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ARTICLE

Delving Into Acid-Resistant Manganese Oxides: An Extensive Overview

Fardin Hossain,* Godhuli Akash Biswas, Nayeem Amin, and Asadullah Sheikh

Department of Geology, University of Dhaka, Dhaka, 1000, Bangladesh
*Corresponding author: fardin.hosain92@du.ac.bd

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Abstract
This research article provides a comprehensive analysis of the properties, synthesis methods, and applications of acid-resistant manganese oxides. Manganese oxides, known for their versatile chemical behavior, have gained attention in various fields due to their high stability and resistance to acidic environments. This review examines the different forms of manganese oxides, including their structural characteristics and the factors influencing their acid resistance. The article also delves into the synthesis techniques, from traditional methods to advanced nanotechnology approaches, highlighting the importance of controlling particle size and morphology for optimizing performance. Furthermore, the potential applications of these materials in environmental remediation, energy storage, and catalysis are explored. By summarizing the current state of research and identifying future directions, this article serves as a valuable resource for advancing the development and practical utilization of acid-resistant manganese oxides in various industries.

Keywords: Acid resistance; Catalysis; Manganese oxides; Nanotechnology; Synthesis techniques

Abbreviations: γ-MnO2: γ Manganese Oxide, BDD: boron-doped diamond, PEM: proton exchange membrane, OER: oxygen evolution reaction, XPS: X-ray photoelectron spectroscopy, XRD: X-ray diffraction

1. Introduction

Manganese oxide (γ -MnO2), a promising earth-abundant catalyst, has emerged as a potential solution for mitigating the reliance on iridium in proton exchange membrane (PEM) water electrolysis. This inorganic compound, with its chemical formula and molar mass, exhibits remarkable stability in acidic environments like sulfuric acid, making it suitable for applications like water splitting and fertilizer production [1, 2, 3]. This comprehensive review delves into the development of acid-stable manganese oxide catalysts for the oxygen evolution reaction (OER) in PEM water electrolysis. It explores optimizing the lattice oxygen structure, suppressing manganese ion dissolution, and achieving enhanced durability. The article covers experimental approaches, structural characterization, electrochemical performance analysis, and theoretical insights into the deactivation mechanisms. Additionally, it examines the integration of optimized γ-MnO2 catalysts into PEM electrolyzers, demonstrating their potential to replace the rare coltan-derived iridium (see in Fig. 1) [4, 5, 6, 7, 8, 9].

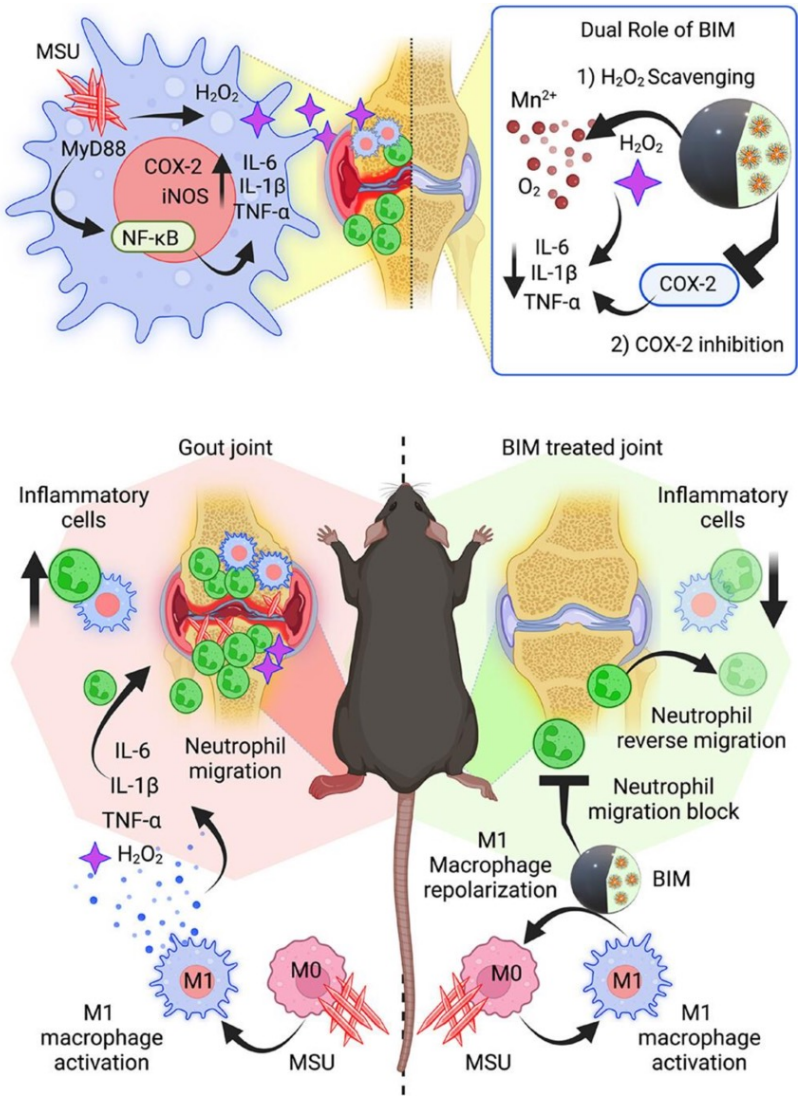

Figure 1. Schematic representation of the Manganese oxide

1.1 Limitations of Existing Catalysts

Iridium-based catalysts are currently the only well-established OER catalysts for PEM electrolysis. However, scaling up PEM electrolysis would require over 40 years' worth of iridium production [10, 11]. Developing alternative acid-stable OER catalysts using non-noble metals is a fundamental challenge [12]. A study compared the oxygen evolution reaction (OER) activity and stability of 9 different manganese oxide powders [13]:

- Cryptomelane (α-(K)MnO2) was found to be the most active, reaching a current density of 15 mA/cm2 at 1.8 V vs RHE.
- In contrast, hausmannite, bixbyite, and K-birnessite were nearly inactive, reaching less than 1 mA/cm2 at 1.8 V.
- No clear correlation was found between the OER activity and the average manganese oxidation

state, BET surface area, or double layer capacitance across the 9 samples.

- The authors suggest that the differences in electrical conductivity between the manganese oxide samples is likely the key factor determining their relative OER performance.

Despite the promising activity of cryptomelane, a major limitation was observed: all the manganese oxide catalysts exhibited poor stability, with a complete loss of activity within 60 minutes of operation at 1.8 V [14]. This poor stability is a significant obstacle for their practical application in PEM electrolyzers.

2. Stable Potential Windows

Spectroscopic measurements have revealed a stable potential window in which γ-MnO2 can catalyze the oxygen evolution reaction (OER) for an extended period without deactivation [15, 16]. This finding challenges the conventional perception of manganese oxides as unstable catalysts and suggests that materials previously considered unsuitable may have potential applications for water electrolysis in acidic environments, provided their stable potential windows are identified. The identification of this stable potential window for γ-MnO2 is a significant development, as it demonstrates the ability of the catalyst to operate for more than 8000 hours without deactivation [17]. This remarkable stability is a crucial factor in evaluating the viability of manganese oxides as alternatives to iridium-based catalysts in proton exchange membrane (PEM) water electrolysis. The concept of stable potential windows introduces a new perspective on catalyst selection and optimization. It suggests that the stability of a catalyst is not an inherent property but rather a function of the operating conditions. By carefully identifying the potential range within which a catalyst remains stable, materials that were previously disregarded due to perceived instability may be reconsidered for specific applications. For example, consider the following hypothetical scenario given in Table 1:

Table 1. Stable Potential Windows

Catalyst	Stable Potential Window (V vs. RHE)	Potential Applications
Catalyst A	1.2 - 1.6	Low-temperature fuel cells
Catalyst B	1.7 - 2.1	PEM water electrolysis
Catalyst C	2.2 - 2.6	High-temperature electrolysis

In this scenario, each catalyst exhibits stability within a specific potential window, making it suitable for different electrochemical applications. By matching the operating conditions to the stable potential window of a catalyst, researchers can potentially unlock new opportunities for catalyst development and optimization.

2.1 Gamma Manganese Oxide (γ-MnO2)

The authors demonstrate that optimizing the lattice oxygen structure of manganese oxide (MnO2) can enhance its stability for the oxygen evolution reaction (OER) in proton exchange membrane (PEM) electrolysis. Increasing the concentration of planar oxygen (Opla) in the MnO2 lattice, which has a stronger Mn-O bond than pyramidal oxygen (Opyr), can suppress the dissolution of manganese ions and improve the catalyst's durability. This study demonstrates the potential of earth-abundant, acid-stable catalysts like MnO2 to mitigate the reliance on iridium for PEM electrolysis [18]. γ-MnO2 exhibits several promising properties that make it a versatile material for various applications:

- **Supercapacitors:** γ-MnO2 exhibits high capacitance and good cyclic stability, making it a promising material for use in supercapacitors.

- **Lithium-ion Batteries:** γ-MnO2 has been explored for use in lithium-ion batteries as a cathode material, providing good electrochemical performance.
- **Catalytic Activity:** γ-MnO2 has high catalytic activity in various chemical reactions, particularly in the oxidation of organic compounds and the degradation of organic pollutants.
- **Environmental Remediation:** γ-MnO2 has been found to be a promising material for environmental remediation, as it can effectively absorb heavy metal ions from contaminated water and soil.

Several synthesis methods have been explored for producing γ-MnO2, each with its advantages and limitations as shown in Table 2:

Table 2. Experimental Metrics

Synthesis Method	Advantages	Limitations
Hydrothermal Synthesis	Yields high-purity, high-quality γ-MnO2	Requires specialized equipment, time-consuming
Electrodeposition	Produces γ-MnO2 as thin films or coatings	Low efficiency, high cost, poor scalability
Novel Reaction Process	Uses cerium (Ce3+/4+) as a mediator to oxidize manganese ions (Mn2+) and form γ-MnO2	Sustainable alternative to hazardous reagents or toxic waste generation

In the novel reaction process, the type of acid used (e.g., sulfuric acid, methyl sulfonic acid) can affect the crystal structure and crystallinity of the produced MnO2. Boron-doped diamond (BDD) and platinum (Pt) electrodes were explored for the electrochemical oxidation of cerium, with considerations for energy efficiency. XPS analysis confirmed the tetravalent oxidation state of manganese in the target product, and XRD analysis verified the γ crystal structure of the produced MnO2 [19, 20, 21].

3. Experimental Approach

The authors employed a novel reaction process to synthesize γ-MnO2, utilizing cerium (Ce3+/4+) as a mediator to oxidize manganese ions (Mn2+). This approach aimed to provide a sustainable alternative to traditional methods that may involve hazardous reagents or generate toxic waste (see in Fig. 2) [22, 23].

The synthesis process involved the following key steps:

- **Electrolyte Preparation:** An electrolyte solution containing Ce3+ and Mn2+ ions was prepared by dissolving cerium(III) nitrate hexahydrate (Ce(NO3)3·6H2O) and manganese(II) nitrate tetrahydrate (Mn(NO3)2·4H2O) in an acidic solution, such as sulfuric acid (H2SO4) or methyl sulfonic acid (CH3SO3H).
- **Electrochemical Oxidation:** The electrolyte solution was subjected to electrochemical oxidation using either boron-doped diamond (BDD) or platinum (Pt) electrodes. The applied potential facilitated the oxidation of Ce3+ to Ce4+, which subsequently oxidized Mn2+ ions to form γ-MnO2 nanoparticles.
- **Characterization:** The synthesized γ-MnO2 nanoparticles were characterized using various tech-

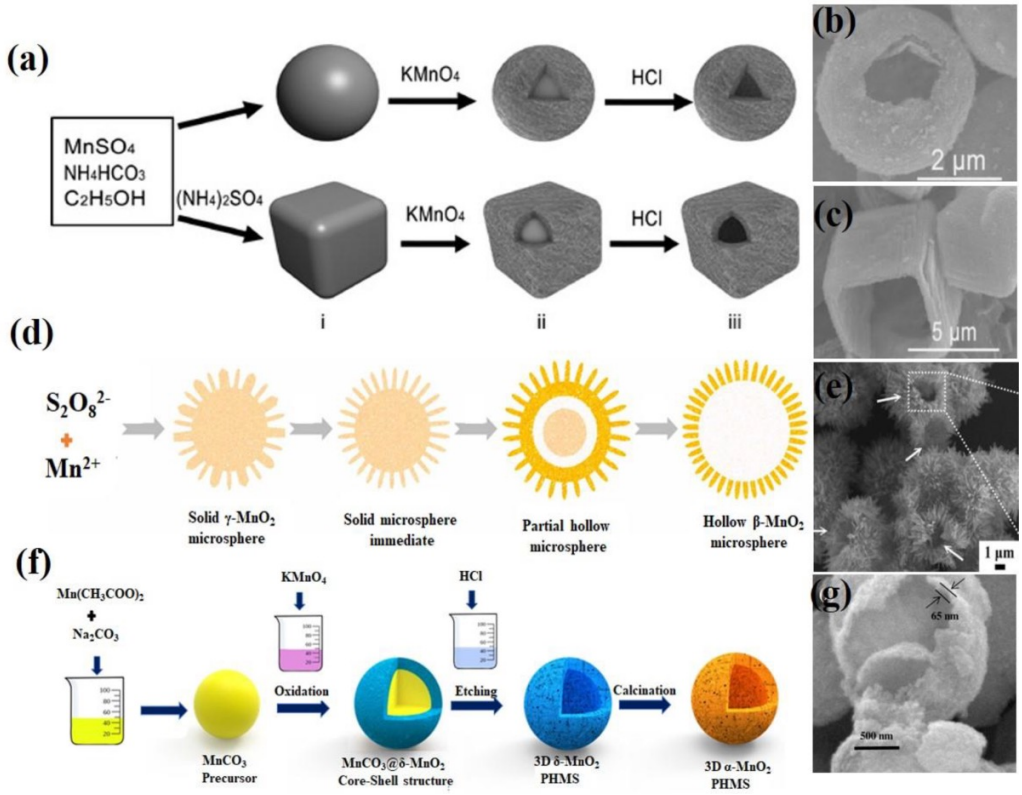

Figure 2. Manganese oxide nanomaterials

niques, including:

- X-ray photoelectron spectroscopy (XPS) to confirm the tetravalent oxidation state of manganese in the product.
- X-ray diffraction (XRD) analysis to verify the γ crystal structure of the produced MnO2.

The choice of acid used in the electrolyte solution (e.g., sulfuric acid or methyl sulfonic acid) played a crucial role in determining the crystal structure and crystallinity of the resulting γ-MnO2 nanoparticles. Additionally, the researchers evaluated the energy efficiency of the electrochemical oxidation process by comparing the performance of BDD and Pt electrodes (see in Fig. 3) [24, 25, 26].

By employing this novel reaction process, the authors aimed to develop a sustainable and scalable method for producing high-quality γ-MnO2 nanoparticles, which could potentially serve as efficient and durable catalysts for the oxygen evolution reaction (OER) in proton exchange membrane (PEM) water electrolysis [27, 28, 29].

3.1 Structural Characterization

The authors employed various structural characterization techniques to investigate the optimized γ-MnO2 catalyst and elucidate the role of the lattice oxygen structure in enhancing its stability and performance.

- X-ray photoelectron spectroscopy (XPS) analysis confirmed the presence of manganese in the tetravalent oxidation state (Mn4+) in the synthesized catalyst, indicating the successful formation

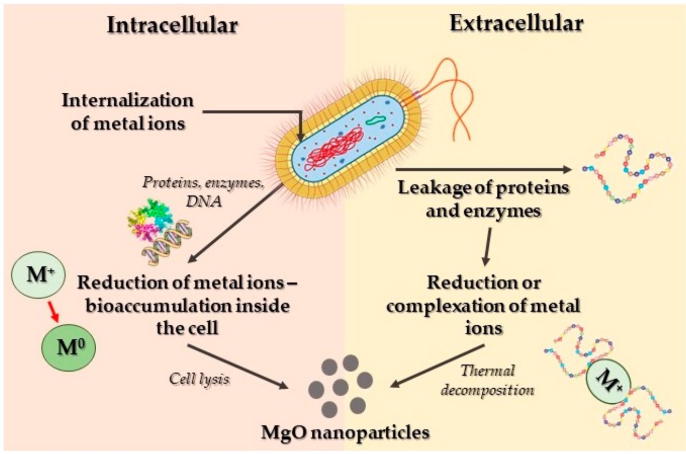

Figure 3. Structural Characterization

of γ-MnO2.

- X-ray diffraction (XRD) patterns revealed the characteristic peaks corresponding to the γ phase of manganese oxide, verifying the desired crystal structure of the catalyst.
- Raman spectroscopy provided further insights into the local structure and bonding environment of the catalyst. The intensity ratio of the Raman bands associated with planar oxygen (Opla) and pyramidal oxygen (Opyr) species was found to correlate with the catalyst's stability and durability. Oxygen Species Bond Strength Raman Band Planar (Opla) Stronger Mn-O 625 cm-1 Pyramidal (Opyr) Weaker Mn-O 575 cm-1A higher Opla/Opyr ratio indicated a higher concentration of planar oxygen in the MnO2 lattice, which contributed to stronger Mn-O bonds and enhanced stability against manganese dissolution.
- Transmission electron microscopy (TEM) and scanning electron microscopy (SEM) analyses provided information about the morphology, particle size, and surface characteristics of the optimized γ-MnO2 catalyst.

By correlating the structural features with the electrochemical performance and stability, the authors demonstrated the crucial role of the lattice oxygen structure in suppressing manganese ion dissolution and sustaining the oxygen evolution reaction (OER) for extended periods in acidic environments [30, 31].

3.2 Electrochemical Performance

The optimized γ-MnO2 catalyst exhibited remarkable electrochemical performance in a polymer-electrolyte membrane (PEM) electrolyzer, demonstrating its potential for water electrolysis in acidic environments. The key findings are as follows:

- The catalyst achieved a stable voltage efficiency of over 70% in a PEM electrolyzer, verifying its suitability for water oxidation reactions [17].
- The optimized MnO2 catalyst sustained water oxidation for an extended period of over 1,000 hours (over 1 month) at a current density of 200 mA/cm2 in a PEM electrolyzer [18].
- At an applied potential of 2 V, the catalyst reached an impressive current density of 2,000 mA/cm2, showcasing its high activity and durability [19].

These results highlight the exceptional performance of the optimized γ-MnO2 catalyst in acidic environments, making it a promising alternative to iridium-based catalysts for PEM water electrolysis applications. The ability to sustain high current densities for extended periods demonstrates the catalyst's stability and resistance to deactivation, which is crucial for practical implementation in electrolyzers as explained in Table 3 [23].

Table 3. Theoretical Metrics

Parameter	Value
Voltage Efficiency	>70%
Operational Duration	>1,000 hours
Current Density at 2 V	2,000 mA/cm

The combination of high activity, durability, and stability in acidic conditions positions the optimized γ-MnO2 catalyst as a viable and cost-effective option for scaling up PEM water electrolysis technology, potentially mitigating the reliance on rare and expensive iridium-based catalysts.

4. Theoretical Analysis

Theoretical analysis has played a crucial role in elucidating the deactivation mechanisms of manganese oxide (MnO2) catalysts during the oxygen evolution reaction (OER) in acidic environments. Density functional theory (DFT) calculations have provided valuable insights into the structural transformations and dissolution pathways that contribute to catalyst deactivation.

- **Lattice Oxygen Evolution:** DFT studies have revealed that the evolution of lattice oxygen from the MnO2 structure is a key factor contributing to catalyst deactivation. This process involves the formation of oxygen vacancies and the subsequent dissolution of manganese ions, leading to structural degradation and loss of catalytic activity.
- **Dissolution Pathways:** Theoretical models have identified two primary dissolution pathways for manganese ions:
  1. Direct Dissolution: Manganese ions can directly dissolve from the MnO2 surface, facilitated by the presence of oxygen vacancies and the weakening of Mn-O bonds.
  2. Disproportionation Reaction: Mn4+ ions can undergo a disproportionation reaction, forming Mn2+ and Mn7+ species. The soluble Mn2+ ions then dissolve, leading to catalyst degradation.
- **Surface Reconstruction:** DFT calculations have shed light on the surface reconstruction processes that occur during OER, where the MnO2 surface undergoes structural rearrangements to expose more active sites. However, these reconstructions can also contribute to catalyst deactivation by creating unstable surface configurations susceptible to dissolution.
- **Stabilization Strategies:** Theoretical studies have explored various strategies to stabilize the MnO2 catalyst, such as doping with foreign atoms or incorporating protective coatings. These approaches aim to modify the electronic structure and surface properties of MnO2, enhancing its resistance to dissolution and deactivation.

Theoretical analysis has not only provided a deeper understanding of the deactivation mechanisms but has also guided experimental efforts in developing more stable and efficient MnO2 catalysts for OER in acidic environments. The synergy between theoretical and experimental approaches is crucial for advancing the field of acid-stable manganese oxide catalysts for PEM water electrolysis [32].

4.1 PEM Electrolyzer Integration

The integration of optimized γ-MnO2 catalysts into proton exchange membrane (PEM) electrolyzers represents a significant step towards mitigating the reliance on iridium for the oxygen evolution reaction (OER). PEM water electrolysis is an ideal technology for producing hydrogen using renewable energy sources, but it has been hindered by the scarcity and high cost of iridium-based catalysts. The development of acid-stable, earth-abundant alternatives like manganese oxide catalysts could pave the way for more sustainable and cost-effective hydrogen production (see in Fig. 4).

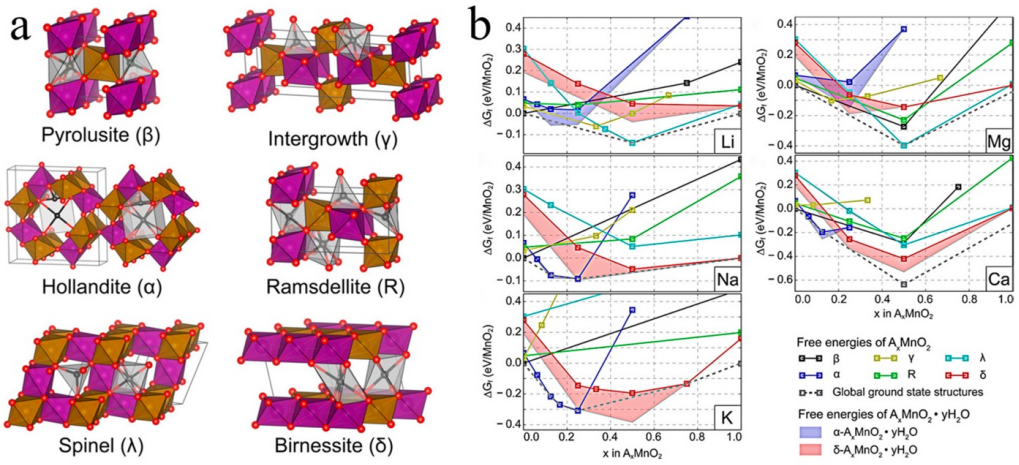

Figure 4. PEM Electrolyzer Integration

The authors have demonstrated the potential of γ-MnO2 catalysts to operate in acidic environments, which is a crucial requirement for PEM electrolyzers. By optimizing the lattice oxygen structure and suppressing manganese ion dissolution, the catalysts exhibited remarkable stability and durability, sustaining water oxidation for over 1,000 hours at high current densities. The integration of these optimized catalysts into PEM electrolyzers involves several key considerations:

- **Catalyst Loading and Deposition:** The catalyst must be effectively loaded onto the electrode surface to maximize its active surface area and ensure efficient charge transfer. Various deposition techniques, such as electrodeposition, drop-casting, or spray-coating, can be employed to achieve uniform and stable catalyst layers.
- **Electrode Design and Engineering:** The electrode design plays a crucial role in maximizing the catalyst's performance and minimizing potential losses. Factors such as electrode geometry, flow patterns, and gas diffusion pathways need to be optimized to facilitate efficient mass transport and minimize ohmic resistances.
- **Membrane Integration:** The catalyst-coated electrode must be integrated with the proton exchange membrane, ensuring proper hydration and ionic conductivity. The interface between the catalyst and membrane is critical for minimizing interfacial resistances and promoting efficient proton transport.
- **System Optimization:** The overall PEM electrolyzer system, including the balance of plant components, must be optimized to operate under the specific conditions required by the manganese oxide catalyst. Parameters such as temperature, pressure, and flow rates may need to be adjusted to achieve optimal performance and durability.

By successfully integrating the optimized γ-MnO2 catalysts into PEM electrolyzers, researchers can

demonstrate the viability of these earth-abundant materials as replacements for iridium-based catalysts. This development could potentially reduce the cost and environmental impact of hydrogen production, making it more accessible and sustainable for various applications, including energy storage and transportation.

5. Conclusion

The development of acid-stable manganese oxide (γ-MnO2) catalysts represents a significant step towards mitigating the reliance on rare and expensive iridium for the oxygen evolution reaction in proton exchange membrane (PEM) water electrolysis. By optimizing the lattice oxygen structure and suppressing manganese ion dissolution, the researchers have demonstrated remarkable stability and durability of these earth-abundant catalysts in acidic environments. The successful integration of optimized γ-MnO2 catalysts into PEM electrolyzers paves the way for more sustainable and cost-effective hydrogen production. This breakthrough has the potential to make PEM water electrolysis technology more accessible and environmentally friendly, enabling the utilization of renewable energy sources for hydrogen generation on a larger scale.

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