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Recent Advances in the Development of Transition Metal-Doped TiO_2-doped Photocatalysts and Electrocatalysts for Hydrogen Evolution Reactions: A Systematic Review

Misriyani*[1], Emil Hardiansyah[2], Isti Yunita[3]
*[1] Department of Biochemistry, Faculty of Medicine, Alkhairaat University, Indonesia
[2] Department of Information Technology Education, Faculty of Teacher Training and Education, Alkhairaat University, Indonesia
[3] Department of Chemistry Education, Faculty of Mathematics and Natural Sciences Universitas Negeri Yogyakarta, Indonesia

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Corresponding Author:

Misriyani,
Department of Biochemistry
Alkhairaat University

Email: misriyani85@gmail.com

ABSTRACT

This study conducted a systematic literature review to evaluate the recent development of transition metal-doped TiO_2 catalysts for the hydrogen evolution reaction (HER). A total of 20 selected articles were analyzed using the PRISMA approach to identify the influence of dopant type, catalyst architecture, and synthesis methods on catalytic performance. The results showed that transition metals such as Fe, Ni, Cu, and Mo significantly enhanced HER activity by introducing active sites, improving electrical conductivity, and suppressing electron–hole recombination. Among them, Fe demonstrated the most consistent performance due to its ability to modify the electronic structure of TiO_2. In addition, catalyst architectures such as heterojunctions, nanocomposites, and core–shell structures improved charge separation and increased active surface area. Synthesis methods, particularly hydrothermal techniques, played a crucial role in controlling crystallinity and dopant distribution. Overall, the enhancement of HER performance was governed by the synergistic interaction between dopant type, catalyst structure, and synthesis strategy. These findings highlighted the importance of integrated material design for advancing TiO_2-based catalysts toward sustainable hydrogen production applications.

Keywords: TiO_2, hydrogen evolution reaction, transition metal doping, photocatalyst, electrocatalyst, systematic literature review

1. INTRODUCTION

The world is currently facing an increasingly urgent dual challenge: the global energy crisis and global warming triggered by increased greenhouse gas emissions due to the uncontrolled use of fossil fuels (Lahkar et al., 2022; Shaban et al., 2024; Sukhadeve et al., 2023). High dependence on conventional energy sources not only threatens economic sustainability due to limited resource reserves, but also poses serious impacts on the environment, such as widespread air and water pollution (Patil et al., 2024; Sukhadeve et al., 2023). In the search for sustainable energy solutions, hydrogen (H_2) is emerging as one of the most promising carriers of clean energy. Hydrogen has a very high mass-based energy density, reaching about three times that of fossil fuels, and only produces water vapor as a byproduct of combustion, so it does not produce carbon emissions (Lahkar et al., 2022; Masihi et al., 2024; Patil et al., 2024).

Nevertheless, the main challenge lies in its production method. Currently, most commercial hydrogen is still produced through the process of steam reforming natural gas, which actually produces large amounts of greenhouse gas emissions (Lahkar et al., 2022; Shaban et al., 2024). Therefore, water splitting technology, both through photocatalytic and electrocatalytic approaches, is the main focus in the development of more environmentally friendly hydrogen production (Mishra et al., 2024; You et al., 2025). Hydrogen Evolution Reaction (HER) is a key process in water breakdown, in which water molecules or protons are reduced to hydrogen gas with the help of a catalyst (Patil et al., 2024; Shaban et al., 2024). Although precious metals such as platinum (Pt) are known to be the most efficient catalysts, their high cost and limited availability are major obstacles in industrial-scale deployment (Danilov et al., 2016; Lahkar et al., 2022; Mishra et al., 2024). Therefore, the development of alternative catalysts that are efficient, economical, and free of precious metals is an important priority in renewable energy research (Ismael, 2020; Lahkar et al., 2022; Shaban et al., 2024).

One of the most studied materials is titanium dioxide (TiO_2), which has advantages in the form of high chemical and thermal stability, non-toxic properties, low production costs, and resistance to corrosion (Bootluck et al., 2021; Ismael, 2020; Lahkar et al., 2022; Sukhadeve et al., 2023). However, pure TiO_2 has fundamental limitations, namely a wide energy band gap (~3.2 eV) so that it is only active under ultraviolet radiation, as well as a high rate of electron-hole pair recombination that inhibits reaction efficiency (Bootluck et al., 2021; Ismael, 2020; Mishra et al., 2024; Sukhadeve et al., 2023).

To overcome these limitations, modification through transition metal doping is one of the most effective strategies. The addition of metals such as iron (Fe), silver (Ag), and nickel (Ni) can narrow the bandgap and form new energy levels that act as electron traps, thereby suppressing charge recombination and increasing catalytic activity (Lahkar et al., 2022; Mishra et al., 2024; Sukhadeve et al., 2023). Among the various dopans, iron (Fe) is one of the most attractive due to its abundant availability, low cost, and relatively low toxicity, thus potentially replacing precious metals in TiO_2-based catalyst systems (Ismael, 2020; Lahkar et al., 2022).

Although various studies have shown an improvement in catalytic performance through transition metal doping, the reported results still vary due to differences in dopane types, synthesis methods, and reaction conditions used. In addition, most studies still focus on the development of materials individually, so they do not provide a comprehensive picture of optimal trends and strategies in the development of TiO_2-based catalysts.

Therefore, a systematic literature review is needed to integrate existing findings and identify the most effective approaches. Based on this background, this study aims to conduct a Systematic Literature Review (SLR) of the latest developments in TiO_2-based photocatalysts and electrocatalysts doped transition metals for hydrogen evolutionary reactions (HERs), with a focus on dopan types, synthesis methods, and catalytic performance reported in various studies.

2. RESEARCH METHOD

This study uses the Systematic Literature Review (SLR) method by following the standard guidelines of PRISMA (Preferred Reporting Items for Systematic Reviews and Meta-Analyses). This approach was chosen to ensure that the process of identification, selection, and evaluation of the literature regarding TiO2 modifications for hydrogen evolutionary reactions is carried out in a transparent, objective, and reproducible manner.

2.1. Article Selection Stages

The literature selection process was carried out through four main stages to capture the most relevant articles with a focus on metal-activated TiO2 materials. The selection flow is detailed as follows:

1. Identification: Initial searches were conducted on major bibliographic databases such as ScienceDirect and Elsevier journals using a combination of keywords such as "Hydrogen Evolution Reaction", "Metal-doped TiO2", and "Electrocatalyst". At this stage, a total of 210 potential articles were found. After manual and automatic duplication checks, a total of 25 articles were deleted, leaving 185 articles for the next stage.
2. Screening: A total of 185 articles were screened by title and abstract. Articles that are not relevant to the focus of clean energy production, such as the use of TiO2 only for wastewater treatment without hydrogen evolution data, are excluded. This stage sets aside 113 articles, leaving 72 articles that are considered to meet the basic criteria.
3. Eligibility: A *full-text review* was conducted on the 72 articles. Strict eligibility criteria are applied, i.e. articles must include quantitative data on HER performance (such as overpotential, hydrogen evolution rate, or quantum efficiency) and an explanation of the charge transfer mechanism. A total of 40 articles were excluded due to lack of in-depth technical data or not using TiO2 as the main material.
4. Included: Based on the feasibility assessment, 20 articles (including the latest literature of 2025) were finally selected to be analyzed in depth in this systematic review.

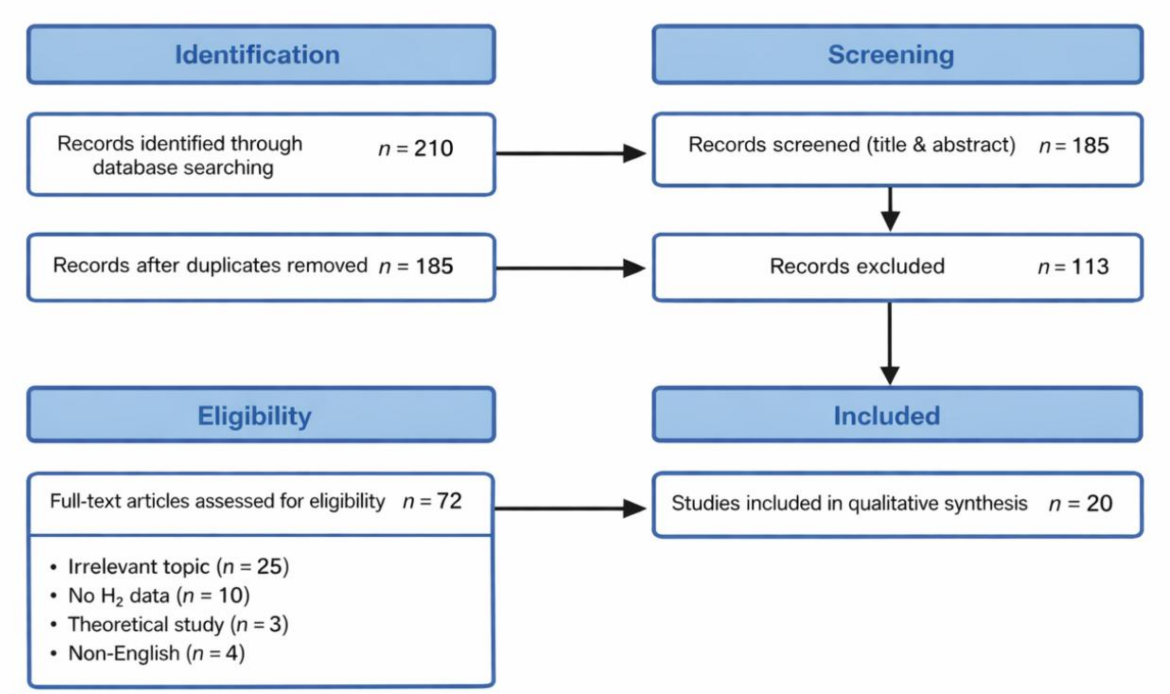

Figure 1. PRISMA flow diagram illustrating the literature screening process used in this systematic review

2.2. PRISMA Flow Summary

The above selection process is summarized in the following table which will be visualized in the form of a PRISMA Flowchart in the final report:

Table 1. Number of Articles in each Selection Stage

Selection Stage	Activity Description	Number of Articles
Identification	Total articles found in the database	210
	Once the duplicate is removed	185
Screening	Based on the suitability of the title and abstract	72
Eligibility	Full text eligibility assessment	32
Included	Final articles analyzed in SLR	20

2.3. Data Extraction and Analysis

Data from 20 selected articles were systematically extracted to compare various key parameters. The parameters taken include:

1. Material Parameters: Identify the type of transition dopan metal used such as Iron (Fe), Silver (Ag), Nickel (Ni), Copper (Cu), or Molybdenum (Mo) as well as the presence of other supporting or functional materials. Some studies integrate conductive carbon materials such as multiwalled carbon nanotubes (MWCNT), reduced graphene oxide (rGO), or utilize industrial waste such as steel slag as a precursor.
2. Catalyst System: Determine the structural configuration of the catalyst, whether it is a nanocomposite (e.g. Fe-doped TiO2/MWCNT), a core-shell structure (TiO2/Fe2O3), a Metal-Organic Framework (MOF) such as MIL-125-Ti, or an advanced heterogeneous system (MoS2/TiO2 and TiO2-Pt/FeHAp).
3. Synthesis Method: Identify the fabrication techniques used, including sol-gel, hydrothermal, electrodeposition, spray pyrolysis, to innovative techniques such as atmospheric plasma spray (APS) or surface modification with argon gas plasma.
4. Catalytic Performance: Focuses on key quantitative metrics to evaluate the efficiency of hydrogen production. This includes the lowest overpotential value (η) at a given current density (e.g. 10 mA/cm2 or 250 mA/cm2) for electrocatalytic testing, as well as the rate of hydrogen evolution (in μmol/h, mmol/h, or μmol/g/h) for photocatalytic systems under visible light irradiation.

Quality analysis is carried out by evaluating the depth of explanation of the reaction mechanism, such as the Volmer-Heyrovsky or Volmer-Tafel pathway, which is the basis for determining the efficiency of the catalyst.

3. RESULTS AND ANALYSIS

The data presented in Table 2 reflect a systematic synthesis of the selected literature based on the PRISMA inclusion criteria. The extraction focus was carried out on four key variables: the type of activator metal, the architecture of the catalyst system, the fabrication method, and the final result in the form of hydrogen evolution rate or electrocatalytic metrics. This parameter was chosen because it directly affects the efficiency of charge transfer and the stability of the material in the long run.

Table 2. Literature Data Extraction: Hydrogen Evolution – Metal-Doped TiO2 / Related Systems

Lead Author, Year	Metals Used	Catalyst System	Synthesis Method	Hydrogen Evolution Rate (or Key Performance)
(Lahkar et al., 2022)	Iron (Fe)	Fe doped TiO2/MWCNT	*Surfactant wrapping in situ sol gel*	Overpotential 221 mV at 10 *mA/cm2 (Electrocatalytic)*
(Sukhadeve et al., 2023)	Silver (Ag) & Iron (Fe)	Ag, Fe co-doped TiO2	*Sol–gel method*	475.56 *μmol/g/h* (2377.82 *μmol/g* in 5 hours)
(Mishra et al., 2024)	Iron (Fe)	Fe-doped TiO2 *Metal-Organic Framework* (MOF)	*Hydrothermal method*	Current 10 *mA/cm2 at potential onset* -0.345 V (Electrocatalytic)
(Patil et al., 2024)	Nickel (Ni) & Iron (Fe)	NiFe2O4/TiO2 *nanocomposite*	*Sol-gel self-ignition method*	146.3 *μmol/g/h* (Photocatalytic)
(Shibli & Sebeelamol, 2013)	Iron (Fe) & Nickel (Ni)	Fe2O3-TiO2 in the Ni-P matrix	Thermal decomposition & *electroless coating*	Overpotential 116 mV at 250 *mA/cm2 (Electrocatalytic)*
(Danilov et al., 2016)	Iron (Fe)	Fe/TiO2 *composite coatings*	*Electrodeposition*	Overpotential -0.47 V at 250 *mA/cm2 (for 5% wt TiO2)*
(Madhumitha et al., 2018)	Iron (Fe)	TiO2/Fe2O3 *core shell particles*	*Co-precipitation method*	2700 *mmol/h* (with EDTA sacrificial agent)
(Sumi et al., 2020)	Iron (Fe) & Nickel (Ni)	Fe2O3-TiO2/rGO-NiP *coating*	*Easy hydrothermal* & *chemical reduction*	Overpotential 96 mV at 10 *mA/cm2 (Electrocatalytic)*

Lead Author, Year	Metals Used	Catalyst System	Synthesis Method	Hydrogen Evolution Rate (or Key Performance)
(Masihi et al., 2024)	Iron (Fe)	Fe3O4 & TiO2 *nanoparticles*	Dark/photo fermentation *hybrid*	Biohydrogen yield 213.66 *mlH2/gVS*
(Narayanan et al., 2024)	Iron (Fe)	NiFe & NiTiO2 *electrocatalysts*	*Atmospheric Plasma Spray* (APS)	Overpotential 298 mV (NiFe) at 20 *mA/cm2 (Electrocatalytic)*
(You et al., 2025)	Molybden (Mo)	MoS2/TiO2 on *stainless steel mesh*	*Sputtering* & *hydrothermal synthesis*	7.0 *µmol/cm2 in 60 minutes (PEC splitting)*
(García-Muñoz et al., 2022)	Iron (Fe) & Platinum (Pt)	*Iron-grafted mesoporous* Pt/TiO2	Calcination & *Iron Grafting*	≈7.5 *µmol/min* (from ethanol dehydrogenation)
(Shu et al., 2023)	Platinum (Pt) & Iron (Fe)	TiO2-Pt/FeHAp *heterojunction*	Hydrothermal & *electrostatic assembling*	3026 *µmol/g/h* (Photocatalytic)
(X. Liu et al., 2022)	Iron (Fe in enzymes)	FeFe-hydrogenase on *black* TiO2 *nanotubes*	Chemical reduction & *enzyme immobilization*	Current 17 *µA/cm2 at -100 mV vs RHE (Electrocatalytic)*
(J. Liu et al., 2021)	Copper (Cu)	TiO2-Cu-Mo2C	Solid phase reaction & *photo-assisted impregnation*	521 *µmol/h* (Photocatalytic, neutral condition)
(Shaban et al., 2024)	Iron (Fe), Co, Ni	Fe-Co-Ni@CF/C-TiO2@NFF	*Spray pyrolysis*	Current 200.8 *mA/cm2 at 1.3 V vs RHE (buffered seawater*)
(Ismael, 2020)	Iron (Fe)	Fe(III) doped TiO2 *nanoparticles*	*Simple precipitation method*	2423 *µmol/h* (for 0.1 mol % Fe)
(Bootluck et al., 2021)	Iron (Fe)	*α*-Fe2O3/TiO2 *nanocomposite*	*Sol-gel precipitation* & *Ar gas plasma treatment*	1.25 *mmol/h* (Photocatalytic)

3.1. Analysis of Dopan Metal Types on TiO_2

Based on the results of the literature review, transition metals such as Fe, Ni, and Ag are the most commonly used dopan in TiO_2 modification to increase HER activity. Among the three, Fe is reported to be the most dominant dopan used in various studies.

Iron (Fe) is the most dominant dopan and has consistently shown increased HER activity compared to other dopans such as Ni and Ag. This advantage is related to its ability to form an effective mid-gap energy state as an electron trap center, thus being able to suppress the rate of electron-hole pair recombination. In addition, the configuration of the Fe electrons allows for stronger interactions with TiO_2 orbitals, which improves the efficiency of charge transfer.

In contrast, Ni plays a more important role in improving the beneficial electrical conductivity of electrocatalytic systems, but its contribution to charge separation in photocatalytic systems is relatively limited. Meanwhile, Ag increases light absorption through the plasmonic effect, but often exhibits lower stability under long-term reaction conditions.

Nonetheless, some studies have shown that the effectiveness of dopan is highly dependent on the method of dopan synthesis and concentration used. Excessive doping can cause the formation of new recombination centers which actually reduce catalytic performance.

Overall, Fe exhibits the most balanced performance in increasing HER activity in TiO_2, but optimization of structure and synthesis methods remains key factors in determining the final performance of the catalyst.

3.2. Influence of Catalyst System Architecture

The catalyst system architecture plays an important role in determining the performance of HER in TiO_2-based materials. Various structures such as nanoparticles, heterojunctions, and core-shells have been developed to optimize catalytic activity. Among these architectures, heterojunction exhibits the most superior performance due to its ability to improve charge separation through the formation of an internal electric field at the material interface. The difference in energy levels between the two semiconductor phases allows for a directional migration of electrons and holes, significantly reducing the rate of recombination.

Compared to single nanoparticles, heterojunctions provide a more efficient charge transfer path, while the core-shell structure offers better stability by protecting the catalyst core from degradation. However, suboptimal shell layer thickness can inhibit charge diffusion and degrade catalytic performance. In addition, nanomaterial structures such as nanotubes and nanosheets provide the added advantage of high surface area, which increases the number of active sites for HER reactions.

Although various architectures have shown significant performance improvements, challenges in structural control, long-term stability, and synthesis complexity remain major barriers in the development of industrially applicable catalysts.

3.3. Effect of Synthesis Method on Catalyst Performance

The synthesis method plays a crucial role in determining the structure and performance of TiO_2-based catalysts. Various methods such as hydrothermal, sol-gel, and coprecipitation have been used to control the morphology, particle size, as well as dopan distribution in materials. Among these methods, hydrothermal synthesis is often reported to result in higher HER performance because it is able to produce good crystallinity, uniform particle size, and high surface area. This characteristic increases the number of active sites and facilitates charge transfer, thereby improving the efficiency of hydrogen evolution reactions.

Compared to hydrothermal methods, sol-gel methods offer more flexible control of chemical composition and doping levels, but often result in structures with lower porosity and crystallinity, which can limit catalytic activity. Meanwhile, the coprecipitation method is relatively simple and economical, but it often results in a less homogeneous dopan distribution.

In addition, the synthesis method also affects the formation of structural defects such as oxygen vacancies, which play an important role in increasing conductivity and catalytic activity. However, each method has limitations, such as process complexity, high energy requirements, and challenges in yield reproducibility. Therefore, the selection of synthesis methods must consider the balance between catalytic performance, process efficiency, and potential scalability for industrial applications.

3.4. Catalytic Performance Trends in HER Systems

In general, the results of the analysis show that modification of TiO_2 through transition metal doping is able to significantly increase HER activity compared to pure TiO_2. This increase is mainly due to several key mechanisms, namely:

1. increased electron-hole pair separation,
2. formation of new active centers on the catalyst surface, and
3. Improved electrical conductivity of the material.

In addition, in electrocatalytic systems, the performance of HER is usually evaluated based on the overpotential value at a given current density. Lower overpotential values indicate better catalytic activity. For example, the Fe_2O_3-TiO_2/rGO-NiP system shows an overpotential of only 96 mV at 10 mA/cm^2, which is one of the best values among the systems analyzed (Sumi, 2019). In photocatalytic systems, performance is usually judged based on the rate of hydrogen evolution (μmol/g/h). Some heterojunction systems show very high performance, such as TiO_2-Pt/FeHAp heterojunction which reaches 3026 μmol/g/h (Shu et al., 2023). This suggests that a combination strategy of metal doping and catalyst structure engineering can provide a significant performance improvement.

The hydrogen evolution reaction (HER) on transition metal-doped TiO_2 generally follows the Volmer–Heyrovsky or Volmer–Tafel mechanism, which involves proton adsorption and electron transfer on the catalyst surface. Transition metal dopants act as active sites that facilitate proton adsorption and enhance electron transfer, thereby accelerating hydrogen generation (Lahkar et al., 2022; Mishra et al., 2024; Shaban et al., 2024). The reaction pathway is illustrated in Figure 2.

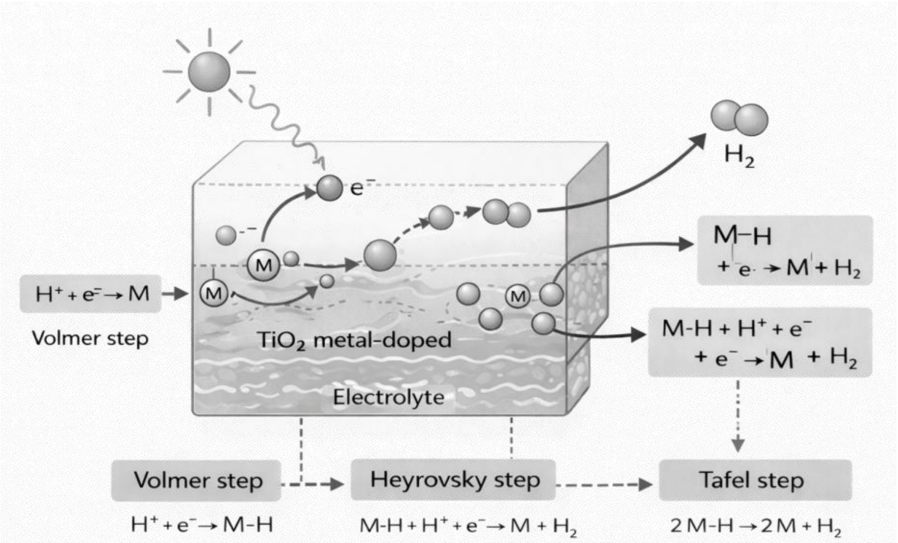

Figure 2. Schematic illustration of the hydrogen evolution reaction (HER) mechanism on transition metal-doped TiO_2 catalysts following the Volmer–Heyrovsky–Tafel pathway

3.5. Research Gap and Future Research Directions

Despite the considerable progress in the development of transition metal-doped TiO_2 catalysts for the hydrogen evolution reaction (HER), several critical research gaps remain that hinder their practical implementation. First, although numerous studies report enhanced catalytic activity through metal doping, systematic investigations that directly correlate dopant-induced modifications in electronic structure—such as band gap tuning, defect state formation, and charge carrier dynamics—with HER performance are still lacking (Ismael, 2020; Mishra et al., 2024). Moreover, existing studies often focus on single-dopant systems and report inconsistent trends regarding dopant effectiveness, highlighting the absence of standardized evaluation frameworks for identifying optimal doping strategies.

Second, the relationship between synthesis methods, catalyst morphology, and catalytic efficiency has not yet been fully elucidated. While techniques such as sol–gel, hydrothermal, and electrodeposition can significantly influence crystal structure, surface area, and defect density, a unified understanding that links synthesis parameters to structural evolution and catalytic performance remains underdeveloped. This limitation restricts the rational design of high-performance catalysts.

Third, most experimental studies are conducted under controlled laboratory conditions with limited operational duration. Consequently, systematic investigations into catalyst stability and degradation mechanisms—such as dopant leaching, surface poisoning, and structural instability during prolonged operation—remain scarce (Shaban et al., 2024). These factors are critical for real-world hydrogen production systems, which require long-term operational reliability under fluctuating environmental conditions.

Furthermore, the integration of TiO_2-based catalysts into scalable hydrogen production systems, including photoelectrochemical water splitting and industrial electrolyzers, is still insufficiently explored. Future research should therefore move beyond conventional single-variable optimization and adopt a more integrated approach, including the development of co-doping strategies, advanced heterostructure engineering, and the application of operando or in situ characterization techniques to better understand reaction mechanisms. In addition, establishing standardized benchmarking protocols will be essential to enable reliable comparison across studies.

Overall, advancing transition metal-doped TiO_2 catalysts toward practical applications requires a paradigm shift from empirical trial-and-error approaches to mechanism-driven catalyst design. Addressing these challenges will be crucial for unlocking the full potential of TiO_2-based materials in sustainable hydrogen production technologies.

4. CONCLUSION

Based on the results of the Systematic Literature Review of 20 articles, it can be concluded that transition metal doping on TiO_2 is an effective strategy to improve catalytic performance in hydrogen evolution (HER) reactions. Dopans such as Fe, Ni, Cu, and Mo contribute to the formation of active sites, increased electron conductivity, as well as the suppression of electron–hole pair recombination. In addition, the design of catalyst architectures—such as heterogeneity, nanocomposites, and core–shell structures—as well as synthesis methods also determine the efficiency of charge transfer and active surface area.

Overall, the improvement in HER performance is the result of synergy between the dopan type, catalyst structure, and synthesis method. These findings confirm that an integrated material design approach is critical in the development of TiO_2-based catalysts to support sustainable hydrogen production in the future.

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