

Green Synthesis and Characterization of Core-Shell Fullerene-Silver Hybrid Nanocomposite by means of Citric Acid

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ABSTRACT

Hybrid nanomaterials combining metals with fullerene provide unique synergistic properties, enabling the development of a new generation of advanced materials. This study reports the green synthesis and comprehensive characterization of a novel core-shell hybrid nanomaterial composed of silver nanoparticles (Ag NPs) encapsulated within a fullerene-citric acid (FCA) nanocomposite matrix. The FCA nanocomposite was first synthesized via two distinct pathways: a conventional thermal method and a greener enzymatic approach utilizing Novozym 435. This FCA product served a dual function as both a reducing agent and a stabilizer in the subsequent synthesis of silver nanoparticles from silver nitrate. Successful formation of the ester bond in FCA was confirmed by FT-IR spectroscopy, showing a characteristic peak around 1730 cm^{-1} . UV-Vis spectroscopy of the final hybrid revealed two key absorption peaks, one at $\sim 268\text{ nm}$ corresponding to the $\pi\text{-}\pi^*$ transitions of the fullerene moiety and a distinct surface plasmon resonance (SPR) band at $\sim 423\text{ nm}$, confirming the formation of Ag NPs. Electron microscopy (FESEM and TEM) analysis demonstrated spherical nanoparticles with an average size of approximately 25 nm and provided clear evidence of the core-shell structure, where Ag NPs form the core surrounded by the FCA shell. The presented methodology offers an efficient route to stable fullerene-metal hybrid nanostructures with potential for advanced applications in catalysis, sensing, and biomedicine.

INTRODUCTION

Carbon nanostructures, particularly fullerenes (C₆₀), have been a cornerstone of advancement in nanotechnology and materials science since the landmark discovery of C₆₀ in 1985 [1, 2]. These cage-like molecules, comprised of networks of sp² hybridized carbon atoms arranged in spherical, elliptical, or tubular configurations, reside at the forefront of interdisciplinary research due to their exceptional electronic, optical, and mechanical properties [3-5]. C₆₀, with its symmetrical structure and high electron affinity, functions as a "super electron-accepting cage," capable of accommodating multiple electrons and serving as a versatile functional unit in the development of advanced materials [6-9]. Its applications span diverse fields from electronics (e.g., photovoltaic devices and field-effect transistors) to biomedicine (e.g., targeted drug delivery, diagnostic agents, and antioxidants) [10-12]. Parallel to these developments, noble metal nanoparticles, especially silver nanoparticles (Ag NPs), have emerged as one of the most utilized nanomaterials due to their

pronounced surface plasmon resonance (SPR), potent antimicrobial activity, and desirable catalytic properties [13, 14]. However, major impediments to the widespread application of Ag NPs are their inherent tendencies toward aggregation and chemical instability under environmental conditions, which lead to a significant decline in their activity and selectivity [15]. To overcome this challenge, stabilization strategies via coating or the creation of core-shell structures using ligands, polymers, or carbon nanostructures have been developed [16, 17]. Among these approaches, the design of hybrid nanocomposites combining metal nanoparticles and carbon nanostructures represents a promising strategy. In such architectures, each component can compensate for the limitations of the other, leading to novel synergistic properties [18, 19]. For instance, coating silver nanoparticles with fullerene derivatives can prevent aggregation, enhance colloidal stability, improve biocompatibility, and, through enabling electron transfer at the interface, augment the catalytic or sensing properties of the hybrid [20-22]. Citric acid, a

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versatile α -hydroxy acid, plays a pivotal role in the green synthesis of silver nanoparticles [23]. This molecule acts not only as a mild and environmentally benign reducing agent but also, through its carboxylate groups, serves as stabilizing ligands that coat nanoparticle surfaces via electrostatic interactions or coordinate bond formation [24]. Conversely, hydroxylated derivatives of fullerene, known as "fullerenol," possess multiple hydroxyl groups that provide a platform for esterification reactions with carboxylic acids [25, 26]. Therefore, citric acid can function as a "molecular bridge" between fullerenol and silver nanoparticles. This covalent linkage (ester bond) leads to the formation of a stable fullerene-citric acid (FCA) nanocomposite, which itself can act dually as a reducing agent for silver ions (Ag^+) and a stabilizing matrix for the formed silver nanoparticles (Ag^0). The synthesis methods for such complex nanocomposites are also of significant importance. Conventional thermal methods may require harsh conditions, whereas enzymatic synthesis using stable lipases (e.g., Novozym 435) offers a "greener" and more selective pathway under mild temperatures [26, 27]. This enzyme can catalyze the esterification between the carboxylic groups of citric acid and the hydroxyls of fullerenol, exemplifying the convergence of nanotechnology and biotechnology. This study, recognizing this scientific gap and opportunity, focuses on the design, synthesis, and comprehensive characterization of a novel core-shell hybrid nanocomposite, wherein a core of silver nanoparticles is encapsulated within a shell of the fullerene-citric acid nanocomposite. We propose and compare two synthetic routes (thermal and enzymatic) for the preparation of the FCA nanocomposite and subsequently employ it as a dual-function agent (reducing/stabilizing) in the generation of silver nanoparticles. Extensive characterization utilizing FT-IR and UV-Vis spectroscopy, alongside Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM), confirms the successful formation of the ester bond, the reduction of silver ions, the emergence of surface plasmon resonance, and the final core-shell morphology. This research paves the way for developing a new generation of stable hybrid nanomaterials with potential applications in advanced fields such as photocatalysis, bio-chemical sensing, and next-generation antimicrobial agents.

MATERIALS AND METHODS

Materials and Equipment

All chemicals were of analytical grade and used without further purification. Polyethylene glycol (MW=400), sodium hydroxide, toluene, citric acid monohydrate, tetrahydrofuran, dichloromethane, and silver nitrate were purchased from Merck, Germany. Fullerene (C_{60}), Novozym 435 (immobilized lipase from *Candida antarctica*), and dialysis bags with a cut-off >2 kDa were obtained from Sigma-Aldrich. Deionized water was produced by a Milli-Q system. Reactions under vacuum were performed using a vacuum pump and a mechanical stirrer. Syntheses were carried out in appropriate glass vials.

Characterization

FT-IR spectra were recorded using a Nicolet 320 spectrophotometer by preparing KBr pellets from dried samples. UV-Vis absorption spectra in the range of 200 to 800 nm were measured using a Shimadzu UV-1800 spectrophotometer from aqueous suspensions of the samples. The morphology and size distribution of the hybrid particles were examined using a Philips XL-30 FESEM field emission scanning electron microscopy (FESEM). The internal structure and confirmation of the core-shell arrangement were performed using a Philips CM120 Transmission Electron Microscope (TEM) operated at an acceleration voltage of 120 kV.

Synthesis of Fullerene-Citric Acid Nanocomposite (FCA)

Thermal Method

This method was based on modified reported protocols [28]. A mixture of fullerenol (0.1 g) and citric acid monohydrate (1 g) was prepared in a dry glass vial. The vial was connected to a system equipped with a vacuum pump and a mechanical stirrer. The mixture was heated under continuous vacuum and stirring first at 120°C for 30 minutes. The reaction temperature was then slowly raised to 130°C and maintained for 1 hour. Finally, the temperature was increased to 140°C , and the reaction continued for an additional 90 minutes at this temperature. Throughout the process, water produced from the esterification reaction was removed every 10 minutes by the vacuum pump. Upon completion, the product was washed with tetrahydrofuran (THF) and collected by centrifugation at 4500 rpm for 5 minutes. The resulting yellow powder was stored in a desiccator.

Enzymatic Method (Catalyzed by Novozym 435)

For a greener synthesis, an enzymatic catalyst was employed [29]. Fullerenol (0.05 g) and citric acid monohydrate (0.5 g) were first mixed and ground together in a porcelain mortar. This mixture was then dispersed in 100 ml of dichloromethane and homogenized by sonication for 15 minutes. Novozym 435 enzyme (5 mg) was added to the mixture, and the suspension was stirred gently at room temperature (25°C) for 72 hours. After the reaction, the immobilized enzyme biocatalyst was separated and recovered by decanting the solution and washing with dichloromethane. The final product was washed with THF and collected by centrifugation at 4500 rpm for 5 minutes. The resulting yellow powder was dried under vacuum.

Synthesis of Fullerene-Silver Nanoparticle Hybrid (Ag@FCA)

The synthesized FCA nanocomposite (0.2 g) was dissolved in 5 ml of deionized water. The solution was placed under vigorous magnetic stirring at 40°C . Then, a silver nitrate solution (0.42 g in 5 ml deionized water) was added dropwise over 15 minutes to the FCA solution. The color change of the solution to brown after approximately 20 minutes indicated the formation of silver nanoparticles. The reaction mixture was stirred for an additional 1 hour

under the same conditions. The hybrid product was first separated by centrifugation at 8000 rpm for 10 minutes and washed with absolute ethanol. For final purification, the product was dissolved in distilled water and dialyzed against 2 liters of distilled water for 24 hours using a dialysis bag. The final hybrid suspension was stored at 4°C (Fig.1)

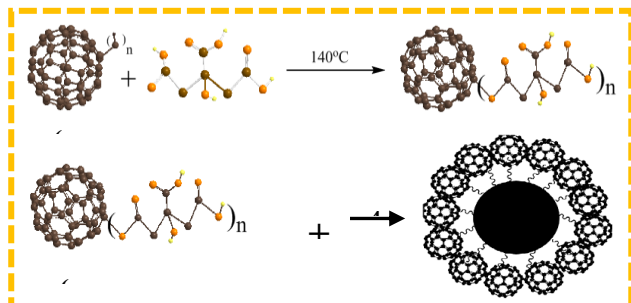


Fig. 1. Schematic of the Synthesis (a) FCA and (b) Ag@FCA

RESULTS AND DISCUSSION

Fig. 2 presents the FT-IR spectroscopy data used to confirm the successful formation of the fullerene–citric acid (FCA) nanocomposite synthesized via two different routes: thermal and enzymatic methods. In panel (a) (thermal synthesis), the FT-IR spectrum of pristine fullerene (C_{60}) exhibits a peak at $\sim 1645\text{ cm}^{-1}$, which is attributed to the $C=C$ stretching vibration of the aromatic carbon framework. The spectrum of fullerenol, a hydroxylated derivative of fullerene, in addition to the $\sim 1645\text{ cm}^{-1}$ band, shows new absorption bands at $\sim 1080\text{ cm}^{-1}$ ($C-O$ stretching), $\sim 1453\text{ cm}^{-1}$ ($C-OH$ vibration), and a broad and intense band at $\sim 3400\text{ cm}^{-1}$ corresponding to the $O-H$ stretching vibration of hydroxyl groups [30, 31]. The FT-IR spectrum of citric acid monohydrate is identified by two characteristic carbonyl bands at ~ 1705 and $\sim 1751\text{ cm}^{-1}$, arising from the $C=O$ stretching vibrations of carboxylic acid groups, along with a broad $O-H$ stretching band around $\sim 3400\text{ cm}^{-1}$ [32]. The spectrum of the FCA nanocomposite (curve D), provides clear evidence for the occurrence of an esterification reaction. The most compelling feature is the appearance of a new and distinct band at $\sim 1730\text{ cm}^{-1}$, which lies in the characteristic region of the ester carbonyl stretching vibration ($C=O$, ester). This band unambiguously confirms the formation of a covalent ester linkage between the carboxyl groups of citric acid and the hydroxyl groups of fullerenol [33]. In addition, the reduced intensity of the broad $O-H$ band at $\sim 3400\text{ cm}^{-1}$ compared to the starting materials indicates the consumption of hydroxyl groups during ester formation. The persistence of the $\sim 1645\text{ cm}^{-1}$ peak further demonstrates that the aromatic fullerene core remains intact throughout the synthesis process.

In panel (b) (enzymatic synthesis catalyzed by Novozym® 435), a similar trend is observed. The FT-IR spectrum of the enzymatically synthesized FCA nanocomposite (curve C) shows the key ester carbonyl band at $\sim 1724\text{ cm}^{-1}$. The presence of this peak, together with the decreased intensity of the $O-H$ stretching region, confirms that the lipase enzyme Novozym® 435

efficiently catalyzes the esterification reaction under mild conditions (room temperature) [34, 35]. The slight shift in the ester carbonyl peak position between the thermal (1730 cm^{-1}) and enzymatic (1724 cm^{-1}) methods may be attributed to differences in the local microenvironment, degree of esterification, or secondary interactions arising from the milder enzymatic conditions.

Overall, Fig. 2 clearly demonstrates that the targeted ester linkage between fullerenol and citric acid is successfully formed via both synthesis routes. The resulting functionalized FCA nanocomposite, possessing suitable surface functional groups, subsequently serves as an effective platform and dual-function agent (reducing and stabilizing) for the synthesis of silver nanoparticles in the next stage of this study.

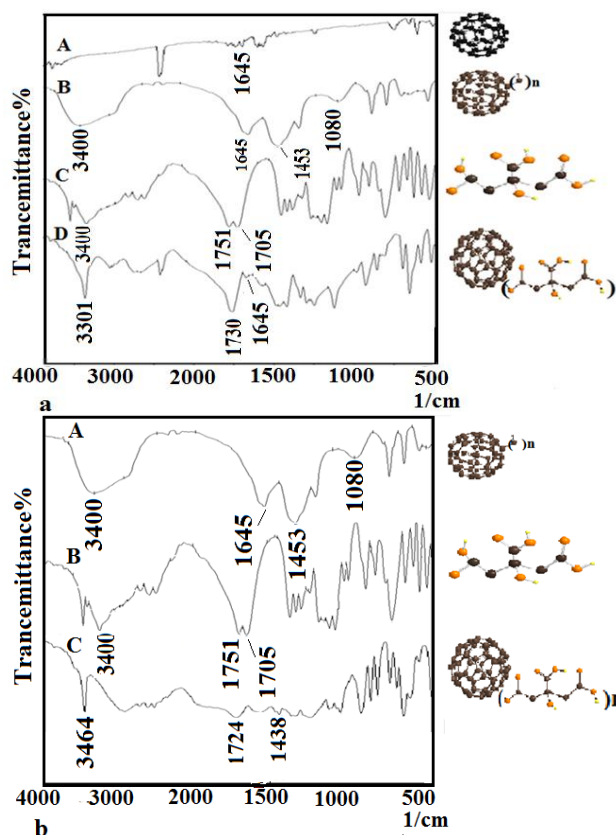


Fig. 2. FT-IR of **a** Thermal method: (A) fullerene C_{60} , (B) Fullerenol, (C) citric acid monohydrate and (D) fullerene-citric acid nanocomposite. **b** enzymatic method: (A) fullerenol, (B) citric acid monohydrate, (C) fullerene-citric acid nanocomposite

Fig. 3 presents the ultraviolet-visible (UV-Vis) absorption spectroscopy data, which are crucial for tracking electronic structure changes and confirming the successful formation of the target nanomaterials at various stages of synthesis.

The absorption spectrum of pure fullerene C_{60} displays two distinct absorption maxima around 286 nm and 332 nm. These characteristic peaks are attributed to $\pi-\pi^*$ electronic transitions within the extensive and conjugated π system of the C_{60} molecule [36]. The high intensity and sharpness of these peaks indicate the purity of the sample and the preservation of the pristine carbon cage structure. In contrast, the spectrum of fullerenol shows significant

changes. The aforementioned peaks are significantly diminished in intensity and shifted towards shorter wavelengths (higher energy). This observation is a direct result of the addition of hydroxyl (-OH) groups to the fullerene core. These groups interfere with the primary π -electronic system, reducing the degree of conjugation and thus weakening the related π - π^* transitions [37]. As expected, citric acid lacks any distinct absorption peaks in the scanned range (approximately 200-800 nm) due to the absence of strong chromophores or a broad conjugated system. The spectrum of the fullerene-citric acid nanocomposite (FCA) shows a broad and weak absorption around 330 nm. The presence of this absorption confirms the continued presence of the π -system derived from fullerene in the nanocomposite architecture. However, its significantly reduced intensity compared to pure C₆₀ provides strong evidence of the successful covalent functionalization of fullerene's surface via ester bond formation, which notably disrupts the original conjugated electronic structure [31].

The UV-Vis spectrum of the final hybrid product provides definitive evidence for the formation of silver nanoparticles. The peak at ~268 nm corresponds to π - π^* transitions associated with the fullerene-based component (shell/matrix) within the hybrid structure. Its presence confirms that the fullerene units remain intact after the silver reduction step and are incorporated into the final architecture. The strong and broad peak at ~423 nm is the most significant spectroscopic evidence in Fig. 3b. The peak indicates a surface plasmon resonance (SPR) band, which is a definitive signature of the formation of colloidal silver nanoparticles (Ag NPs) [30]. The SPR phenomenon occurs when the collective oscillation of conduction electrons at the nanoparticle surface is excited by light of resonant frequency. The exact position (λ_{max}) of the SPR band is highly sensitive to the size, shape, dielectric environment, and degree of aggregation of the metal nanoparticles [38]. The observed band at ~423 nm, along with its relatively broad profile, is consistent with the SPR reported for spherical silver nanoparticles stabilized by organic ligands, suggesting a relatively narrow to moderate size distribution in the synthesized nanoparticles [33]. The simultaneous presence of both characteristic peaks the fullerene-based transition in the UV region and the SPR band for silver nanoparticles in the visible region, in a single spectrum provides compelling spectroscopic evidence for the formation of an integrated hybrid material, rather than a mere physical mixture of its components. This finding strongly supports the core-shell design hypothesis, where the FCA nanocomposite acts as a dual-function agent (reducing and stabilizing), facilitating the formation and encapsulation of silver nanoparticles within its matrix [39]. Minor shifts or changes in the fullerene-related peak in the hybrid, compared to the precursors, may further indicate electronic or physical interactions between the FCA shell and the core silver nanoparticles. In summary, the UV-Vis spectral analysis in Fig. 3 convincingly traces the synthesis pathway from the clear electronic modulation of the fullerene core upon functionalization (in fullerenol and

FCA) to the definitive emergence of the SPR band of silver nanoparticles in the final hybrid. These data, in powerful collaboration with the FTIR results (Fig. 2) and electron microscopy (Fig. 4), provide a multifaceted confirmation of the successful synthesis of the target core-shell hybrid nanostructure (AgNP@FCA).

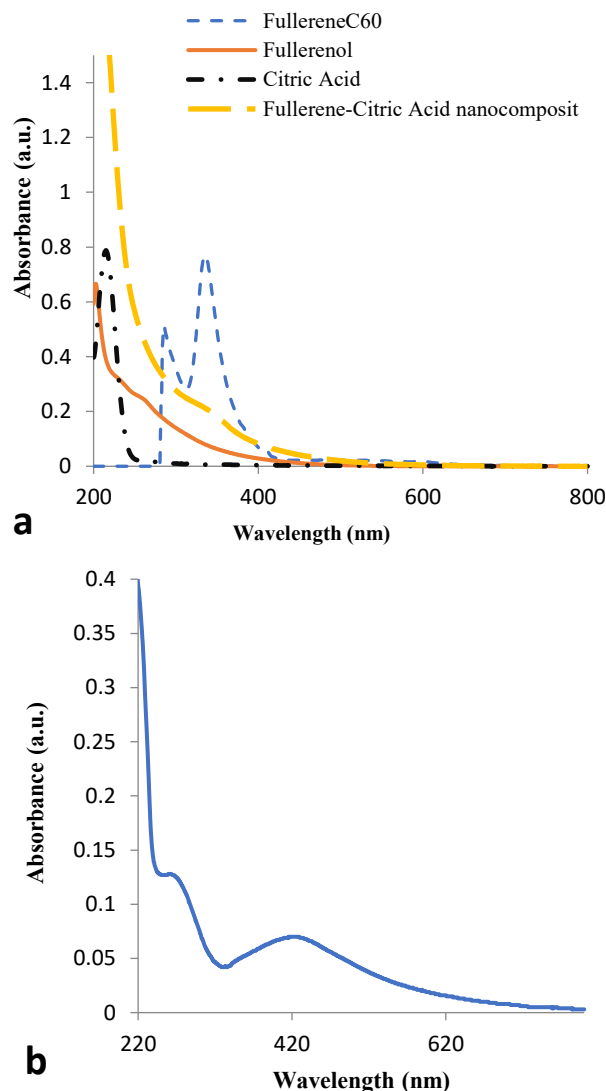


Fig. 3. UV-Vis absorption (a) C₆₀ (dash line), Fullerenol (solid line), Citric Acid (dash dot line), fullerene-citric acid nanocomposite (long dash line) (b) fullerene-Ag nanoparticle hybrid

Fig. 4 presents FESEM and TEM images of the final fullerene-silver nanoparticle hybrid, providing vital information on the morphology, size, and internal structure of the synthesized nanomaterial. The FESEM image provides an overview of the hybrid particle morphology and distribution. As observed, the particles exhibit a spherical and relatively uniform shape. Significant agglomeration is not evident, indicating the effective role of the fullerene-citric acid (FCA) nanocomposite as a stabilizing agent. The functional groups on FCA likely prevent particle adherence through steric hindrance or electrostatic repulsion. Measurements from the SEM image indicate that the average size of these hybrid

nanoparticles is approximately 25 nanometers. The relative uniformity in particle size aligns with the moderately narrow width of the surface plasmon resonance (SPR) band observed in the UV-Vis spectrum (Fig. 3b).

The TEM image, which allows for higher-resolution examination of internal structure, provides direct and clear evidence of a core-shell architecture. This image clearly demonstrates a distinct contrast between two regions with different electron densities. The darker central areas correspond to the silver nanoparticles (Ag NPs), where the higher electron density of silver relative to the surrounding carbonaceous or organic matrix leads to stronger electron scattering and therefore higher contrast in the TEM image [31]. In contrast, the brighter peripheral regions represent the fullerene-citric acid nanocomposite (FCA) shell or matrix. Owing to its lower electron density, this organic-carbonaceous layer allows greater electron transmission, resulting in a noticeably brighter appearance around the dark nanoparticle cores.

This distinct core-shell arrangement clearly demonstrates that the silver nanoparticles (the core) have been successfully encapsulated by the FCA nanocomposite network (the shell) during the synthesis process. This visual observation directly supports the synthesis design hypothesis, wherein FCA acts both as a reducing agent and as a template/stabilizer for the formation of the silver nanoparticles. The average overall size of the core-shell particles in the TEM image is also estimated to be around 25 nm, showing good agreement with the size obtained from the FESEM image and validating the measurement consistency.

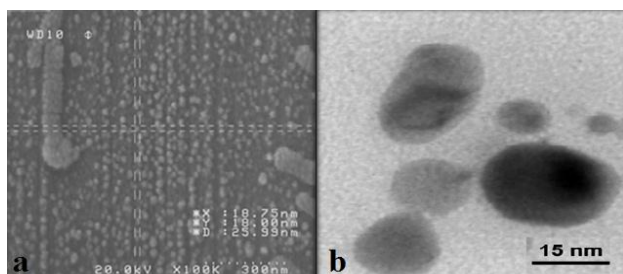


Fig. 4. (a) FESEM and (b) TEM images of fullerene-Ag nanoparticle hybrid

CONCLUSIONS

This study successfully reports the synthesis of a novel core-shell hybrid nanostructure consisting of silver nanoparticles (core) encapsulated within a fullerene-citric acid nanocomposite (shell). The key intermediate, the FCA nanocomposite, was synthesized via an esterification reaction between fullerenol and citric acid using two distinct methods: thermal and enzymatic (catalyzed by Novozym 435). FT-IR spectra conclusively confirmed the formation of the covalent ester bond, evidenced by the characteristic peak in the range of 1730-1724 cm^{-1} . This functionalized nanocomposite subsequently served a dual role as an effective green reducing agent and a stabilizing matrix for the synthesis of silver nanoparticles. UV-Vis spectroscopic evidence, demonstrating the concurrent

presence of the fullerene-derived π - π^* transition peak (~ 268 nm) and the distinctive surface plasmon resonance (SPR) band of silver nanoparticles (~ 423 nm), verified the hybrid's formation and the existence of metallic nanoparticles. FESEM and TEM images directly revealed spherical morphology, a relatively uniform distribution with an average particle size of approximately 25 nm, and, most importantly, the core-shell architecture. The FCA shell effectively prevented the aggregation of silver nanoparticles, providing desirable colloidal stability. In summary, this research presents an efficient and relatively green strategy for designing and fabricating stable metal-fullerene hybrid nanocomposites. The resulting materials, combining the unique properties of silver nanoparticles (e.g., plasmonic activity, antimicrobial effect) and fullerene derivatives (e.g., electron-accepting capability, biocompatibility), hold significant potential for advanced applications in fields such as photocatalysis, sensing, and biomedicine (e.g., drug delivery or antimicrobial agents).

Data Availability Statement

The data that support the results of this study are available from the corresponding author upon reasonable request.

Conflicts of Interest

There are no conflicts to declare.

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