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Sol-Gel Production Of Unmodified And Lanthanum-Modified $BiFeO_3$ Nanomaterials And Photoelectrochemical Studies Of Hydrogen Evolution

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In this study, pure and lanthanum-doped bismuth ferrite ($La_xBi_{1-x}FeO_3$,x=0.00 and 0.10) nanomaterials were synthesized using a simple and cost-effective sol–gel method. This technique offers better control over particle size, morphology, and phase purity of the prepared materials. The structural, morphological, and optical properties of the synthesized samples were investigated using various characterization techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), and UV–visible spectroscopy. The XRD analysis confirmed the successful formation of the

$BiFeO_3$ phase and indicated improved phase purity in the lanthanum-doped sample. SEM micrographs revealed noticeable changes in surface morphology after doping, showing reduced agglomeration and more uniform particle distribution.The photocatalytic performance of both pure and doped materials was evaluated under UV–visible light irradiation. The experimental results showed that the synthesized materials exhibit good photocatalytic activity for hydrogen generation through photoelectrochemical water splitting. Furthermore, the lanthanum-doped $BiFeO_3$ sample demonstrated enhanced photocatalytic “performance compared to the pure $BiFeO_3$ material. This improvement is mainly attributed to better structural properties, reduced impurity phases, and improved charge carrier separation in the doped material. The rate of hydrogen evolution during the photoelectrochemical process was measured using the inverted burette method. Overall, the results suggest that lanthanum-doped $BiFeO_3$ is a promising material for efficient solar-driven hydrogen production and renewable energy applications.

Keywords: $BiFeO_3$, Photocatalysts, perovskite, Scanning electron micrograph, surface morphology.

1. Introduction

Solar energy is one of the most abundant and sustainable energy resources available on Earth. With the continuous depletion of fossil fuels and the increasing environmental problems caused by their use, the development of renewable and clean energy technologies has become a global priority. Among the various renewable energy sources, solar energy is particularly attractive because it is inexhaustible, environmentally friendly, and widely available. One promising approach for utilizing solar energy is its conversion into chemical energy in the form of hydrogen fuel. Hydrogen is considered an ideal clean energy carrier because it produces only water as a by-product during combustion and has a high energy density. Therefore, developing efficient methods for hydrogen production using solar energy has gained significant attention in recent years. Photoelectrochemical (PEC) water splitting is an important technique that enables the direct conversion of solar energy into hydrogen by splitting water molecules into hydrogen and oxygen using semiconductor materials.[1]

In photoelectrochemical systems, semiconductor photocatalysts play a crucial role because they absorb solar radiation and generate electron–hole pairs that drive the water splitting reaction. However, the efficiency of PEC hydrogen production largely depends on the optical and electronic properties of the photoelectrode materials. An ideal photocatalyst should possess several key characteristics, including a suitable band gap for visible light absorption, high chemical stability in aqueous environments, efficient charge separation, and low recombination rates of photogenerated charge carriers. Many semiconductor materials have been investigated as photocatalysts for PEC water splitting. Among them, titanium dioxide (TiO_2) is one of the most extensively studied materials due to its excellent chemical stability, low cost, and resistance to photocorrosion. However, TiO_2 has a relatively large band gap of about 3.2 eV, which restricts its absorption mainly to the ultraviolet region of the solar spectrum. Since ultraviolet light constitutes only a small fraction of solar radiation, the overall solar-to-hydrogen conversion efficiency of TiO_2-based systems remains limited. This limitation has encouraged researchers to explore alternative semiconductor materials with narrower band gaps that can effectively utilize visible light.[2-3]

In recent years, bismuth ferrite ($BiFeO_3$) has attracted considerable attention as a potential photocatalyst for photoelectrochemical applications. $BiFeO_3$ is a multiferroic perovskite oxide material that exhibits both ferroelectric and antiferromagnetic properties at room temperature. One of the major advantages of $BiFeO_3$ is its relatively narrow band gap of approximately 2.2–2.3 eV, which allows it to absorb a significant portion of visible light. In addition, $BiFeO_3$ possesses excellent chemical stability and resistance to photocorrosion, making it a suitable candidate for photocatalytic and photoelectrochemical processes. Despite these promising properties, the practical performance of pure $BiFeO_3$ as a photocatalyst is often limited by several factors, including rapid recombination of photogenerated electron–hole pairs, poor charge transport properties, and the presence of secondary impurity phases during synthesis. These limitations reduce the overall efficiency of hydrogen generation in PEC systems.

To overcome these challenges and enhance the photocatalytic performance of $BiFeO_3$, various modification strategies have been explored. One effective approach is the doping of $BiFeO_3$ with suitable elements. Doping can modify the electronic structure of the host material, improve charge carrier mobility, suppress recombination of electrons and holes, and adjust the band gap to enhance light absorption. Among the different dopants investigated, lanthanum (La) has been widely studied due to its ability to improve the structural stability and electrical properties of $BiFeO_3$. Substitution of La^{3+} ions at the Bi^{3+} site can reduce the formation of impurity phases, alter the crystal structure, and improve the morphology of the material. These modifications can significantly enhance the photocatalytic and photoelectrochemical performance of the material, leading to improved hydrogen evolution efficiency.Another important factor influencing the properties of photocatalytic materials is the synthesis method. Different synthesis techniques can lead to variations in particle size, morphology, crystallinity, and phase purity, which directly affect the photocatalytic activity. Among various synthesis methods, the sol–gel technique is widely used for the preparation of nanostructured materials because it offers several advantages such as low processing temperature, good compositional control, high purity, and uniform particle distribution. The sol–gel method also allows easy incorporation of dopant ions into the host lattice, making it particularly suitable for preparing doped photocatalytic materials.In this study, pure $BiFeO_3$ and lanthanum-doped $BiFeO_3$ nanomaterials were synthesized using a simple sol–gel method. The structural, morphological, and photoelectrochemical properties of the prepared materials were investigated using techniques such as X-ray diffraction (XRD) and scanning electron microscopy (SEM). Furthermore, the photocatalytic performance of the synthesized materials was evaluated through photoelectrochemical measurements under illumination. The primary objective of this work is to compare the hydrogen generation efficiency of pure and La-doped $BiFeO_3$ and to understand the effect of lanthanum doping on the structural and photocatalytic properties of $BiFeO_3$ for potential applications in solar hydrogen production.[4]

2. Synthesis of Pure and La-Doped $BiFeO_3$

Pure bismuth ferrite ($BiFeO_3$) and lanthanum-doped bismuth ferrite (La–$BiFeO_3$) nanomaterials were synthesized using a simple and cost-effective sol–gel method. All the chemicals used in the synthesis process were of analytical grade with a purity of 99.9% and were used without further purification. The required precursor materials included bismuth nitrate pentahydrate [$Bi(NO_3)_3 \cdot 5H_2O$], iron nitrate nonahydrate [$Fe(NO_3)_3 \cdot 9H_2O$], lanthanum nitrate hexahydrate [$La(NO_3)_3 \cdot 6H_2O$], distilled water, and a few drops of concentrated nitric acid (HNO_3) to maintain solution stability.[5]

For the synthesis of pure $BiFeO_3$ (BFO), a stoichiometric amount of bismuth nitrate pentahydrate was first dissolved in 50 mL of distilled water under continuous stirring. The solution was heated to approximately 80 °C to ensure complete dissolution of the precursor. Since bismuth tends to volatilize during the high-temperature synthesis process, an additional 5 wt% of bismuth nitrate was added to compensate for possible bismuth loss during annealing. After about ten minutes of stirring, the required stoichiometric quantity of iron nitrate nonahydrate was gradually added to the solution. To stabilize the solution and prevent precipitation, a few drops of concentrated nitric acid were introduced while maintaining continuous stirring.The resulting homogeneous solution was further heated to around 120 °C with constant stirring until it gradually transformed into a viscous gel. This gel formation indicates the completion of the sol–gel process, where the metal ions are uniformly distributed at the molecular level. The obtained gel was then dried in an air oven to remove excess solvent and obtain a dry precursor powder.The dried powder was ground thoroughly using a mortar and pestle to achieve uniform particle distribution. The powder was then pressed into pellet form to facilitate heat treatment.

These pellets were subsequently annealed in a furnace at 500 °C for two hours to promote crystallization and formation of the $BiFeO_3$ phase.For the synthesis of lanthanum-doped $BiFeO_3$ (BLFO), a similar procedure was followed. In this case, an appropriate stoichiometric amount of lanthanum nitrate hexahydrate was added along with the precursor solution to achieve a doping concentration of 10% lanthanum at the bismuth site. The remaining steps, including gel formation, drying, pelletization, and annealing, were carried out under identical conditions to ensure consistent synthesis parameters. This method resulted in the successful formation of both pure and La-doped $BiFeO_3$ nanomaterials suitable for further structural and photoelectrochemical investigations.[6]

$BiFeO_3$ as a Photocatalyst for Hydrogen Production

$BiFeO_3$ (Bismuth Ferrite) is a promising semiconductor material belonging to the perovskite oxide family. It crystallizes in a rhombohedral structure with the R3c space group and exhibits a relatively narrow band gap of about 2.2–2.3 eV, which allows it to absorb a significant portion of visible light from the solar spectrum. Due to these properties, $BiFeO_3$ has attracted considerable attention as a potential photocatalyst for photoelectrochemical (PEC) water splitting and solar hydrogen production.

In PEC systems, $BiFeO_3$ is commonly used as a photoanode material. When the material is exposed to light irradiation, photons with sufficient energy excite electrons from the valence band to the conduction band, generating electron–hole pairs. These charge carriers participate in oxidation and reduction reactions at the electrode–electrolyte interface, leading to the splitting of water molecules into hydrogen and oxygen.

The generated electrons move toward the cathode to produce hydrogen, while holes participate in oxygen evolution reactions.Despite these advantages, the photocatalytic efficiency of pure $BiFeO_3$ is often limited by several factors. These include structural defects, formation of secondary impurity phases such as $Bi_{25}FeO_{40}$, and rapid recombination of photogenerated electron–hole pairs. Therefore, improving the structural stability and charge separation efficiency of $BiFeO_3$ remains essential for enhancing its overall photocatalytic performance in hydrogen generation applications.[7]

Modification Strategies for $BiFeO_3$

Various strategies have been developed to improve the photocatalytic performance of $BiFeO_3$, particularly for photoelectrochemical water splitting. One effective method is metal ion doping, which alters the structural and electronic properties of the material.

Metal Ion Doping:

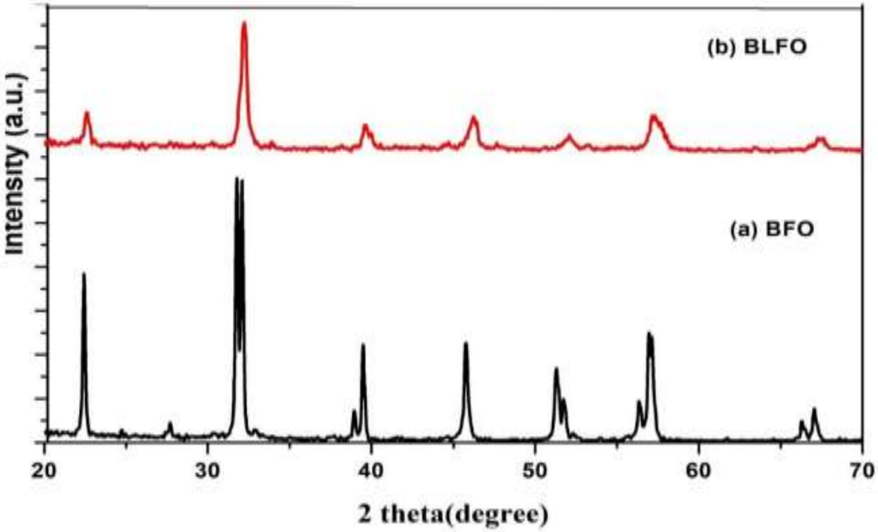
Doping $BiFeO_3$ with rare-earth elements such as lanthanum (La), neodymium (Nd), samarium (Sm), and gadolinium (Gd) has been widely studied. These dopant ions substitute Bi^{3+} ions in the crystal lattice and modify the structural and electronic characteristics of the material. Among these elements, lanthanum doping has shown particularly promising results. The incorporation of La^{3+} ions reduce lattice distortion, improves crystallinity, and suppresses the formation of unwanted impurity phases.

Additionally, La doping enhances the separation and mobility of photogenerated electron–hole pairs, which reduces recombination losses. As a result, La-doped $BiFeO_3$ exhibits higher photocurrent density and improved photocatalytic activity, leading to enhanced hydrogen evolution efficiency compared to pure $BiFeO_3$.[8]

2. Result and discussion

3.1 X-Ray Diffraction (XRD)

The X-ray diffraction (XRD) patterns of pure $BiFeO_3$ (BFO) and lanthanum-doped $BiFeO_3$ (BLFO) are presented in Figure 1. The diffraction peaks observed for the pure BFO sample confirm the successful formation of the $BiFeO_3$ phase with a rhombohedral crystal structure belonging to the R3c space group. However, a small impurity peak was detected near $2\theta \approx 27°$, which corresponds to the oxygen-rich secondary phase $Bi_{25}FeO_{40}$. In contrast, the XRD pattern of the La-doped BFO sample shows noticeable structural changes. Two high-intensity peaks associated with the Bi site merged into a single peak after the incorporation of 10% lanthanum. This indicates a structural transformation from rhombohedral symmetry to orthorhombic symmetry due to La substitution. Additionally, no impurity peaks were observed in the doped sample, indicating improved crystallinity and phase purity.[9-10]



The X-ray diffraction (XRD) patterns of pure $BiFeO_3$ (BFO) and lanthanum-doped $BiFeO_3$ (BLFO) are shown in Figure 1. The XRD profile of pure BFO, presented in Fig. 1(a), confirms the successful formation of the $BiFeO_3$ phase. The diffraction peaks can be indexed to a rhombohedral crystal structure with the R3c space group [12]. However, along with the main phase, a few minor impurity phases were also observed. A small intensity peak appearing near $2\theta \approx 27°$ corresponds to the oxygen-rich impurity phase $Bi_{25}FeO_{40}$. The XRD pattern of La-doped BFO is shown in Fig. 1(b). After doping with 10% lanthanum, two high-intensity peaks corresponding to the Bi site merged into a single peak. This merging indicates that La doping causes a structural transition from rhombohedral to orthorhombic symmetry [11]. Additionally, no impurity phases were observed in the doped sample, suggesting improved phase purity.

3.2 Scanning Electron Microscopy (SEM)

The surface morphology of pure $BiFeO_3$ (BFO) and lanthanum-doped $BiFeO_3$ (BLFO) was examined using scanning electron microscopy (SEM), and the obtained micrographs are shown in Figure 2(a) and 2(b). The SEM image of pure BFO reveals that the particles are highly agglomerated and irregular in size and shape. This agglomeration may occur due to strong particle–particle interactions during the synthesis process. In contrast, the SEM image of the La-doped BFO sample shows noticeable changes in the particle morphology. The particles appear more uniform, relatively spherical in shape, and less agglomerated compared to the pure BFO sample. These observations suggest that the incorporation of lanthanum influences the nucleation and crystal growth process during synthesis. As a result, the surface morphology of the material is significantly modified. Since surface morphology plays an important role in photocatalytic activity, these structural improvements may enhance the overall photocatalytic performance of the doped material.[12]

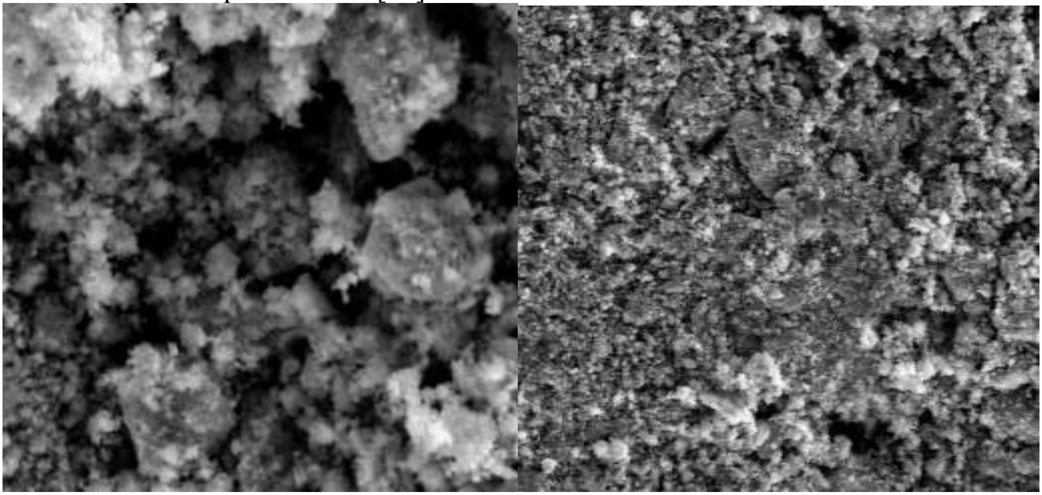


Fig 2 (a)Scanning electron micrograph (SEM) of pure BFO

Fig;2 (b)Scanning electron micrograph (SEM) of La doped BFO

The synthesized material was analyzed using scanning electron microscopy (SEM) to study its surface morphology, as shown in Figure 2(a) and 2(b). The SEM images of pure $BiFeO_3$ (BFO) reveal that the particles are agglomerated and distributed in different sizes. This clustering of particles may occur due to strong interactions between the nanoparticles during the synthesis process. After doping with lanthanum, noticeable changes in morphology were observed. The La-doped BFO particles appear less agglomerated and exhibit a more spherical shape compared to the pure sample. This indicates that the incorporation of lanthanum influences the nucleation, growth, and crystallization process of the material. The SEM observations demonstrate that introducing a small amount of dopant into the host lattice can significantly modify the surface morphology. Since surface structure plays a crucial role in photocatalytic reactions, such morphological changes can positively influence the photocatalytic performance of the material.

4. Photoelectrochemcal measurements

The photoelectrochemical (PEC) performance of pure $BiFeO_3$ (BFO), La-doped $BiFeO_3$ (BLFO), and their graphene composite thin films was evaluated using a standard three-electrode system. The prepared thin films were used as photoanodes and were fixed onto Perspex sheets containing a circular opening of 1 cm diameter using Araldite adhesive. The adhesive was allowed to cure completely to ensure proper attachment and stability of the films during measurements. To establish an electrical connection, a copper wire was attached to the back side of the Ti foil supporting the thin film using silver paint, which provided an effective ohmic contact.The photoelectrochemical experiments were carried out using a three-electrode configuration consisting of the prepared BFO-based photoanodes as the working electrode, a platinum foil with a diameter of 1 cm as the counter electrode, and a saturated calomel electrode (SCE) as the reference electrode. The electrolyte used for the measurements was a 0.5 mol/L NaOH aqueous solution, which provides a suitable alkaline environment for the electrochemical reactions.For illumination, a 150W, xenon (Xe) lamp was used as the light source to simulate the solar spectrum during the measurements. The photoelectrochemical measurements were performed using a PG302B potentiostat/galvanostat to record the current–voltage characteristics of the photoanodes under both dark and illuminated conditions. The rate of hydrogen evolution was determined by collecting the gas produced at the cathode using an inverted burette method. The measurements were carried out at an applied potential of 0 V versus the saturated calomel electrode (SCE). [14]

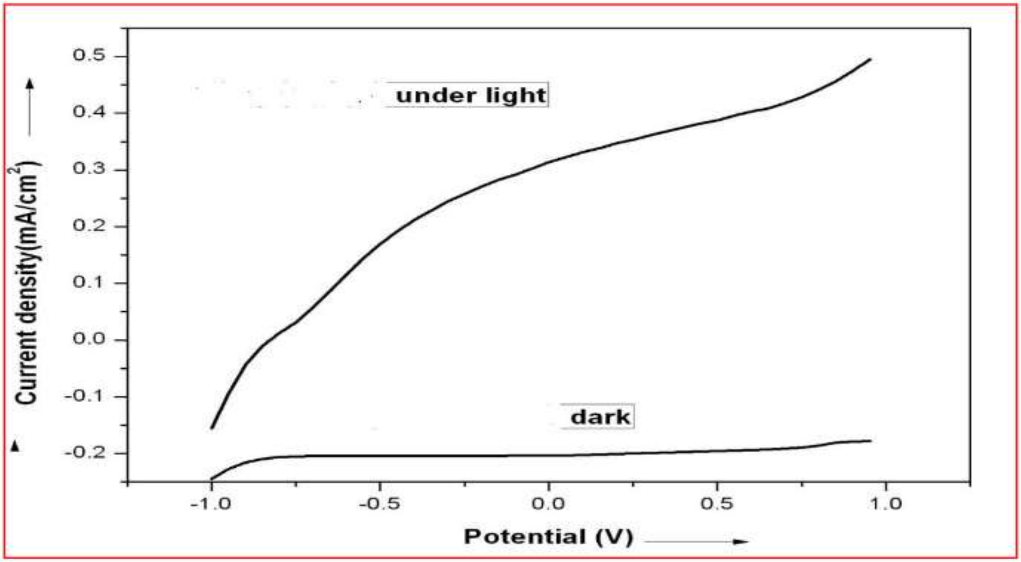


Fig.3 Photoelectrochemical I-V measurements (a) under dark condition (b) under illumination of BLFO

The photoelectrochemical I–V characteristics of the BLFO photoanode are shown in Figure 3. Under dark conditions, the current density is very low, indicating minimal charge carrier activity in the absence of light. However, when the photoanode is exposed to solar illumination, a significant increase in current density is observed. This enhanced photocurrent response indicates that the photoanode effectively absorbs light and generates charge carriers. The increase in current density reflects the strong photoactivity of the semiconductor material and suggests its suitability for solar hydrogen production. Higher photocurrent density is directly related to increased hydrogen evolution during the photoelectrochemical process [12]. The improved solar spectral response of the La-doped BFO photoanode may be attributed to the improved crystal structure and the absence of impurity phases, which enhance charge separation and reduce recombination losses.

5. Photoelectrochemical I–V Measurements

The photoelectrochemical current–voltage (I–V) characteristics of the lanthanum-doped $BiFeO_3$ (BLFO) photoanode are shown in Figure 3. These measurements were carried out to evaluate the photoresponse and hydrogen generation capability of the synthesized material. Under dark conditions, the observed current density is very low, indicating that very few charge carriers are generated in the absence of light. However, when the photoanode is illuminated with light, a significant increase in current density is observed. This rise in photocurrent demonstrates the strong photoactivity of the semiconductor material under illumination.

The increase in photocurrent indicates that the BLFO photoanode efficiently absorbs solar radiation and generates electron–hole pairs, which participate in the photoelectrochemical water splitting process. Efficient separation and transport of these photogenerated charge carriers lead to enhanced hydrogen evolution at the cathode. Therefore, higher current density directly corresponds to a higher rate of hydrogen production.[13]

The improved performance of the La-doped $BiFeO_3$ photoanode can be attributed to several factors. Lanthanum doping enhances the crystallinity of the material, reduces the presence of impurity phases, and improves charge carrier separation by minimizing recombination losses. As a result, the doped material exhibits a better solar spectral response and improved photoelectrochemical performance compared to the pure $BiFeO_3$ sample. These characteristics make La-doped $BiFeO_3$ a promising material for efficient solar hydrogen production.[11]

Conclusions

In this study, pure $BiFeO_3$ (BFO) and lanthanum-doped $BiFeO_3$ (BLFO) Nanomaterials were successfully synthesized using a simple and cost-effective sol–gel method. This synthesis technique proved to be efficient in producing materials with controlled composition and improved structural properties. The prepared samples were characterized using various analytical techniques to examine their structural, morphological, and photoelectrochemical properties.

The X-ray diffraction analysis confirmed the formation of the $BiFeO_3$ phase with good crystallinity. In the case of the pure BFO sample, a small impurity phase was detected, whereas the La-doped sample showed improved phase purity with no noticeable impurity peaks. This indicates that lanthanum doping plays an important role in stabilizing the crystal structure and improving the overall structural quality of the material. The scanning electron microscopy (SEM) images revealed significant changes in surface morphology after lanthanum doping. Pure BFO particles were found to be highly agglomerated and irregular in shape, while the La-doped BFO particles appeared more uniform, less agglomerated, and relatively spherical in shape. Such improvements in morphology can enhance the surface area and active sites available for photocatalytic reactions.

The photoelectrochemical measurements demonstrated that the La-doped BFO photoanode exhibits a much higher current density under illumination compared to dark conditions. This enhanced photocurrent response indicates that the doped material has a stronger solar spectral response and improved photoactivity. The increase in current density suggests more efficient generation and separation of photogenerated charge carriers, which directly contributes to enhanced hydrogen evolution during the photoelectrochemical water-splitting process.the results of this study demonstrate that lanthanum doping significantly improves the structural, morphological, and photoelectrochemical properties of $BiFeO_3$. The enhanced photocurrent and improved material properties suggest that La-doped $BiFeO_3$ is a promising photoanode material for efficient solar-driven hydrogen production and future renewable energy applications.

Challenges and Future Perspectives

Although $BiFeO_3$-based photocatalysts have shown significant potential for solar-driven hydrogen production, several challenges still limit their practical application. One of the main issues is the low quantum efficiency, which reduces the overall conversion of solar energy into hydrogen. In addition, instability under long-term illumination can degrade the material performance during prolonged photoelectrochemical operation. Another major challenge is the difficulty in controlling phase purity during synthesis, as unwanted secondary phases may form and negatively affect photocatalytic activity. Furthermore, rapid recombination of photogenerated electron–hole pairs remains a critical factor that reduces the efficiency of the water-splitting process.To overcome these limitations, future research should focus on developing improved material design strategies. The fabrication of multi-component heterostructures can enhance charge separation and extend light absorption. Integrating $BiFeO_3$ with two-dimensional (2D) materials, such as graphene or transition metal dichalcogenides, may significantly improve electrical” conductivity and electron transport. Additionally, optimizing nanostructure morphology can increase surface area and provide more active catalytic sites. Improvements in charge transport mechanisms are also essential for enhancing photocurrent generation. Advanced synthesis techniques, combined with theoretical modeling and computational studies, may further improve the solar-to-hydrogen conversion efficiency of $BiFeO_3$-based photocatalysts, enabling their practical use in sustainable energy technologies.

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