

Article

Morphological Impact of Iron Tungstate on Electrochemical Properties in the Oxygen Evolution Reaction

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Abstract

In this study, the influence of nanostructure and morphology was used as a comparative strategy to investigate the material's performance. For this purpose, two distinct morphologies, one of which is novel for iron tungstate (nanoplatelets) and obtained by the hydrothermal method, were tested to verify their influence on the catalytic activity, reaction kinetics, and operational stability of the oxygen evolution reaction. Although the self-organized rosette and nanoplatelet morphologies maintain a monoclinic ferberite-type structure, the 2D nanoplatelet architecture shows an almost 30% reduction in catalytic activity and a 16% increase in reaction rate in basic medium (1 M KOH), in addition to four times higher turnover frequencies (TOF). Furthermore, the new morphology (nanoplatelets) provides higher charge density, lower charge transfer resistance, and favorable band edge positions, resulting in greater oxidation capacity (10 times more). Regarding the stability factor, both morphologies show a slight degradation over 1000 cycles, but the nanoplatelet morphology maintains almost constant activity, while the self-organized rosette morphology shows a performance drop of approximately 25%. These results demonstrate that morphological engineering is an effective means of improving electrocatalytic performance.

Keywords: iron tungstate; morphological engineering; oxygen evolution reaction; electrocatalysis; benchmarking

1. Introduction

The rapid expansion of renewable energy systems has led to unprecedented growth in installed capacity, with clean energy sources accounting for 40.9% of global electricity production in 2024 [1]. Despite this progress, the gap towards achieving the Paris Agreement targets remains substantial, highlighting the need for efficient energy storage and conversion technologies [2,3].

Hydrogen is widely regarded as a key energy vector for decarbonization. However, conventional production routes require harsh operating conditions (700–1000 °C) and up to 120 bar, resulting in high energy consumption [4–6]. In this context, electrochemical and photoelectrochemical water splitting have emerged as sustainable routes for hydrogen production [7]. The oxygen evolution reaction (OER), which occurs at the anode, is widely recognized as the kinetically limiting step due to its complex four-electron transfer mechanism [8]. Therefore, the development of efficient and stable electrocatalysts is essential to reduce the required overpotential and increase overall energy efficiency. Although



Academic Editor: Jiefang Zhu

Received: 19 May 2026

Revised: 30 June 2026

Accepted: 9 July 2026

Published: 13 July 2026

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ruthenium- and iridium-based oxides are currently the benchmarks for the OER, their high cost, scarcity, and limited durability hinder large-scale industrial deployment [9]. Transition metal oxides, including Fe_2O_3 and WO_3 , have been extensively studied as anodic materials, making tungstates promising candidates for OER catalysis [8–11].

Various types of electrocatalysts abundant on Earth have been presented, including nanostructured transition metal phosphides, chalcogenides, carbides, borides, nitrides, oxides, and (oxy)hydroxides, among others, and have been tested in water splitting (OER and HER), as can be seen. An extensive review of different classes of OER catalyst materials has been reported in the literature [12–19]. In all these studies, one can find a multitude of different materials used as electrocatalysts, the vast majority of which are based on transition elements. Among the inorganic materials of the metallic oxide class, metallic tungstate has stood out significantly in the last fifteen years. Over the last decade, the family of metal tungstates (MWO_4 , where $\text{M} = \text{Fe}, \text{Co}, \text{Zn}$, among others) has attracted growing interest due to its structural versatility and tunable physicochemical properties, which enable applications across a wide range of fields. Studies on NiWO_4 and ZnWO_4 , for instance, demonstrate that optical and electronic properties depend on electronic structure and crystalline defects. Furthermore, ZnWO_4 -based systems have been extensively investigated for photocatalysis, as well as for composites and advanced ceramics, where phase composition, morphology, and crystallinity directly influence the material's optical, mechanical, and functional properties. These results highlight the versatility of tungstates and confirm the increasing interest in this class of compounds for applications in photonics, sensors, photocatalysis, and energy conversion [20–22]. The crystalline structure of tungstate is a peculiar characteristic, with tungstates generally exhibiting tetragonal scheelite or monoclinic wolframite structures [23–25]. Among wolframite-type tungstates, iron tungstate (FeWO_4) has attracted increasing attention, showing a growth trend similar to that of metallic tungstates in general. Tungstates are particularly relevant in environmental remediation and renewable energy systems, and more recently in electronic devices, such as materials for resistive memory [11,25–34]. However, although the number of studies on tungstates has increased significantly in recent years, their specific application in water electrolysis (HER/OER) is still relatively low when compared to other classes of materials [12,13,15,16,18,19].

Among them, iron tungstate (FeWO_4) is particularly attractive due to its chemical stability and self-passivation behavior in both acidic and alkaline media, which are desirable properties for an OER electrocatalyst, unless photoelectrochemical operation is intended [10]. Adak et al. (2022) highlighted FeWO_4 as a promising structure for water electrolysis, showing superior performance to some noble metal-based electrocatalysts [35]. Several studies demonstrate that combining FeWO_4 with co-catalysts or forming heterojunctions ($\text{FeWO}_4/\text{NiFeOOH}$ [36], $\text{FeWO}_4\text{-Ni}_3\text{S}_2$ [27], Ni-NPs@FeWO_4 [37], $\text{FeWO}_4\text{-WO}_3/\text{NF}$, $\text{FeWO}_4/\text{Fe}_2\text{O}_3$ [38] and $\text{FeWO}_4/\text{FeOOH}/\text{NiOOH}$) significantly improves its electrocatalytic activity for the OER [39]. Furthermore, incorporating a third element into the FeWO_4 crystal structure has proven to be an effective strategy to increase catalytic activity in alkaline media and reduce overvoltage. Nakayama et al. (2020) synthesized $\text{Co}_{0.5}\text{Fe}_{0.5}\text{WO}_4$, obtaining superior performance even to that of the reference catalyst RuO_2 [9]. Luo et al. (2018) deposited $\text{Co}_{0.5}\text{Fe}_{0.5}\text{WO}_4$ in situ on carbon nanotubes dispersed in carbon paper and presented an overvoltage of 290 mV and a Tafel slope of 42 mV dec^{-1} [40]. In turn, Shao et al. (2018) synthesized $\text{Co}_{0.7}\text{Fe}_{0.292}\text{WO}_4$ using the hydrothermal method and observed superior electrocatalytic performance when compared to CoWO_4 and FeWO_4 supported on glassy carbon [41].

Recent studies have primarily focused on enhancing FeWO_4 performance through doping strategies [9] or with different types of heterojunctions [26,27,42]. However, beyond

compositional engineering, the morphology of the electrocatalyst plays a critical role in determining OER activity, as it directly influences accessibility to active sites, charge transport and interfacial kinetics [43,44]. In particular, our group recently demonstrated that controlling the morphology of FeWO_4 simply by changing the precursor's physical state during hydrothermal synthesis enables the design of specific nanostructures with distinct structural and optical properties [45]. Despite these advances, the intrinsic relationship between FeWO_4 morphology and its electrocatalytic performance remains insufficiently understood, especially in the absence of doping or composite effects. According to Maya et al. [30], iron tungstate nanoparticles exhibit lower resistance to electron transfer than 1D structures, while additional enhancements, including a substantial decrease in overpotential, have been reported when combining FeWO_4 with nickel nanoparticles. However, these approaches do not isolate the sole effect of morphology.

As is known, iron tungstate exhibits overpotentials that are still considered high when compared to other semiconductor materials used in the OER, but these values vary considerably depending on the reaction system and its own structural and electrochemical properties [9,35,41]. Therefore, iron tungstate was used as a model material, since it is a stable semiconductor with a well-defined crystalline structure and measurable electrocatalytic activity, thus allowing for observation of the possible effects that morphological modifications may have on its properties and application. The structural stability and sensitivity to morphological modifications exhibited by iron tungstate make it a good candidate for fundamental studies of the correlation between morphology and electrochemical performance. Furthermore, novel morphologies of iron tungstate (nanoplatelets and self-organized rosettes) were used for these purposes. Therefore, this study aims to understand how changes in morphology can influence the structural and electrochemical properties of the material and its performance in the oxygen evolution reaction at low temperatures and in an alkaline medium. Thus, by fixing the chemical composition, the substrate, and the test conditions, it is possible to analyze in isolation the effect of morphology on charge transfer, the electrochemically active area, the roughness factor, and oxygen production from the evolution reaction. This knowledge can then be applied to the development of more efficient semiconductor systems. Therefore, iron tungstate possesses the necessary criteria and several advantages for studies related to its structure and properties [39,45,46].

In this work, we address this gap by investigating the OER performance of pure FeWO_4 with two distinct morphologies (self-organized rosette structures and nanoplatelets) synthesized via a controlled hydrothermal route. By avoiding doping or heterojunction formation, this study aims to decouple and elucidate the intrinsic role of morphology in governing electrocatalytic activity.

Electrochemical performance was systematically evaluated using key metrics, including overpotential at $10 \text{ mA}\cdot\text{cm}^{-2}$, Tafel slope, and stability, following established benchmarking criteria [12]. In addition, carbon paper was intentionally selected as the substrate to minimize extrinsic enhancements associated with highly conductive supports (e.g., nickel foam or vitreous carbon) [35], thereby ensuring that the observed performance differences arise primarily from the FeWO_4 morphology itself. Through this approach, this work provides a direct structure–property–performance correlation, offering new insights into the design of efficient, morphology engineered tungstate-based electrocatalysts for oxygen evolution.

2. Results

2.1. Structural and Morphological Characterizations of FeWO_4

The crystalline structure and morphology of the synthesized FeWO_4 were first examined to establish a robust foundation for interpreting their electrocatalytic behavior.

Figure 1 presents the XRD patterns and SEM images of the two morphologies, namely self-organized rosette structures and nanoplatelets. The synthesis methodology and detailed growth mechanisms have been previously reported and validated in our earlier work [45].

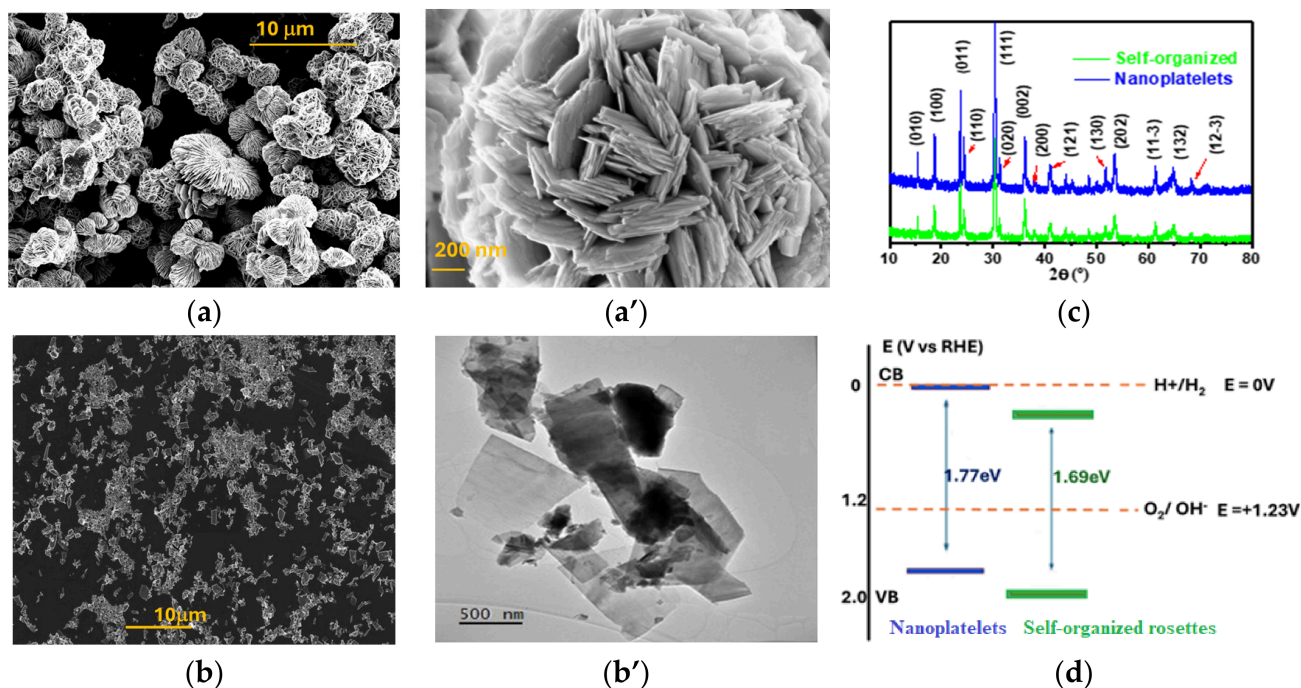


Figure 1. SEM of iron tungstate: self-organized rosettes ((a) (2 kx), (a') (40 kx)) and nanoplatelets ((b) (2 kx), (b') (10 kx)); XRD (c); and band edge positions (CB and VB) of iron tungstate (self-organized rosettes and nanoplatelets) (d).

Both samples exhibit diffraction peaks consistent with the monoclinic ferberite phase with the P2/c space group (ICSD 15193) along the [101] zone axis, confirming phase purity regardless of morphology. For both morphologies, electron diffraction patterns obtained from elementary grains reveal monocrystalline FeWO_4 particles [45]. The refined lattice parameters (self-organized rosettes: $a = 4.7332 \text{ \AA}$, $b = 5.7102 \text{ \AA}$, $c = 4.9774 \text{ \AA}$, $\beta = 90.3336^\circ$; and nanoplatelets: $a = 4.7310 \text{ \AA}$, $b = 5.7083 \text{ \AA}$, $c = 4.9763 \text{ \AA}$, $\beta = 90.2918^\circ$) show negligible differences, indicating that variation in morphology does not alter the bulk crystal structure. Such structural invariance is essential, as it ensures that any differences in electrochemical performance arise primarily from morphological effects rather than crystallographic or compositional variations. To provide a clear basis for the current electrocatalytic investigation, the main physical properties of the two morphologies, nanoplatelets and self-organized rosettes, are summarized in Table 1.

Table 1. Summary of the structural and optical properties of FeWO_4 morphologies.

Properties	Self-Organized Rosettes	Nanoplatelets
Density (g cm^{-3})	7.49	7.50
Crystallite size (nm)	67.8	87.3
BET surface area ($\text{m}^2 \text{g}^{-1}$)	6.80	12.65
Band Gap, E_g (eV)	1.69	1.77
Conduction band (eV)	0.28	−0.05
Valence band (eV)	1.97	1.72

In contrast, significant differences are observed at the micro- and nanoscale. SEM analysis reveals that the self-organized rosette morphology consists of aggregated structures, whereas the nanoplatelets exhibit a well-defined two-dimensional architecture. This mor-

phological distinction is directly reflected in the specific surface area, with the nanoplatelets displaying nearly twice the BET surface area ($12.65 \text{ m}^2 \cdot \text{g}^{-1}$) compared to the self-organized rosette structures ($6.80 \text{ m}^2 \cdot \text{g}^{-1}$). Such an increase is expected to enhance the density of accessible active sites and facilitate electrolyte penetration, both of which are key parameters governing OER activity. Crystallite size analysis further supports this distinction, with larger crystallites observed for the nanoplatelets (87.3 nm) compared to the self-organized rosettes morphology (67.8 nm). While larger crystallites are often associated with reduced surface area, the present results indicate that the nanoplatelet architecture compensates through its anisotropic geometry, maximizing exposed surface planes. This suggests that not only the surface area but also the nature of exposed facets may contribute to catalytic performance.

As summarized in Table 1, the optical properties suggest that the nanoplatelets have a slightly wider bandgap (1.77 eV) [45]. Both morphologies exhibit a valence band edge (EVB) that is positively shifted relative to the O_2/OH^- redox potential (Figure 1c). Despite the small difference in band gaps, a detailed analysis shows that the EVB energy of the nanoplatelets (1.72 eV) is closer to the target redox potential (O_2/OH^-) than that of the self-assembled structures (1.97 eV). This optimized energy alignment ensures a sufficient thermodynamic driving force for water oxidation. Furthermore, the increased current density enhances the nanoplatelets' oxidation capacity for the oxygen evolution reaction (OER).

Overall, these results demonstrate that while the bulk crystal structure remains unchanged, the morphology-driven modifications induce significant differences in surface area, active site accessibility, and electronic properties. These features are expected to directly influence charge transfer processes and catalytic activity, providing the basis for the electrochemical behavior discussed in the following sections.

2.2. Electrolyte Screening and Initial OER Assessment

The electrocatalytic response of FeWO_4 was first evaluated in different electrolyte environments in order to identify the most suitable conditions for OER investigation and to probe the influence of pH on catalytic behavior. Figure 2 presents the linear sweep voltammetry (LSV) recorded in acidic (0.5 M H_2SO_4 , pH 0.5), inert (1 M Na_2SO_4 , pH 4) and alkaline (1 M KOH, pH 14) media, while the corresponding overpotentials at 10 mA cm^{-2} are summarized in Table 2. This measurement corresponds to the anodic current density evolution as a function of the applied potential. The current was divided by the area of the support used as the working electrode to present the LSV results as current density, and the potential was converted to the scale based on the standard hydrogen potential (RHE). The LSV curves showed similar profiles for all three electrolytes, with negligible faradaic current below 1.4 V. The initiation potential, which is defined as the point at which the current deviates significantly from its base value, is minimal in the case of a KOH electrolyte. Consequently, as the applied potential increases, the current density grows rapidly, resulting in the fastest kinetics.

Table 2 presents the overpotential results calculated from the LSV data according to the electrolytic solutions tested in the OER. At the commonly used reference current density $j = 10 \text{ mA cm}^{-2}$ [47], the overpotential corresponds to the additional voltage that must be applied beyond the thermodynamic potential for the electrolysis of water ($\approx 1.23 \text{ V}$ at 25°C , vs. RHE); it overcomes kinetic and transport losses [48].

A strong dependence of the OER activity on the nature of the electrolyte environment is observed. The overpotential decreases significantly from 720 mV in Na_2SO_4 and 670 mV in H_2SO_4 to 430 mV in KOH, clearly demonstrating that alkaline conditions promote more favorable reaction kinetics.

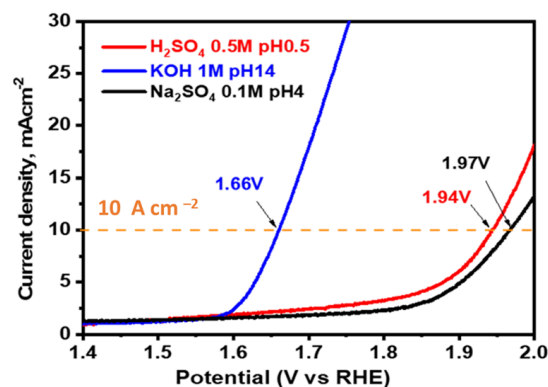


Figure 2. LSV performed on iron tungstate with a scan rate of $10 \text{ mV} \cdot \text{s}^{-1}$.

Table 2. Overpotential according to electrolyte solutions for the OER.

Solution	Concentration, M	pH	Overpotential, mV vs. RHE
H ₂ SO ₄	0.5	0.5	670
Na ₂ SO ₄	1	4	720
KOH	1	14	430

This behavior can be attributed to the OER mechanism in alkaline media, where the reaction proceeds via OH^- adsorption and subsequent formation of oxygenated intermediates at the catalyst surface. Although there is no direct evidence of the adsorption energy of surface intermediates, this mechanistic hypothesis is considered plausible, being consistent with the band-edge positions obtained in relation to the O_2/OH^- redox potential. The oxygen evolution reaction (OER) process has a standard thermodynamic potential of 1.23 V vs. RHE. For the reaction to initiate, the holes (h^+) generated in the valence band (E_{VB}) of the nanoplatelets must possess energy exceeding the water oxidation potential. Analyzing the band alignment makes it possible to evaluate the gap between the material's valence band level and the OER potential in order to determine the theoretical overpotential. The closer the valence band is to the ideal position, the lower the energy barrier for the formation of intermediates ($^*\text{OH}$, $^*\text{O}$, $^*\text{OOH}$), thereby increasing the current density for a given overpotential [49–51].

The high availability of OH^- ions in KOH facilitates these elementary steps, reducing kinetics barriers associated with charge transfer and intermediate formation. In contrast, under acidic conditions, the limited availability of reactive hydroxyl species and slower interfacial kinetics results in higher overpotentials. The higher overpotential observed in Na_2SO_4 compared to H_2SO_4 indicates that OER activity is not solely governed by hydroxide ion concentration. It reflects the limited reactivity of sulfate electrolytes, which neither provide sufficient OH^- for alkaline pathways nor promote efficient proton-coupled electron transfer as in strongly acidic media, resulting in slower interfacial kinetics.

Beyond the electrolyte effect itself, these results also provide insight into the intrinsic properties of FeWO_4 . The improved performance in alkaline media suggests that the material exhibits more efficient charge transfer and surface reactivity when interacting with hydroxide ions, which is consistent with the expected behavior of transition metal oxides in the OER.

Based on these findings, 1 M KOH was selected as the optimal electrolyte for all subsequent electrochemical analyses (LSV, EIS, and stability tests) because it provided the lowest overpotential to reach a current density equal to 10 mA cm^{-2} . This choice ensures that the catalytic performance is evaluated under conditions in which kinetic limitations are minimized, allowing for a more reliable comparison of the intrinsic effect of morphology on OER activity.

2.3. Morphology-Dependent Electrocatalytic Performance

2.3.1. OER Activity of FeWO₄ Morphologies

The intrinsic electrocatalytic activity of FeWO₄ was further evaluated in 1 M KOH, selected as the optimal electrolyte based on the previous analysis. As can be seen in Figure 3a, the polarization curves reveal a pronounced dependence of OER activity on morphology. To reach the benchmark current density of 10 mA·cm^{−2}, the nanoplatelet morphology requires a significantly lower potential (1.66 V vs. RHE) compared to the self-organized rosette structure (1.82 V vs. RHE), corresponding to overpotentials of 430 mV and 590 mV, respectively (Figure 3b).

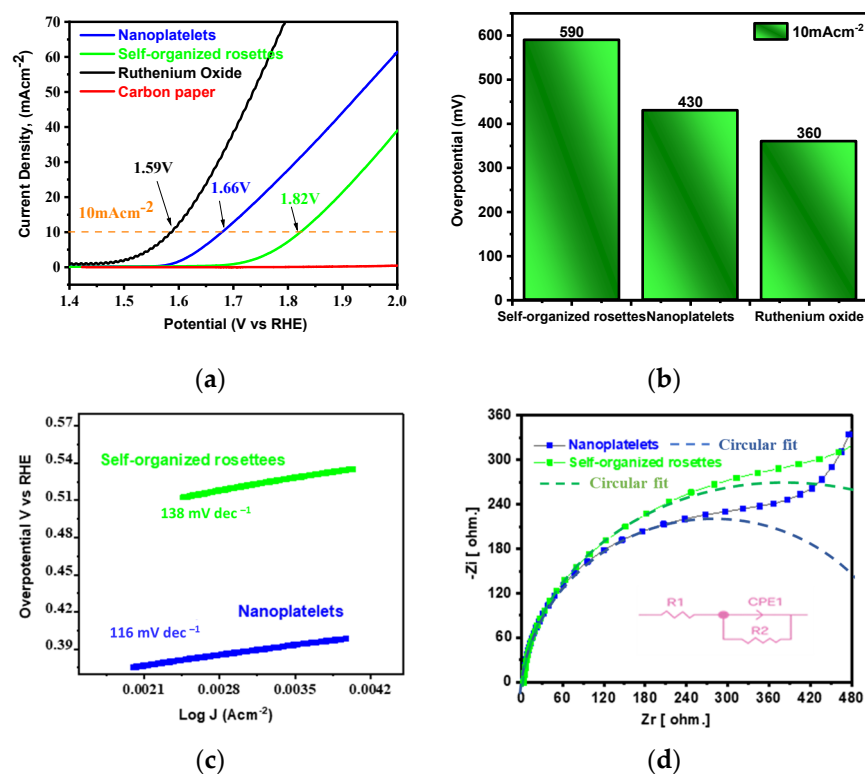


Figure 3. Electrochemical analysis of FeWO₄ (self-organized rosettes and nanoplatelets): (a) LSV, (b) Overpotential, (c) Tafel slope and (d) EIS; inset corresponds to the equivalent circuit.

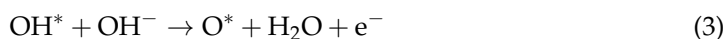
This substantial decrease of 138 mV reflects a morphology-driven enhancement of the catalytic interface for the nanoplatelet architecture, which provides increased accessibility to active sites and promotes more efficient interaction with OH[−] species, which are directly involved in the OER mechanism under alkaline conditions.

2.3.2. Kinetic Analysis and Reaction Mechanism

To gain further insight into reaction kinetics, Tafel analysis was performed based on the LSV data. The OER mechanism under alkaline conditions is represented by Equation (1), which involves the following four fundamental steps (Equations (2)–(5)).

The electrode kinetics and the possible mechanisms involved in the OER process were investigated using Tafel analysis. The OER mechanism under alkaline conditions is represented by Equation (1), which involves the following four fundamental steps (Equations (2)–(5)) [52,53].





In these equations, * indicates the catalyst active site, and OH*, O*, and HOO* represent the species adsorbed at the active catalytic site. To better understand the OER mechanisms, Tafel slopes provide further insight into the reaction kinetics beyond LSV analysis. The extracted values (Figure 3c) are 116 mV dec^{−1} for the nanoplatelets and 138 mV dec^{−1} for the self-organized rosettes morphology. The lower slope observed for the nanoplatelets indicates faster reaction kinetics and a reduced energy barrier for the rate-determining step.

Both values remain close to ~120 mV·dec^{−1}, suggesting that the first electron transfer step, corresponding to OH[−] adsorption (Equation (2)), is likely the rate-determining step for both morphologies. The reduced slope for the nanoplatelets indicates that this elementary step is kinetically facilitated, which is consistent with improved surface reactivity and active site accessibility.

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The equivalent circuit included in the Nyquist diagram of EIS (inset of Figure 3d) provides a better understanding of the charge transfer resistance and interfacial electron transfer behavior of the catalysts. R1 (solution resistance) represents the overall ohmic resistance of the system, R2 (charge transfer resistance, R_{ct}) corresponds to the resistance associated with electron transfer at the electrode/electrolyte interface, and CPE1 (constant phase element) represents the CPE, which is used to model the non-ideal capacitive behavior of the electrical double layer. This equivalent circuit generally describes a system dominated by a single main electrochemical process: ionic conduction in the electrolyte (R1), formation of the electrical double layer (CPE1), and charge transfer at the electrode/electrolyte interface (R2). The presence of a single semicircle in the Nyquist diagram is generally associated with a single charge transfer process.

2.3.3. Charge Transfer Properties

Electrochemical impedance spectroscopy (EIS) provides complementary information about charge transfer processes at the electrode/electrolyte interface. As can be seen in Figure 3d, the Nyquist plots show a clear reduction in semicircle diameter for the nanoplatelet morphology, corresponding to a lower charge transfer resistance (R_{ct} = 0.52 kΩ) compared to the self-organized rosette structure (0.72 kΩ). This decrease in R_{ct} directly reflects more efficient electron transfer kinetics, which is a key factor governing OER activity. The consistent trends observed in EIS, Tafel slope, and overpotential results clearly indicate that the nanoplatelet morphology enhances both interfacial charge transfer and OER kinetics.

2.3.4. Integrated Structure–Property–Performance Relationship

Taken together, these results demonstrate that the improved OER performance of FeWO₄ nanoplatelets originates from a combination of factors induced by morphology: (i) increased density of accessible active sites, (ii) enhanced OH[−] adsorption and activation, and (iii) reduced charge transfer resistance. The convergence of independent electrochemical descriptors (overpotential, Tafel slope and R_{ct}) provides strong evidence that morphology acts as a primary factor governing catalytic performance in this system.

2.3.5. Stability Assessment

Beyond activity and kinetics, the operational stability of the electrocatalysts is a critical parameter for practical applications and must be evaluated to validate the relevance of any observed performance differences. The stability of both morphologies was evaluated through accelerated cycling (1000 cycles) and chronopotentiometry measurements over 14 h at a fixed current density of $10 \text{ mA} \cdot \text{cm}^{-2}$ (Figure 4a,b), which state that durations greater than 12 h are generally considered stable materials [13,35,43].

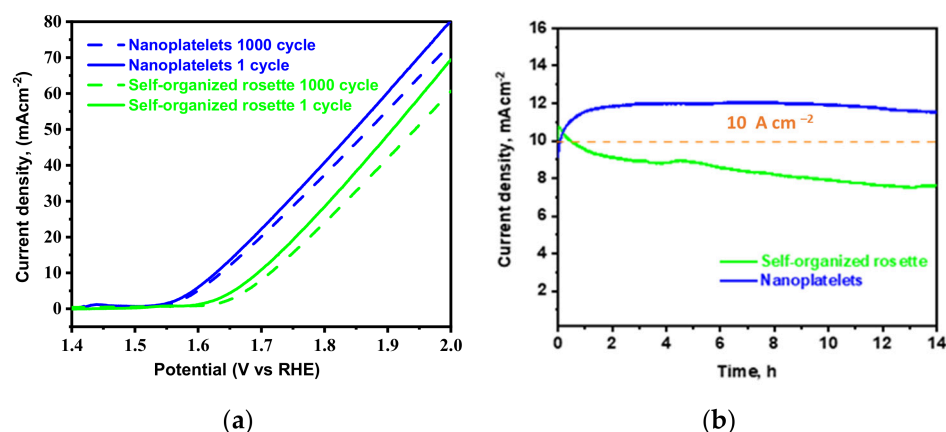


Figure 4. Electrochemical analysis of FeWO_4 (self-organized rosettes and nanoplatelets): (a) Stability (1000 cycles) and (b) Chronopotentiometry (14 h).

Relatively good LSV stability is observed after 1000 cycles. In the case of the tungstate with nanoplatelet morphology, the results indicate relatively good stability over this period (14 h). In the first few minutes there is an increase in current density, but it stabilizes and remains practically constant over time. In this case, a significant increase in oxygen can be observed at the beginning, but it stabilizes over time. However, for the self-organized rosette morphology, there is a 25% reduction over the 14 h period. This indicates that tungstate with a nanoplatelet morphology is more stable.

Both morphologies exhibit relatively good stability during cycling, with minimal deviation between initial and post-cycling LSV curves (Figure 4a), indicating structural robustness under repeated operation. However, significant differences emerge during long-term operation. The nanoplatelet morphology maintains a nearly constant current density over 14 h, after a short initial stabilization period, whereas the self-organized rosette structure exhibits a progressive decay of approximately 25%. This enhanced stability of the nanoplatelets can be attributed to their morphology, which likely promotes more uniform current distribution and improved structural integrity during OER. The combination of higher activity and superior stability further reinforces the role of morphology as a key determinant of overall electrocatalytic performance.

To verify the chemical integrity of the FeWO_4 electrodes, the electrolyte was analyzed using inductively coupled plasma (ICP) spectroscopy after the stability test. Measurements were taken for 100 g of catalyst dispersed in 100 mL of solution. For both morphologies, no significant trace of tungsten was detected compared to the reference values (noise). For iron cations, the measurements at pH 5 and 10 show mean values of 14 and 95 $\mu\text{g/L}$, respectively, for nanoplatelet and self-organized rosette samples. These values remain relatively negligible, particularly for nanoplatelets. However, at pH 3 in an acidic medium, the concentrations were high, exceeding 500 $\mu\text{g/L}$, reflecting the instability of both catalysts in acidic media. The low concentrations of iron ions in the solution indicate that leaching through the cycles is negligible for nanoplatelets. The ability to maintain effi-

ciency over multiple cycles demonstrates the ferberite's stability and efficiency for practical photodegradation applications.

After the stability tests, scanning electron microscopy analyses were performed and no significant morphological variations were observed after the reaction tests, as shown in Figure 5. Post-reaction SEM analysis revealed the preservation of the initial morphology for both iron tungstate morphologies tested. These results support the conclusion that the material exhibits good short-term operational stability.

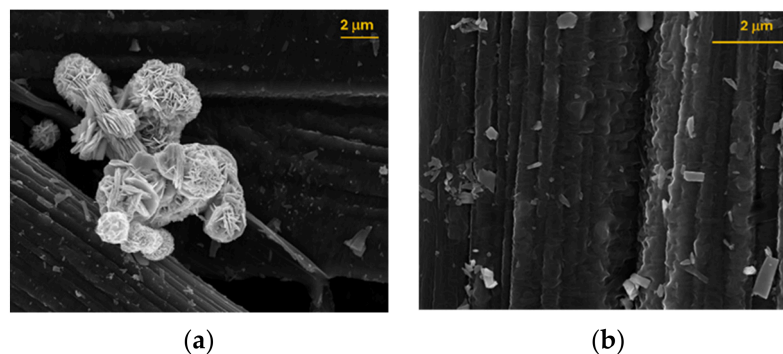


Figure 5. SEM images of both morphological types after reaction-stability tests. (a) Self-organized rosettes and (b) nanoplatelets.

2.4. Surface Accessibility, Intrinsic Activity, and Oxygen Production

2.4.1. Electrochemically Active Surface Area (ECSA)

The electrochemically active surface area (ECSA) was estimated from cyclic voltammetry measurements performed in the non-faradaic region, as shown in Figure 6a,b. The plots of current density versus scan rate revealed a linear relationship for both morphologies, confirming that the measured current originates from double-layer charging and validating the extraction of the double-layer capacitance (C_{dl}) [54].

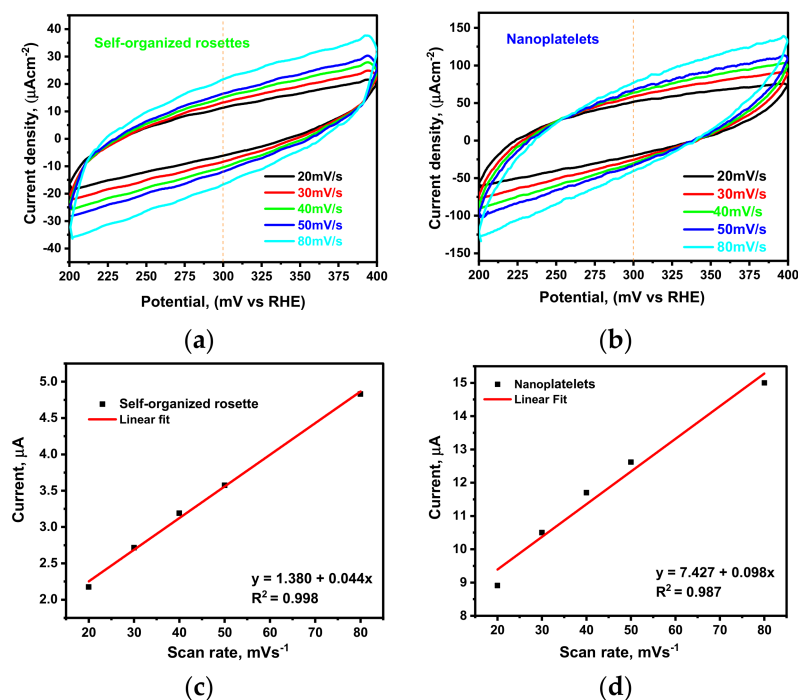


Figure 6. Cyclic voltammetry measurements in the non-faradaic region for FeWO_4 self-organized rosette structures (a) and nanoplatelets (b) recorded at different scan rates, and corresponding capacitive current density versus scan rate plots for self-organized rosette structures (c) and nanoplatelets (d), was obtained at the median voltage indicated by the dashed line.

The nanoplatelet morphology exhibits a significantly higher slope, corresponding to a larger C_{dl} and, consequently, a higher ECSA (2.45 cm^2) compared to the self-organized rosette structure (1.10 cm^2). This result indicates a greater density of electrochemically accessible active sites [55], consistent with the higher BET surface area and the two-dimensional architecture discussed in Section 2.1 [55].

2.4.2. Correlation Between ECSA, Intrinsic Catalytic Activity and Oxygen Production

Finally, the theoretical amount of O_2 generated, estimated from the charge transferred according to Faraday's law assuming 100% Faradaic efficiency (mmoles h^{-1} , C_{dl} (mF), ECSA (cm^2), RF (dimensionless), and TOF (s^{-1}) using 1 M KOH pH 14 as the electrolyte solution as a function of the iron tungstate morphology type, is presented in Figure 7. In order to decouple the contribution of surface effects from intrinsic catalytic activity, and correlate these parameters with oxygen evolution performance, the turnover frequency (TOF), roughness factor (RF) and oxygen production rate were evaluated for both FeWO_4 morphologies (Figure 7). Nanoplatelet morphology exhibits a markedly higher TOF compared to the self-organized rosette structure, indicating that each site is intrinsically four times more active. This result demonstrates that the superior electrocatalytic performance of the nanoplatelets is not solely a consequence of a larger electrochemically accessible surface area but also arises from more favorable catalytic properties at the active site level. Such enhancement may originate from morphology-induced modifications of the surface structure, including differences in exposed crystallographic facets, defects, and local electronic environment, which can facilitate the adsorption and conversion of OER intermediates. In addition, the roughness factor values further support the beneficial effect of the nanoplatelet morphology, indicating a larger effective electrochemically active interface.

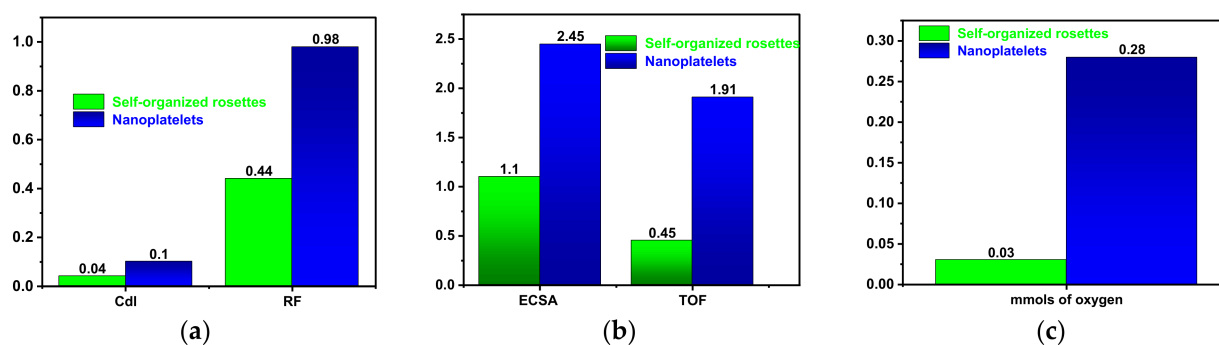


Figure 7. (a) C_{dl} (mF) and RF (dimensionless), (b) ECSA (cm^2) and TOF (s^{-1}), and (c) O_2 production (mmols h^{-1}) using 1 M KOH pH 14 as the electrolyte solution.

The combined increase in ECSA, RF and TOF is directly reflected in the oxygen evolution rate. As shown in Figure 7, nanoplatelet morphology achieves an oxygen production rate of 0.28 mmol h^{-1} , whereas the self-organized rosette structures produce only 0.03 mmol h^{-1} . This nearly 10-fold increase in oxygen production significantly exceeds the variation expected solely from the ECSA enhancement ($\sim 2.2\times$), further supporting the existence of a substantial intrinsic activity improvement in the nanoplatelet morphology ($\sim 4.2\times$).

Overall, these results establish a clear structure–property–performance relationship in which morphology governs OER activity through a dual effect: (i) increasing the density of accessible sites (ECSA effect) and (ii) enhancing the intrinsic catalytic activity of these sites (TOF effect). The convergence of independent electrochemical descriptors, including overpotential, Tafel slope, charge transfer resistance, ECSA and TOF, provides strong evidence that morphology acts as a primary factor governing catalytic performance in this system. Certainly, it demonstrates that nanoplatelet morphology does not merely provide a

larger reactive surface but fundamentally modifies the catalytic behavior of FeWO_4 , leading to a substantial improvement in oxygen evolution efficiency.

3. Discussion

3.1. Morphology-Induced Modulation of Structural and Electronic Properties

Although both FeWO_4 samples crystallize in the same monoclinic ferberite structure (P2/c space group), the change in morphology induces significant modifications in their surface and electronic characteristics. Beyond surface accessibility, morphology acts as a fundamental lever for modifying intrinsic kinetic properties. In particular, the nanoplatelet morphology exhibits a substantially larger specific surface area ($12.65 \text{ m}^2 \cdot \text{g}^{-1}$) than the self-organized rosette structures ($6.80 \text{ m}^2 \cdot \text{g}^{-1}$), together with a well-defined two-dimensional architecture. Such a morphology is expected to enhance availability of reactive surface centers and facilitate electrolyte diffusion at the catalyst/electrolyte interface. The improved intrinsic activity of the nanoplatelet morphology may also be associated with preferential exposure of specific crystallographic planes, particularly the [101] axis orientation previously identified for this morphology, as presented in greater detail in our previous study [45].

Another important observation concerns the optical properties. Nanoplatelet morphology exhibits a slightly wider band gap (1.77 eV) compared to the self-organized rosette structures (1.69 eV). Despite this increase, both morphologies maintain valence band positions sufficiently positive relative to the O_2/OH^- redox potential, ensuring the thermodynamic feasibility of oxygen evolution. Moreover, the slightly more positive valence band position observed for the nanoplatelets suggests a stronger oxidative capability, which may contribute to enhanced OER kinetics.

Importantly, these electronic differences occur simultaneously with the morphological evolution, indicating that morphology not only modifies the geometric surface characteristics of FeWO_4 but also influences its interfacial electronic behavior. The synergy between favorable band alignment, enhanced charge-transfer properties, and increased active site accessibility provides a coherent explanation for the superior electrocatalytic activity observed for the platelet morphology.

3.2. Influence of the Electrolyte on OER Activity

The electrolyte-dependent electrochemical measurements further highlight the importance of interfacial reaction conditions in governing OER activity. Among the investigated electrolytes, 1 M KOH exhibited the best electrocatalytic performance, whereas moderate and strongly acidic media resulted in significantly higher overpotentials. This behavior is consistent with the generally more favorable OER kinetics in alkaline media, where OH^- ions directly participate in the adsorption and oxidation steps involved in oxygen evolution. In contrast, acidic environments can alter the adsorption energetics of oxygenated intermediates and limit the efficiency of interfacial charge-transfer processes. The superior activity observed in KOH therefore indicates that FeWO_4 operates more efficiently under alkaline conditions, where the reaction pathway and surface chemistry are kinetically more favorable. These findings are consistent with previous studies reported by Li et al. [47], who demonstrated that alkaline electrolytes significantly reduce the overpotential of water electrolysis compared with acidic media. Their work showed that 1 M KOH provided the lowest overpotential among several investigated electrolyte configurations, emphasizing the critical role of electrolyte pH in governing electrocatalytic efficiency.

It should be noted that in this work, the electrolyte screening also established the experimental framework used for morphology comparison. By selecting the electrolyte providing the most favorable kinetics, the subsequent analyses could more reliably isolate the intrinsic influence of morphology on OER performance.

3.3. Morphology-Dependent Electrocatalytic Activity of FeWO_4

The electrochemical analyses consistently demonstrate that the nanoplatelet morphology exhibits superior OER performance compared to the self-organized rosette structures. The lower overpotential, smaller Tafel slope, reduced charge-transfer resistance, higher electrochemically active surface area, and larger turnover frequency collectively indicate that morphology plays a central role in governing the catalytic behavior of FeWO_4 .

The decrease in charge-transfer resistance confirms that the nanoplatelet morphology facilitates faster interfacial electron-transfer kinetics [27], which directly contributes to improved electrocatalytic activity. This interpretation is supported by the simultaneous reduction in both overpotential and Tafel slope [52,53], indicating that the enhanced catalytic behavior does not originate from a single electrochemical parameter but from an overall improvement of the reaction kinetics at the catalyst/electrolyte interface.

Notably, the Tafel slopes obtained for both morphologies (116 and 138 $\text{mV}\cdot\text{dec}^{-1}$) remain close to the characteristic value of 120 $\text{mV}\cdot\text{dec}^{-1}$ generally associated with the first electron-transfer step of OH^- adsorption being the rate-determining step [46]. This suggests that both catalysts follow a similar OER mechanism, while the nanoplatelet morphology accelerates the kinetics of the elementary reaction steps [56]. However, this work does not present direct evidence regarding adsorption energetics or surface intermediates; the interpretations are based on electrochemical parameters, particularly the Tafel slope.

The Tafel slope describes the rate at which current density increases with applied potential. The smaller the Tafel value, the smaller the potential increment required to increase the current by an order of magnitude, indicating faster charge transfer kinetics. Looking at the table, iron tungstate, which has a Tafel slope of 48.8 mV dec^{-1} , exhibits better kinetics than the other iron tungstate morphologies presented in the same table. Values close to 40–60 mV dec^{-1} are frequently associated with OER mechanisms in which electron transfer occurs rapidly and the rate-limiting step involves surface chemical transformations of the adsorbed intermediates ($^*\text{OH}$, $^*\text{O}$, and $^*\text{OOH}$). This is a typical example where a low Tafel slope does not necessarily imply lower overpotential. In other words, despite the low Tafel value, the corresponding overpotential value is high (459 mV), demonstrating that even with intrinsically favorable kinetics for the OER, the number of available active sites is probably limited, or the electrochemically active area may be limited, as there may be mass transport limitations.

In the case of the particle morphology (60 mV dec^{-1}), this sample presents both a low Tafel slope and a lower overpotential (403 mV), suggesting that this morphology provides greater electrical connectivity between particles, lower charge transfer resistance, and a more homogeneous distribution of catalytic sites. Self-organized rosettes exhibit the worst kinetic performance (highest Tafel slope and highest overpotential), which may be associated with internal regions less accessible to the electrolyte. That is, not all surface area is necessarily electrochemically active. Meanwhile, the nanoplatelet morphology (116 mV dec^{-1}) shows intermediate performance. The overpotential of 430 mV is relatively close to that of the particle morphology, but the Tafel slope is significantly higher. In this case, the reaction achieves 10 mA cm^{-2} with a relatively moderate overpotential but requires larger potential increments to further increase the current. This is characteristic of systems where the active area is high, but electron transfer remains limited. However, the first two samples were measured on glassy carbon and the others on carbon paper. Therefore, part of the observed difference may not be solely associated with the morphological change in the iron tungstate, as the substrate influences the overall electrical resistance, catalyst adhesion, effective exposed area, current distribution, and the electrical contact between catalyst and collector. Thus, a direct comparison between the obtained Tafel and overpotential values using different substrates presented in the literature cannot be made,

as the substrate also contributes to the electrocatalytic response. But this is a potential difference between the two morphologies presented in this work, where all conditions are exactly the same, with only morphology as a variable.

The ECSA and TOF analyses provide further insight into the origin of the enhanced activity. The platelet morphology exhibits both a larger electrochemically accessible surface area and higher intrinsic catalytic activity per active site. Importantly, the increase in oxygen production largely exceeds the increase in ECSA alone, demonstrating that the performance enhancement cannot be explained solely by surface-area effects.

Such behavior strongly suggests that the two-dimensional nanoplatelet architecture modifies the catalytic properties of the FeWO_4 surface itself. In addition to increasing the density of accessible active sites, nanoplatelet morphology may promote more favorable exposed crystallographic facets, local electronic environments, and adsorption energetics of OER intermediates. Similar morphology-dependent improvements in charge-transfer properties and catalytic activity have previously been reported for FeWO_4 -based systems and related tungstate electrocatalysts [35,57].

3.4. Comparison with Reported Tungstate-Based Electrocatalysts

Table 3 aims to provide a broader overview of the diversity of tungstate-based OER catalysts and to highlight the influence of experimental conditions on the electrochemical parameters reported in the literature, particularly regarding the various tungstate morphologies and the different substrates used as the working electrode. Our primary objective at this stage of the work is to independently evaluate the effect of iron tungstate morphology on OER activity under strictly identical experimental conditions, comparing the two proposed morphologies (platelets and self-organized structures), given that both FeWO_4 samples were synthesized, deposited, and tested under the same conditions, with morphology being the only intentionally varied parameter.

Table 3. Tungstate oxide electrocatalysts, Tafel slope and overpotential at 10 mA cm^{-2} .

Electrocatalyst	Morphology	Tafel Slope (mV dec^{-1})	Overpotential (10 mA cm^{-2})	Substrate	Study
ZnWO_4	rods	155	537	nickel foams	[58]
CoWO_4	cubes	82	450 mV	glassy carbon	[59]
CoWO_4	spheres	46 ± 1	388 ± 2	glassy carbon	[60]
CoWO_4	spheres	44 ± 1	336 ± 3	nickel foam	
NiWO_4	spheres	40 ± 1	507 ± 3	glassy carbon	
NiWO_4	spheres	34 ± 1	363 ± 5	nickel foam	[61]
CuWO_4	spheres	83	242	nickel foam	
FeWO_4	rods	54	285	nickel foams	[35]
FeWO_4	particle	60	403	glassy carbon	[41]
FeWO_4	-	48.8	459	glassy carbon	[9]
FeWO_4	self-organized rosettes	138	553	carbon paper	Present Work
FeWO_4	nanoplatelets	116	430	carbon paper	Present Work

To further evaluate the relevance of the present results, the electrocatalytic performance of FeWO_4 was compared with previously reported wolframite-type tungstate electrocatalysts, including ZnWO_4 , CoWO_4 , NiWO_4 , CuWO_4 , and FeWO_4 systems investigated under alkaline conditions (1 M KOH) (Table 3). The comparison reveals that catalysts deposited on highly conductive substrates such as nickel foam generally exhibit lower overpotentials and Tafel slopes due to improved electrical conductivity and enhanced catalyst adhesion [59]. For instance, CuWO_4 deposited on nickel foam exhibits particularly low overpotential values [61], while spherical structures of CoWO_4 and NiWO_4 also

show enhanced performances when supported on conductive substrates. In the specific case of FeWO_4 , previous studies reported significant improvements when evolving from particle-like structures to anisotropic morphologies such as nanorods [35]. This trend is consistent with the present work, where the nanoplatelet morphology clearly outperforms the self-organized rosette structures. However, a direct comparison is limited by differences in the substrate, charge, electrolyte, and normalization protocol.

It is important to emphasize, however, that the present performances were obtained using carbon paper, which is less conductive and catalytically less active than commonly used substrates such as nickel foam. This choice minimizes substrate-induced enhancement effects and highlights the intrinsic contribution of morphology to OER activity. Under these more conservative experimental conditions, the results reinforce the conclusion that morphology engineering alone can significantly enhance the intrinsic electrocatalytic properties of FeWO_4 , even in the absence of doping, heterojunction formation, or highly conductive supports.

Overall, the present work highlights morphology engineering as an effective strategy for optimizing tungstate-based electrocatalysts and provides a clearer understanding of the intrinsic structure–property–performance relationship governing OER activity in FeWO_4 .

4. Materials and Methods

4.1. Synthesis Methodologies and Characterizations of Iron Tungstate

The iron tungstate nanostructures used in this study were synthesized by the hydrothermal method following the procedure optimized and reported in our previous publication [45]. In brief, the FeWO_4 powders were prepared using a mixture of sodium tungstate dihydrate and iron (II) sulfate heptahydrate precursors. First, the mixture was diluted in 100 mL of water and stirred for 10 min at room temperature and an acidic pH (pH 1). Then, after stirring, solid oxalic acid was added. Finally, the mixture was transferred to an autoclave, heated to 200 °C for 24 h and then cooled naturally. The precipitate was collected, washed several times with water, and dried at 80 °C for 24 h. The obtained FeWO_4 powders were structurally investigated by an Empyrean Panalytical diffractometer, in the classic θ – 2θ mode, from 10° to 80° with step size of 0.003° and scan speed of 0.004° s^{−1}.

The morphology of the different powders was observed by transmission electron microscopy (TEM) and characterized by electron diffraction (ED) using a 200 kV FEI Tecnai G2 transmission electron microscope (Thermo Fisher Scientific, Waltham, MA, USA). The chemical composition was determined by X-ray energy-dispersive spectroscopy (EDS), while the surface morphology was studied with a Zeiss SUPRA 40 Vp scanning electron microscope (SEM, GEMINI, Oberkochen, Germany). Nitrogen physisorption isotherms were measured at 77 K using a Quantachrome Autosorb iQ AG surface area (USA). The Brunauer–Emmett–Teller (BET) method was used to deduce the specific surface area from the obtained isotherms. Finally, the optical properties were studied by UV–Vis spectroscopy, in diffuse reflectance modes DRS, using a Shimadzu 2600 UV/Vis spectrophotometer (Shimadzu Corporation, Kyoto, Japan). These results are fully consistent with our earlier structural findings [45].

To verify the preservation of morphology after exposure during a long reaction period, scanning electron microscopy analyses without any specific prior treatment were performed for both iron tungstate morphologies after electrochemical reaction tests.

4.2. Electrochemical Measurements and Electrodes

Electrocatalytic tests were performed in a reaction system consisting of a three-electrode electrochemical cell connected to a potentiostat and a computer, as shown in

Figure 8. The working electrode surface and the Ag/AgCl (3 M) reference electrode are positioned close to each other in the cell to minimize ohmic drop. All electrochemical properties and measured reactions were performed at room temperature, without any type of purge (gas (N₂, Ar)), additives, or contaminants. To prepare the working electrode, 5 mg of iron tungstate powder was dispersed in 1 mL of ethanol and sonicated for 30 min to ensure complete dispersion. It was then deposited on a carbon paper-based support (Toray Carbon Paper, TGP-H-60, 19 × 19), which was heated to 80 °C for approximately 2 h for complete evaporation of the ethanol used in the dispersion step (Figure 8b).

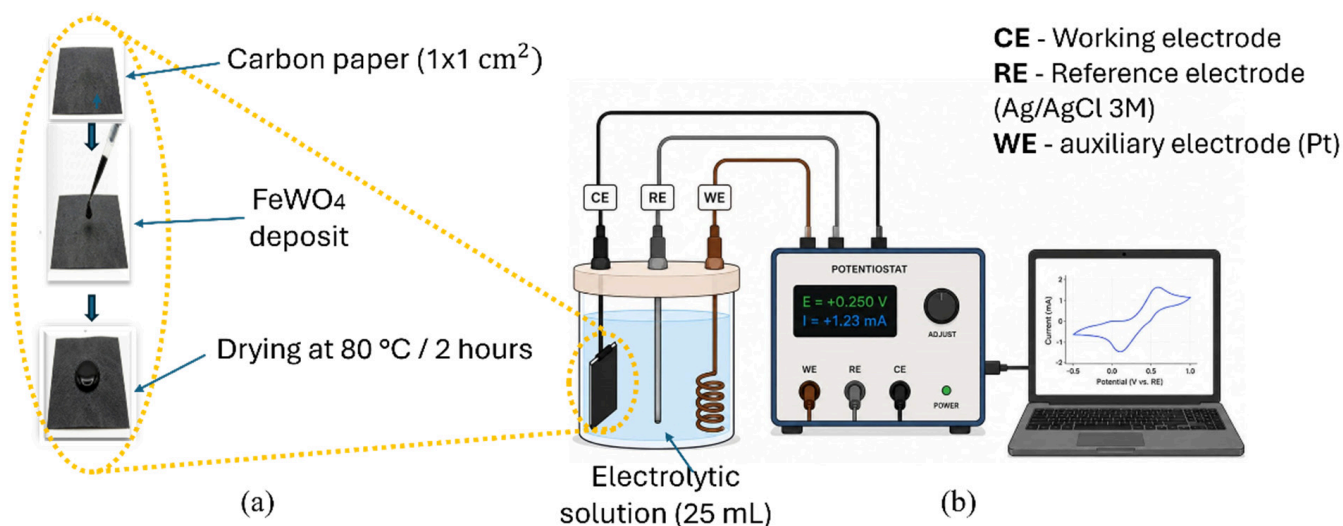


Figure 8. Electrochemical system: (a) Preparation of the working electrode and (b) electrolysis device.

All electrochemical characterizations (linear voltammetry, cyclic voltammetry, EIS, photocurrent, Mott–Schottky, chronoamperometry, and stability) were conducted using an OrigaLys (OrigaLys ElectroChem SAS, Lyon, France) potentiostat/galvanostat workstation, in a standard three-electrode configuration. All analyses were performed at room temperature.

To evaluate the performance of the materials synthesized for the oxygen evolution reaction (OER), a comprehensive electrochemical study was conducted, beginning with the selection of an optimal electrolyte. Potential candidates, including potassium hydroxide (KOH), sulfuric acid (H₂SO₄), and sodium sulfate (Na₂SO₄), were screened to identify the solution with the highest ionic mobility, which is essential for reducing thermodynamic barriers and energy consumption during water electrolysis. The solutions and their respective concentrations to be tested were determined based on the literature (Table 4).

Table 4. Electrolytic solutions tested at the OER.

Solutions	Concentration, M	pH	Studies
H ₂ SO ₄	0.5	0.5	[62,63]
Na ₂ SO ₄	1	4	[64,65]
KOH	1	14	[66]

Once the electrolyte had been defined, the catalytic activity was measured using linear sweep voltammetry (LSV) at a scan rate of 10 mV s^{−1} over a potential range of 1.0 to 2.0 V (relative to the RHE). All LSV data were iR-corrected and the raw current measurements were normalized to current density (*j*) by dividing them by the working electrode area [39], where E_{Ag/AgCl} is 0.197 V vs. NHE at 25 °C.

The overpotential (η) for the OER of the different morphologies (self-organized rosettes and nanoplatelets) was determined from the LSV measurement for the equivalent current

of 10 mA cm^{-2} and then calculated using Equation (6) [19]. To evaluate the reaction kinetics and identify the rate-limiting step, Tafel slopes were generated by plotting the overpotential (η) against the log of the current density (j) (Equation (7)). The resulting Tafel slope and the overpotential required to reach a reference current density of 10 mA cm^{-2} were used as primary efficiency indicators. Subsequently, the electrical characteristics of the electrode interface were further probed using Electrochemical Impedance Spectroscopy (EIS) by applying a 5 mV AC signal across a frequency range of 10 kHz to 0.01 Hz at a constant 1.55 V. The charge transfer resistance was quantified.

To estimate the electrochemically active surface area (ECSA) and the roughness factor (RF), CV was performed in the non-faradaic region (200–400 mV vs. Ag/AgCl) at varying scan rates (20, 30, 40, 50 and 80 mV s^{-1}). For this estimate, Equation (8) was used [35]. The double-layer capacitance (Cdl) was derived from the slope of the current density versus scan rate, which provided a basis for calculating the total available active sites. Here, Cs is the specific capacitance of standard electrode materials measured per unit surface area (0.04 mF cm^{-2}) [37].

4.3. Electrolyte Screening and OER Performance Evaluation

Finally, the production of oxygen during OER was estimated based on the total charge passed through the system, using Equations (9) and (10) [67], where I is the current (A), t is the time (s), n is the number of electrons transferred (four for OER), and F is the Faraday constant ($F = 96,485 \text{ C mol}^{-1}$). While overpotential and current density describe the electrode's general activity, the turnover frequency (TOF) was calculated to determine each individual active site's catalytic power. The roughness factor (RF) was determined for both morphologies, according to Equation (11) [68], where $A_{\text{geometric}}$ is the geometric area and the electrochemically active surface area (ECSA) is given by Equation (12).

To estimate the turnover frequency (TOF), cyclic voltammetry measurements were performed at various scan rates within a non-faradaic region, where the current contribution is predominantly capacitive and redox processes are negligible. Initially, the capacitive current (Δi) was calculated using the relationship $\Delta i = (i_a - i_c)/2$, where i_a and i_c are the anodic and cathodic currents, respectively, obtained at a fixed potential (300 mV). Subsequently, the capacitive current values were plotted against the scan rate; the slope of the resulting line represents the double-layer capacitance (Cdl), assuming a linear dependence between capacitive current and scan rate. The electrochemically active surface area (ECSA) was estimated from the electric double-layer capacitance (Cdl) using Equation (13) and a specific capacitance value (Cs) of 0.04 mF cm^{-2} , a value widely used in the literature for metal surfaces and metal oxides in alkaline media [69]. Although specific capacitance can vary depending on chemical composition, surface roughness, porosity, and the electrolyte used, the choice of 0.04 mF cm^{-2} allows for a consistent comparison among different materials reported in the literature [39,69]. Next, the number of surface active sites (Ns) was estimated using Equation (13), assuming a surface density of active sites (Ds) of $1.0 \times 10^{16} \text{ sites cm}^{-2}$, a value frequently used to represent the surface atomic density of compact metal oxides. This approximation assumes a homogeneous distribution of catalytic sites across the electrochemically accessible surface and that the entire ECSA contributes to the reaction. Finally, the TOF was calculated using Equation (14), where J corresponds to the current density obtained during the oxygen evolution reaction (OER), A is the geometric area of the electrode, F is the Faraday constant ($96,485 \text{ C mol}^{-1}$), and the factor of 4 represents the number of electrons required to form one O_2 molecule. It is worth noting that the TOF obtained via this method represents an apparent estimate, as the actual number of catalytically active sites cannot be determined directly from Cdl measurements. Thus, the calculation relies on approximations widely accepted in the

literature and is particularly useful for relative comparisons between samples prepared and evaluated under identical experimental conditions.

To evaluate long-term durability, the electrode performance was verified through 1000 continuous cycles and a 14 h chronoamperometry (CA) stability test at a fixed current density of $10 \text{ mA} \cdot \text{cm}^{-2}$, ensuring the electrodes could withstand prolonged operational stress. To assess the stability of the material and the risk of metal ions leaching during electrochemical testing, the electrolyte was analyzed using inductively coupled plasma mass spectrometry (ICP-MS) after the tests.

$$\eta_{\text{OER}} = E(\text{RHE}) - 1.23 \quad (6)$$

$$\eta_{\text{OER}} = b * \log j + a \quad (7)$$

$$\text{ECSA} = \frac{\text{Cdl}}{\text{Cs}} \quad (8)$$

$$n(\text{mol}) = \frac{Q}{n * F} \quad (9)$$

$$Q = I * t \quad (10)$$

$$\text{RF} = \frac{\text{ECSA}}{A_{\text{geometric}}} \quad (11)$$

$$\text{Ns} = \text{ECSA} \times \text{Ds} \quad (12)$$

$$\text{ECSA} = \frac{\text{Cdl}}{\text{Cs}} \quad (13)$$

$$\text{TOF} = \frac{J * A}{4 * F * \text{Ns}} \quad (14)$$

5. Conclusions

This study confirms that anisotropic morphology engineering is a key factor in optimizing the electrocatalytic performance of FeWO_4 in the oxygen evolution reaction (OER). Although both studied morphologies maintain the ferberite monoclinic structure, the nanoplatelet morphology exhibits superior catalytic efficiency compared to self-organized rosette structures, due to a synergistic combination of intrinsic geometric and electronic advantages. The nanoplatelet morphology's slightly wider bandgap (1.77 eV) and more positive valence band position enhance its oxidative capability, thereby accelerating OER kinetics and reducing charge-transfer resistance. Its 2D form provides a surface area that is almost double that of self-organized rosette structures, creating more active sites and enabling the electrolyte to flow more easily across the preferentially exposed (100) plane of the surface.

Electrolyte screening identified 1 M KOH solution as the optimal medium, where high facilitates alkaline OER kinetics and interfacial OH^- adsorption. Under these conditions, the nanoplatelet morphology achieves lower overpotentials and reduced Tafel slopes ($116 \text{ mV} \cdot \text{dec}^{-1}$), indicating faster electron transfer kinetics during the rate determining step of OH^- adsorption. The significantly higher TOF ($\times 4.2$) for the nanoplatelets confirms that the 2D structure modifies the catalytic nature of the surface itself, promoting more favorable adsorption energetics for OER intermediates. These findings establish FeWO_4 nanoplatelets as a high-performance, stable, and cost-effective candidate for alkaline water electrolysis, offering a clear pathway for future design strategies in transition metal tungstate systems.

Author Contributions: Conceptualization, Investigation, Supervision, Writing—review and editing, M.A. and C.L.; Investigation, Data curation, Formal analysis, Writing—original draft preparation, Y.E., A.G.d.S. and V.C. All authors have read and agreed to the published version of the manuscript.

Funding: This study is supported by the Departmental Council of Var (CD83), the urban community of Toulon Provence Mediterranean and University of Toulon for their financial support within the framework of the “NanoCat”–“Disolar, Acte no: CO 2023-1273” and “NSPEC, TPM, Acte No 24/150” projects.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Data are available upon request.

Acknowledgments: The authors gratefully acknowledge the Regional Council of Provence-Alpes Cote d’Azur, Departmental Council of Var (CD83), the urban community of Toulon Provence Mediterranean TPM and University of Toulon for their financial support. A. GOMES DOS SANTOS thanks UFRSA/CCEN/DCME for taking care of her delegation within the PhD framework.

Conflicts of Interest: The authors declare that there are no conflicts of interest.

References

1. Graham, E.; Fulghum, N.; Altieri, K. *Global Electricity Review 2025*; Ember: London, UK, 2025.
2. Executive Summary—Renewables 2024—Analysis. Available online: <https://www.iea.org/reports/renewables-2024/executive-summary> (accessed on 1 March 2026).
3. Creamer, T. Cost-Competitiveness to Underpin Surge in Global Renewables Roll-out to 2030—IEA. Available online: <https://www.engineeringnews.co.za/article/cost-competitiveness-to-underpin-surge-in-global-renewables-roll-out-to-2030---iea-2024-10-09> (accessed on 1 March 2026).
4. Oliveira, M.L.M.; Alves, C.M.A.C.; Andrade, C.F.; de Azevedo, D.C.S.; Lobo, F.L.; Fuerte, A.; Ferreira-Aparicio, P.; Caravaca, C.; Valenzuela, R.X. Recent Progress and Perspectives on Functional Materials and Technologies for Renewable Hydrogen Production. *ACS Omega* **2025**, *10*, 3282–3303. [CrossRef] [PubMed]
5. Tabu, B.; Akers, K.; Yu, P.; Baghirzade, M.; Brack, E.; Drew, C.; Mack, J.H.; Wong, H.-W.; Trelles, J.P. Nonthermal Atmospheric Plasma Reactors for Hydrogen Production from Low-Density Polyethylene. *Int. J. Hydrogen Energy* **2022**, *47*, 39743–39757. [CrossRef]
6. Alves, C.; Castro, G.; Coelho, R.; Hocevar, L.; Alves, C.; Castro, G.; Coelho, R.; Hocevar, L. Hydrogen Technologies: Recent Advances, New Perspectives, and Applications. In *Hydrogen Technologies—Advances, Insights, and Applications*; IntechOpen: London, UK, 2024; ISBN 978-1-83769-603-1.
7. Subba, C.B.; Mattipally, P.; Sivakumar, J.; Laref, A.; Yvaz, A.; Rai, D.P.; Pachau, Z. 2D MXene-Based Photocatalysts for Efficient Water Splitting and Hydrogen Evolution: A Brief Review. *J. IRAN. CHEM. SOC* **2025**, *22*, 1825–1848. [CrossRef]
8. Righi, G.; Fabris, S.; Piccinin, S. Oxygen Evolution Reaction on the Fe₃O₄(001) Surface: Theoretical Insights into the Role of Terminal and Bridging Oxygen Atoms. *J. Phys. Chem. C* **2021**, *125*, 18752–18761. [CrossRef]
9. Nakayama, M.; Takeda, A.; Maruyama, H.; Kumbhar, V.; Crosnier, O. Cobalt-Substituted Iron-Based Wolframite Synthesized via Polyol Route for Efficient Oxygen Evolution Reaction. *Electrochem. Commun.* **2020**, *120*, 106834. [CrossRef]
10. Zhou, L.; Shinde, A.; Suram, S.K.; Stein, H.S.; Bauers, S.R.; Zakutayev, A.; DuChene, J.S.; Liu, G.; Peterson, E.A.; Neaton, J.B.; et al. Bi-Containing n-FeWO₄ Thin Films Provide the Largest Photovoltage and Highest Stability for a Sub-2 eV Band Gap Photoanode. *ACS Energy Lett.* **2018**, *3*, 2769–2774. [CrossRef]
11. Liu, Z.; Ma, H.; Zhou, T.; Yang, J.; Yu, T. One-Pot Synthesis of NiFe Foam-Supported FeWO₄/Ni₃Se₂ Nanocomposites and Their Enhanced Electrocatalytic Performance for Oxygen Evolution Reaction. *J. Alloys Compd.* **2024**, *1002*, 175294. [CrossRef]
12. Plevová, M.; Hnát, J.; Bouzek, K. Electrocatalysts for the Oxygen Evolution Reaction in Alkaline and Neutral Media. A Comparative Review. *J. Power Sources* **2021**, *507*, 230072. [CrossRef]
13. Higareda, A.; Hernández-Arellano, D.L.; Ordoñez, L.C.; Barbosa, R.; Alonso-Vante, N. Advanced Electrocatalysts for the Oxygen Evolution Reaction: From Single- to Multielement Materials. *Catalysts* **2023**, *13*, 1346. [CrossRef]
14. Tan, D.; Xiong, H.; Zhang, T.; Fan, X.; Wang, J.; Xu, F. Recent Progress in Noble-Metal-Free Electrocatalysts for Alkaline Oxygen Evolution Reaction. *Front. Chem.* **2022**, *10*, 1071274. [CrossRef] [PubMed]
15. Roger, I.; Shipman, M.A.; Symes, M.D. Earth-Abundant Catalysts for Electrochemical and Photoelectrochemical Water Splitting. *Nat. Rev. Chem.* **2017**, *1*, 0003. [CrossRef]
16. Chen, D.; Chen, C.; Baiye, Z.M.; Shao, Z.; Ciucci, F. Nonstoichiometric Oxides as Low-Cost and Highly-Efficient Oxygen Reduction/Evolution Catalysts for Low-Temperature Electrochemical Devices. *Chem. Rev.* **2015**, *115*, 9869–9921. [CrossRef] [PubMed]

17. Zhao, S.; Yang, Y.; Tang, Z. Insight into Structural Evolution, Active Sites, and Stability of Heterogeneous Electrocatalysts. *Angew. Chem. Int. Ed.* **2022**, *61*, e202110186. [[CrossRef](#)] [[PubMed](#)]
18. Gao, L.; Cui, X.; Sewell, C.D.; Li, J.; Lin, Z. Recent Advances in Activating Surface Reconstruction for the High-Efficiency Oxygen Evolution Reaction. *Chem. Soc. Rev.* **2021**, *50*, 8428–8469. [[CrossRef](#)] [[PubMed](#)]
19. Raveendran, A.; Chandran, M.; Dhanusuraman, R. A Comprehensive Review on the Electrochemical Parameters and Recent Material Development of Electrochemical Water Splitting Electrocatalysts. *RSC Adv.* **2023**, *13*, 3843–3876. [[CrossRef](#)] [[PubMed](#)]
20. Alina, A.K.; Kadyrzhanov, K.K.; Kozlovskiy, A.A.; Konuhova, M.; Popov, A.I.; Shlimas, D.D.; Borgekov, D.B. Microcompósitos WO₃ /ZnWO₄ Com Potencial Aplicação Em Fotocatálise. *Opt. Mater.* **2024**, *150*, 115280. [[CrossRef](#)]
21. Kuzmin, A.; Pankratov, V.; Kalinko, A.; Kotlov, A.; Shirmane, L.; Popov, A.I. UV-VUV Synchrotron Radiation Spectroscopy of NiWO₄. *Low Temp. Phys.* **2016**, *42*, 543–546. [[CrossRef](#)]
22. Lisitsyn, V.M.; Karipbayev, Z.T.; Lisitsyna, L.A.; Dauletbekova, A.K.; Tupitsyna, I.A.; Akilbekov, A.T.; Zdorovets, M.V. Effects of Doped Oxygen on ZnWO₄ Crystal Luminescence. *Key Eng. Mater.* **2016**, *712*, 345–350. [[CrossRef](#)]
23. Shim, H.-W.; Cho, I.-S.; Hong, K.S.; Cho, W.I.; Kim, D.-W. Li Electroactivity of Iron (II) Tungstate Nanorods. *Nanotechnology* **2010**, *21*, 465602. [[CrossRef](#)] [[PubMed](#)]
24. Rajagopal, S.; Nataraj, D.; Khyzhun, O.Y.; Djaoued, Y.; Robichaud, J.; Mangalaraj, D. Hydrothermal Synthesis and Electronic Properties of FeWO₄ and CoWO₄ Nanostructures. *J. Alloys Compd.* **2010**, *493*, 340–345. [[CrossRef](#)]
25. Patil, A.R.; Dongale, T.D.; Rajpure, K.Y. Metal Tungstates for Resistive Memory Applications: A Mini Review. *Curr. Appl. Phys.* **2025**, *71*, 70–79. [[CrossRef](#)]
26. Yu, T.; Wang, Z.; Tan, K.; He, H.; Yin, S. Novel Hierarchical FeWO₄–FeNi₃ Heterostructure Nanosheets for Highly Active Water Electrolysis at Large Current Density. *Mater. Today Phys.* **2023**, *35*, 101138. [[CrossRef](#)]
27. Wang, Z.; Qian, G.; Yu, T.; Chen, J.; Shen, F.; Luo, L.; Zou, Y.; Yin, S. Carbon Encapsulated FeWO₄–Ni₃S₂ Nanosheets as a Highly Active Catalyst for Overall Water Splitting at Large Current Density. *Chem. Eng. J.* **2022**, *434*, 134669. [[CrossRef](#)]
28. Nguyen, A.Q.K.; Pham, T.T.T.; Nguyen, O.T.K.; Nguyen, B.N.; Pham, N.S. One-Step Hydrothermal Synthesis of Magnetic Iron Tungsten Oxides for Degradation of Sulfamethoxazole in Oxone and Hydroxylamine System. *Mater. Lett.* **2025**, *385*, 138192. [[CrossRef](#)]
29. Xu, L.; Ge, Y.-D.; Zhou, X.-Y.; Xing, M.-Y.; Wu, X.-Q.; Wang, Y.; Zhang, L.; Wang, X. Study on the Sonocatalytic Removal of Tetracycline by an Type-II Heterojunction CuS/FeWO₄. *J. Alloys Compd.* **2025**, *1015*, 178826. [[CrossRef](#)]
30. Maya, A.A.S.; Akhtar, P.; Hossen, M.A.; Alam, M.J.; AlMohamadi, H.; Ahmed, Y. Heterogeneous Fenton-Assisted Antibiotic Removal from Wastewater: Effect of FeWO₄ Nanomaterial Morphology across Four Fenton Processes. *Water Res. X* **2025**, *29*, 100354. [[CrossRef](#)]
31. Saranya, A.; Klan, M.R.; Ahmad, N.; Srivastava, P.; Poonguzhali, S.; Oh, T.H.; Vignesh, S.; Hartati, S.; Jothimani, K. Enhanced Photocatalytic Properties of Visible Light-Responsive ZnS/FeWO₄ Composites for Degradation of Tetracycline and *Escherichia coli* Inactivation. *J. Alloys Compd.* **2025**, *1010*, 176983. [[CrossRef](#)]
32. Xian, T.; Sun, C.; Ma, K.; Di, L.; Pan, L.; Sun, X.; Yang, H. Boosted Photo Catalytic Performance of Ternary S-Scheme AuAg@FeWO₄/Bi₂O₃ Heterojunction via Synergy of Photothermal-Assisted and LSPR Effects. *J. Environ. Chem. Eng.* **2025**, *13*, 115610. [[CrossRef](#)]
33. Xu, L.; Wang, X.-F.; Liu, B.; Sun, T.; Wang, X. Fabrication of Ferrous Tungstate with Enhanced Sonocatalytic Performance for Meloxicam Removal. *Colloids Surf. A Physicochem. Eng. Asp.* **2021**, *627*, 127222. [[CrossRef](#)]
34. Ahamad, T.; Naushad, M.; Alshehri, S.M. Analysis of Degradation Pathways and Intermediates Products for Ciprofloxacin Using a Highly Porous Photocatalyst. *Chem. Eng. J.* **2021**, *417*, 127969. [[CrossRef](#)]
35. Adak, M.; Rajput, A.; Mallick, L.; Chakraborty, B. Electrochemically Robust Ferberite (FeWO₄) Nanostructure as an Anode Material for Alkaline Water- and Alcohol-Oxidation Reaction. *ACS Appl. Energy Mater.* **2022**, *5*, 5652–5665. [[CrossRef](#)]
36. Reddy, I.N.; Veerla, S.C.; Akkinapally, B.; Dhanasekar, M.; Shim, J.; Bai, C. Unveiling the Electrochemical Kinetics of an FeWO₄–NiFeOOH Anode: Electrolyte Effects on Energy Conversion. *Crystals* **2025**, *15*, 345. [[CrossRef](#)]
37. Paudel, D.R.; Kafle, R.; Dhital, N.; Singh, M.; Salokhe, B.S. Nickel Nanoparticles Infused Iron Tungstate Nanocomposite for Clean Energy Carrier Hydrogen Generation via Water Splitting. *Adv. Mater. Interfaces* **2025**, *12*, e00644. [[CrossRef](#)]
38. Li, R.; Zhang, H.; Chen, J.; Zhang, K.; Li, W.; Feng, X.; Zhang, H.; Abdoukader, I.D.; Zhang, X.; Zhang, T. Interfacial Engineering of FeWO₄/Fe₂O₃ Homometallic Heterojunctions for Synergistic Electrocatalytic Water Splitting. *Inorg. Chem. Front.* **2023**, *10*, 3675–3685. [[CrossRef](#)]
39. Ta, X.M.C.; Nguyen, T.K.A.; Bui, A.D.; Nguyen, H.T.; Daiyan, R.; Amal, R.; Tran-Phu, T.; Tricoli, A. Optimizing Surface Composition and Structure of FeWO₄ Photoanodes for Enhanced Water Photooxidation. *Adv. Mater. Technol.* **2023**, *8*, 2201760. [[CrossRef](#)]
40. Luo, W.; Wang, J.; Hu, J.; Ji, Y.; Streb, C.; Song, Y.-F. Composite Metal Oxide–Carbon Nanotube Electrocatalysts for the Oxygen Evolution and Oxygen Reduction Reactions. *ChemElectroChem* **2018**, *5*, 2850–2856. [[CrossRef](#)]

41. Shao, W.; Xia, Y.; Luo, X.; Bai, L.; Zhang, J.; Sun, G.; Xie, C.; Zhang, X.; Yan, W.; Xie, Y. Structurally Distorted Wolframite-Type $\text{Co}_x\text{Fe}_{1-x}\text{WO}_4$ Solid Solution for Enhanced Oxygen Evolution Reaction. *Nano Energy* **2018**, *50*, 717–722. [\[CrossRef\]](#)
42. Mohamed, M.J.S. High Bifunctional Electrocatalytic Activity of $\text{FeWO}_4/\text{Fe}_3\text{O}_4/\text{NrGO}$ Nanocomposites towards Electrolyzer and Fuel Cell Technologies. *J. Electroanal. Chem.* **2021**, *897*, 115587. [\[CrossRef\]](#)
43. Jansi Rani, B.; Ravi, G.; Yuvakkumar, R.; Praveenkumar, M.; Ravichandran, S.; Muthu Mareeswaran, P.; Hong, S.I. Bi_2WO_6 and FeWO_4 Nanocatalysts for the Electrochemical Water Oxidation Process. *ACS Omega* **2019**, *4*, 5241–5253. [\[CrossRef\]](#) [\[PubMed\]](#)
44. Zhang, L.; Quan, Z.; Wang, Y.; Li, H.; Yang, X. Construction of Flower-like FeCo_2O_4 Nanosheets on Ni Foam as Efficient Electrocatalyst for Oxygen Evolution Reaction. *Coatings* **2023**, *13*, 1875. [\[CrossRef\]](#)
45. Gomes dos Santos, A.; Elaadssi, Y.; Chevallier, V.; Leroux, C.; Lopes-Moriyama, A.L.; Arab, M. Insights of Nanostructured Ferberite as Photocatalyst, Growth Mechanism and Photodegradation Under H_2O_2 -Assisted Sunlight. *Molecules* **2025**, *30*, 4026. [\[CrossRef\]](#) [\[PubMed\]](#)
46. Shinagawa, T.; Garcia-Esparza, A.T.; Takanabe, K. Insight on Tafel Slopes from a Microkinetic Analysis of Aqueous Electrocatalysis for Energy Conversion. *Sci. Rep.* **2015**, *5*, 13801. [\[CrossRef\]](#) [\[PubMed\]](#)
47. Li, W.; Tian, H.; Ma, L.; Wang, Y.; Liu, X.; Gao, X. Low-Temperature Water Electrolysis: Fundamentals, Progress, and New Strategies. *Mater. Adv.* **2022**, *3*, 5598–5644. [\[CrossRef\]](#)
48. Pollet, B.G.; Kalanur, S.S. Applications of Ferric Oxide in Water Splitting by Electrolysis: A Comprehensive Review. *Molecules* **2024**, *29*, 4990. [\[CrossRef\]](#) [\[PubMed\]](#)
49. Man, I.C.; Su, H.-Y.; Calle-Vallejo, F.; Hansen, H.A.; Martínez, J.I.; Inoglu, N.G.; Kitchin, J.; Jaramillo, T.F.; Nørskov, J.K.; Rossmeisl, J. Universality in Oxygen Evolution Electrocatalysis on Oxide Surfaces. *ChemCatChem* **2011**, *3*, 1159–1165. [\[CrossRef\]](#)
50. Jin, S.; Kevin, M.; Hubert A, G.; John B, G.; Yang, S.-H. A Perovskite Oxide Optimized for Oxygen Evolution Catalysis from Molecular Orbital Principles. *Science* **2011**, *334*, 1383–1385. [\[CrossRef\]](#) [\[PubMed\]](#)
51. Grätzel, M. Photoelectrochemical Cells. *Nature* **2001**, *414*, 338–344. [\[CrossRef\]](#) [\[PubMed\]](#)
52. Gerken, J.B.; McAlpin, J.G.; Chen, J.Y.C.; Rigsby, M.L.; Casey, W.H.; Britt, R.D.; Stahl, S.S. Electrochemical Water Oxidation with Cobalt-Based Electrocatalysts from pH 0–14: The Thermodynamic Basis for Catalyst Structure, Stability, and Activity. *J. Am. Chem. Soc.* **2011**, *133*, 14431–14442. [\[CrossRef\]](#) [\[PubMed\]](#)
53. Hanan, A.; Solangi, M.Y.; Lakhan, M.N.; Alhazaa, A.; Shar, M.A.; Laghari, A.J.; Soomro, I.A.; Abro, M.I.; Kumar, M.; Aftab, U. $\text{CoSe}_2/\text{Co}_3\text{O}_4$ Nanostructures: A Promising Catalyst for Oxygen Evolution Reaction in Alkaline Media. *Catal. Commun.* **2024**, *186*, 106830. [\[CrossRef\]](#)
54. Li, X.; Chang, S.; Wang, Y.; Zhang, L. Silver-Carbonaceous Microsphere Precursor-Derived Nano-Coral Ag Catalyst for Electrochemical Carbon Dioxide Reduction. *Catalysts* **2022**, *12*, 479. [\[CrossRef\]](#)
55. Liu, H.-J.; Zhang, S.; Chai, Y.-M.; Dong, B. Ligand Modulation of Active Sites to Promote Cobalt-Doped 1T-MoS₂ Electrocatalytic Hydrogen Evolution in Alkaline Media. *Angew. Chem. Int. Ed.* **2023**, *62*, e202313845. [\[CrossRef\]](#) [\[PubMed\]](#)
56. Holewinski, A.; Linic, S. Elementary Mechanisms in Electrocatalysis: Revisiting the ORR Tafel Slope. *J. Electrochem. Soc.* **2012**, *159*, H864. [\[CrossRef\]](#)
57. Rajagopal, V.; Mehla, S.; Jones, L.A.; Bhargava, S.K. Nanoengineered Cobalt Electrocatalyst for Alkaline Oxygen Evolution Reaction. *Nanomaterials* **2024**, *14*, 946. [\[CrossRef\]](#) [\[PubMed\]](#)
58. Brijesh, K.; Bindu, K.; Shanbhag, D.; Nagaraja, H.S. Chemically Prepared Polypyrrole/ ZnWO_4 Nanocomposite Electrodes for Electrocatalytic Water Splitting. *Int. J. Hydrogen Energy* **2019**, *44*, 757–767. [\[CrossRef\]](#)
59. AlShehri, S.M.; Ahmed, J.; Ahamad, T.; Arunachalam, P.; Ahmad, T.; Khan, A. Bifunctional Electro-Catalytic Performances of CoWO_4 Nanocubes for Water Redox Reactions (OER/ORR). *RSC Adv.* **2017**, *7*, 45615–45623. [\[CrossRef\]](#)
60. Srirapu, V.K.V.P.; Kumar, A.; Srivastava, P.; Singh, R.N.; Sinha, A.S.K. Nanosized CoWO_4 and NiWO_4 as Efficient Oxygen-Evolving Electrocatalysts. *Electrochimica Acta* **2016**, *209*, 75–84. [\[CrossRef\]](#)
61. Khan, M.; Ahmed, M.M.; Akhtar, M.N.; Sajid, M.; Riaz, N.N.; Asif, M.; Kashif, M.; Shabbir, B.; Ahmad, K.; Saeed, M.; et al. Fabrication of $\text{CuWO}_4/\text{MIL-101 (Fe)}$ Nanocomposite for Efficient OER and Photodegradation of Methylene Blue. *Heliyon* **2024**, *10*, e40546. [\[CrossRef\]](#) [\[PubMed\]](#)
62. Ye, Z.-G.; Meng, H.-M.; Sun, D.-B. New Degradation Mechanism of $\text{Ti}/\text{IrO}_2 + \text{MnO}_2$ Anode for Oxygen Evolution in 0.5 M H_2SO_4 Solution. *Electrochimica Acta* **2008**, *53*, 5639–5643. [\[CrossRef\]](#)
63. Qin, Y.; Yu, T.; Deng, S.; Zhou, X.-Y.; Lin, D.; Zhang, Q.; Jin, Z.; Zhang, D.; He, Y.-B.; Qiu, H.-J.; et al. RuO_2 Electronic Structure and Lattice Strain Dual Engineering for Enhanced Acidic Oxygen Evolution Reaction Performance. *Nat. Commun.* **2022**, *13*, 3784. [\[CrossRef\]](#) [\[PubMed\]](#)
64. Molina-Lozano, A.E.; Lanza, M.R.V.; Ortiz, P.; Cortés, M.T. Photoelectrochemical and Structural Insights of Electrodeposited CeO_2 Photoanodes. *Surfaces* **2024**, *7*, 898–919. [\[CrossRef\]](#)
65. Khan, M.A.; Varadhan, P.; Ramalingam, V.; Fu, H.-C.; Idriss, H.; He, J.-H. Importance of Oxygen Measurements during Photoelectrochemical Water-Splitting Reactions. *ACS Energy Lett.* **2019**, *4*, 2712–2718. [\[CrossRef\]](#)

66. Ali, A.; Najaf, D.; Nazir, A.; Haider, A.; Iqbal, M.; Alwadai, N.; Kausar, A.; Ahmad, A. Fabrication of Efficient Electrocatalysts for Electrochemical Water Oxidation Using Bimetallic Oxides System. *ACS Omega* **2023**, *8*, 9539–9546. [[CrossRef](#)] [[PubMed](#)]
67. Bedoch, A.M.; Koga, G.Y.; Nogueira, R.P.; Zepon, G. On the electrochemical hydrogenation of Nb: An insight into the effect of hydrogen absorption on the kinetics of the hydrogen evolution reaction. *Electrochimica Acta* **2021**, *389*, 138626. [[CrossRef](#)]
68. Elaadssi, Y.; Ouardi, M.E.; Madigou, V.; Jada, A.; Arab, M. Hydrogen and Oxygen Evolution Reactions for High-Performance Electrocatalytic Water Splitting by Tuning Cobalt Content in Cobalt Ferrite. *Fuel* **2026**, *418*, 138788. [[CrossRef](#)]
69. Shang, X.; Yan, K.-L.; Rao, Y.; Dong, B.; Chi, J.-Q.; Liu, Y.-R.; Li, X.; Chai, Y.-M.; Liu, C.-G. In Situ Cathodic Activation of V-Incorporated Ni_x S_y Nanowires for Enhanced Hydrogen Evolution. *Nanoscale* **2017**, *9*, 12353–12363. [[CrossRef](#)] [[PubMed](#)]

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