observed in the 2θ ≈ 30–50° range can be associated with partially crystalline phases of chitosan [7]. In the XRD patterns of the nanocomposites, the characteristic semi-crystalline peak of chitosan is retained; however, compared to pristine chitosan, a noticeable reduction in peak intensity and partial peak broadening are observed. These changes can be attributed to the physical presence of carbon nanotubes between chitosan chains, which restricts the regular packing of the polymer chains and consequently leads to a reduction in the degree of crystallinity of chitosan. The characteristic graphitic (002) reflection of carbon nanotubes at 2θ ≈ 26° appears significantly weakened or masked by the background in the nanocomposite XRD patterns. This behavior can be explained by the good dispersion of carbon nanotubes within the chitosan matrix and the physical wrapping of polymer chains around the CNT surfaces. It should be emphasized that the carbon nanotubes used in this system were not chemically functionalized, and the observed structural modifications arise solely from physical interactions. Overall, the XRD results confirm that the carbon nanotubes were successfully incorporated into the chitosan matrix, and that the CS/MWCNT nanocomposite structure is primarily governed by physical interactions between the polymer chains and the nanotubes.

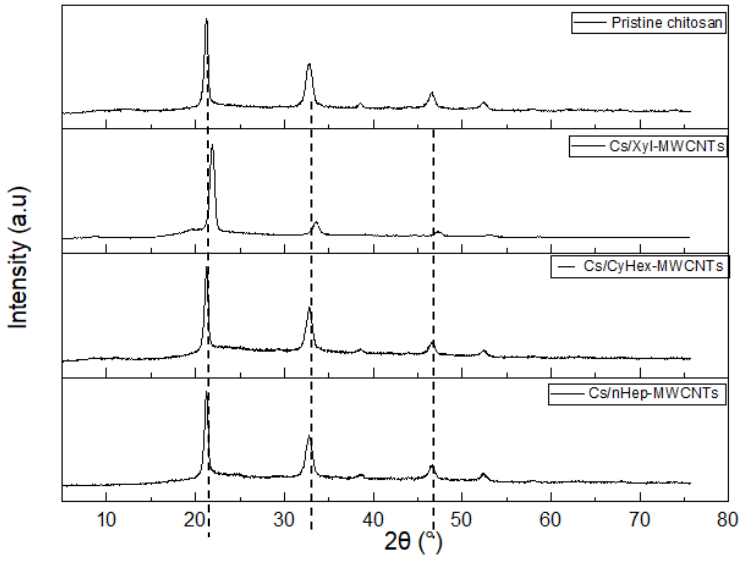

Figure 2. XRD patterns of pristine chitosan (CS) and CS/MWCNT nanocomposites prepared using pristine Xyl-MWCNTs, CyHex-MWCNTs and nHep-MWCNTs previously reported in Ref. [6].

The pristine chitosan exhibits a characteristic semi-crystalline diffraction peak at $2\theta \approx 19–21°$. This peak is retained in all CS/MWCNT nanocomposites, while a noticeable reduction in intensity and peak broadening is observed, indicating a decrease in chitosan crystallinity due to physical interactions and wrapping of CS chains around the carbon nanotubes. The dashed lines highlight the positions of the main diffraction features. The attenuation of the CNT-related (002) reflection (~26°) is attributed to good dispersion and polymer coverage within the chitosan matrix rather than chemical functionalization.

4. Conclusions

In this study, green polymer nanocomposites were fabricated by incorporating multi-walled carbon nanotubes synthesized from three different carbon sources into a chitosan matrix. The XRD results indicated that the carbon nanotubes were effectively incorporated and well distributed within the chitosan matrix without undergoing chemical functionalization, while preserving their graphitic structure. Although the characteristic semi-crystalline peak of chitosan was retained in the diffraction patterns of the nanocomposites, the observed reduction in peak intensity and peak broadening indicate that the regular packing of chitosan chains was physically restricted by the presence of carbon nanotubes. The potential formation of a percolative conductive network within the chitosan matrix through physical wrapping and weak intermolecular interactions between carbon nanotubes and polymer chains highlights the potential of these systems for electrical applications. The obtained results not only elucidate the influence of the carbon source on the structural and functional properties of the nanocomposites but also contribute significantly to the design of environmentally sustainable conductive materials.

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