

Functions of CNMs in Photocatalytic H₂ Generation

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To improve photocatalysis, scientists are turning to carbon-based nanomaterials (CNMs) such as graphene, carbon nanotubes (CNTs), graphitic carbon nitride (g-C₃N₄), fullerene (C₆₀), carbon dots (CDs), and carbon nanofibers (CNFs), etc. Carbon-based nanomaterials show exceptional physicochemical properties, including thermal and chemical stability, high surface area, and better electrical, mechanical, and optical properties. Green energy must replace fossil fuels, and hydrogen is a prime choice. Photocatalytic water splitting (PWS) under solar irradiation could address energy and environmental problems.

semiconductor

photocatalysis

water splitting

carbon nanomaterials

1. Structural Support to Improve Structure Sustainability

In semiconductor photocatalysis, different carbon compounds such as CNTs, CDs, graphene, GO, and activated carbon (AC) serve as structural support [1][2][3][4][5]. Carbon nanomaterials offer a larger surface site for the dissemination and immobilization of nanoparticles (NPs). These inert and thermally stable CNMs maintain their structure and properties when coupled with NPs. Numerous defect and active sites (oxygen-based functional groups) present in carbon materials can be exploited to initiate the development and anchoring of homogenous nanoparticles. The carbon materials' light weight is essential for their use as structural support. It is now also possible to produce activated carbon, carbon fibers, CNTs, and graphene on a considerable scale and inexpensively due to the copious amount of carbon in the earth's crust. The presence of these carbon components can change nucleation and growth rates, slowing down semiconductor nanoparticle aggregation and strengthening their architectures [6][7][8]. This section discusses how carbon-based materials stabilize nanostructures. Thus, structural stability applies to component geometries and morphologies. Extremely small nanoparticles, clusters, or other nanostructures that are dispersed and stabilized can substantially increase structural strength. As a result, semiconductor nanoparticles possess a large surface area, numerous surface defects, and a superior contact interface with carbon materials for increased charge transfer. Therefore, carbon-based hybrid semiconductor photocatalysts are more effective at producing H₂ than pure semiconductor photocatalysts. The surface chemistry of carbon materials is greatly impacted by interactions between semiconductor nanoparticles and carbon material. Wang et al. [3] proposed that hydroxyl, carboxyl, carbonyl, and epoxy groups increased the hydrophilic property of the CNTs surface by first treating them with HCl and HNO₃. The Zn_xCd_{1-x}S nanoparticles were then supported by the functionalized CNTs using a solvothermal technique with the help of the raw ingredients including zinc acetate,

cadmium chloride, and thiourea. The well-organized Zn_{0.83}Cd_{0.17}S nanoparticles on the CNTs surface have a diameter of about 100 nm. If CNTs were not there, these nanoparticles would stick together. The excellent dispersion improves the interfacial surface of the Zn_{0.83}Cd_{0.17}S/CNT photocatalyst. Since the conduction band of the photocatalytic material is more populated than the Fermi level of CNTs, photoexcited electrons will move to the surface of the nanotubes. Thus, the aforesaid phenomenon can efficiently separate photoinduced charge carriers at the photocatalyst's interface. In addition, CNT-incorporated Zn_{0.83}Cd_{0.17}S nanocomposite has a lower band gap value than pure Zn_{0.83}Cd_{0.17}S. Under 300 to 800 nm illumination, Zn_{0.83}Cd_{0.17}S/CNT nanocomposite generates 6.03 mmol·h⁻¹g⁻¹ of H₂, which is 1.5 times more than the pristine Zn_{0.83}Cd_{0.17}S.

Li et al. [9] prepared CdS-coupled graphene nanosheets via a hydrothermal approach using GO as supporting material, Cadmium acetate for Cd²⁺ precursor, and DMSO as the solvent as well as source of S²⁻. Solvothermal heating turned GO into graphene with CdS clusters on its surface. The interactions between nanoparticles and graphene can be strengthened by physical adsorption, the electrostatic force of attractions, and charge transference. The homogenous CdS cluster distribution on graphene, the CdS/graphene photocatalyst, has a greater specific surface area. Uniform CdS cluster distribution is attributed to the more effective transport of photoexcited charges to graphene. At 1.0 wt% graphene, 1.12 mmol·h⁻¹ of hydrogen is generated, which is 4.87 times than the neat CdS. Shen et al. [10] also used rGO additive to stabilize the very thin nanorods of Zn_{0.5}Cd_{0.5}S with a corresponding upsurge in photocatalytic H₂ evolution.

2. Cocatalyst

Cocatalysts in a photocatalytic system enhance H₂ evaluation efficacy. Metals such as Pt, Au, or Pd have a more significant work function than semiconductors, making them excellent cocatalysts. Cocatalysts improve charge separation, provide catalytic sites, reduce over-potential, and minimize H₂ activation energy [11][12]. Noble metals are expensive and rare. Thus, economical cocatalysts are needed for economic photocatalytic systems. Carbon compounds are effective cocatalysts for photocatalytic H₂ evaluation [13][14]. The Fermi level of carbonaceous compounds is lesser than CB but RP is greater than H⁺/H₂. Carbon compounds have advantages similar to noble metal cocatalysts, with increased active sites and a localized photothermal effect owing to increased surface area and extended light absorption intensity [15][16]. Khan et al. [13] fabricated a CNTs/CdS/TiO₂/Pt photocatalyst via the hydrothermal method. Pt was designed onto TiO₂ via photodeposition whilst CdS via hydrolysis. This photocatalyst generates H₂ at a greater rate under visible radiations, using sodium sulfide and sulfate sacrificial agents. In the first step, the electrons and holes are separated between the TiO₂ and CdS interface. The photoelectron moves from the TiO₂ to the CNT surface and then Pt catalyst. The Pt nanoparticles work as cocatalysts due to their lower Fermi level of CNTs as compared to TiO₂. Both Pt and CNTs are promising photocatalysts and can improve the photo-catalytic H₂ evaluation of CdS/TiO₂. Preferably, using 0.4 and 4 wt.% of Pt and CNTs, respectively, enhances the photocatalytic performance by 50%. A graphene/MoS₂/TiO₂(GMST) photoccomposite was prepared hydrothermally using Na₂MoO₄, H₂NCSNH₂, and graphene oxide precursors at 210 °C and utilized as an efficient material for photocatalytic H₂ production [17]. It was exposed by using ethanol as a sacrificial agent and TiO₂ as the photocatalyst while graphene and MoS₂ functioned as cocatalysts. The GMST photocatalyst exhibited a high rate

(~165.3 mol·h⁻¹) of H₂ generation at 5.0 and 0.5 wt.% of graphene and MoS₂, respectively, having 9.7% quantum efficiency (QE) at 365 nm [17]. A hybrid rGO/Zn_xCd_{1-x}S photocatalyst prepared first via coprecipitation and then a hydrothermal reduction approach elevates the cocatalytic performance for H₂ production [18]. A high rate of H₂ evaluation (~1824 mol·h⁻¹g⁻¹) was observed for this optimized photocatalyst using 0.25 wt.% of rGO, which has 23.4% apparent QE at 420 nm under solar irradiation. Liu et al. integrated NH₃-treated CDs into g-C₃N₄ by heating CDs and urea for 3 h at 550 °C, where the carbon dots were first synthesized electrochemically and then hydrothermally treated with ammonia. This g-C₃N₄/CDs hybrid photocatalyst was found to be efficient in WS under solar light for the evolution of H₂ and O₂. A maximum QE of 16% was achieved using a g-C₃N₄/CD catalyst at 420 ± 20 nm. Actually, the photocatalytic action of g-C₃N₄ at the first stage caused water to be split into H₂O₂ and H₂, then, at the subsequent stage, the carbon dot-catalyzed breakdown of H₂O₂ into H₂O and O₂.

3. Photosensitization

Carbon compounds with semiconducting or dye-like characteristics can function as a photosensitizer in some cases, enhancing the photoresponse of a broad bandgap of the semiconductor photocatalyst by inducing additional photogenerated electrons [19][20][21]. The emission of photoelectrons from the material can overlap with semiconductor absorption and are responsible for the transfer of the resonance energy from the carbon material to the coupled semiconductor. As the result, the LUMO of the carbon material becomes more negative in comparison to the coupled semiconductor's CB. Wang et al. [22] prepared a graphene/ZnS (G-ZS) photocatalyst via the hydrothermal method for photocatalytic H₂ evolution under visible light. A graphene-supported ZnS photocatalyst evaluated 7.42 mol·h⁻¹g⁻¹ hydrogen at 0.1 wt.% graphene, which was found to be 8 times that of the pristine ZnS. Since visible light cannot excite ZnS, the photoelectron most likely originates from graphene and migrates to the CB of ZnS [19]. The rGO/Pt/TiO₂ photocatalyst is an excellent photosensitizer and evaluates H₂ at a rate of 11.24 mmol·h⁻¹g⁻¹. Carbon dots are excellent candidates for use as photosensitizers in photocatalytic reactions due to their high photo-absorption and photoluminescence properties. For instance, Martindale et al. [23] fabricated a carbon dot (photosensitizer)-based Ni-bis- (diphosphine) (NiP) photocatalyst for H₂ generation under visible light. To produce this photocatalyst, 30 nmol of NiP and 2.2 nmol of CD are used in 0.1M EDTA solution with pH 6; a photocatalyst g-CQDs/NiP produces H₂ at 398 mol·h⁻¹g⁻¹. The apparent QE at 360 ± 10 nm was observed ~1.4%. Carbon dots can convert near-infrared photons for semiconductor photocatalysis due to their photoluminescence up-conversion property [21][24]. Hydrogenated TiO₂(H-TiO₂)-based photocatalysts were prepared with carbon quantum dots (CQDs) under bath reflux. This photocatalyst is UV-visible-NIR compatible and showed remarkable photocatalytic H₂ production capability under the illumination of a 300 W Xe arc lamp. For this photocatalytic system, Pt was used as the cocatalyst and methanol as the sacrificial agent across a wide spectral range. The higher H₂ generation rate of 7.42 mmol·h⁻¹g⁻¹ was observed in comparison to H-TiO₂ nanobelts (6.01 mmol·h⁻¹g⁻¹) [24].

4. Photocatalyst

Carbon-based nanomaterials are effective photocatalysts for H₂ generation, according to theory and experiment [25] [26] [27], owing to the semiconducting properties of CDs, CNTs, graphene, GO, rGO, and C₆₀. Such a nanocarbon of the semiconductor sort can have a greater number of negatively charged LUMO sites than H⁺/H₂ RP. Reduced graphene oxide rGO is a commonly used photocatalyst [28] [29], as its CB minimum consists of anti-bonding π^* orbital; at pH = 0, it has a potential of -0.52 eV [30]. Density functional theory (DFT) studies explain the electronic structure of graphene oxide by alternating the relative ratio of -OH and epoxy groups present on the surface. The resulting electronic structure shows the photocatalytic hydrogen, and oxygen evaluation reaction occurs [31]. Teng's group reported that GO synthesized by a modified Hummers' method had an apparent direct E_g value of 3.3–4.3 eV and an indirect E_g of 2.4–3.0 eV. Graphene oxide (GO) is comprised of graphene molecules of different oxidation stats and can produce H₂ under UV/Visible illuminations. In a 20% methanol aqueous solution with 0.5g GO and no cocatalyst, mercury lamp irradiation yielded 17,000 mmol·h⁻¹ H₂ [27]. Meng et al. [29] prepared a p-MoS₂/n-rGO photocatalyst with p-type MoS₂ deposited on the surface of n-type rGO. Under solar irradiation and ethanol as sacrificial agents, this photocatalyst exhibited higher H₂ evaluation activity than bare MoS₂ and MoS₂/rGO. This is because, as shown by the photoelectrochemical experiment, the p-MoS₂/n-rGO junctions are very good at separating charges. Zhu's team compared H₂ production activity for neat CDs and CD-based composite materials [26] [32] [33]. An H₂ evaluation at a 423.7 mol·h⁻¹g⁻¹ was achieved using carbon dots in pure water without needing a cocatalyst. Carbon dots were created hydrothermally from MWCNTs oxide [26]. In methanol sacrificial agent and Pt cocatalyst, a hybrid carbon nanodot/WO₃ photocatalyst produces H₂ at 1330 mol·h⁻¹g⁻¹ under xenon lamp irradiation [32].

5. Band Gap Narrowing Effect

Chemical bonds can form when semiconductor photocatalysts and carbon compounds make strong contacts (e.g., metal O C bonds). Chemical bonding reduces photocatalyst band gaps, increasing H₂ production [34] [35]. Ye et al. [36] reported the hydrothermal fabrication of graphene/CdS (0.01:1) and CNTs/CdS (0.05:1) hybrid materials with the photocatalytic H₂ evaluation of and 52 mol·h⁻¹, respectively. The addition of graphene or CNTs into CdS resulted in the narrowing of the E_g values of these hybrid photocatalysts and consequently led to superior photocatalytic H₂ generation as compared to pristine CdS. This was in addition to the benefit of more effective charge separation. Bi₂WO₆ is often not used for water-splitting H₂ generation, due to its smaller RP and comparatively less negative CB. However, coupling Bi₂WO₆ nanosheets with graphene, the CB of Bi₂WO₆ becomes more negative, and the feasibility of photocatalytic H₂ evaluation increases [37]. In a typical experiment [38], Bi₂WO₆ nanoparticles were produced via sonication onto the graphene's sheets using GO, HNO₃, (NH₄)₁₀W₁₂O₄₁, and Bi(NO₃)₃·5H₂O, followed by calcination at 450°C for 3h in an inert environment. Raman and XPS research validated the chemistry between Bi₂WO₆ and graphene. After coupling with graphene, Mott–Schottky calculations showed that the RP of CB of Bi₂WO₆ increased from +0.09 V to -0.30 V as compared to the standard hydrogen electrode. As a result, 0.03 g of Bi₂WO₆/graphene photocatalyst was used to produce 159.2 mol·h⁻¹ of H₂ at 420 nm in an aqueous solution of methanol.

In conclusion, in several aspects, carbon materials can significantly enhance the H₂ reaction rate compared to semiconductor photocatalysts. The inclusion of various carbonaceous materials coupled with semiconductor photocatalysts can result in a variety of proficient effects including structural support for improved structure constancy, electron collection, the reduction of the recombination rate of photo-stimulated charge pairs, photocatalyst, E_g narrowing outcome cocatalyst, and photosensitization. It is essential to remember that certain carbon materials can potentially play many functions throughout the entire photocatalytic process. Moreover, due to some limitations imposed by its structure and properties, a particular type of carbon material might not be able to fulfill all the functions. The potential characteristics of various CNMs are summarized in **Table 1**.

Table 1. Role of carbon nanomaterials in photocatalysis.

Function Type	Carbon Nanotubes	Graphene	Fullerenes	Graphene Oxide	Graphitic Carbon Nitride	Carbon Quantum Dots
Supporting material	✓	✓	✗	✓	✓	✓
Photocatalyst	✓	✓	✓	✓	✗	✓
Cocatalyst	✓	✓	✓	✓	✓	✓
Photosensitizer	✗	✓	✓	✓	✓	✓
Bandgap narrowing effect	✓	✓	✗	✓	✓	✓

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