

CHAPTER I

INTRODUCTION

1.1 Significance of the study

As societies undergo industrialization and population growth, the demand for energy resources such as fossil fuels, including coal, oil, and natural gas, has been significantly increased. These resources have been extensively combusted for electricity generation, transportation, and various industrial processes. As a consequence, toxic gases released from industrial activities and vehicular emissions have been introduced into the atmosphere, contributing substantially to air pollution. This pollution has been associated with respiratory illnesses and other adverse health effects among populations residing in contaminated environments (Dockery and Pope, 1994; Organization, 2020). In parallel, the direct discharge of industrial effluents and organic waste into natural water bodies has been widely practiced, leading to severe water pollution. As a result, the prevalence of water-borne diseases has been increased, and access to clean and safe drinking water has been critically limited for millions of people worldwide (Organization, 2023; Schwarzenbach, Egli, Hofstetter, Von Gunten and Wehrli, 2010). In addition to air and water pollution, the continuous accumulation of carbon dioxide (CO₂) in the atmosphere has been identified as a major contributor to global warming and climate change. These phenomena have produced profound impacts on natural ecosystems, global climate patterns, and sea-level rise (Change, 2013). In recognition of the urgency of these environmental challenges, the period from 2021 to 2030 has been designated by the United Nations as the “Decade on Ecosystem Restoration.” This global initiative highlights the necessity of mitigating environmental degradation and restoring damaged ecosystems (Canton, 2021). Achieving ecological sustainability has therefore been identified as a critical global

challenge, requiring strategies that satisfy present societal needs while preserving the capacity of future generations to meet their own demands (Brundtland, 1987).

Among renewable energy sources, the Sun has been recognized as an abundant and powerful source of energy, with an energy output far exceeding current global consumption. Solar energy has been widely regarded as a key component in the transition toward sustainable and environmentally friendly energy systems. Solar radiation can be harvested through multiple technologies, including photovoltaic systems, which directly convert sunlight into electricity. In addition, photoelectrochemical and photocatalytic processes have been developed to utilize solar energy for applications such as water splitting, hydrogen production, and environmental remediation, including the degradation of organic pollutants (Fujishima and Honda, 1972; Hoffmann, Martin, Choi and Bahnemann, 1995; Irshad et al., 2022; Lewis and Nocera, 2006).

Photocatalysis is defined as a process in which a chemical reaction is accelerated by a catalyst under light irradiation, where light serves as the primary energy source (Fujishima et al., 1972). This process is governed by the interaction between incident photons and the photocatalyst material, which enables various chemical transformations to occur. In photocatalytic systems, a photocatalyst material plays a central role by absorbing photons and generating photogenerated electron–hole pairs. These charge carriers migrate to the surface of the photocatalyst, where they participate in redox (reduction–oxidation) reactions involving electron transfer, ultimately driving chemical conversions (Hoffmann et al., 1995). Photocatalysis has been widely investigated for a range of environmentally beneficial applications, including the degradation of water pollutants and the conversion of abundant and stable molecules such as H_2O , CO_2 , and N_2 (Chong, Jin, Chow and Saint, 2010; Hisatomi, Kubota and Domen, 2014; Lewis et al., 2006). Through these processes, valuable products such as hydrogen gas (H_2), hydrocarbon fuels (e.g., methane and methanol), and ammonia (NH_3) can be produced. In particular, CO_2 reduction and nitrogen fixation via photocatalytic routes have been recognized as promising strategies for environmental remediation and sustainable chemical production (Chen, Shen, Guo and

Mao, 2010; Wang and Domen, 2019). Collectively, these applications represent viable approaches for environmental purification and resource sustainability.

However, many conventional photocatalysts exhibit high activity only under ultraviolet (UV) light irradiation, which imposes a significant limitation on practical applications due to the low UV fraction in natural solar radiation (Linsebigler, Lu and Yates Jr, 1995). Consequently, the development of photocatalytic materials capable of efficiently utilizing visible light has become a major research focus. Among the investigated materials, $\text{PbBi}_2\text{Nb}_2\text{O}_9$ has been identified as a promising visible-light-responsive photocatalyst. Its electronic structure can be effectively tuned through elemental substitution or doping, enabling enhanced photocatalytic performance for a wide range of applications (Zhang and Zhu, 2012; Zou, Ye, Sayama and Arakawa, 2001).

In this work, the photocatalytic activity of $\text{PbBi}_2\text{Nb}_2\text{O}_9$ was enhanced through cerium (Ce) doping. The primary objective of this study was to systematically investigate the effects of Ce incorporation on the crystal structure, electronic properties, and photocatalytic performance of $\text{PbBi}_2\text{Nb}_2\text{O}_9$ -based materials. Particular emphasis was placed on evaluating the photocatalytic degradation of methylene blue as a model reaction to elucidate the role of Ce doping in improving photocatalytic efficiency.

1.2 Photocatalysis

Photocatalysis is a chemical process in which a photocatalyst is employed to accelerate and facilitate chemical reactions under light irradiation. This process has attracted significant attention in the field of sustainable and green chemistry due to its strong potential for addressing environmental challenges, including water purification and the production of clean and renewable fuels (Fujishima et al., 1972; Hoffmann et al., 1995; Irshad et al., 2022).

Photocatalytic reactions are surface-driven processes that occur on the surface of the photocatalyst material. The fundamental photocatalytic mechanism begins with the absorption of photons by the photocatalyst. For effective initiation of photocatalytic reactions, the energy of the absorbed photons must be equal to or

greater than the band gap energy of the photocatalyst ($h\nu \geq E_g$). Upon photon absorption, electrons are excited from the valence band to the conduction band, resulting in the formation of photogenerated electron–hole pairs (e^- and h^+). These charge carriers subsequently migrate to the surface of the photocatalyst, where they participate in redox reactions with adsorbed species, leading to the degradation of pollutants or other chemical transformations (Chen and Mao, 2007; Schneider et al., 2014). The overall photocatalytic degradation process is schematically illustrated in Figure 1.1.

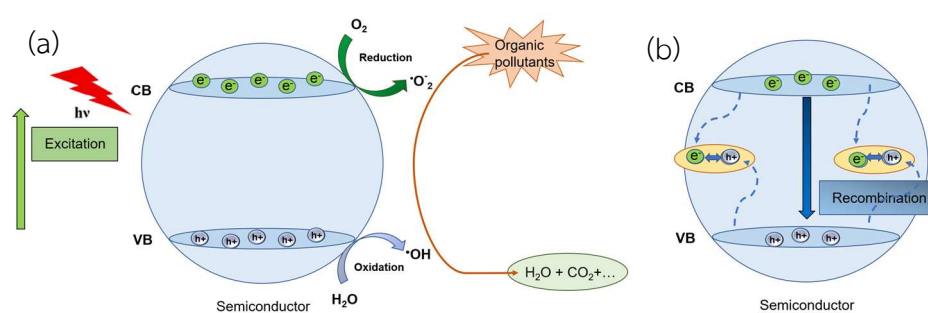
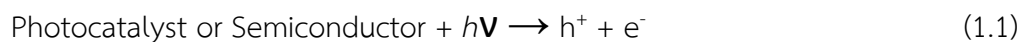


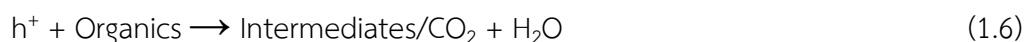
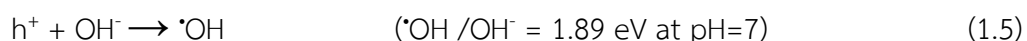
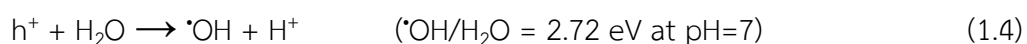
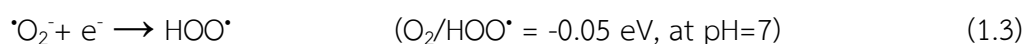
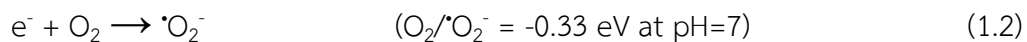
Figure 1.1 Photocatalytic degradation process (a), e^- and h^+ recombination (b).

The photogenerated electron–hole pairs are highly reactive species and serve as the key intermediates in photocatalytic reactions. Upon light irradiation, photoexcitation occurs when a semiconductor photocatalyst absorbs photons with energy equal to or greater than its band gap energy, resulting in the generation of free charge carriers, as expressed in Equation (1.1):

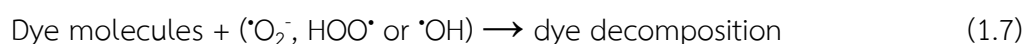


Once generated, the electron–hole pairs are able to migrate to the surface of the photocatalyst, where they participate in a series of redox (reduction–oxidation) reactions with adsorbed species. The photogenerated electrons can react with molecular oxygen adsorbed on the catalyst surface, leading to the formation of superoxide radicals ($\cdot O_2^-$) and hydroperoxyl radicals ($HOO\cdot$), while the photogenerated

holes can oxidize either organic pollutants directly or surface-adsorbed water molecules and hydroxide ions to produce highly reactive hydroxyl radicals ($\cdot\text{OH}$) (Kong, Yang, Rui and Ji, 2019). These elementary reactions are described in Equations (1.2) - (1.6):



In the context of photocatalytic degradation, the reactive oxygen species (ROS) generated on the photocatalyst surface subsequently react with dye molecules. Through these reactions, complex organic dye molecules are broken down into smaller intermediates and, in some cases, completely mineralized into CO_2 and H_2O , as illustrated in Equation (1.7):



The nature of the pollutant species present in the reaction system plays a crucial role in determining the dominant photocatalytic degradation pathway. Cationic organic dyes, such as methylene blue (MB) (Houas et al., 2001; Jing, Wang, Zhang, Wang and Li, 2014; Vasiljevic et al., 2020) and Rhodamine B (RhB) (Vijaya Sankar and Ashok, 2020), are generally degraded predominantly through reduction processes, in which photogenerated electrons participate in the reduction of the positively charged dye molecules. In contrast, anionic dyes, such as methyl orange (MO) (Cerrato, Gaggero, Calza and Paganini, 2022), are more commonly degraded via oxidation pathways, where photogenerated holes or hydroxyl radicals act as the primary oxidizing agents.

One of the major challenges in photocatalysis is the recombination of photogenerated electron-hole pairs, as described in Equation (1.8). When recombination occurs prior to charge carrier participation in surface redox reactions, the absorbed photon energy is dissipated as heat or light, resulting in a significant loss of photocatalytic efficiency. Therefore, suppressing charge carrier recombination and promoting efficient charge separation are critical objectives in the design and optimization of high-performance photocatalysts.



1.3 Photocatalyst

The core component of the photocatalytic process is the photocatalyst material, which is most commonly a semiconductor. Semiconductor materials are employed because of their ability to absorb light energy and generate photogenerated electron-hole pairs that drive subsequent redox reactions. Widely studied photocatalysts include titanium dioxide (TiO₂), zinc oxide (ZnO), and a variety of metal oxides and sulfides, owing to their suitable electronic structures, chemical stability, and relatively low cost (Fujishima et al., 1972; Hoffmann et al., 1995; Linsebigler et al., 1995).

In semiconductor photocatalysts, the absorption of photons promotes electrons from the valence band to the conduction band when the photon energy is equal to or greater than the band gap energy of the material. Consequently, the magnitude of the band gap determines the range of light wavelengths that can be absorbed and utilized for photocatalytic reactions. Many conventional photocatalysts, such as TiO₂ and ZnO, possess wide band gaps and are therefore primarily active under ultraviolet (UV) irradiation, which represents only a small fraction of the solar spectrum (Chen et al., 2007).

To enhance photocatalytic efficiency and extend light absorption into the visible region, various modification strategies have been widely adopted. These strategies include surface modification, elemental doping, coupling with co-catalysts, and the construction of nanostructured materials. Doping with metal or non-metal

elements is often employed to tailor the electronic structure and reduce the band gap energy, thereby improving visible-light absorption and charge carrier separation (Asahi, Morikawa, Ohwaki, Aoki and Taga, 2001; Kudo et al., 1988). The introduction of co-catalysts can further enhance surface reaction kinetics by providing additional active sites and facilitating charge transfer. In addition, the fabrication of nanostructured photocatalysts can increase the specific surface area and shorten charge carrier diffusion distances, which helps suppress electron–hole recombination and improve overall photocatalytic performance (Schneider et al., 2014).

1.4 Perovskite material

Perovskite materials have attracted significant attention in the fields of materials science, physics, and photovoltaics due to their remarkable physicochemical properties and broad potential for energy and environmental applications. These materials are defined by a characteristic crystal structure known as the perovskite structure, which is typically described by the general chemical formula ABX_3 . In this structure, the B-site cation is located at the center of an octahedron formed by X-site anions, while the A-site cation occupies the interstitial spaces between the octahedra. The A-site is commonly occupied by alkaline earth metals or lanthanides, the B-site by transition metals, and the X-site by an anion, most frequently oxygen (Irshad et al., 2022). A schematic illustration of the perovskite crystal structure is shown in Figure 1.2.

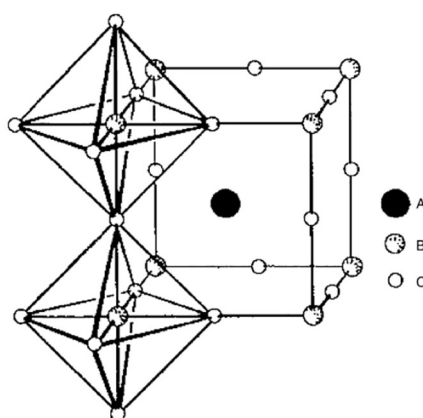


Figure 1.2 Perovskite structure illustrated for ABO_3 (Li, Soh and Wu, 2004).

In recent years, perovskite materials have also been increasingly explored in the field of photocatalysis due to their unique and tunable properties. These materials have been demonstrated to possess adjustable band gap energies, strong light absorption capabilities, and favorable charge carrier transport characteristics, which are critical factors for efficient photocatalytic performance (Kudo and Miseki, 2009). Similar to conventional semiconductor photocatalysts, perovskite materials can absorb photons to generate electron–hole pairs that subsequently participate in surface redox reactions.

One of the most attractive features of perovskite materials is the high degree of compositional flexibility offered by the ABX_3 structure. By substituting different elements at the A, B, and X sites, the electronic structure and band gap energy of perovskite materials can be systematically tuned. This tunability enables the design of perovskite photocatalysts with absorption edges that match specific regions of the solar spectrum, thereby enhancing visible-light-driven photocatalytic activity. In addition, many perovskite materials exhibit strong absorption in the visible and near-infrared regions, allowing efficient utilization of a broad range of solar wavelengths. The relatively high charge carrier mobility reported in many perovskite systems further contributes to improved charge separation and reduced recombination losses during photocatalytic reactions (Snaith, 2013).

Despite these advantages, the practical application of perovskite materials in photocatalysis remains challenged by their limited stability under operational conditions. Many perovskite materials are sensitive to moisture, thermal stress, and prolonged light irradiation, which can lead to structural degradation and performance loss. As a result, considerable research efforts have been directed toward improving the chemical and structural stability of perovskite photocatalysts through compositional engineering, surface modification, and the development of protective architectures (Li et al., 2016; Niu, Guo and Wang, 2015; Zhou et al., 2020).

Although perovskite-based photocatalysis is still a relatively emerging research area, it has demonstrated significant promise as an environmentally friendly and efficient approach for addressing energy conversion and environmental remediation challenges. Ongoing research is focused on optimizing perovskite compositions,

enhancing long-term stability, and improving scalability to facilitate their practical deployment in real-world photocatalytic applications.

1.5 Sol-Gel method

The sol-gel method is a versatile and widely used chemical process for the synthesis of various materials, including nanomaterials and catalysts. It is a technique that involves the transformation of a solution (sol) into a gel-like or solid material (gel) through controlled chemical reactions. The sol-gel process can be used to create a wide range of materials with diverse properties and applications (Vasiljevic et al., 2020; Vijaya Sankar et al., 2020; Wu et al., 2012).

1.5.1 The basic steps in the Sol-Gel process

1. The process begins with the preparation of a solution, often containing metal alkoxides, metal chlorides, or other precursor compounds. This solution, known as a sol, typically consists of metal ions or molecules dispersed in a solvent.

2. The sol undergoes a chemical reaction, typically hydrolysis and condensation reactions, which result in the formation of a three-dimensional network of interconnected particles or polymers. This network transforms the liquid sol into a gel, which is a semi-solid or solid-like material.

3. After gelation, the material is allowed to age or mature for a period of time, during which further chemical reactions and structural changes can occur. Following aging, the gel is carefully dried to remove the solvent, leaving behind a solid material.

4. Depending on the desired properties and applications of the material, heat treatment (calcination) may be performed at elevated temperatures. This step can help to enhance the crystallinity and stability of the final product.

1.5.2 The key advantages of the Sol-Gel method

1. The sol-gel process allows for precise control over the composition and uniform distribution of components in the material.

2. It can be used to produce a wide range of materials with different compositions and properties.

3. The sol-gel method often yields highly pure materials with low levels of impurities.

4. The structure and properties of the final material can be controlled by adjusting parameters such as precursor composition, solvent, pH, and processing conditions.

5. The sol-gel process can be used to create nanomaterials and nanocomposites with unique properties.

1.6 Doping photocatalyst

Doping in photocatalysts refers to the deliberate incorporation of foreign atoms, ions, or molecules (dopants) into the crystal lattice of a photocatalytic material. This strategy is widely employed to tailor the structural, electronic, and surface properties of photocatalysts in order to enhance their performance in light-driven chemical reactions. Through controlled doping, the intrinsic limitations of pristine photocatalysts, such as wide band gap energy and rapid charge carrier recombination, can be effectively mitigated. One of the primary objectives of doping is the modification of the band gap energy of the photocatalyst. By introducing suitable dopant species, the electronic structure of the host material can be altered, allowing light absorption to be extended into the visible region of the solar spectrum, which constitutes the majority of solar irradiation. As a result, the photocatalyst becomes more efficient in harvesting solar energy and generating photogenerated charge carriers under visible-light illumination (Asahi et al., 2001; Di Valentin, Pacchioni and Selloni, 2005).

In addition to band gap engineering, doping can significantly influence charge carrier dynamics. Dopant atoms can introduce localized energy levels within the band gap or induce lattice distortions, which can act as electron or hole trapping sites. These features promote the separation and migration of photogenerated electron-hole pairs while suppressing their recombination, which is a major loss pathway in photocatalytic systems. Consequently, enhanced charge carrier lifetime and mobility can be achieved, leading to improved photocatalytic activity. Furthermore, doping can modify the surface chemistry of photocatalysts by introducing new active sites or altering surface adsorption characteristics. Such changes can improve the interaction between the photocatalyst surface and reactant molecules, thereby enhancing reaction selectivity

and catalytic efficiency for specific photocatalytic processes, including pollutant degradation and redox reactions (Chong et al., 2010).

Doping with rare earth metal ions has been extensively investigated as an effective approach to enhance the photocatalytic performance of perovskite-based materials. Rare earth elements, such as cerium (Ce), lanthanum (La), and europium (Eu), are known to possess unique electronic configurations with partially filled 4f orbitals, which can impart distinctive optical, electronic, and catalytic properties to perovskite photocatalysts. In particular, the coexistence of multiple oxidation states (e.g., $\text{Ce}^{3+}/\text{Ce}^{4+}$) can facilitate efficient charge transfer and act as redox centers, thereby further improving photocatalytic efficiency (Wendari et al., 2021).

1.7 References

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