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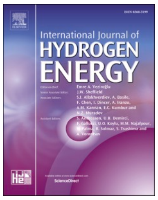

Electrode modifications with electrophoretic deposition methods for water electrolyzers

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ABSTRACT

This review covers the methods of hydrogen production through water electrolysis, encountered problems, used catalysts, methods of preparing these catalysts, and especially the method of electrode modification using the electrophoretic deposition technique. The methods of hydrogen production mechanism, electrode and electrocatalyst structures, and electrode modification techniques are classified based on types of the electrolyzers namely Alkaline Water Electrolysis (AEL), Anion Exchange Membrane (AEM) Water Electrolysis, Proton Exchange Membrane Electrolysis (PEMEL) and Solid Oxide Electrolysis (SOEL). The mechanisms of hydrogen evolution and oxygen evolution reactions, which are crucial in hydrogen production, have been provided in all types of electrolyzers. The parameters influencing these reactions on the anode and cathode electrodes have been discussed. This review summarizes and compares important parameters that show the catalyst's performance in detail. The fundamental principles of the electrophoretic deposition technique have been explained, and examples of electrocatalysts prepared using this technique have been provided.

1. Energy

Energy is becoming a fundamental phenomenon worldwide today with the increasing use of technology [1]. As the human population continues to rise, so does the consumption of fuels such as coal, oil, and natural gas, playing a substantial role in contributing to pollution. Traditional energy sources such as oil, natural gas, and coal, which account for 80% of our energy consumption, are running out [2–4]. Moreover, conventional energy resources give rise to environmental concerns, including climate change and global warming, which have both immediate and long-term repercussions. Climate change is directly responsible for an annual death toll of 160,000 people, according to the World Health Organization (WHO). The effects of climate change manifest in various ways, leading to natural disasters like floods, droughts, and significant shifts in atmospheric temperatures [5,6]. When these fuels are burned for energy, they release pollutants into the atmosphere, including carbon dioxide (CO_2), which is a greenhouse gas that contributes to climate change, as well as other contaminants like sulfur dioxide (SO_2), nitrogen oxides (NOx), and particulate matter, which can have adverse effects on air quality and human health.

Furthermore, the pollution from burning fossil fuels can lead to various environmental problems, including air pollution, water pollution, and soil contamination [7]. It can also harm human health, causing respiratory problems, cardiovascular diseases, and other health issues. It contributes to the proliferation of certain diseases within communities, particularly malaria, malnutrition, and diarrhea, creating epidemic conditions [5,6]. Thus, the need for clean energy sources is growing day by day. Although fossil fuels have been used widely since the Industrial Revolution, to address this issue, research has been carried out to reduce our reliance on fossil fuels and transition to cleaner, renewable, sustainable, green, and alternative energy resources, such as solar energy, wind energy, hydroelectric energy, nuclear power in recent years [8–11]. Renewable energy sources like solar, hydro, and wind power are inherently abundant but are susceptible to significant fluctuations [12]. The existing problems in producing, converting, and storing energy in a renewable and sustainable manner hinder the provision of environmentally friendly, sustainable energy production at a low cost [13–15]. Efficient utilization of these energy sources necessitates the presence of effective electrochemical energy conversion and storage systems [16]. Hence, there is a particular focus on developing new technologies for storing renewable energies. Electrochemical technologies are well-suited for utilizing surplus electricity, which is expected to lead to

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greater electrification of chemical processes. In this regard, there are various approaches to harness surplus electricity electrochemically. The most widely acknowledged and discussed solutions include storing it as chemical energy carriers like hydrogen through water electrolysis and using electrochemical energy storage devices such as batteries. Electrolysis is recognized as a versatile and efficient approach to storing and converting energy, enabling the storage of electrical energy in hydrogen for subsequent electricity generation. Electrolysis, within the realm of energy production, is a technique that employs electrical energy to generate hydrogen gas or other valuable chemical substances from water or various chemical compounds. It serves to store energy through chemical potential [12,17].

2. Hydrogen energy

Hydrogen, the Earth's third most abundant element, obtained as a secondary energy source using renewable energy sources such as biomass, sunlight, wind energy, and ocean thermal energy, is more of an energy carrier than an energy source [18]. The hydrogen industry primarily focuses on producing and utilizing hydrogen for purposes other than serving as an energy carrier or fuel for energy generation. Instead, the annual production of nine million tons of hydrogen was predominantly directed towards chemical, petroleum refining, metals, and electronics applications. Ongoing hydrogen initiatives emphasizing its role as an energy carrier or primary fuel necessitate advancements in multiple industry sectors, encompassing production, transportation, storage, conversion, and end-user applications. The concept of a hydrogen economy, serving as both an energy carrier and storage solution, represents the enduring objective of numerous nations. It promises to bolster energy security while delivering environmental and economic advantages. Hydrogen technologies have the potential to mitigate carbon emissions when hydrogen is generated from renewable sources or nuclear energy (specifically nuclear-thermochemical processes). Additionally, hydrogen produced from fossil fuels with carbon capture and sequestration can contribute to carbon emission reduction efforts [19–22].

Examining the enhanced utilization efficiency of hydrogen further solidifies its position as the optimal choice for transportation fuel, as hydrogen energy is both portable and storable. This is among the factors explaining why hydrogen is the preferred fuel for space programs worldwide despite its higher cost than fossil fuels. All fuels, except hydrogen, can be converted through a single combustion process. Hydrogen, on the other hand, boasts five distinct conversion pathways. In addition to traditional flame combustion, it can be transformed directly into steam, converted into heat via catalytic combustion, serve as both a heat source and heat sink through chemical reactions, and be directly converted into electricity through electrochemical processes. In essence, hydrogen proves to be the most versatile fuel source. In almost every application, hydrogen can be converted into the desired energy form more efficiently than alternative fuels [23–25]. Recent research efforts are directed towards generating renewable energy, with a primary focus on harnessing hydrogen, mainly through hydroelectricity. Electrolytic hydrogen (H_2) is a viable alternative to replace fossil fuels in the energy sector [26,27].

Furthermore, its global development is focused on its use in fuel cell technology, a high-energy and clean fuel that only emits water and water vapor into the atmosphere [17,28,29]. The environmentally friendly aspect of H_2 utilization is enhanced by producing water as a non-toxic by-product, mainly using renewable energy sources like solar, wind, and hydroelectric power [30]. Hydrogen energy is regarded as a clean and futuristic technology that contributes to both lowering energy demand and mitigating environmental pollution [31]. It boasts numerous advantages compared to conventional energy sources, and its clean nature allows for innovative storage methods. Hydrogen is 2.5 times more efficient than gasoline, and 1 kg of hydrogen can store more energy than 2.75 kg of gasoline [32].

In contrast to fossil fuels, hydrogen is not naturally abundant. Nevertheless, it can be generated from various primary energy sources and subsequently utilized as a fuel, either through direct combustion in an internal combustion engine or within a fuel cell, resulting in the production of only water as a by-product [33–36]. It can be seen in Table 1 that being the sole carbon-free energy source and possessing the highest energy content when compared to any known hydrogen, it is universally recognized as an environmentally friendly secondary form of renewable energy, serving as an alternative to fossil fuels [17,37,38].

3. Hydrogen production

The primary challenge associated with using hydrogen gas as a fuel is its scarcity in nature and the requirement for cost-effective production methods [40]. Hydrogen production processes can be broadly categorized into two main groups based on the raw materials employed: conventional and renewable technologies. Fig. 1 illustrates the different methods for hydrogen production [41].

Water is the most abundant raw material that can be used to produce hydrogen, which is considered the cleanest renewable energy source for humanity. Therefore, the 'water splitting' method is highly practical for hydrogen production. This method is divided into three categories: electrolysis, thermolysis, and photo-electrolysis. Electrolysis types, which are used in various applications and play significant roles in many industrial and scientific fields such as hydrogen production, energy storage, chemical manufacturing, and water purification, can be divided into two categories such as electrolysis processes in chemical energy carriers like hydrogen–water electrolysis and electrolysis processes in electrochemical energy storage devices.

Firstly, there are three primary technologies used for water electrolysis: alkaline water electrolysis (AEL), proton exchange membrane (PEM) (or polymer electrolyte membrane (PEMEL)) electrolysis, and solid oxide electrolysis (SOEL) (Table 2). Numerous electrolyte systems have been developed for water electrolysis, as shown in Fig. 2. Among these, AEL and PEMEL have achieved high technical readiness levels [48–51]. Despite extensive discussions about water electrolysis as a crucial technology for producing pure hydrogen using renewable electricity, the current reality is that less than 4% of hydrogen production originates from electrolysis. Most hydrogen is not derived from water electrolysis but from chlor-alkali electrolysis, where hydrogen is a by-product of chlorine production [12,52]. Secondly, various electrochemical energy storage devices are considered in the context of intermediate energy storage from renewable sources. Rechargeable batteries like Li-ion and redox flow batteries are among the familiar options. The acid-base flow battery is a particularly intriguing solution that relies on the principle of reverse electrodialysis with bipolar membranes. During the charging process, the system functions in an electrolysis mode. This involves hydrogen and oxygen evolution reactions (OER) at the cathode and anode. In this process, acid and base solutions are regenerated in their respective compartments. Like conventional water electrolysis, these two half-reactions occur under distinct pH conditions, involving both acidic and alkaline environments [12,53].

Water is recognized as one of the Earth's most abundant natural resources, and the production of H_2 can be accomplished through water-splitting techniques like thermolysis, electrolysis, and photolysis. In the

Table 1
Higher and lower heating values for various fuels [39].

Fuel	State at ambient temperature and pressure	HHV (MJ/kg)	LHV (MJ/kg)
Hydrogen	Gas	141.9	119.9
Methane	Gas	55.5	50
Ethane	Gas	51.9	47.8
Gasoline	Liquid	47.5	44.5
Diesel	Liquid	44.8	42.5
Methanol	Liquid	20	18.1

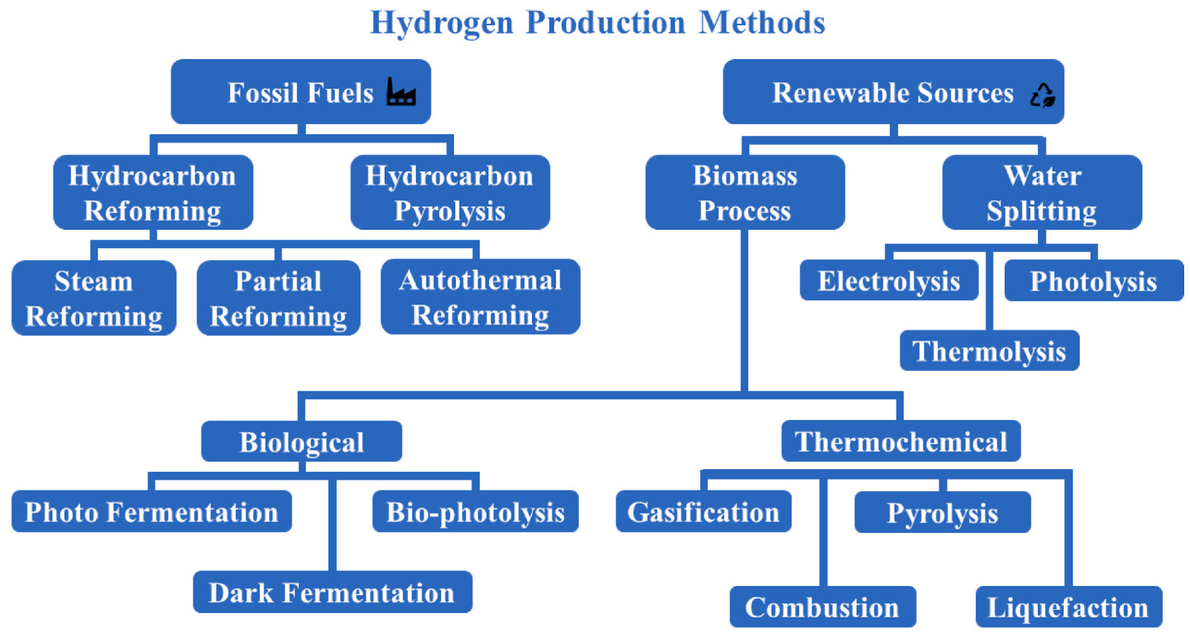


Fig. 1. Hydrogen production methods [41].

Table 2
Contrast among diverse water electrolyzer variants [42].

Detail	PEM	AWE	AEM	SOE
Capacity	trade	trade	trade	trade
Charger carrier	H^+	OH^-	OH^-	O^{2-}
Electrolyte	Solid polymer	Aqueous solution KOH/ NaOH	Solid polymer	Solid ceramic
Working aqueous	Pure water	High con. solution	Pure water or low cons. solution	Steam
Cathode material	Pt; Pt=C	Ni alloys	Ni, Ni-Fe, $NiFe_2O_4$	Nicerments
Temperature (°C)	70–90	65–100	50–70	650–1000
Working pressure	15–30 bar	2–10 bar	up to 35 bar	<30 bar
Efficiency, HHV	67–84%	62–82%	-	~90%
Cell voltage	1.80 2.40 V	1.80 2.40 V	~1.85 V	0.95 1.30 V
Current density	0.6–2 A/ cm^2	0.2 0.4 A/ cm^2	0.1–0.5 A/cm^2	0.3 1 A/cm^2
Startup duration	<15 min	15 min	-	>60 min
Stack lifetime	<40,000 h	<90,000 h	>10,000 h	<40,000 h
Energy consumption, kWh/Nm^3	4,5–7,5	4,5-7	~4,8	2,5-3,5
Estimated cost by 2050.	~$750/ kWch	~$600/ kWch	–	~$200/ kWch
Advantages	oBasic and compact design. oFast advance and feedback. oHigh purity of hydrogen.	oCapital cost is low. oHaven't noble material. oStable.	oCheap. oSuitable for load fluctuation. oA mixture of the advantages of PEM and AWE.	oCheap. oHaven't novel material. oHigh efficiency. oCan be used as a fuel cell.

electrolysis process, applying a direct electric current to the system causes the reactant, water, to dissociate into H_2 and O_2 [41]. The water electrolysis processes were given in (1-3).

$$\text{Anode: } H_2O \rightarrow ½\, O_2 + 2H^+ + 2e^- \tag{1}$$

$$\text{Cathode: } 2H^+ + 2e^- \rightarrow H_2 \tag{2}$$

$$\text{Overall: } H_2O \rightarrow H_2 + ½\, O_2 \tag{3}$$

Water electrolysis experiments primarily hinge on three key parameters: temperature, electrolyte, and pH. Water electrolysis technologies can be categorized into three temperature ranges.

- low-temperature processes ($T < 150\ °C$),
- medium-temperature processes ($200\ °C < T < 600\ °C$),
- high-temperature processes ($T > 600\ °C$).

Regarding pH, the half-cell reactions exhibit variations corresponding to the pH level of the electrolyte [43]. Material selection and operational conditions may vary within these systems, but the underlying operating principles remain consistent. Water electrolysis can be employed at both low and high temperatures across various types (Fig. 3).

Biohydrogen from renewable biomass is the ultimate green hydrogen technology in different hydrogen production methods [55]. Producing hydrogen using fermentative bacteria, photosynthetic bacteria, or algae is an eco-friendly and energy-efficient process [56]. Microbial electrochemical systems (MESs) are innovative technologies that harness microbial electrochemical processes to generate bioenergy and produce valuable chemicals [57–66]. MES generates electrical energy by converting the chemical energy into waste lignocellulosic biomass and wastewater. This conversion happens through a redox (reduction-oxidation) process, which is facilitated by biological catalysts. MES technology is an interdisciplinary field that merges concepts from materials science, electrochemistry, and chemical engineering microbiology [67]. Examples of these systems include microbial desalination cells (MDCs), sediment microbial fuel cells (SMFCs), microbial fuel cells (MFCs) [55], microbial electrolysis cells (MECs) [68], microbial electrosynthesis cells (MESCs), and microbial reverse electrodialysis cells (MRCs) among others [69–72]. MFCs are notable for generating electricity by breaking down organic matter in wastewater with the help of microbial catalysts in the anode, among MESs [71,73,74]. This approach offers a sustainable alternative to direct fuel combustion by converting the chemical energy in organic waste into bioelectricity [75]. MECs, like MFCs, also treat wastewater by removing organic matter, but they differ in that they

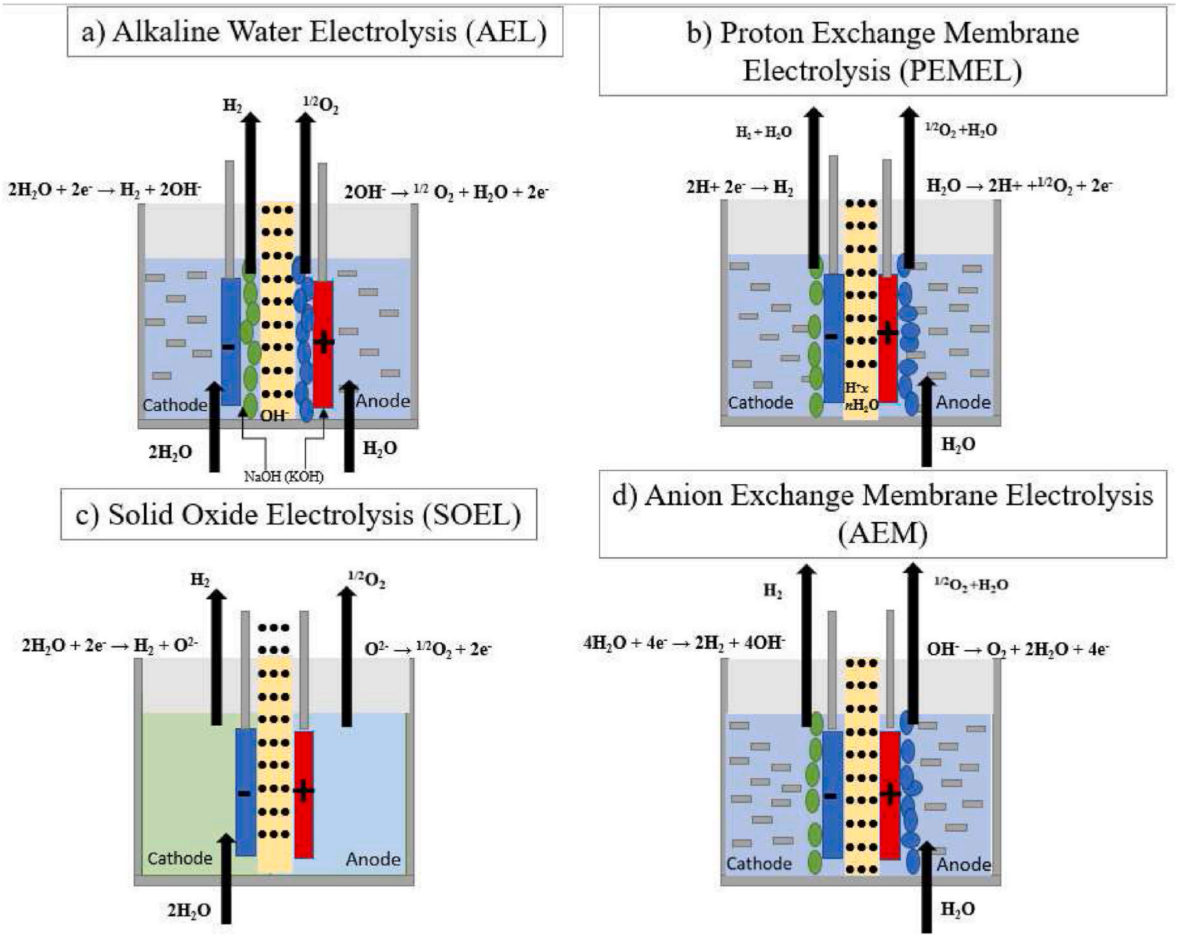


Fig. 2. Schematic diagram of a) AEL, b) PEMEL, c) SOEL, and d) AEM [43–47].

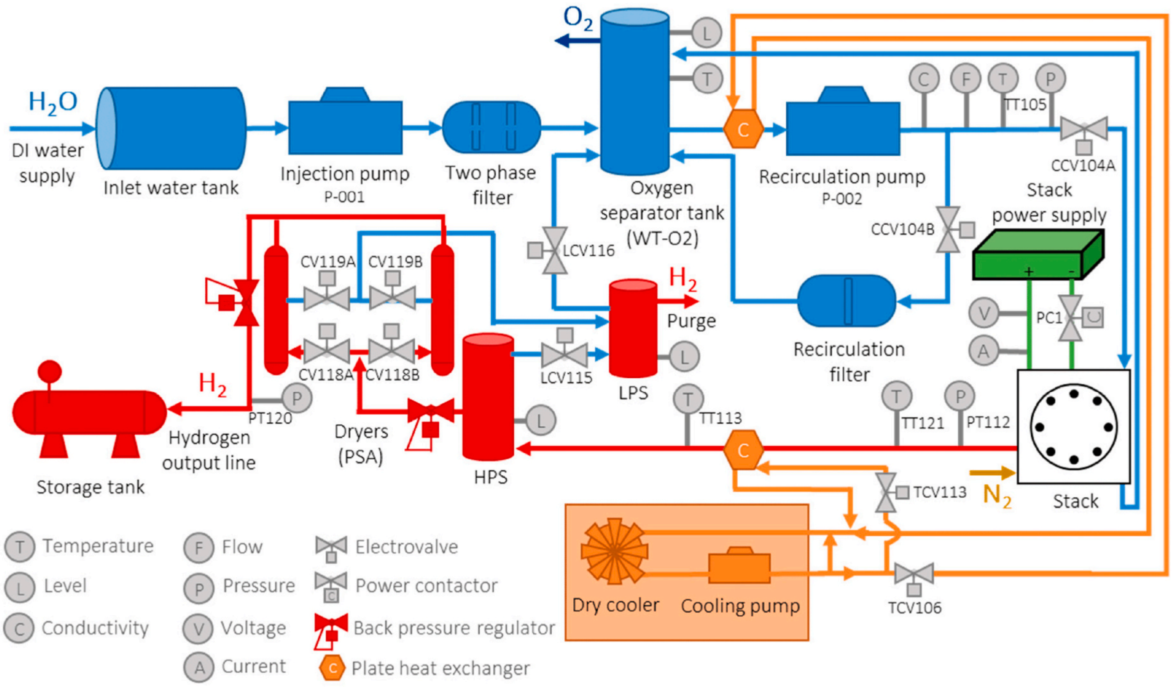


Fig. 3. Flow diagram of the water electrolysis process [54].

produce green hydrogen gas at the cathode [68,76]. The electrodes are crucial components of MESs, as most biotic and abiotic electrochemical reactions occur at the anode and cathode [77,78]. Electrode linear sweep voltammetry (LSV) tests in MESs are typically conducted using a three-electrode setup, which includes a working electrode, a reference electrode, and a counter electrode [79–81]. The counter electrode facilitates reactions opposite to those occurring at the working electrode. The selection of the counter electrode depends on the specific electrode reaction type. For anode LSV tests, it is important to consider the cathode material used during MEC operation. Stainless steel counter electrodes have shown higher reproducibility in achieving maximum current compared to platinum electrodes [71].

3.1. Alkaline water electrolysis (AEL)

Water electrolysis in an alkaline environment was first proposed by Troostwijk and Diemann in 1789 and became the first method used commercially for hydrogen production worldwide. The working principle of this method is based on the formation of hydrogen and hydroxyl ions by reducing water in the cathode reaction. Hydroxyl ions pass through the porous diaphragm, allowing the anode reaction to occur. Hydrogen gas output occurs on the cathode side, while water and oxygen gas output occurs on the anode side. Fig. 2a shows a schematic representation of water electrolysis in an alkaline environment [46].

Alkaline electrolysis occurs at temperatures as low as 30–80 °C in the electrolyte (such as KOH/NaOH). In the alkaline water electrolysis process, cell voltage (V) current density (mA/cm^2) asbestos diaphragm, and nickel materials are used as electrodes. The diaphragm located in the middle of the cell, in addition to separating the cathode and anode from each other, prevents them from interfering with the electrolysis process by removing the produced gases from the electrodes. There are negative aspects of electrolysis in alkaline environments, such as low restrictive current densities (below 400 mA/cm^2), low operating pressure, and low energy efficiency. A new approach in recent research is the development of anion exchange membranes consisting of polymers with anionic conductivity instead of an asbestos diaphragm [44,46].

Alkaline water electrolysis can be considered a well-positioned water electrolysis, especially since it does not require noble metal materials compared to other electrolyzers and can operate at relatively low temperatures. However, operating at a low current density requires a larger and thicker structure than similar technologies. Due to this disadvantage, with the development of materials with higher conductivity and thinner diaphragms, it may now be possible to develop alkaline electrolysis systems that can operate efficiently at significantly higher current densities thanks to advanced electrode coatings and innovative cell structures [44,46]. In their study on the performance of alkaline electrolyzers, **De Groot, et al., 2022** systematically investigated the optimum operating conditions according to temperature, pressure, and diaphragm thickness. They carried out optimization studies on their systems with a temperature range of 60–100 °C, a pressure range of 1–50 bar, and a diaphragm thickness of 0.1–0.5 mm. Studies have shown that both higher temperature and smaller diaphragm thickness improve the current-voltage curve and therefore enable a higher nominal current density [82]. **De Groot, et al., 2021** demonstrated the development potential of alkaline technology by using a Zirfon diaphragm in an alkaline electrolyzer due to its low restrictive current densities. With the study, a potential as low as 1.72 V was reached at 1 A/cm^2 with a thin Zirfon-type separator without the use of noble metals, and high current density alkaline electrolyzers were developed with the optimization of the zero gap configuration [83]. **Li Dan, et al., 2020** examined Pt atoms immobilized on a NiRu hydroxide layer (Pt/NiRu-OH) with high catalytic activity for the hydrogen evolution reaction (HER) in the AWE application (overpotential of 38 mV at 10 mA cm^{-2}) [84]. Functionalized NiRu-OH was converted from NiRu layered double hydroxide via Ru leaching and self-restructuring under oxygen evolution reaction (OER) conditions. As a result, NiRu-OH could further accelerate the dissociation of water, while the vacancies resulting from Ru dissolution managed to immobilize single Pt atoms, showing extremely high HER activity. The excellent catalytic activity was attributed to the synergistic effect of highly dispersed single Pt atoms and NiRu-OH in the dual-site alkaline HER mechanism. In the work of **Razmjooei et al., 2020** a rational methodology was developed to achieve promising performance from non-noble metal-based NiMo alloys as catalysts of slow HER in alkaline electrolytes. This was achieved by fabricating the catalyst layer as binder-free electrodes via atmospheric plasma spraying and precise control of processing conditions to correlate fabrication parameters with the structure properties and performance of the electrodes. It has been shown that by increasing the input plasma power and flow rate, the powders achieve higher velocity and momentum in the plasma during flight, and the resulting electrode coatings exhibit thinner and larger amounts of pores and appropriate Ni and Mo content after activation, resulting in increased HER activity [85].

For AWE, the bubbles of porous electrodes seriously interfere with the kinetics of the decomposition reaction of water. **Bi Songhu et al., 2023** prepared a porous NM/PN/NiFeO x electrode by a two-stage electrodeposition method. It has been stated that it shows good OER performance at high current density with a potential of 1.45 V at 500 mA cm-2 current density and 1.48 V at 1000 mA cm-2 current density. Moreover, this is due to the good electro-catalytic performance of both nano-cone structured NiFeOx. as well as attributed to the good bubble removal performance of the porous nickel (PN) interlayer with curved pore channels. In alkaline water electrolysis (AWE), hydrogen production efficiency is extremely low because electrodes are expensive and inefficient [86]. In the study, **Zhou et al., 2022** used a kind of high entropy alloy (HEA), bulk AlCoCrFeNi, as the efficient electrode for AWE. The results show that HEA processed by 5-min rapid anodization can achieve ultra-high catalytic activities for hydrogen and oxygen formation reactions with low extreme potentials of 880 and 845 MV to achieve current densities of −500 and 500 mA cm^{-2}, respectively [87].

Alkaline water electrolysis boasts a rich history within the chemical industry. Its utilization for energy conversion in the hydrogen energy system is gaining considerable interest, leading to the development of advanced electrolyzers. The current trajectory of advancement focuses on achieving higher efficiency through the implementation of high-temperature and high-pressure operations [47]. A straightforward approach, Alkaline Water Electrolysis stands out but entails relatively higher costs. When the demand for hydrogen is relatively modest, on-site water electrolysis can prove to be a cost-effective choice compared to alternative methods. This method is environmentally friendly and yields hydrogen gas with a purity exceeding 99.989% [88].

While Alkaline electrolysis cells (AECs) have seen the most advancement in practical applications, the emergence of exceptionally active and dynamically stable electrodes utilizing noble metals like ruthenium and iridium, along with highly proton-conductive PFSA membranes, has substantially enhanced the efficiency of acidic electrolysis when compared to alkaline systems.

Economical electrocatalysts derived from transition metal oxides are commonly utilized materials for the HER in alkaline conditions. Among these, nickel stands out as the predominant electrocatalyst for the HER, primarily due to its cost-effectiveness and strong chemical stability in alkaline environments. However, these electrodes encounter issues of deactivation, as the HER efficiency of Ni cathodes diminishes significantly over prolonged continuous operation, with fresh electrodes exhibiting higher activity [89]. Various metals and metal oxides have been suggested as alloying agents with nickel to deter the formation of nickel hydrides, thereby enhancing stability. Examples include Fe [90], Cu [91], Ti [92], Ru [93], Mo [94], Cr [95], Pd [95], W [96], Co [97], P [98], Sn [99], IrO_2 [100], RuO_2 [100], MoO_2 [101], and CeO_2 [102]. Coating the surface of Ni foam with Pd and Ru nanoparticles has been demonstrated to augment the catalytic activity of the initial material for the HER in an alkaline solution [103]. Cobalt is considered a viable material for alloying with nickel. In this regard, the HER activities of various electrodeposited Ni-Co alloys were examined, revealing enhanced activity in comparison to pure Ni and Co electrocatalysts [104]. While the HER activity of Fe is inherently higher than that of Ni, the instability of Fe in alkaline environments renders it unsuitable for commercial applications in alkaline electrolysis. Nevertheless, promising results emerge when combining Fe with Ni, with Ni—Fe alloys demonstrating superior HER activity compared to the individual parent materials [105]. Table 3 outlines the current state of development for the primary material families in electrocatalysis for the HER in alkaline media [106].

3.2. *Anion exchange membrane (AEM) water electrolysis*

AEM water electrolysis technology has been developing for hydrogen production in recent years. In the past few years, many research organizations/institutions have been actively working on the development of AEMWE due to its low cost and high performance compared to other traditional electrolysis technologies. AEM water electrolysis technology includes all the advantages of conventional alkaline water electrolysis. However, the main difference between alkaline and AEM water electrolysis is that traditional diaphragms (asbestos) are replaced with an anion exchange membrane (quaternary ammonium ion exchange membranes) in alkaline water electrolysis. They show lower ohmic losses because the anion exchange membrane is thinner than traditional membranes (AWE). AEM water electrolysis offers various advantages, such as using cost-effective transition metal catalysts instead of noble metal catalysts and using distilled water/low-concentrated alkaline solution (1 M KOH) as an electrolyte instead of highly concentrated (5 KOH solution). Moreover, it has high performance due to high conductivity and advanced OH-transfer. But despite these important advantages, AEM-based electrolysis still lacked ionomer structure and membrane stability at high temperatures, so it needed further research/improvement towards MEA stability and cell efficiency, which are more necessary for large-scale or commercial applications [46].

The use of a thin anion exchange membrane (AEM) has been suggested for high performance at low cost in AEM electrolyzers. Advantages offered by thin membranes include reduced mass transfer resistance and ohmic resistance. A membrane electrode assembly (MEA) with a thinner membrane will have improved hydroxide ion transfer due to the shorter ion transfer path. **Vincent et al., 2018** have demonstrated the high AEM electrolyzer performance and relatively high stability of ultrathin A-901 and A-201 membranes (approximately 10 and 30 μm, respectively) when used as electrolytes [107]. **Pushkareva et al., 2020** studied AEM water electrolysis using non-valent catalysts and found that the catalyst layer structure significantly affects the AEM electrolyzer performance [108]. According to **Carbone et al., 2020** a specific membrane-electrode assembly based on the FAA3-50 anionic membrane and the $NiMn_2O_4$ anode catalyst has been developed. It was found that the ionomer with FAA3-50 alkaline membrane and $NiMn_2O_4$ anode catalyst showed excellent AEM performance at 80 °C and reached 530 mA/cm^2 at 2 V [109]. In the **Park et al., 2020** study, it was applied as a cathode to a single-cell AEM electrolyzer by electro-precipitating Co_3S_4 nanolayers on Ni foam and obtained a high current density of 431 mA/cm^2 at 2 V, which was highly accessible by the electrolyte for the hydrogen evolution reaction (HER) [110]. **Zhiani et al., 2017** reported that Ni foam (NF) was modified with a catalyst with a low content of Pd, Ni, Fe, and Co metal nanoparticles (PdNiFeCo/C-Ceria-NF) and evaluated as a cathode in an alkaline anion exchange membrane (AAEM) water electrolyzer. High catalytic activity has been observed in the hydrogen formation reaction (HER) [111].

Table 3
Current progress in the primary material categories for electrocatalysis in the alkaline media of the HER [106].

Material	Activity	Stability	Status
Raney Ni	Sufficient activity	Deactivation after intermittent operation	Commercially used
Ni–Co, Ni–Fe	High activity, which can be further improved upon alloying with rare earths	Better stability than Raney Ni, but still not optimal	Laboratory applications
$NiFe_2O_4$	Very high activity	Long term stability	Applied in lab-scale electrolysis with polymeric membrane
Ni–Mo	Very high activity	Long term stability	Pyrophoric material: inappropriate for commercialization
(Ni,Co)–W	High activity	Unknown	Laboratory applications
Co_2Si	Very high activity	Unknown	Laboratory applications
Ni_3N	High activity	Unknown	Laboratory applications

3.3. *Proton Exchange Membrane Electrolysis (PEMEL)*

The first PEM electrolysis was introduced by Grubb in the 1950s and then developed commercially by General Electric in 1966. Electrolysis technology with PEM is like PEM fuel cell technology, in which solid polysulfonate membranes (Nafion, Fumapem) are used as electrolytes (proton conductors). These proton exchange membranes have many advantages such as low gas permeability, high proton conductivity (0.1 ± 0.02S cm-1, low thickness (20–300 μm), and high-pressure processes.

In terms of sustainability and environmental impact, electrolysis with PEM is one of the appropriate methods for converting renewable energy into high-purity hydrogen. This promising method has great advantages such as compact design, high current density (over 2 A cm^{-2}), high efficiency, fast response, small coating area, operation at low temperatures (20–80 °C), and ultra-pure hydrogen production. Catalysts containing noble metals such as Pt/Pd developed for the hydrogen formation reaction (HOR) at the cathode in PEM electrolysis show high activity. The catalysts containing noble metals such as Pt/Pd show high activity IrO_2/RuO_2 catalysts developed for oxygen formation reaction (OOR) on the anode side have made this method more expensive than alkaline water electrolysis. Therefore, one of the main challenges in electrolysis with PEM is to reduce the production cost and maintain high efficiency. The research conducted to improve electrolysis components with PEM brings this technology closer to commercial [46]. In the electrolysis method with PEM, water is pumped to the anode side, causing the formation of oxygen (O_2), protons (H^+), and electrons (e^-). The protons pass through the proton-conducting membrane to the cathode side, while the electrons exit the anode from the external power circuit, which provides the driving force (cell voltage) for the reaction. On the cathode side, protons and electrons combine to produce hydrogen by the mechanism shown in Fig. 2b.

The hydrogen evolution reaction (HER) is very important for hydrogen production in PEM electrolyzers, which are very effective in carbon-independent energy conversion. On the other hand, PEM cathode electrodes used in electrolyzers contain the expensive and rare Pt (Platinum) noble metal, making it difficult to commercialize hydrogen production. Therefore, much research is being done to reduce the temperature, develop alternative catalysts that do not contain Pt or a reduced amount of Pt, and preserve the activity of the electrode and its stability in an acidic environment. In the same way, they are precious metals in the electrodes (Ir, Ru) used in oxygen evolution reactions (OER). There are many studies available to reduce the cost of a PEM electrolyzer and improve its intrinsic activity [44].

The problem for the hydrogen evolution reaction (HER) in the electrolyzer catalyst is the development of highly activated and stable catalysts. **Zhong et al., 2020** developed a method to immobilize atomically dispersed Pt atoms onto carbon nanotubes (Pt-CNTs) for electrocatalytic acidic hydrogen formation [112]. The electrocatalytic activity can be increased and improved by minimizing the amount of Pt loading in the catalyst. In another study, **Morozan et al, 2020** carried out the synthesis and characterization of non-valuable bimetallic iron-molybdenum sulfide bio-inspired electrocatalysts for the hydrogen evolution reaction (HER) and observed that FeMoS increased HER performance [113]. **Yang et al., 2020** proposed a strategy to activate transition metal carbides (TMC) (Co, Fe, Cr) and further improve the hydrogen evolution reaction performance of these TMCs. The superior performance of nickel-adsorbed (Ni-TMC) catalysts on TMC has been demonstrated in both alkaline and acidic environments [114]. **Hu et al., 2021** investigated the effect of charge transfer and proton penetration on the hydrogen evolution reaction mechanisms of the non-noble metals Ni and Cu in an acidic electrolyte on graphene layers. It has been shown that increasing the number of graphene-coated layers in the catalyst significantly changes the hydrogen evolution reaction performances of Ni and Cu [115]. In a study conducted **Yoo et al., 2021** encapsulation of the Pt nano-catalyst with a carbon layer containing N (nitrogen) to improve the catalytic activity and stability in the hydrogen evolution reaction was observed to have a positive effect on both the activity and stability of the catalyst. Due to the acidic and strongly oxidative environments, developing high-activity and robust electrocatalysts for OER in PEM electrolyzers is challenging. For this reason, materials for various catalysts have been investigated to optimize the activity of OER and reduce the amount of precious metals [116]. Reducing the particle size or preparing the ultrafine structure is beneficial in improving the catalytic activity by maximizing the surface area. In the study conducted by **Fu et al., 2018** ultrathin monodisperse Ir nanoparticles with high dispersion were successfully synthesized by a colloidal method. Due to the ultrafine particle size and uniform distribution, this catalyst has been shown to improve its catalytic performance for the acidic oxygen formation reaction [117]. In the work of **Xue et al., 2018** carbon nanocassettes were supplemented with ultrathin iridium nanocrystals. The formation of an active and stable electrocatalyst for the oxygen formation reaction in an acidic environment has been observed [118].

Shan et al., 2021 also reported that short-range regular Ir single atoms integrated into the cobalt oxide spinel structure exhibit much higher acidic OER activity and excellent stability [119]. **Velasco-Vélez et al., 2019** prepared electrochemically active Ir nanoparticles on graphene and reported that it exhibited improved acidic OER activity and stability [120]. **Forgie et al., 2010** compared pure ruthenium (Ru) with Ru-Co, Ru-Cu, and Ru-Ir for catalyst activity and reported a significant improvement in oxygen evolution reactions [121]. **Hartig-Weiss et al., 2020** synthesized an iridium oxide catalyst supported on antimony-doped tin oxide for high oxygen evolution reaction activity in an acidic environment. It has been reported that the synthesized acidic OER exhibits performance and robust stability [122].

Fig. 2b illustrates the fundamental structure and operational principle of a PEM-based electrolyzer. The system comprises a polymer membrane that permits only protons to pass, an anode catalyst layer where water undergoes decomposition through an electrochemical reaction resulting in oxygen formation, a cathode catalyst layer where hydrogen is produced, and electrode layers responsible for facilitating the transmission and removal of liquids and gases to the catalyst surface concurrently with the application of electricity [47]. Proton exchange membrane electrolytic cell (PEMEC) systems offer compactness and rapid start-up capabilities, even at room temperature [123].

The water introduced into the cell via the anode inlet undergoes dissociation into hydrogen ions (H^+, Proton) and oxygen gas within the anode catalyst layer. As oxygen gas is released from the anode outlet to the external environment, hydrogen ions traverse the Nafion membrane, receiving electrons in the cathode catalyst layer and transforming into hydrogen gas. Oxygen is produced in the anode region because of the above reaction, influenced by the ruthenium and iridium catalyst layer on the membrane surface. Hydrogen moves towards the cathode region, donating electrons (H^+). In this transition, some water migration occurs due to the structure of the Nafion membrane. Through the application of electricity to the cell, hydrogen ions in the cathode region acquire electrons, resulting in the production of hydrogen gas [47].

The reactions involved in this process are outlined below:

$$\text{Anode reaction: } 2H_2O \rightarrow O_2 + 4H^+ + 4e^- \tag{4}$$

$$\text{Cathode reaction: } 4H^+ + 4e\text{-} \rightarrow 2H_2 \tag{5}$$

$$\text{Total reaction: } H_2O \rightarrow H_2 + \tfrac{1}{2}O_2 \tag{6}$$

Most studies on the HER for PEM electrolyzers use Pt-based materials [50,51,124–126], and currently, carbon-supported highly dispersed Pt nanoparticles serve as the standard electrocatalysts for PEM electrolysis [51]. In the search for highly active HER electrocatalysts made from abundant earth materials, recent proposals have highlighted sulfides, carbides, and nitrides as promising alternatives to Pt cathodes. Among these newly developed materials, molybdenum sulfides, primarily MoS_2, stand out as a promising class for HER electrocatalysis. They exhibit relatively high activity (though still lower than Pt), excellent stability, and cost-effectiveness, given that molybdenum is approximately 104 times more abundant than Pt [127–130]. Recently, nitrogen-doped carbon-encapsulated Co nanoparticles have been employed as potential catalysts for the HER across a broad pH range. The study revealed that the strong interactions between Co nanoparticles and the carbon nano-shells on the surface led to elevated HER activities [131].

Currently, used Pt-based catalysts exhibit high activity, but their high cost and scarcity due to their noble metal nature hinder their development. Therefore, to meet industrial requirements, the construction of a highly effective, economical and durable electrocatalyst for HER with energy-saving performance at all pH conditions remains a significant challenge for hydrogen production via electrochemical water splitting [151]. Carbides, phosphides, sulfides, borides and their heterostructures have a large specific surface area that enables them to contact electrolytes faster and increase the charge transfer rate, resulting in highly active sites with more internal atoms. Table 4 list the efficiency of different HER electrocatalysts in a 0.5 M H_2SO_4 environment. Similarly, industries use the basic medium for water electrolysis and the proton concentration in the electrolyte decreases during practical applications. Consequently, it is necessary to develop an electrocatalyst that can be operated over a wide pH range [152].

Huo et al. produced a porous structured Ni_2P/Ni foam catalyst for a universal pH hydrogen evolution reaction. The electrocatalyst showed excellent durability in acidic, alkaline and neutral environments [153]. Wei et al. La and Yb-doped MoP electrocatalysts have been prepared for hydrogen evolution in a wide pH range. The results show low overpotential for acidic, alkaline and buffer solutions [154]. Jirkovský et al. investigated the effect of a wide pH range on hydrogen evolution using a Co–Mo-S_x chalcogen-based electrocatalyst. The electrocatalyst exhibits highly active and stable hydrogen evolution activity [155]. However, these methods need a long procedure that requires high temperature, vacuum, high pressure and the application of a surfactant or binder. The refinement of an electrocatalyst that can be effortlessly synthesized and produced is imperative to develop large-area and binder-free electrodes for hydrogen evolution reactions over a wide pH range. A comparison of HER activity of different electrocatalysts at universal pH is tabulated in Table 5 [152].

Electrochemical processes involve redox reactions that occur at the interface of electrodes and electrolytes. The stability and functionality of electrodes across a wide pH range are crucial for various applications, including sensors, batteries, and fuel cells. The key elements affecting electrode stability and ion transport kinetics include firstly, it is the electrode material. As electrode materials; metals, common electrode materials like platinum, gold, and silver have varying stability in different pH environments. For example, platinum is highly stable across a wide pH range, while silver tends to dissolve in acidic conditions. Metal oxides, materials such as titanium dioxide and zinc oxide are stable in alkaline environments but may dissolve in acidic conditions. Carbon-based materials, graphite, graphene, and carbon nanotubes are generally stable across a wide pH range but may undergo oxidation at

Table 4
Assessment of the efficiency of different HER electrocatalysts in a 0.5 M H_2SO_4 environment.

Catalyst	Tafel slope (mV/dec)	Exchange current density (mA/cm^2)	Current density i (mA/cm^2)	Overpotential at current density i (mV)	Reference
Amorphous MoS_3 film	40	0.0013	2	170	[132]
MoS_2 nanoparticles on graphene	41	–	10	150	[133]
Defect-rich MoS_2 nanosheets	50	0.009	10	180	[134]
Pt–MoS_2 nanosheets on carbon fibers	53.6	–	10	35	[135]
CoS_2 nanowire	51.6	0.015	10	145	[136]
FeP on candle soot	58	0.22	10	112	[137]
FeP nanowires[a]	38	0.42	10	55	[138]
CoP on carbon nanotubes	57	0.13	10	115	[139]
CoP nanowires	51	0.288	10	67	[140]
CoP nanoparticles	50	0.13	20	85	[141]
Ni_5P_4 nanocrystals	42	0.057	10	105	[142]
NiP_2 on carbon nanospheres	46	0.49	–	–	[143]
Mo/P/S	50	0.2	–	–	[144]
WP_2 nanorods	52	0.013	10	148	[145]
C_3N_4 layers on nitrogen-doped graphene	49	0.43	10	80	[146]
Mo_2C–WC nanowire	56	0.0047	80	184	[147]
$Co_{0.6}Mo_{1.4}N_2$	–	0.000015	10	200	[148]
Mo_2N	100	–	10	381	[149]
Mo_2C	56	–	10	198	[149]
Mo_2C on carbon nanotubes	55	0.014	1	63	[150]

[a] In 0.1 M $HClO_4$.

Table 5
Assessment of the efficiency of different HER electrocatalysts in acidic, alkaline, and neutral environments.

Electrocatalyst	Substrate	Overpotential (mV@10 mA/cm^2)	Tafel Slope (mV/dec)	Reference
CuS/NiO	Ag-modified cellulose paper	363[a]	119	[151]
		290[b]	90	
		312[c] (@20 mA/cm^2)	194	
CuS	Ag-coated paper	79[a]	128	[156]
		258 (3 M NaCl)	205	
		375[b]	316	
Co-CW	Wood	271[b]	275	[157]
Ni-CW		230[b]	207	
Co/Ni-CW		157[b]	68	
NiCoFe phosphate	Ni Foam	231[b]	86	[158]
NOPHC10-900′s	Glassy carbon	280[b]	120	[159]
Ni NWs	PC/Au/Ni	275[b]	119	[160]
$Ni_{68}Co_{32}$ NWs	PC/Au/Ni	256[b]	94	
Ni_6CO_{94} NWs	PC/Au/Ni	231[b]	105	
$Co_{0.75}Fe_{0.25}$-NC	Glassy carbon	202[b]	67.9	[161]
Pd@NLS	Glassy carbon	250[b]	251	[162]
Ni_3FeN NPs	Carbon cloth	238[b]	46	[163]
$NiFe_2O_4$	Glassy carbon	300[b]	125	[164]
NiFe LDH-NS@DG	Ni foam	300[b]	110	[165]
Cu_2CoSnS_4	Whatman filter paper	233[a]	110	[152]
		310[b]	122	
		261[c]	273	
CONC/GD	Copper Foil	340[a]	138	[166]
		284[b]	115	
		368[c]	207	
Ni_2P NPs @ NPC	Glassy carbon	200[a]	–	[167]
		251[b]	–	
		433[c]	–	

*Electrolytes:
[a] – 0.5 M H_2SO_4.
[b] – 1 M KOH.
[c] – 1 M PBS.

high anodic potentials. Conducting polymers: Polymers like polyaniline and polypyrrole exhibit pH-dependent redox behavior, which can affect their stability and conductivity. Secondly, it is the Electrode-Electrolyte Interface. At the electrode-electrolyte interface, the electric double layer and surface adsorption are examined. Electric double layer, the structure of the electric double layer changes with pH, affecting the distribution of ions near the electrode surface and the potential drop across the interface. Surface adsorption, pH influences the adsorption of ions and molecules on the electrode surface, which can either passivate or activate the surface. For example, in alkaline solutions, hydroxide ions can adsorb on metal surfaces, altering their electrochemical behavior [168]. The third is the electrochemical reactions. Hydrogen Evolution Reaction (HER) and Oxygen Evolution Reaction (OER), both HER and OER are highly pH dependent. HER is more favorable in acidic conditions, while OER occurs readily in alkaline conditions. The stability of the electrode material is critical for these reactions. Reversible Hydrogen Electrode (RHE), the potential of the RHE varies with pH, influencing the electrochemical window and the stability of electrodes in different pH conditions. Fourth, it is the passivation and corrosion. Passivation, the formation of a stable oxide layer on the electrode surface can protect it from further corrosion. For instance, aluminium forms a protective oxide layer in neutral and slightly alkaline conditions. Corrosion, in acidic environments, metals like iron and zinc undergo rapid corrosion, while in alkaline environments, metals like aluminium and magnesium are more prone to corrosion [169]. Finally, it is the ion transport kinetics. Ion transport kinetics is essentially the rate at which ions migrate between the electrode and the electrolyte solution. Efficient ion transport is vital for optimal performance in electrochemical devices. Here are some key factors influencing ion transport kinetics; electrode material properties, the porosity, and conductivity of the electrode material play a critical role. Porous materials allow for better electrolyte penetration and ion diffusion, while high conductivity facilitates faster ion movement. Electrolyte composition, the ionic strength, and the viscosity of the electrolyte solution affect ion mobility. Higher ionic strength generally leads to faster ion transport, while increased viscosity hinders ion movement. Applied potential, the applied potential can influence the rate of ion transport by affecting the concentration of ions at the electrode surface. Presence of diffusion limitations, in some cases, the rate of ion transport may be limited by the diffusion process within the bulk solution or the electrode material. This can lead to a concentration gradient near the electrode surface, hindering further ion transfer [170]. The stability and functionality of electrodes in a wide pH range depend on a complex interplay of material properties, electrochemical reactions, and ion transport kinetics. Understanding these factors and their interactions is crucial for designing durable and efficient electrochemical systems for various applications [168].

Notably, molecular catalysts embedded within a polymer matrix have demonstrated outstanding molecular catalysis capabilities for the hydrogen evolution reaction (HER) [171–173]. Extensive research has been conducted on transition metal complexes with various ligands, exploring their potential as homogeneous and/or heterogeneous electrocatalysts for the hydrogen evolution reaction (HER). The rich redox properties in these complexes motivate the in-depth study. Complexes exhibiting metal-based reduction processes before HER have generally demonstrated commendable electrocatalytic activity. Porphyrin and phthalocyanine derivatives have been explored as promising candidates for molecular catalysis in this context [174–176]. Phthalocyanines with metal-based reduction processes have been identified as promising cathode electrocatalysts for enhancing the reaction kinetics of the HER, thanks to their higher activities (Fig. 4) [177]. The catalytic activity of the phthalocyanine complexes experienced a substantial enhancement upon their incorporation with Nafion [178].

To comprehensively assess the hydrogen evolution reaction (HER) activity of distinct electrode materials, it is essential to comprehend the mechanism through which HER takes place. Typically, a Tafel plot is employed to ascertain the mechanism and assess the efficiency of the catalytic reaction [180]. The more the Tafel slope decreases, the higher the activity towards the HER [123]. The small overpotential observed in the electrodes, a remarkable 33-fold increase in the current density of the hydrogen evolution reaction (HER), signifies the superior catalytic performance of these electrodes [179]. The electrocatalytic activities of the electrodes decline with increases in the pH of the solution [179].

3.4. Solid oxide electrolysis (SOEL)

Solid oxide electrolysis (SOE) was first introduced by Donitz and Erdle in the 1980s (Carmo et al., 2013). It has attracted attention due to the conversion of electrical energy into chemical energy in the solid oxide electrolysis method and the more efficient production of ultrapure hydrogen. Since solid oxide electrolysis takes place at high pressure and high temperatures such as 500–850 °C, water is used in the form of steam in this method. O^{-2} ions are the charge carriers in solid oxide electrolysis processes [44]. The working principle of SOE is shown in Fig. 2c. Some ceramic proton-conducting materials have been developed for solid oxide fuel cells, and research is ongoing. Developing a stable working system is necessary for the large-scale commercialization of the solid oxide electrolysis (SOE) method. Due to solid oxide water electrolysis's high operating temperatures, thermal stability is prioritized in electrode selection. For this reason, the state-of-the-art material in the hydrogen formation reaction consists of a ceramic-metal(cermet) consisting of a yttria-stabilized zirconia (YSZ) electrolyte and nickel, a non-precious metal catalyst with high electronic conductivity. It is usually referred to as Ni-YSZ [44]. In the same way, the catalyst used for the oxygen formation reaction must contain excellent electrocatalytic activity, high mixed ionic-electronic conductivity, and appropriate chemical and thermal compatibility with the electrolyte over time. Co-based and Sr-doped perovskites in catalyst production are often used as O_2 electrode materials due to their higher electrocatalytic performance. The latest technology is perovskite (La, Sr) (Co, Fe) O_3-based, and $La_{0.8}Sr_{0.2}MnO_{3-\delta}$-based materials with optimized microstructures (particle size and volumetric content in single layers) [181]. In addition to electrode mechanical strength and thermal and chemical stability, adequate durability is critical. For this reason, many studies are possible for electrode improvements. In the study conducted by **Deka et al., 2019** the performance of nickel-doped on lanthanum strontium ferritin ($La_{0.7}Sr_{0.2}FeO_3$) as a SOEC cathode was investigated. It has been found that $La_{0,7}Sr_{0,2}FeO_3$ for hydrogen production has a lower faradaic efficiency than the Ni-doped cathode, where the efficiency reaches 100% [182]. In the study conducted by **Tan et al., 2022,** it was investigated silver and gadolinium-doped cerium (Ag-GDC) as an electrode material for high-performance energy production and water electrolysis. Conventional hydrogen electrode, Ni-YSZ, and oxygen electrode, LSCF ($La_{0,6}Sr_{0,4}Co_{0,2}Fe_{0,8}O_{3-\delta}$)-GDC were tested for comparison purposes. It has been found that it is more active as a solid oxide electrode than Ag-GDC, Ni-YSZ, and LSCF-GDC [183]. In the study conducted by **Alizad Farzin et al., 2022** the electrocatalytic activity of $Sr_2FeMo_{0.5}Mn_{0.5}\ O_6$ (SFMM) double perovskite is carefully evaluated as a candidate to replace the state-of-the-art Ni/YSZ fuel electrode. Improving the catalytic activity with increasing oxygen partial pressure is an important feature of the developed electrode, making this electrode a promising candidate for solid oxide electrolysis application [184]. **Yu et al., 2020** adopted the method of adding Fe to Ni to produce Ni-Fe bimetal to improve the performance of a Ni-based cathode in their study. The study has proven that Ni-Fe bimetal can improve the performance of a Ni-based cathode in the appropriate proportion and has also shown that this material acts as an excellent cathode for SOEC at medium temperatures [185]. **Neofytidis et al., 2019** focused on a study on how the contribution of Au/Mo to NiO/GDC powder can affect the electrolysis of solid oxide cell water through its modification. In particular, the triple cathode cell performed significantly better than (Ni-, Au-, Mo-) binary electrodes and Ni/GDC. The superior performance of the ternary sample has been attributed primarily to the enrichment of the surface with Au and the bulk phase with Mo through the formation of the Ni-Au-Mo solid solution [186].

The Solid Oxide Electrolyzer Cell (SOEC) stands out as a promising technology for ensuring sustainable development in the future. It enables the recycling of CO_2 into viable fuels and holds significant potential for the hydrogen economy. Solid Oxide Electrolyzers represent the technology with the least commercial development. Operating at significantly elevated temperatures compared to other technologies, they consume substantially less electricity owing to their superior energy conversion efficiency. Nevertheless, the high-temperature operation necessitates the use of specialized materials capable of withstanding the conditions of the process [187]. Solid oxide electrolyzers (SOELs)

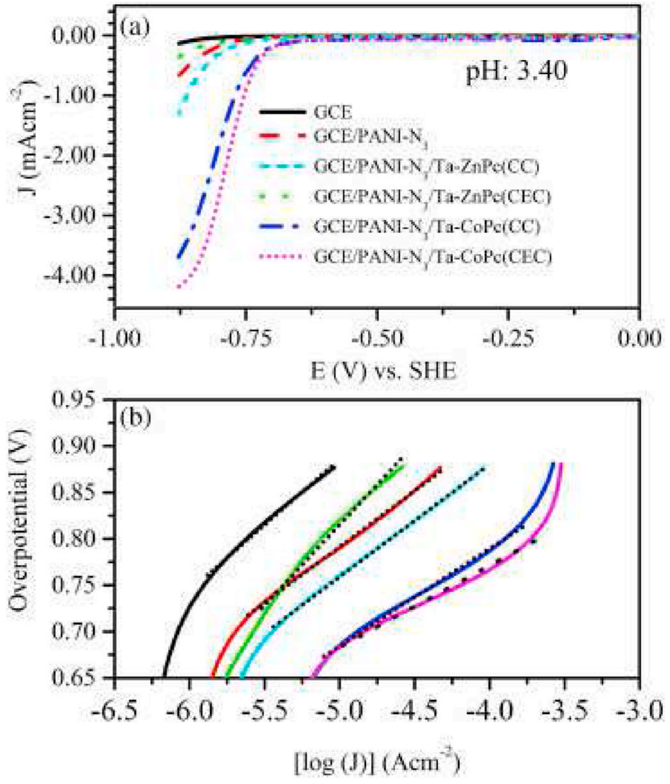


Fig. 4. a) LSVs and b) Tafel plots of various modified electrodes in acidic media [179].

commonly function at temperatures exceeding 500 °C, utilizing water in the form of steam. Traditionally, SOE technology relies on O_2 conductors. Nevertheless, in recent years, various ceramic proton conductors have emerged and been employed in solid oxide fuel cells [106]. When it comes to operational conditions and the reactions occurring at the electrode surface, the materials employed in these distinct systems must possess specific properties. The technology has the capability to expand into the simultaneous co-electrolysis of H_2O and CO_2. The resulting product from this co-electrolysis is syngas, which can be further processed to generate synthetic fuel [188,189]. Consequently, solid oxide electrolyzers present the potential for chemical energy storage/carrier when converting renewable energy or surplus energy from fossil power plants into hydrogen or syngas [190].

The hydrogen electrode in large-scale Solid Oxide Electrolyzers (SOEs) is commonly made of ceramic-metallic cermet Ni-YSZ, where YSZ refers to ZrO_2 stabilized with 8 mol% Y_2O_3 [191–194]. Reports indicate a decline in the performance of Ni-YSZ during extended steam electrolysis, attributed to cracks in Ni-YSZ [195], as well as Ni aggregation resulting from elevated temperature and humidity [196]. Consequently, endeavors have concentrated on innovating materials with superior activity and stability to substitute Ni-YSZ cathodes in Solid Oxide Electrolyzers (SOEs). Initially, the concept of combining highly dispersed Ni with samaria-doped ceria was introduced [197], but current research endeavors primarily focus on the advancement of novel perovskite materials. Cathodes for steam Solid Oxide Electrolyzers (SOEs) are frequently explored in the form of substituted lanthanum chromates [198]. In the pursuit of enhanced electrocatalytic activity, the incorporation of active Fe [199] or Ni [200] into the lanthanum composite has been investigated. The introduction of Ni into the perovskite-based composite led to a notable 20% rise in Faradaic efficiency [106]. Recently, there was a proposal for a new fuel electrode for steam electrolysis in oxygen-ion solid oxide electrolyzers featuring a perovskite chromate cathode with nickel nanoparticles securely anchored [201]. The experimental results demonstrated that Fe-doped titanate with A-site deficiency and B-site excess has the potential to serve as a cathode for oxygen-ion conducting Solid Oxide Electrolyzers (SOEs). The incorporation of Fe resulted in an enhanced electrocatalytic activity for the titanate, like the improvements observed in the cases of chromate doping [199–201]. To ensure a substantial length of three-phase boundaries (TPBs) and to improve adhesion to the electrolyte, hydrogen electrodes are commonly made from nickel-ceramic cermets rather than pure nickel [202,203].

3.5. The microbial electrochemical systems (MESs)

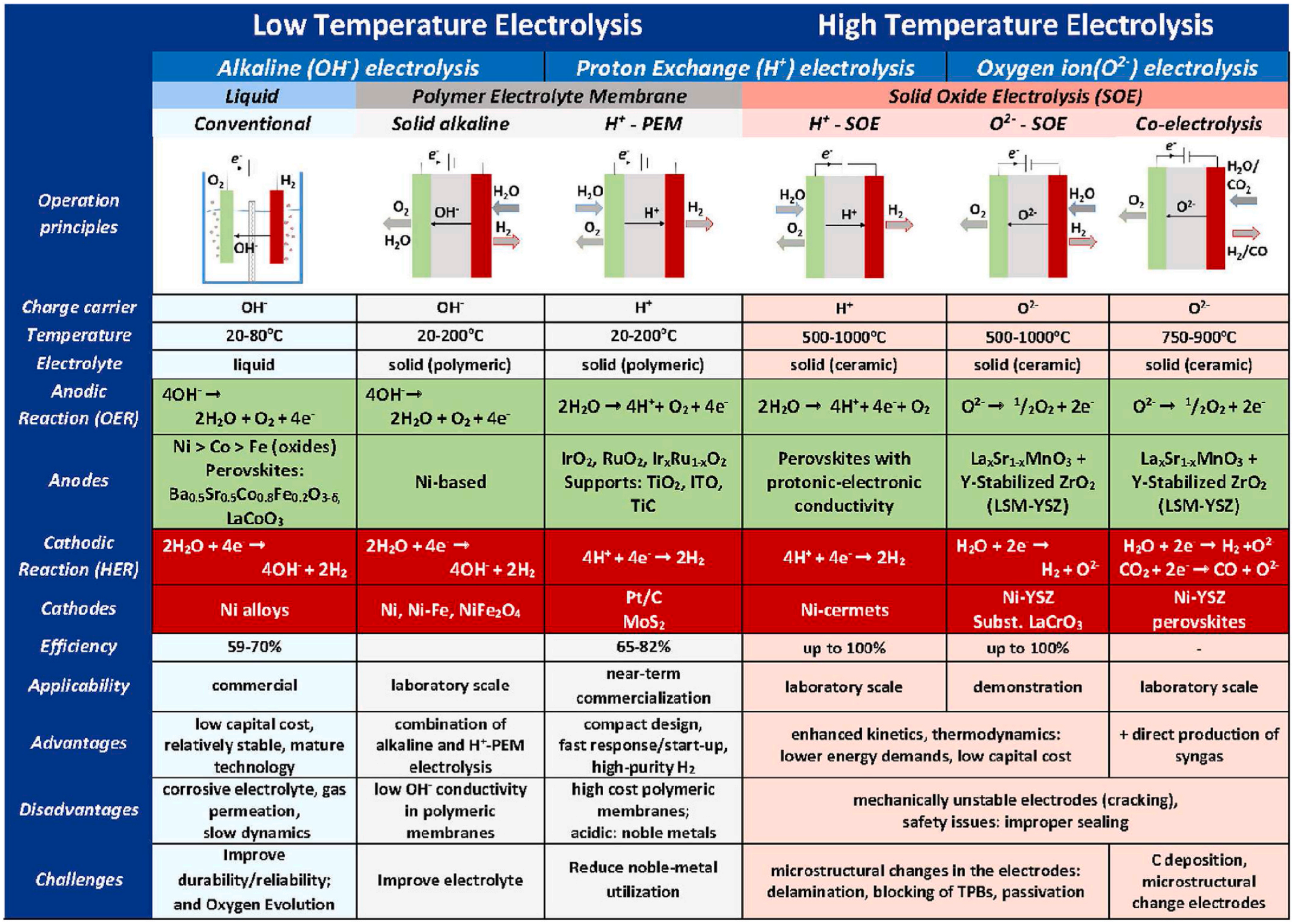
The Microbial Electrolysis Cell (MEC) is an environmentally friendly technology that can extract electricity and hydrogen from wastewater [204]. MEC offers a promising solution for wastewater treatment and simultaneous energy recovery [205–209]. The electrohydrogenesis technique in microbial electrolysis cells (MECs) can yield higher quantities of hydrogen gas than fermentation, with greater energy efficiency than traditional water electrolysis. Hydrogen production in MEC can be considered green hydrogen [210]. Various factors influence cell performance in MEC, such as applied potential, current density, temperature, pH, substrate, and scale [57,67]. The cathode plays a crucial role in MECs for gas production, as it can generate hydrogen and other valuable products. Finding a cathode material with excellent catalytic activity for HER is essential. Choosing the suitable cathode catalyst, along with the cathode material and binder, is crucial for MECs to produce hydrogen efficiently, as it is the primary site for HER [211]. Previous reports have highlighted Pt as a preferred catalyst due to its exceptional catalytic activity for the hydrogen evolution reaction (HER). However, its commercial application is constrained by high costs and susceptibility to fouling. Finding cost-effective alternative materials with comparable HER capabilities is essential to ensure the economic viability of the process without relying on Pt [212]. Nickel (Ni)-based materials have been selected as favorable electrocatalysts to replace Pt due to their comparable catalytic activity and lower cost [80,213,214]. Modifying the cathode materials could enhance hydrogen production in MECs. A study that used nickel as an additive in modified cathodes, using PVDF or PTFE as binders in activated carbon-based cathodes, showed that Ni/AC/PTFE combines the catalytic activity of nickel with the high cathode capacitance and porosity of PTFE. Ni shows promise as a viable catalyst for HER in terms of coulombic efficiency, energy efficiency, hydrogen production, and cost-effectiveness [68].

MFCs generate renewable bioelectricity from wastewater, positioning them as one of the most significant environmental technologies addressing water and energy crises. However, electrogenic microbial communities' inconsistent and slow activity poses a substantial challenge for achieving accurate and reproducible measurements in MFCs. MFCs utilize microorganisms within the anode chamber to break down organic substances, transferring electrons to the anode [215]. Electrons generated by bacteria from the substrates are transferred to the anode (negative terminal) and flow to the cathode (positive terminal) through a conductive material with a resistor or when operated under a load. Conventionally, current is considered to flow from the positive terminal to the negative terminal, which is opposite to the actual direction of electron flow. The microbial generation of electrons at the anode and their subsequent consumption at the cathode, both sustainable processes, are the defining characteristics of MFCs. The effectiveness of MFCs applications in wastewater treatment hinges on factors such as the concentration and biodegradability of organic matter in the influent, wastewater temperature, and the absence of toxic chemicals. Despite the construction of MFCs using various materials and configurations expanding, further improvements in MFC designs are necessary before a marketable product can be realized [216]. The fluctuating and sluggish kinetics of electrogenic microbial cells pose significant challenges for achieving accurate and consistent measurements in MFCs. Enhancing the reproducibility of MFC data requires constructing MFCs that are as identical as possible and employing standardized measurement techniques. These challenges can be addressed by utilizing systems with low internal resistance and minimal systematic differences, such as single-chamber cubic MFCs. Due to its lower internal resistance and reduced variability compared to other MFC systems, it consistently produces more reproducible results. Additionally, there is a need to advance methods for reproducible measurements [217]. Although MFCs are not generally intended for hydrogen production, they can be modified or integrated into hybrid systems to produce hydrogen. Microbial Electrolysis Cells (MECs) are better suited for more direct and efficient hydrogen generation. A coupled MEC-MFC system for biological hydrogen production from waste consists of an MEC for electrolysis and an MFC with an air cathode supplying the additional electricity needed. The main advantage of this setup is that it uses the electric energy generated in-situ, allowing for hydrogen production without the need for an external power source [56,218].

Microbial desalination cells (MDCs), which utilize microbial consortia to remove ions from low-salinity aqueous solutions by converting chemical energy, have been studied extensively. MDCs are designed similarly to MFCs (Microbial Fuel Cells) but include a desalination chamber between the anode and cathode. This chamber is separated from the anode by an anion exchange membrane (AEM) and from the cathode by a cation exchange membrane (CEM) [219]. MDCs can be integrated with MECs to generate hydrogen and valuable chemicals in an energy-efficient manner while simultaneously treating wastewater and desalinating water. This integration is achieved by dividing the cell into three chambers using ion exchange membranes [220]. A reactor configuration known as the Microbial Electrolysis and Desalination Cell, which enables simultaneous hydrogen production, desalination, and wastewater treatment, was developed by Luo et al. [220]. Hydrogen production in this system can be enhanced by increasing the external voltage [215,221].

A microbial reverse electrodialysis cell (MRC) represents an innovative microbial electrochemical system comprising an anode chamber, a cathode chamber, and a reverse electrodialysis (RED) stack [222,223]. This technology integrates a microbial fuel cell (MFC) with a RED stack to harness electricity from the salinity gradient between seawater and freshwater, simultaneously enabling wastewater treatment [224]. Reverse electrodialysis (RED) utilizes membrane-based technology to directly convert chemical potentials into electricity by facilitating ion transport [225]. The RED stack is constructed by alternately stacking anion exchange membranes (AEMs) and cation exchange membranes (CEMs), separated into high-concentration (HC) and low-concentration (LC) cells [226,227]. The integration of MEC and RED is called a microbial reverse-electrodialysis electrolysis cell (MREC). Hydrogen gas can be generated through electrochemical processes in MRECs [228]. A limitation of using MRECs for hydrogen gas production is the necessity for nearby organic matter and seawater sources, which restricts the process to coastal areas, rendering it impractical for inland regions. Another potential drawback of this method is seawater, which can lead to biofouling of the membranes unless the water is treated similarly to the processes used in reverse osmosis desalination systems [229].

Microbial Electrosynthesis (MES) refers to the process where microorganisms, utilizing a biocathode, convert electrical energy into high energy-density chemicals (fuels) or their precursors. MES presents an innovative and desirable method for generating valuable products from electricity and wastewater [72]. MES is an innovative technology that delivers electrons to microorganisms through an electric current within an electrochemical cell, utilizing a biocathode composed of a biofilm and cathode. These microorganisms function as biocatalysts, harnessing the electrons to convert carbon dioxide into valuable industrial products, including transportation fuels [230,231]. Cathode catalysts for hydrogen production in MES include platinum, which, despite its effectiveness, is economically unfeasible due to its high cost. Consequently, alternative materials such as stainless steel and nickel are preferred for their affordability, stability, and low overpotentials. These materials are more efficient than platinum for hydrogen production because they operate at lower voltages and are cost-effective. However, while initially effective, enzymatic biocathodes are unstable and lose catalytic activity over time. Research has demonstrated that hydrogen production can be efficiently catalyzed by immobilizing the necessary enzymes on carbon electrodes, ensuring a more sustainable catalytic process [72,232–236]. The performance of microbial electrosynthesis (MES) can be significantly enhanced by utilizing a HER catalyst. This improvement is achieved by constructing an electrode modified with molybdenum carbide (Mo_2C), an active HER electrocatalyst. Electrochemical studies have indicated that Mo_2C can facilitate the production of H_2, support biofilm formation, and monitor the mixed culture of microbes. Mo_2C exhibits an electronic structure akin to platinum, making it a high-performing HER electrocatalyst. Incorporating molybdenum carbide into carbon felt (Mo_2C-CF) leads to a substantial increase in H_2 evolution in MES, with an average rate 12.7 times higher than that of carbon felt without Mo_2C. Furthermore, Mo_2C aids in regulating the mixed culture of microbes in biofilms and planktonic cells during microbial electrosynthesis. Integrating molybdenum carbide into the carbon-felt cathode is a critical, rapid, and straightforward approach



	Low Temperature Electrolysis			High Temperature Electrolysis		
	Alkaline (OH^-) electrolysis		Proton Exchange (H^+) electrolysis		Oxygen ion(O^{2-}) electrolysis	
	Liquid	Polymer Electrolyte Membrane		Solid Oxide Electrolysis (SOE)		
	Conventional	Solid alkaline	H^+ - PEM	H^+ - SOE	O^{2-} - SOE	Co-electrolysis
Operation principles						
Charge carrier	OH^-	OH^-	H^+	H^+	O^{2-}	O^{2-}
Temperature	20-80°C	20-200°C	20-200°C	500-1000°C	500-1000°C	750-900°C
Electrolyte	liquid	solid (polymeric)	solid (polymeric)	solid (ceramic)	solid (ceramic)	solid (ceramic)
Anodic Reaction (OER)	$4OH^- \rightarrow 2H_2O + O_2 + 4e^-$	$4OH^- \rightarrow 2H_2O + O_2 + 4e^-$	$2H_2O \rightarrow 4H^+ + O_2 + 4e^-$	$2H_2O \rightarrow 4H^+ + 4e^- + O_2$	$O^{2-} \rightarrow {}^1/_2O_2 + 2e^-$	$O^{2-} \rightarrow {}^1/_2O_2 + 2e^-$
Anodes	Ni > Co > Fe (oxides) Perovskites: $Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-\delta}$, $LaCoO_3$	Ni-based	IrO_2, RuO_2, $Ir_xRu_{1-x}O_2$ Supports: TiO_2, ITO, TiC	Perovskites with protonic-electronic conductivity	$La_xSr_{1-x}MnO_3$ + Y-Stabilized ZrO_2 (LSM-YSZ)	$La_xSr_{1-x}MnO_3$ + Y-Stabilized ZrO_2 (LSM-YSZ)
Cathodic Reaction (HER)	$2H_2O + 4e^- \rightarrow 4OH^- + 2H_2$	$2H_2O + 4e^- \rightarrow 4OH^- + 2H_2$	$4H^+ + 4e^- \rightarrow 2H_2$	$4H^+ + 4e^- \rightarrow 2H_2$	$H_2O + 2e^- \rightarrow H_2 + O^{2-}$	$H_2O + 2e^- \rightarrow H_2 + O^2$ $CO_2 + 2e^- \rightarrow CO + O^{2-}$
Cathodes	Ni alloys	Ni, Ni-Fe, $NiFe_2O_4$	Pt/C MoS_2	Ni-cermets	Ni-YSZ Subst. $LaCrO_3$	Ni-YSZ perovskites
Efficiency	59-70%		65-82%	up to 100%	up to 100%	-
Applicability	commercial	laboratory scale	near-term commercialization	laboratory scale	demonstration	laboratory scale
Advantages	low capital cost, relatively stable, mature technology	combination of alkaline and H^+-PEM electrolysis	compact design, fast response/start-up, high-purity H_2	enhanced kinetics, thermodynamics: lower energy demands, low capital cost		+ direct production of syngas
Disadvantages	corrosive electrolyte, gas permeation, slow dynamics	low OH^- conductivity in polymeric membranes	high cost polymeric membranes; acidic: noble metals	mechanically unstable electrodes (cracking), safety issues: improper sealing		
Challenges	Improve durability/reliability; and Oxygen Evolution	Improve electrolyte	Reduce noble-metal utilization	microstructural changes in the electrodes: delamination, blocking of TPBs, passivation		C deposition, microstructural change electrodes

Fig. 5. An overview of the fundamental features of water electrolysis technologies [106].

that effectively enhances the MES system. To develop an efficient H_2 catalyst, Mo_2C shows advantages in hydrogen evolution even under acidic conditions, despite typically being neutral. Thus, active HER catalysts like Mo_2C boost hydrogen release, promoting the growth of mixed microbial culture biofilms and leading to a higher CO_2 reduction rate [72,234,237].

3.6. Comparison of electrolysis methods

Each electrolysis type comes with its own set of advantages and disadvantages, yet all three technologies are attractive, and hold promise for sustainable energy applications (Fig. 5).

3.7. Hydrogen evolution reaction (HER) in water splitting process

Hydrogen, the Earth's most prevalent element, is typically bound with other elements and consistently exists within water, biomass, and fossil hydrocarbons. The production of hydrogen gas currently stems from diverse primary sources, including natural gas, naphtha, heavy oil, water, and coal. At present, industrial facilities worldwide generate approximately 55 million metric tons of hydrogen annually, experiencing a demand growth of approximately 5% each year [88]. Considering the energy crisis and environmental pollution resulting from the combustion of fossil fuels, significant progress has been made in the recent development of the hydrogen evolution reaction (HER) using solar light [29,238–240]. Water electrolysis can yield exceptionally pure hydrogen; however, a significant drawback lies in the elevated energy consumption associated with this process [17,241]. To minimize production expenses by decreasing the overpotential (the potential difference between reversible reduction potential and practical discharge potential) in the hydrogen evolution reaction (HER), diverse electrocatalytic materials have been extensively examined as optimal solutions [242–245]. There are two types of catalysts, electrocatalysts, and photocatalysts, which are used in hydrogen evolution reactions [88]. The basic properties of electrocatalysts are listed in Table 6 and illustrated in Fig. 6.

3.7.1. Cathode electrocatalysts for hydrogen evolution reaction (HER)

Enhancing the specific performance of electrodes through catalyst utilization is crucial for effectively reducing production costs [242]. Electro-catalysts for the hydrogen evolution reaction play a crucial role in lowering the energy barrier and enhancing process efficiency. Additionally, the catalytic capability is highly influenced by both hydrogen adsorption energy and the kinetic energy barrier, variables that can be altered by the catalyst atom.

Table 6
Criteria for selecting anode and cathode materials [246].

Surface area and porosity	The surface area of the electrodes greatly limits the output power of MFCs. The Ohmic losses are proportional to the resistance of the circuit an electrode The simplest way to reduce resistance is to expand the effective surface area while maintaining the volume constant improves efficiency. Furthermore, a large surface area allows more reaction sites, which improves electrode kinetics. However, porosity reduces electrical conductivity [247,248].
Electrical conductivity	After passing through the anode, electrons released by microbes must travel along an external circuit. The electron flow is caused by the electrode material's high electrical conductivity [249].
Stability and durability	The reducing and oxidizing environment in an MFC can cause material swelling and decomposition. The large surface roughness increases the material's durability while it may increase the likelihood of fouling and, as a result, may decrease the MFC's long-term performance. Therefore, electrode material should be durable as well as stable in an acidic and a basic environment [250].
Cost and accessibility	To be commercially viable, MFC must be low-cost, long-lasting, and readily available. Some metals, such as platinum, are extremely expensive, in addition to being non-durable and unsustainable. Materials made of non-precious metals, such as composites could be used as a replacement. In the future, precious metals will be used in electrodes [251,252].

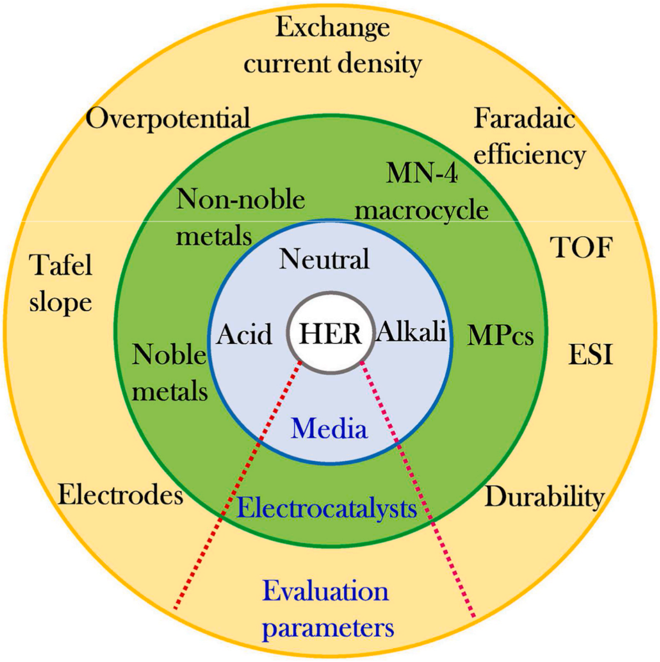


Fig. 6. Outline of the electrolyte media, various electrocatalysts, and evaluation parameters for HER [177].

Historically, catalysts based on noble metals, like platinum catalysts, have demonstrated impressive efficiency and catalytic activity for the hydrogen evolution reaction (HER) with reduced overpotentials. Despite being benchmark catalysts for HER, the widespread application of these precious metal-based catalysts is hindered by their high cost and limited reserves [253–255]. Numerous studies explore various alternative catalysts and electrode materials in synthetic routes, aiming to foster the advancement of next-generation electro-catalysts [256–258].

Nevertheless, there are limited instances of effective homogeneous catalysts for this reaction (Fig. 7). In the pursuit of new catalysts for the hydrogen evolution reaction (HER), research efforts have concentrated on a diverse array of functional materials, including nickel and cobalt-based alloys [259,260], MoSx [261], graphene [261], cobalt clathrochelates [262], iron hydrogenase [260,263], glyoxime and tetraimine complexes [264], and metal phthalocyanine complexes (Table 7) [265,266].

3.7.2. Cathodic HER mechanisms under alkaline and acidic conditions

The initiation of the hydrogen evolution reaction (HER) under alkaline conditions involves two distinct processes: adsorption and dissociation. In these processes, H* is generated because of the breakdown of the H-O-H bond [279,280]. The step responsible for producing H* is identified as the rate-determining step, demanding additional energy for the formation of this H* proton. HER predominantly occurs through two mechanisms: the **Volmer-Tafel mechanism** or the **Volmer-Heyrovsky mechanism**, with Volmer representing the adsorption step and Tafel and Heyrovsky representing the dissociation step [281]. The reactions occurring in an alkaline medium (Fig. 8) are outlined as follows equations:

$$\text{Volmer reaction: } H_2O + e^- \rightarrow H^* + OH^- \tag{7}$$

$$\text{Heyrovsky reaction: } H^* + H_2O + e^- \rightarrow H_2 + OH^- \tag{8}$$

$$\text{Tafel reaction: } H^* + H^* \rightarrow H_2 \tag{9}$$

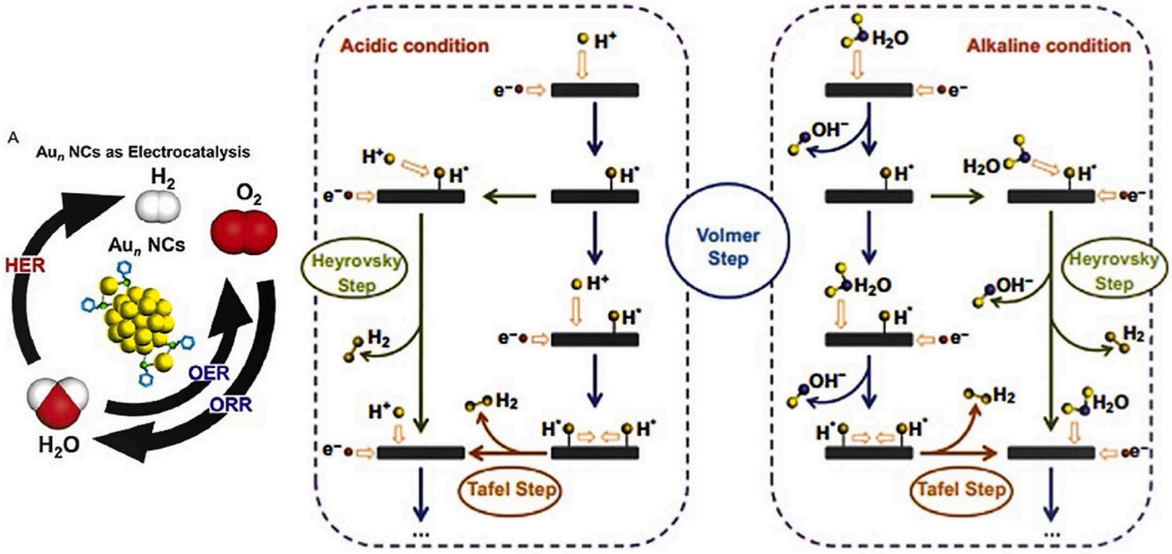


Fig. 7. Schematic representation of the role of catalyst in hydrogen production [246].

Table 7
HER catalysts and their resultant hydrogen production rates [246].

HER Catalyst	Cathode material	Hydrogen Production Rate ($m^3H_2\ m^{-3}\ d^{-1}$)	Reference
Pt	Carbon paper	0.3	[267]
Pd	Carbon paper	0.68	[267]
NiFeLDH	Nickel foam	2.0	[267]
Fe/Fe_3C (N-doped)	Graphite carbon	0.02	[268]
MoS_2	Carbon nanotubes	0.01	[269]
Nickel Hollow fiber	Hollow fiber membrane	0.2	[270]
Ni mesh	–	4.2	[271]
NiFe double hydroxide	Nickel foam	2	[79]
$NiCl_2$	Stainless steel mesh	0.3	[272]
Ni-Graphene oxide	Carbon film	4.2	[273]
NiMo	Carbon fiber-weaved cloth	–	[274]
NiW	Carbon fiber-weaved cloth	–	[274]
Ni 210	Carbon Cloth	–	[213]
Ti mesh		–	[275]
Ti plate		0.052	[276]
NiOX	Stainless steel	0.76	[277]
Ni foam	Stainless steel	50	[214]
Ni 201		0.38 ± 0.04	[278]
Ni 400		0.41 ± 0.10	[278]
Ni 625		0.79 ± 0.27	[278]
Ni HX		0.55 ± 0.11	[278]

Here, the asterisk * denotes the empty site on the catalyst, and H* represents the hydrogen atom absorbed onto that site.

In an acidic environment, the cathodic reaction involves the reduction of hydronium ions to form gaseous hydrogen molecules. This reaction typically takes place at the reference hydrogen electrode (RHE). The reactions are outlined with equations below:

$$\text{Volmer reaction: } H_3O^+ + e^- + * \rightarrow H^* + H_2O \tag{10}$$

$$\text{Heyrovsky reaction: } H^* + H_3O + e^- \rightarrow H_2 + H_2O \tag{11}$$

$$\text{Tafel reaction: } H^* + H^* \rightarrow H_2 \tag{12}$$

Equation (10) entails the reduction of a proton at an active site of a catalyst. Meanwhile, Equations (11) and (12), corresponding to the Heyrovsky and Tafel reactions, respectively, are employed for the generation of hydrogen gas. The Heyrovsky reaction involves electron transfer, while the Tafel reaction involves the recombination of two absorbed protons [282]. It is noteworthy that the kinetics of the hydrogen evolution reaction (HER) significantly rely on the electrode material. For instance, the kinetics are slower on a Hg electrode compared to Pt, which is renowned as one of the fastest electrocatalytic processes [283].

3.8. *Oxygen evolution reaction (OER) in water splitting process*

Water electrolysis can generally be divided into two half-cell reactions: hydrogen formation reaction (HER) and oxygen formation reaction (OER). HER is the reaction in which water is reduced at the cathode to produce H_2, and OER is the reaction in which water is oxidized at the anode to produce O_2. One of the critical barriers that protects the splitting of water from practical use is the slow reaction kinetics of OER and HER due to high extreme potential, which is a measure of kinetic energy barriers. Therefore, catalysis plays an important role in both OER and HER. Moreover, OER greatly affects the overall energy efficiency because slow OER kinetics causes a large excess potential, so many efficient catalysts are needed. Highly effective catalysts are required to minimize the excessive potential of OER and HER for efficient H_2 and O_2 production.

As mentioned earlier, OER is the other important half-reaction in the water-splitting reaction. This reaction occurs at the anode and involves a four-electron transfer process that requires a high excess potential compared to HER. It is known that OER is the biggest bottleneck in improving the overall efficiency of the electrochemical water separation process. Therefore, it is imperative to search for high-efficiency OER catalysts that can effectively reduce the kinetic limitation. Significant progress has been made in recent developments towards the understanding of the mechanism of OER towards the rational design of OER electrocatalysts. It is widely accepted that OER can progress through two different mechanisms, namely conventional adsorbate evolution (AEM) and lattice oxygen-mediated mechanism (LOM), which are discussed in two of the following subsections [284].

3.8.1. *Anodic oxygen evolution reaction (OER) mechanism of adsorbate evolution (AEM)*

For OER, the adsorbate evolution mechanism (AEM) has

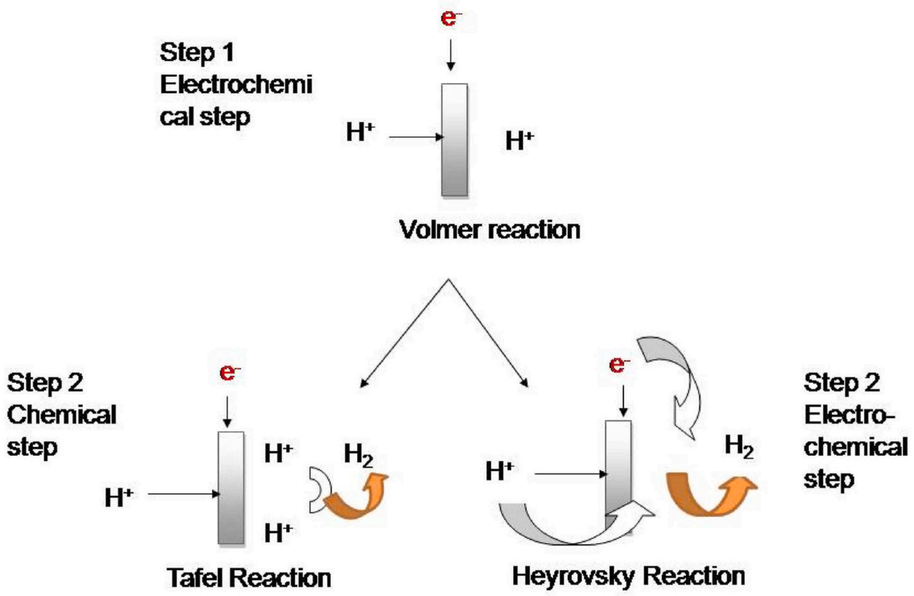


Fig. 8. Mechanism of the hydrogen evolution reaction under alkaline conditions [246].

traditionally been used to describe various reaction steps. In AEM, the reaction typically involves the transfer of four compatible protons and electrons, where the metal centers are the active site (M) and produce oxygen molecules from water in acidic and alkaline media. First, M − OH is formed by adsorbing hydroxide anions to the active site of the metal. Then M − OH forms M − O after deprotonation. One way of this is that M − O reacts with OH− to form the intermediate M–OOH and produces O_2 through decontonation of M–OOH by regeneration of the active site. The other way involves combining the two M − O types and converting them to O_2 along with the regeneration of the M active site, which is thought to have a large activation barrier. The OER(AEM) reaction steps are shown in the equations below [284];

$$OH^- + M \rightarrow M - OH + e^- \tag{13}$$

$$M - OH + OH^- \rightarrow M - O + H_2O + e^- \tag{14}$$

$$M - O + OH^- \rightarrow M - OOH + e^- \tag{15}$$

$$M - OOH + OH^- \rightarrow O_2 + H_2O + e^- + M \tag{16}$$

For the electrocatalysis of OER, a detailed understanding of the binding strength of the reaction intermediates on the electrode surface is crucial for improving the overall OER performance, since the binding strength is an important parameter governing the overpotential of the reaction. Catalysts play a very important role in lowering the kinetic barrier in electrolytic water separation. The evaluation of the performance of a catalyst for electrocatalytic water separation is based on various basic parameters in terms of activity, stability, and efficiency. The activity is characterized by excessive potential, Tafel slope, and exchange current intensity. Stability is characterized by changes in the overpotential or current over time.

Overpotential, the hydrogen evolution reaction can theoretically occur according to the standard hydrogen electrode potential. It starts at 0 V. However, due to losses on the catalyst/electrolyte surface, this value shifts to more negative potentials. The difference is defined as overpotential. When evaluating the performance of catalysts, 10 mA cm^{-2} to produce current density. The required overpotential values are compared. Alternating current density is defined as the current density at the equilibrium potential. The rate of electron transfer between the catalyst and the electrolyte gives information about. In other words, it is an indicator of the intrinsic activity of the catalyst. Tafel slope expresses the linear change of the logarithmic value of the current with the overpotential (η). The fact that the tafel slope is small means that the reaction takes place quickly. For example, the Tafel slope of platinum, the best-known catalyst for hydrogen formation reaction, is around 30 mV dec^{-1}. In addition, the Tafel slope is the reaction mechanism and the step that limits the reaction it gives information about you. It is obtained by fitting the Tafel curve to the Tafel equation given below.

$$\eta = b \log(j/j0) \ (1.10) \tag{17}$$

Here, η denotes the excess potential, b denotes the Tafel slope, j denotes the current density, and jo denotes the alternating current density.

Stability, High acidic or basic electrolytes with a pH of 0 or 12 are preferred in electrolysis cells. The stability test provides an estimate of whether the catalyst will be stable for HER of the structural and catalytic properties of the electrode. To characterize the electrode stability, the change of the potential over time can be measured at a current density of 10 mA cm^{-2} [284].

There are current reviews that discuss the structure, morphology, composition, active site of catalysts, and their correlation with activity and stability for HER and OER in both acidic and alkaline media. These studies are particularly driven by the growing need for active, stable, and low-cost electrocatalysts for efficient hydrogen production from electrocatalytic water decomposition. There are many challenging areas for the development of active, stable, and low-cost electrocatalysts. Firstly, most of the effective OER catalysts, such as Ir and Ru-based electrocatalysts, exhibit a high dissolution resistance in the acidic state, while most of the non-noble metal-based electrocatalysts cannot survive under these conditions. In this context, it is the development of stable and robust non-noble metal OER electrocatalysts with high activity and long-term stability performance in an acidic environment. Secondly, non-inert metal-based electrocatalysts such as carbides, phosphides, and chalcogenides attract great interest due to their high performance for OER in an alkaline environment, while catalysts undergo composition and structural transformation during OER conditions. Therefore, the identification of real active sites is still challenging, and the development of techniques for detecting real active sites that will guide the design and preparation of optimal catalysts is challenging. Thirdly, the knowledge about the catalytic mechanisms of many electrocatalysts, especially transition metal-based catalysts for HER in the alkaline state, is quite limited compared to HER in the acidic state. Therefore, determining the factors that govern the catalytic mechanism of HER in an alkaline environment is an important challenge.

Despite the many advantages of PEM electrolyzers, especially for OER catalysts that experience acidic and strong oxidative environments at the same time, it is hampered by the extremely exorbitant cost and excessive degradation of electrocatalysts under an acidic environment. To solve these problems, major research efforts have been directed towards reducing the use of noble metals and using noble metal-free catalysts, increasing the intrinsic catalytic performance and the durability of catalysts in acidic electrolytes. The limited operating time in alkaline water electrolysis leads to many start-up and shutdown cycles, which may exceed the maximum start-up/stop number defined by the manufacturer, and therefore may reduce the expected system life or warranty contracts. Basically, the electrodes are affected by repetitive start/stop behavior, and the electrode degradation is accelerated. Nickel electrodes are known to degrade significantly after 5000 to 10,000 start/stop cycles. This problem can be solved by the development of stable electrode compositions or self-repairing electrode surfaces. In addition, although nickel is the most widely used electrode material, it provides very high over potentials for oxygen and hydrogen formation reactions. Therefore, electrocatalytic materials are added to the electrodes. Iron is a cost-effective catalyst for the oxygen formation reaction. Molybdenum reduces the overvoltage for the formation of hydrogen at the cathode. Moreover, it causes high gas pollution due to operations at low current densities. This situation can be prevented by reducing the overall cell area (overloading) or dividing the system into several small blocks. The application of electrolyzers with smaller electrode fields also limits the maximum load compared to larger systems, while partial system operation is improved. Solid oxide electrolysis is also known as high temperature or vapor electrolysis due to the conversion of gaseous water into hydrogen and oxygen at temperatures between 700 and 900 °C. Theoretically, close to 100% stack efficiency is possible due to positive thermodynamic effects on power consumption at high temperatures. However, increasing thermal demand requires a suitable source of waste heat from the chemical, metallurgical or thermal power generation industry for economical operation. Moreover, the corrosive environment has some problems, such as requiring further material development [52].

There are two main types of OER electrocatalysts: noble metal-based electrocatalysts and non-noble metal-based electrocatalysts. For noble metal-based electrocatalysts, Ru and Ir-based catalysts are considered the most advanced electrocatalysts, especially for OER in acidic electrolytes, which have a greater dissolution resistance compared with other metals. To reduce the high price, improve the electrocatalyst activity, and stability, and even strengthen the dissolution resistance in an acidic environment, there are various strategies for designing and optimizing the catalyst composition, structure, and morphology. Besides Ir and Ru, other noble metals such as Rh, Au, Pt, and Pd-based catalysts have also been developed as two- or three-function electrocatalysts with promising performance for OER, HER, and oxygen reduction reaction (ORR). For non-noble metal-based catalysts, oxide and (oxy)hydroxide electrocatalysts abundant in the soil, studies are available for OER, especially for Ni-Fe based oxide and (oxy)hydroxide, some of which are the most common OER.

Noble metal and metal oxide electrocatalysts have long been recognized as the most powerful electrode materials in OER. RuO_2 and IrO_2, which are considered the most advanced electrocatalysts for OER, take part. However, for concerns such as the high cost and serious dissolution of RuO_2 and IRO_2, modification of catalysts has been carried out, making composition and structure/morphology optimization possible. Various strategies have been proposed to improve the electrocatalyst activity and stability and reduce the high cost [284]. Heteroatom doping for adjusting the composition of IrO_2-based OER electrocatalysts has aroused great interest. However, different guest atoms created different energy fields for the host system. **Su et al., 2018** Cu-doped RuO_2 with hollow porous polyhedral morphology consisting of ultra-small nanocrystals were prepared by thermal decomposition of Ru-modified metal-organic framework (MOF) derivatives. The catalyst has shown excellent stability in acidic electrolytes and good catalytic activity in OER [285].

Alloying Ru or Ir with other transition metals has been an intriguing method for designing OER catalysts that can effectively change the electronic structure and optimize the adsorption energy of reaction intermediates. Decoupling of Ru or Ir with other transition metals has been a key step in the development of OER catalysts. **Wang et al., 2019** designed a class of Ir-M (M=Ni, Co, Fe) catalysts with a unique network structure consisting of nonporous nanowires intertwined with a self-templating strategy. The results show a property related to the transition metal. Compared with Ir-Fe NPs and Ir-Co NPs, Ir-Ni NPs were confirmed by the results to exhibit the best OER activity with the lowest excess potential [286].

Surface structure modifications play an extremely important role in uncovering catalytically active regions and exploiting the interfacial effect. Morphology regulation as an aspect of surface structure modification is gaining more and more attention. For example, hollow nanoparticles such as nanocafes, nanocouples, and nanoframes have been proven effective for increased catalytic activity; these reflect their most open structures with increased catalytically active sites. According to **Park et al., 2017** demonstrated that an Ir-based multimetallic bilayer nanoscale (DNF) electrocatalyst can be created by a simple, one-step synthesis, and revealed that the IrNiCu DNF structure retains a rhombic dodecahedral morphology after strong acid etching. The IrNiCu catalyst for OER has been shown to exhibit excellent electrocatalytic activity and durability [287].

Non-noble metal-based OER electrocatalysts have attracted a significant amount of research interest due to their low cost and abundant supply. Efforts are increasingly being made to search for efficient, noble metal-free OER electrocatalysts. In recent years, significant progress has been made in demonstrating superior catalytic activity comparable to noble metal catalysts. **Zhang et al., 2016** prepared a gelled FeCoW oxyhydroxide (W, Fe doped CoOOH, G-FeCoW) with homogeneous metal distribution by sol-gel production. FeCoW oxyhydroxide has been observed to exhibit the lowest excess potential. This performance is superior to the performance of the reference Ni-Fe-based catalyst [288]. Most non-precious metal catalysts contain metal oxides and (oxy)hydroxides. Recently, many other promising electrocatalysts have been exhibiting excellent OER catalytic activities consisting of transition metal phosphides, sulfides, and selenides. However, these compounds exhibit unsatisfactory stability under high oxidative potential in alkaline solutions. Therefore, the understanding of the chemical structure of the actual active sites is of great interest in the development of catalysts related to OER. **Xu et al., 2016** synthesized nanostructured nickel iron diselenide ($Ni_x Fe_1 - Se_2$) has shown excellent OER activity in place of a highly active nickel-iron oxide catalyst [289].

In particular, the OER is the bottleneck in the water electrolysis process due to its slow kinetics, insufficient stability of some catalysts, and in alkaline electrolysis also additional ohmic losses from bubble separation. Electrodes for alkaline electrolyzers can be made of networks and expanded metals or metal foams as a substrate coated with different active catalysts such as nickel-, iron-, cobalt-based oxides, spinel-type oxides, or perovskites. In particular, the combination of nickel or nickel hydroxide and a second active catalyst (Fe, Co, Cr, Mn) improves the electrode performance, where iron shows the highest catalytic activity towards OER. **Koj et al., 2019** demonstrated that laser-structured iron-nickel electrode alloy improved electrolytic activity for oxygen generation in alkaline water electrolysis [290]. Recently, a new OER mechanism involving lattice oxygen species has been proposed, namely the lattice oxygen-mediated mechanism (LOM). In LOM, the lattice oxygen on the catalyst participates directly in the oxygen formation reaction. The participation of lattice oxygen can be considered an alternative reaction route and sometimes the optimal one for alloy catalysts in gas-phase catalytic oxidation reactions. **Mefford et al., 2016** study, a primary reaction pathway was proposed in which lattice oxygen participates in the OER reaction through the reversible formation of oxygen vacancies on the surface. A series of cobaltite perovskite structures are presented and the relationship between oxygen vacancies, metal-oxygen covalency, and OER activity is demonstrated. Therefore, the increased covalence between the metal-oxygen bond exhibited a higher vacancy concentration in the catalyst, which could be controlled by substituting lower valence Sr^{2+} into the $La_{1-x}Sr_xCoO_{3-\delta}$ (LSCO) structure. Modeling and experimental data results show a direct relationship between oxygen vacancies, oxygen diffusion rate, and OER activity [291].

3.9. Comparison of the performance of electrocatalysts in HER and OER

It is possible to use many materials such as noble metals, non-noble metal materials, metal carbides, and perovskites as electrocatalysts in the anode and cathode electrodes in the electrolysis of water for hydrogen production. Catalysts of especially Ni (nickel) and Pt (platinum) elements are encountered in both anode and cathode catalysts. There are many studies on nickel and nickel alloys. The AWE technology is compatible with non-noble metal catalyst electrodes. Transition metal-based catalysts incorporating cobalt and nickel are the most used compositions for anodic and cathodic electrodes, respectively (Table 8).

Xu et al. (2022) developed a highly efficient bifunctional electrocatalyst of 2D metal-organic framework (MOF)-derived NiCoP nanoflakes ($NiCo_{nf}$ -P) for overall water splitting in alkaline media. The developed electrocatalyst performance was studied by three-electrode cell assembly with linear sweep voltammetry (LSV) method at the potential cycling rate of 2 mV s−1 in a 1 M KOH electrolyte solution. The developed Nikon-P was shown better electrocatalytic activity with a lower onset potential of 37 mV and 1.435 V than commercial 5% Pt/C (43 mV) and IrO_2 (1.504 V) at the current density of 100 mV cm^{-2} towards the HER and OER. In addition, $NiCo_{nf}$ -P was shown lower overpotential of 119 mV and 315 mV than commercial 5% Pt/C (291 mV) and IrO_2 (400) to achieve the current density of 100 mV cm^{-2} towards the HER and OER [307]. In addition, the performance of the NiCo-P electrocatalyst HER and OER developed has also been studied in an alkaline electrolyzer; it shows excellent electrocatalytic activity with a lower cell voltage of 1.94 V at a working current density of 1 A cm^{-2} [284].

Hydrogen production on an industrial scale, especially through the electrolysis of water, is an important component in the transition to sustainable energy systems. The efficiency and stability of the electrodes used in electrolyzers are very important for cost-effective hydrogen production. Recent advances in electrode preparation techniques and materials have significantly improved the performance and durability of electrolyzers. Firstly, improvements in the electrode material are encountered. Interestingly, Nickel-based electrocatalysts along with non-metallic elements such as oxides, hydroxides, chalcogenides, and phosphides show great promise as a bifunctional electrocatalyst to simultaneously catalyze HER and OER in alkaline solutions owing to superior electron-enriched catalytic sites. These electrocatalysts; conductivity, excellent water dissociation capabilities and H^+ adsorption ability can significantly improve catalytic performance and stability. Sharma et al. demonstrate the manufacture of a highly flexible and efficient catalytic current collector by controlled current-free coating of Ni-metallic nanoparticles together with Ni_xP_y on a cotton-polyester fabric. Thus, an advanced approach to water and urea electrolysis for H_2 production on an industrial scale is shown, which addresses the challenges in energy and environmental sustainability [308]. Wang et al., developed a multi-element transition metal phosphide (V-CoP/Ni_2P/NF) self-supporting electrocatalyst via V-doping and interface engineering for highly efficient hydrogen evolution reaction. This work provided a facile strategy to rationally synthesize 3D porous heteroatom-doped metal phosphides as stable, effective, and pH universal catalysts for HER [309]. Joshi et al., efficient hybrid water separation and direct electro-oxidation of organic dye from wastewater using copper cobalt sulfide ($CuCo_2S_4$) nanolayers have been achieved. Significant water separation activity has been achieved by using urea and organic dye (methylene blue) as supporting electrolytes to prevent delayed OER kinetics. Thus, wastewater treatment can be realized through a circular economy strategy by combining the goals of environmental improvement (dye degradation) and renewable energy production (hydrogen evolution) [310]. The current state of knowledge in the industrial-scale production of hydrogen highlights important advances in electrode materials and preparation techniques. Precious metals such as platinum and iridium remain the gold standard for catalytic performance, but cost-effective alternatives such as nickel-based alloys, transition metal oxides, carbon-based and metal sulfur-based materials are increasingly being complemented or replaced. Moreover, electroplating techniques have recently become very important in the creation of high-performance, durable electrodes. Ongoing research is aimed at further improving the stability, durability, and cost-effectiveness of these materials to facilitate the widespread adoption of hydrogen production technologies [311].

Table 8

Comparison of HER, OER, and overall water splitting performance of NiCoP-based electrocatalysts in 1.0 M KOH alkaline electrolyte.

Catalyst	HER		OER			
	η mV@mA cm^{-2}	Tafel slope (mV dec^{-1})	η mV@mA cm^{-2}	Tafel slope (mV dec^{-1})	Overall Water Splitting (V@ mA cm^{-2})	References
s	138@10	124.1	270@10	65.7	1.59@10	[292]
Ti_3C_2@mNiCoP	127@10	103	237@10	104	1.57@10	[293]
CoNiP/NF	147 @ 10	51	234@10	47	1.62@10	[294]
NiCoP/CNF	130 @ 10	83	268@10	83	1.65@10	[295]
NiCoP NSA	133 @ 10	68.6	308@50	–	1.77@50	[296]
$Ni_{1-x}Co_xP$ films	82 @ 10	43	239@10	45	1.57@10	[297]
NiCoP	71 @10 149 @ 100	57	268@10	71	1.57@10	[298]
NiCoP	47 @ 10	64.1	238@10	96	1.52@10	[299]
Ni/NiCoP	90 @ 10	95	260@20	80	1.57@10	[300]
NiCoP/CC	82 @ 10	63	250@10	67	1.558@10	[301]
NiCoP/NF	78 @ 10	78	262@10	75	1.6@10	[302]
NiCoP/NF	32 @ 10	37	280@10	87	1.58@10 1.82@100 1.98@200	[303]
Nest-like NiCoP/CC	62 @ 10	68.2	242@10	64.2	1.52@10 1.77@100	[304]
N-NiCoP/NCF	78 @ 10	83.17	225@10	66.94	1.56@10 1.56@20	[305]
NiCoP@NC. NA/NF	37 @ 10	53.9	305@50	70.5	1.70@100 1.76@200	[306]

4. Electrode modification techniques

In addition to the effects of catalyst materials in hydrogen production, electrode preparation techniques are also important factors affecting cost, stability, and durability. The biggest obstacle to the mass production of electrolyzer cells lies in the high production costs. In particular, the manufacturing cost of electrocatalyst layers represents more than 50% of the total cost of cell stacks. In addition, if the water electrolysis method is used for hydrogen production, the importance of cost-effective electrocatalysts will increase even more. As a result, for the commercialization of electrolysis cells, it is necessary to implement a cost-effective and simple manufacturing process for the manufacture of electrocatalyst layers with excellent performance. An electrocatalyst layer is a composite material consisting of a catalyst material supported on a catalyst support. The main role of the catalyst material is the oxidation or reduction of gases or liquids injected into fuel cell assemblies. In addition, the catalyst fixed to the catalyst material must serve the purpose of conducting electrons. The performance of the electrocatalyst layer depends on the efficiency of the catalyst and the catalyst support [312]. For practical applications, it is common knowledge that an electrocatalyst should be applied as a coating on an electrode, and these catalysts should act as active catalysts for the hydrogen evolution reaction (HER) and oxygen evolution reaction (OER). Therefore, research on catalysts shows that the techniques used for immobilization in the construction of modified electrodes are important parameters that affect the activity of the catalyst [50,313]. To prepare catalyst-modified electrodes, several techniques such as cast film coating, dip coating, immobilization into polymeric matrixes, electrodeposition, physical adsorption, thermal evaporation, chemical bath deposition, spray pyrolysis, hydrothermal, Langmuir Blodget film coating and electrophoretic deposition can be used (Fig. 9) [175,314–317].

Physical vapor deposition (PVD) is one of the most prominent techniques for thin film deposition that includes the condensation of vaporized materials which are stemmed from solid/liquid sources onto various substrates. The most preferred PVD processes are evaporation and sputtering. The steps used in PVD can be entitled as (i) evaporation/sputtering of components to produce vapor phase, (ii) supersaturation of the vapor phase in an inert atmosphere to promote the condensation of metal particles, (iii) consolidation of the nanocomposite by thermal treatment under inert atmosphere. PVD has some advantages such as the usability of all organic and inorganic materials, yet its disadvantages cover the high cost of the process, low output yield, and complexity of the process [318].

Chemical vapor deposition (CVD) is a versatile method in which thin films of elemental and compound semiconductors as well as metal alloys are grown on a suitable substrate by chemically reacting a volatile compound of the material to be deposited with other gaseous reactants to produce a nonvolatile solid. CVD involves several well-established processes, which are metal-organic CVD (MOCVD), plasma-enhanced CVD (PECVD), low-pressure CVD (LPCVD), laser-assisted CVD (LACVD), aerosol-assisted CVD (AACVD). The type of process is selected based on the type of precursors and deposition conditions. CVD is different from PVD in terms of chemical interaction between a gas mixture and the material surface, which in turn, results in the chemical decomposition of gas constituents forming a solid film deposit. In CVD, the surface temperature is high, and the reaction occurs under an inert atmosphere using gas precursors. In the literature, CVD is generally used to form 2D materials with large areas, especially graphene. Hexagonal boron nitrides and metal dichalcogenides are also 29 produced and investigated by this technique. The disadvantages of the CVD technique are listed as the requirement of an appropriate substrate, and high levels of temperature and pressure limits. Furthermore, high production cost is another challenge [318].

Chemical bath deposition (CBD) encompasses different routes to construct thin films by immersing a substrate in a liquid solution bearing precursors. The reaction usually occurs at low temperatures (30-80 °C). Sulfide precursors used in CBD are generally thiourea, thioacetamide, and thiosulphate. This method is advantageous since it doesn't require sophisticated instrumentation or other expensive types of equipment. The most important parameters to consider are the interrelated effects of solution temperature, pH, and concentration of the precursors. There are various studies regarding the metal chalcogenide formation by CBD technique [318].

Spin coating is a common method to produce thin films of desired thickness which is based on spinning a solution of semiconductors dripped on the surface of a suitable substrate on a rotating disc. The thickness of the film is controlled by adjusting the concentration and viscosity of the solution or rotation speed. Solvent selection is another important issue to consider. In this method, aggregation of the particles can be a problem [318].

Considering the abundance of paints and varnishes included in spray devices that can be purchased in stores all over the world, the spray-

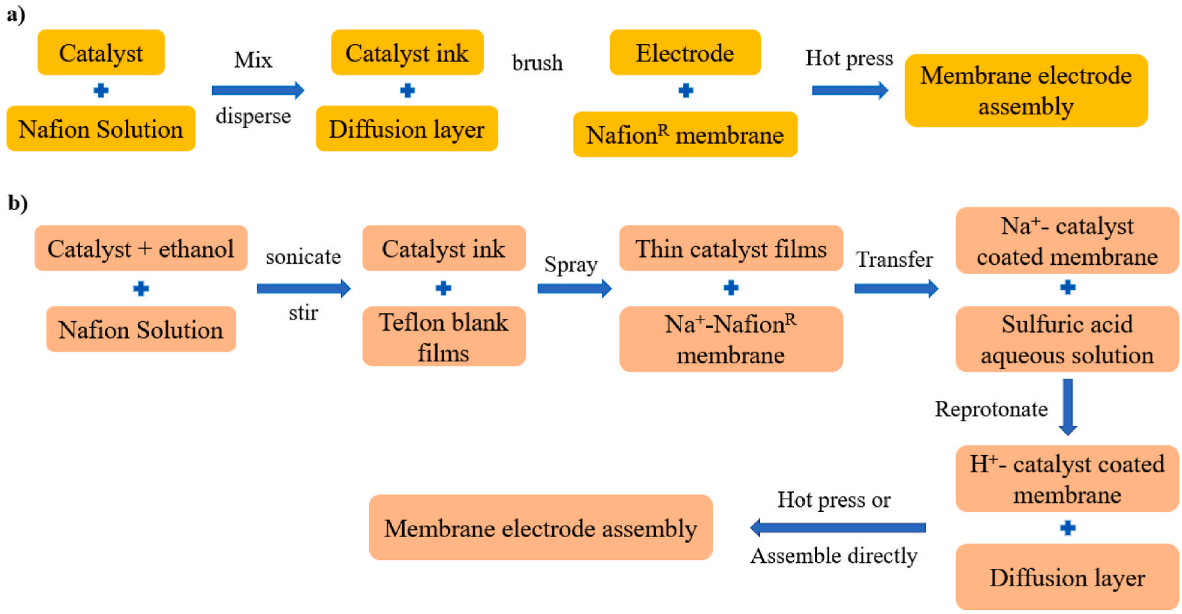


Fig. 9. Schematic of electrode preparation processes for both (a) the conventional method and (b) the decal transfer method [320].

coating technique may be one of the most useful for covering surfaces. There are also other variants of the procedure, including spray-coating with plasma, thermal spray, and powder, among others. The traditional spray-coating method is still the most often reported due to its low cost and lack of specialized equipment, though. Some equipment is connected to compressors for photocatalytic coating so that the solution exits at the desired speed and pressure. Similar to the dip-coating technique, the solution must be correctly prepared before being loaded into the spray [319]. In studies on photoelectric devices, the ultrasonic spray coating process (USCP) has proven appealing due to its high material efficiency, cheap manufacturing costs, and suitability to simplify production. By integrating an extra-low surface tension diluent and a surface tension control mechanism, a high-quality polymer anode buffer layer, and a tiny molecule emission layer are effectively achieved by USCP.

Blade coating offers the benefit of large-area homogeneity, little material waste, interlayer dissolution prevention, compatibility with roll-to-roll manufacturing, and more efficient use of active material while still allowing for the preparation of well-defined films. The blade coating method's quick-drying step prevents the traditional solvent annealing procedure from slowing down manufacturing throughput [319]. Roll-coating is a pre-metered coating that applies coating liquid to a substrate using a succession of rollers. The amount of coating material provided to the substrate is practically independent of the characteristics and structures of the fabric because a metered layer of the coating liquid is first generated on the roller surface before it is transferred to the substrate. Precise control is achievable and is mainly governed by the rheology of the fluid and the relative speed of two spinning surfaces. A single revolving roller is used in the most basic roll-coating setup. The roller's upper portion is in touch with the substrate, while its lower half is submerged in a coating liquid bath. A portion of the liquid film formed by the coating liquid on the roller surface is transferred from the roller surface to a substrate as the roller rotates. Hydrodynamics controls the quantity of coating that is applied to the substrate. The elements affecting coating thickness include substrate speed, roller rotation speed, and the rheological characteristics of the coating fluid. One roller serves as both a metering and an application device in this configuration. By adding additional rollers, more accurate control is made possible. A well-known and efficient method for making solar cells is roll-to-roll (R-2-R). Additionally, several film-forming techniques, including inkjet printing, vacuum deposition, doctor-blading, slot-die coating, and chemical vapor deposition, can be used in the production of R-2-R. Flexible substrates are required for the production of R-2-R [319].

In the dip coating method, a substrate is dipped into a bath with a precursor solution, remains inside for some time, and is then pulled up from the solution with a constant velocity. After the excess liquid from the substrate is drained, the solvent evaporates and forms a thin film. The parameters affecting the process are immersion time, number of dipping cycles, temperature, withdrawal speed, solution composition, etc. Dip coating is more appropriate to be applied in lab-scale applications like spin coating since it doesn't require any sophisticated equipment. The main drawbacks of this deposition technique are its inefficiency in terms of time and its ability to block the screen. Additionally, some materials can hardly be deposited using this technique [316].

4.1. Electrodeposition

Electrodes can be fabricated by several preparation methods, such as physical vapor deposition, chemical vapor deposition, chemical bath deposition, spin coating, dip coating, and electrodeposition [316]. Among all the methods, electrodeposition stands out as particularly noteworthy due to its economic advantages, ease of operation on large-area substrates, continuous processing capability, and the straightforward control it provides over morphology. Moreover, electrodeposition is a solution-based technique that can be conveniently conducted at low temperatures and exhibits a rapid growth rate [321, 322]. Electrochemical deposition, also known as electrodeposition, is a widely recognized and straightforward process for growing thin films. It entails the creation of a thin film on a substrate material through the electrochemical reduction of metal ions from an electrolyte. To facilitate this process, an electrical current is applied using a potentiostat. The electrode where thin film formation transpires is the cathode, where the reduction of cations from the target material within the electrolyte occurs [323]. A platinum foil or wire is employed as the counter electrode (anode) to complete the circuit by introducing electrons into the electrolyte (Fig. 10). At the atomic level, the electrodeposition process comprises a series of steps, including:

- ion transportation to the electrode surface,
- charge transfer processes,
- the incorporation of atoms into the crystal lattice.

Various electrodeposition modes exist, such as potentiostatic, galvanostatic, pulsed potential, and pulsed current, as well as cyclic voltammetry. In the potentiostatic mode, a constant direct current (DC) potential is applied, while the galvanostatic mode utilizes a constant current [324]. Cyclic voltammetry electrodeposition is among the most promising techniques for acquiring high-quality data concerning electrochemical processes and behaviors. It swiftly discloses the redox potentials of electro-active species and their respective positions [325]. The implementation of the cyclic voltammetric method is simplified, as it can be executed at room temperature, with controlled scan rates, within a specific potential window range, and with minimal reliance on additives in the electrolytic solution. Electrodeposition offers advantages by enabling the production of thin films with thicknesses ranging from several microns to several hundred microns. When compared to other deposition methods, electrodeposition is preferred due to its scalability, cost-effectiveness, and capability to coat large surface areas while maintaining precise control over composition [326,327].

Electrochemical deposition or electrodeposition is regarded as a simple and well-known process of film growth which involves the formation of a thin film onto a substrate material via the electrochemical reduction of metal ions from an electrolyte. For this process, electrical current is applied by a potentiostat. The electrode where thin film formation occurs is the cathode, where the reduction of cations of target material from an electrolyte takes place. A Pt foil or wire is used to serve as the counter electrode (anode), and it closes the circuit by injecting electrons into the electrolyte. On an atomic level, the electrodeposition

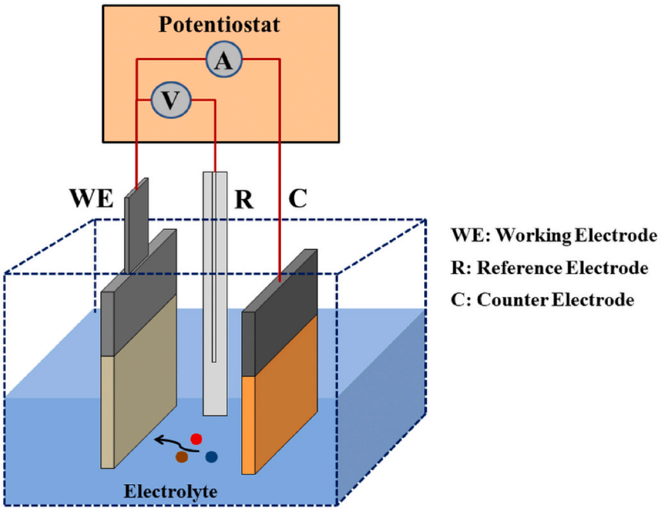


Fig. 10. A general representation of electrodeposition technique [328].

process includes a series of steps as follows: (i) ion transportation to the electrode surface, (ii) charge transfer processes, and (iii) atom incorporation into the crystal. There is a variety of electrodeposition modes: potentiostatic, galvanostatic, pulsed potential or pulsed current, and cyclic voltammetry. In potentiostatic mode, constant direct current (DC) potential is applied, whereas constant current is used in galvanostatic mode. The pulsed potential and pulsed current modes use non-DC signals. Cyclic voltammetry electrodeposition is one of the most promising electrodeposition methods for obtaining high-quality data on electrochemical processes and behaviors. It reveals the locations of the redox potentials of electro-active species instantly. Each of these modes requires different conditions. The galvanostatic mode requires high temperatures and pre-conditioning of the electrolytic bath by adjusting pH, concentrations, precursor concentration, and the use of complexing agents. Nevertheless, the deployment of the cyclic voltammetric method is easier, since it can be performed at room temperature within controlled scan rates in a potential window range without the need for too many additives in the electrolytic bath. Electrodeposition is beneficial since it allows the fabrication of thin films with thicknesses up to several hundred microns. However, this number is limited to only a few microns when the PVD or CVD technique is used. Diverse nanostructures such as nanorods, nanowires, nanotubes, nanosheets, and composite nanostructures can be formed by electrodeposition. In comparison to alternative deposition procedures, electrodeposition is favored because of its scalability, economy, and ability to cover a broad surface area while yet allowing precise composition control [316].

4.1.1. Electrophoretic deposition (EPD)

Electrophoretic deposition (EPD) is a versatile technique that attracts attention due to its ease of use and performance in depositing high-quality layers at room temperature. The principle of this technique is based on deposition from a fixed colloidal suspension, on a direct or alternating current. Using relatively simple and low-cost equipment, the EPD technique allows the deposition of layers with controlled microstructures at the nanoscale.

In comparison with other electrocatalyst layer deposition techniques such as drop-casting, the EPD technique offers clear advantages for the control of the thickness and packing density of the electrocatalyst layers. Moreover, due to the dense packing density, the electrocatalyst layers deposited by EPD can achieve increased conductivity and efficiency [312]. The biggest obstacle to the mass production of electrolyzer cells lies in the high production costs. In particular, the manufacturing cost of electrocatalyst layers represents more than 50% of the total cost of cell stacks. In addition, if the water electrolysis method is used for hydrogen production, the importance of cost-effective electrocatalysts will increase even more. As a result, for the commercialization of fuel cells, it is necessary to implement a cost-effective and simple manufacturing process for the manufacture of electrocatalyst layers with excellent performance. An electrocatalyst layer is a composite material consisting of a catalyst material supported on a catalyst support. The main role of the catalyst material is the oxidation or reduction of gases or liquids injected into fuel cell assemblies. In addition, the catalyst fixed to the catalyst material must serve the purpose of conducting electrons. The performance of the electrocatalyst layer depends on the efficiency of the catalyst and the catalyst support [312].

In comparison with other electrocatalyst layer deposition techniques such as drop-casting, the EPD technique offers clear advantages for the control of the thickness and packing density of the electrocatalyst layers. Moreover, due to the dense packing density, the electrocatalyst layers deposited by EPD can achieve increased conductivity and efficiency [312]. In 2004, Moriwaka et al. suggested employing electrophoresis for catalyst layer deposition. This technique harnessed surface charges on particles, offering versatility by applying them to various membrane materials. An additional benefit was the elimination of the conventional hot-pressing step, a common feature in many fabrication methods for membrane electrode assemblies (MEAs). Following the fine-tuning of the electrophoresis process for their specific membrane and catalyst blend, the researchers scrutinized the resulting catalyst layer [329]. In 2006, Munakata et al. investigated various factors influencing the electrophoretic deposition process, such as the catalyst mixture composition, including Nafion content, electric field intensity, and deposition time, with a focus on their impact on the performance of the membrane electrode assembly (MEA). Their efforts resulted in the attainment of a 76% platinum utilization rate [330]. The same research team conducted an in-depth examination of the membrane electrode assembly (MEA) and determined that electrophoresis led to a greater electrochemically active surface area compared to the decal method [331]. Yu et al. introduced a novel electrophoresis deposition technique that combines electrophoresis and electrodeposition. This method employs an electric field to regulate the size of platinum particles during electrodeposition. The researchers achieved precise control over the deposition of platinum nanoparticles on the electrode, maintaining a size range of 3–4 nm [332,333]. Adilbish et al. extended their exploration of this technique. They experimented with different duty cycles and identified that a 25% duty cycle (on a 1-min scale) produced the most favorable surface morphology for platinum clusters. Following the optimization of the duty cycle, they explored the impact of deposition time on platinum deposition on the electrode. They also examined how deposition time influenced the electrochemically active surface area. Their findings indicated an increase in hydrogen oxidation activity over time, but the electrochemically active surface area decreased after 10 min [334]. Felix et al. enhanced the electrophoresis process through a detailed examination of Nafion content, solution pH, and field strength. Single-cell testing revealed subpar performance, which was attributed to Nafion being an unsuitable ionomer for high-temperature proton exchange membrane fuel cells (PEMFCs) [335].

Recently, the electrophoretic deposition (EPD) technique has gained attention as an appealing method for fabricating electrodes [336,337]. Electrophoretic deposition presents an appealing method for generating thin films and coatings, eliminating the necessity for a binding agent [338–340]. Electrophoretic deposition stands out as a versatile and user-friendly technique, garnering attention for its ability to deposit high-quality layers efficiently. This method proves to be a valuable strategy for layer fabrication on conductive substrates at room temperature [312]. It operates by depositing charged particles from a stable colloidal suspension onto the substrate using either direct or alternating current, facilitating precise control over layer thickness [341–344]. The EPD technique, known for its cost-effectiveness and colloidal-based process, has found practical application in crafting functional layers for Li-ion batteries, supercapacitors, fuel cells, and Hydrogen production systems [345–347]. High-efficiency electrocatalysts can be produced through the process of electrophoretic deposition, specifically for applications involving alcohol oxidation, oxygen reduction, hydrogen evolution, and oxygen evolution reactions [312]. With the use of relatively simple and affordable equipment, EPD enables the deposition of layers with controlled microstructures at the nanoscale. This deposition process relies on several key control parameters, including thickness, time, solution composition, binders, surfactants, applied voltage, and current. The flexibility provided by these parameters makes EPD an appealing choice for fabricating high-performance electrocatalyst layers, a topic thoroughly explored in the following section [347–349]. Fig. 11 displays a schematic representation of the EPD process.

The application of the EPD technique in crafting the electrocatalyst layer allows for the direct placement of active particles onto conductive substrates. This process, in turn, provides control over both the conductivity and the packing density of the layers, crucial factors influencing the efficiency of electrocatalyst layers [351,352]. Additionally, the use of binder-free layers, created through the EPD technique, enhances the performance of electrocatalyst layers by ensuring that the entire layer is active and actively participates in electrochemical reactions [353,354]. The EPD technique is suitable for relatively brief processing durations due to its reliance on simple and cost-effective

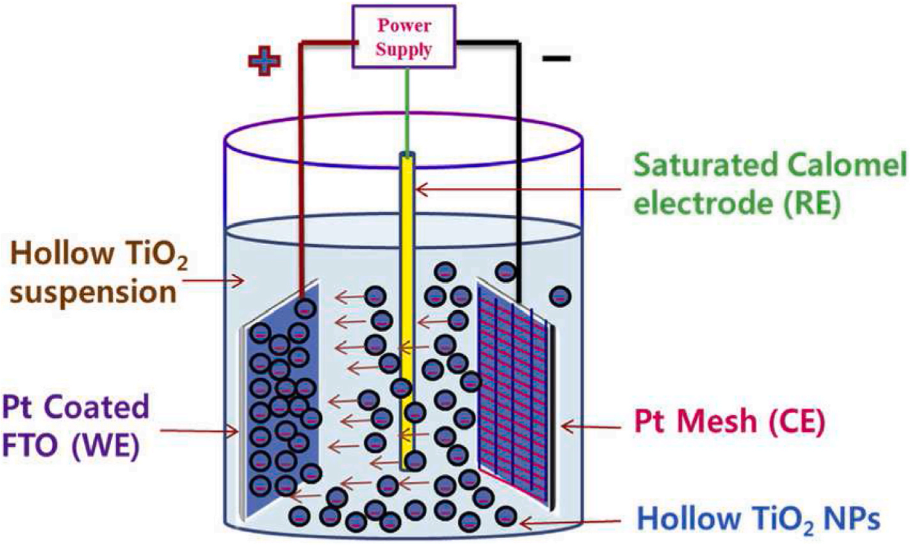


Fig. 11. A scheme of the electrophoretic process [350].

equipment. Beyond these advantages, the technique also offers the potential for scaling up to larger dimensions [349,355]. The EPD technique allows for the manipulation of specific nanostructures, such as materials based on graphene. These materials leverage distinctive electrical and mechanical properties, making them highly practical for the development of exceptionally efficient electrocatalyst layers [312]. Fig. 12 illustrates the layer structures deposited through both the EPD and drop-casting techniques.

Despite being introduced approximately two centuries ago, the mechanism of the EPD technique remains incompletely understood, primarily due to the multitude of parameters influencing the EPD process [347,349,356]. The EPD technique is a solution-based procedure that utilizes an appropriate electric field to convey charged particles in suspension and adhere them to conductive substrates. The deposition of these charged particles can be executed on any conductive substrate without restrictions on surface flatness or size. This process boasts a high deposition rate and ensures good uniformity of the layers [357–359]. Selecting an appropriate solvent, categorized as either aqueous or non-aqueous, significantly determines the attainment of a suitable particle dispersion for the layer [356,360]. Water is a well-established solvent in the EPD technique, featuring both advantages and disadvantages. The use of aqueous solutions in EPD necessitates low voltage. Water is highly favored for the EPD process due to its desirable attributes related to health, safety, environmental impact, and cost. However, a drawback arises from the electrolysis of water at elevated voltages, leading to the evolution of H_2 and O_2 at the EPD electrodes. The generation and trapping of H_2 gas at the cathode and O_2 gas at the anode pose challenges to the uniformity of the deposited layers [361–363].

Achieving successful development of suspensions for electrophoretic deposition relies on adopting a systematic approach to create

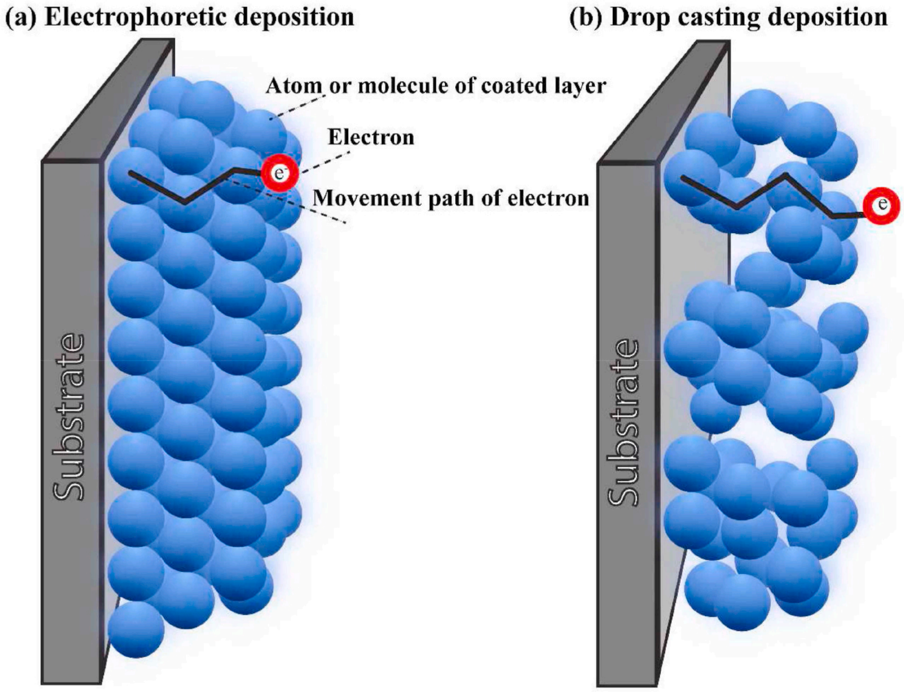


Fig. 12. Schemes of the layer morphology formed by EPD and drop-casting techniques [312].

suspensions with particles exhibiting a high zeta potential, while simultaneously maintaining low ionic conductivity. A prerequisite, though not the sole factor, for attaining a high zeta potential is a substantial surface charge. Particles in suspension will respond to the electric field only if they bear a charge. Four mechanisms have been identified for the development of charge on particles [364]:

- selective adsorption of ions onto the solid particle from the liquid,
- dissociation of ions from the solid phase into the liquid,
- adsorption or orientation of dipolar molecules at the particle surface,
- electron transfer between the solid and liquid phases resulting from differences in work function.

Recent studies have demonstrated that electrophoretic deposition (EPD) is a straightforward and effective method for preparing nanoparticles or nanocomposites. The traditional EPD process is fundamentally a two-step procedure comprising electrophoresis and deposition. Initially, the support and the metal particles in a liquid suspension are compelled to migrate toward the electrode under the influence of an electric field. Subsequently, the particles accumulate at the electrode, forming a cohesive deposit [365].

Table 9 provides a synopsis of electrophoretic deposition parameters documented in the literature, encompassing electrode materials, deposition time, deposition voltage, and electrode separation.

4.1.2. Examples of electrocatalysts fabricated by electrophoretic deposition

In the realm of energy storage, many studies on electrophoretic deposition focus on creating composite films involving active materials with rGO/GO, CNT/MWCNT (Fig. 13). This is attributed to their ability to be readily charged, forming a stable suspension owing to the abundance of surface functional groups [383–386]. EPD has proven to be an effective synthesis method for depositing highly adherent and porous NVP carbonaceous material, specifically carbon nanotube (CNT) and reduced graphene oxide (rGO), onto an aluminum current collector [337]. The electrophoretic deposition technique, based on suspension, has effectively been utilized to produce $Na_3V_2(PO_4)_3$ cathodes in combination with diverse carbonaceous materials. The NVP-carbon black electrode grown through EPD exhibits enhanced specific capacity, cycling stability, and rate capability, attributed to the uniform and porous microstructure of the deposited film. The introduction of rGO or CNT notably enhances the electrochemical properties of the NVP cathode [337].

Electrophoretic deposition is acknowledged as a straightforward and effective method for producing films of carbon nanotubes (CNT) and carbon nanofibers (CNF) that exhibit high homogeneity and suitable roughness [367,387–389]. The application of electrophoretic deposition on metal substrates has been investigated for CNFs. The resulting CNF deposition exhibits a structure dominated by mesopores and macropores, particularly advantageous for liquid catalytic reactions [390]. By using the electrophoretic deposition technique, a graphene oxide (GO) film was generated on fluorine-doped tin oxide (FTO) from a dispersed GO mixture. A glass cell with two electrodes was assembled using a cleaned FTO positive electrode and a stainless-steel negative electrode, maintaining a 1 cm distance between them. The cell was filled with a 4 mg mL^{-1} exfoliated GO solution, and a voltage of 10 V was applied across the electrodes. To achieve a uniform thin layer of GO nanosheets

Table 9
Summary of electrocatalysts fabricated at low temperatures under different EPD conditions [312,367].

Electrode properties	EPD Parameters							Reference
	Constant voltage	Deposition time	Distance between electrodes	Over potential	Tafel Slope	Stability	HER/ OER	
Stainless steel (1 × 1 × 0.2 cm^3)	5–50 V	0.5–10 min	20 mm	20 mV	–	Electrostatically stabilize	HER	[368]
$Cu(OH)_2$ and CuO nanobelts	10 V (DC)	5–60 s	1 cm	370 mV–500 mV	57 mV/dec	Excellent stability	OER	[369]
FeOOH and $Ni(OH)_2$ with Ni foam coating	–	5 min	1 cm	207 mV	70 mV/dec	Excellent activity and stability	OER	[370]
Carbon Fibre paper Electrodes (CFE) (2.25 × 2.25 × 0.6 cm^3)	~40 V	2 min	~5 mm	200–400 mV	30–180 mV/dec	Electrochemical stability	HER	[371]
Co_3O_4 nanoparticles	300 (DC)	30 s	3–6 mm	10 mV	96 mV/dec	Good stability	OER	[372]
$MoSe_2$ with carbon cloth coating	100 V (DC)	30 min	1 cm	282 mV	100 mV/dec	Good stability	HER	[373]
$MoSe_2$ with carbon cloth coating	100 V (DC)	60 min	1 cm	220 mV	76 mV/dec	Good stability	HER	[373]
$MoSe_2$ with carbon cloth coating	100 V (DC)	120 min	1 cm	321 mV	89 mV/dec	Good stability	HER	[373]
$MoSe_2$ with carbon cloth coating	100 V (DC)	180 min	1 cm	366 mV	102 mV/dec	Good stability	HER	[373]
Nonvertical MoS_2 NFs/FTO	8 V	4 min	1 cm	490 mV	143 mV/dec	Good stability	HER	[374]
Vertical MoS_2/FTO	20 V	4 min	1 cm	370 mV	102 mV/dec	Good stability	HER	[374]
Vertical MoS_2 QDs/NFs/FTO	20 V	4 min	1 cm	250 mV	74 mV/dec	Good stability	HER	[374]
Ni-Graphene sheets composite	10 V (DC)	30 min	1 cm	65 mV	54 mV/dec	Long-term stability	HER	[375]
CuSe with Ni foam coating	1.68 V	2 min	0.288 nm	297 mV–162 mV	89 mV/dec	Long-term stability	HER	[376]
Nitrogen-doped porous carbon/Co/CoP with carbon paper coating	160 V	5 min	0.24–0.28 nm	208 mV–350 mV	239 mV/dec	High electrocatalytic activity and stability	HER	[377]
$ZnCo_2O_4$ with Ni coating	10-30 (DC)	1–5 min	15 mm	700 mV	70–130 mV/dec	Mechanical stability	OER	[378]
Bulk $MoSe_2$	5 V	12 min	5 mm	340 mV	58.8 mV/dec	Excellent stability	HER	[379]
$MoSe_2$ nanosheets	5 V	17 min	5 mm	280 mV	53.8 mV/dec	Excellent stability	HER	[379]
$MoSe_2$-MoO_x	5 V	17 min	5 mm	210 mV	49.9 mV/dec	Excellent stability	HER	[379]
Coating of electrode with iron phosphide (FeP) nanoparticles deposited on macroporous carbon paper (CP)-at various nanoparticles concentrations	500 V DC	3.3–20 min	5 mm	38 mV	49.4–100.6 mV/dec	Long-term stability	HER	[380]
$BiSbSe_3$ nanoparticles	7.5 V	10 min	1 cm	72 mV	31.6 mV/dec	Excellent stability	HER	[381]
Cobalt, nickel and iron (Co-Ni-Fe) oxide on coatings on the AISI 304	20 V	30–60 s	1 cm	204 mV	52–59 mV/dec	Excellent and long-term stability	OER	[382]

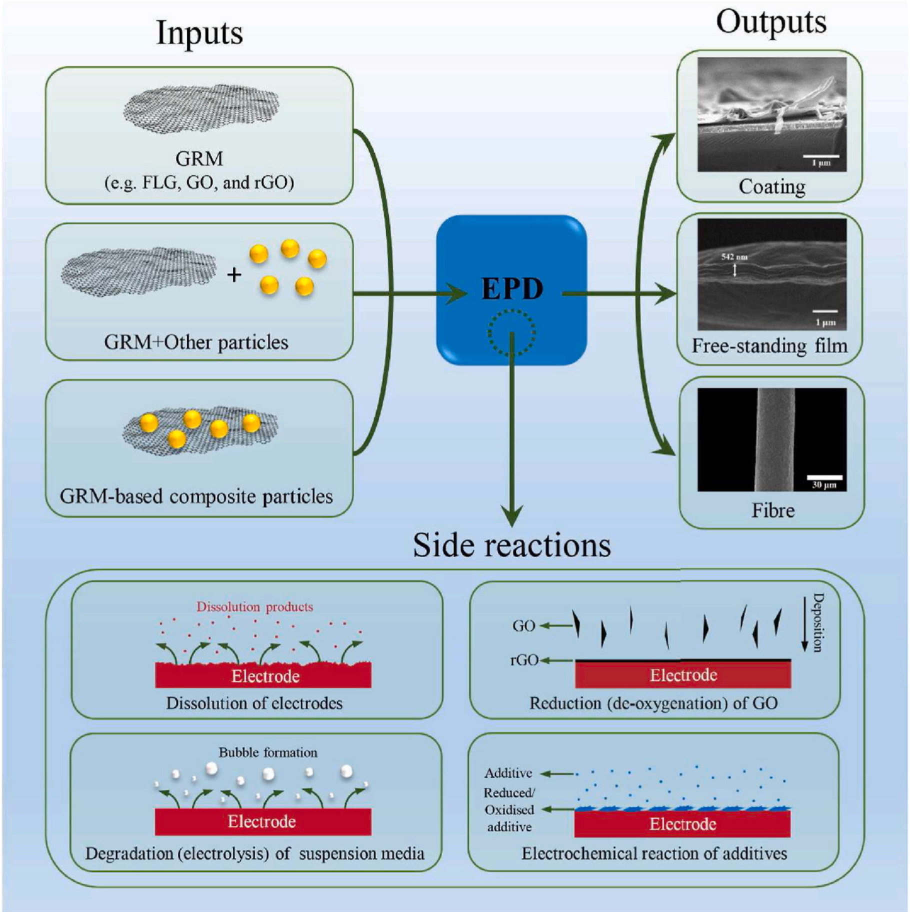


Fig. 13. An overview of inputs, outputs, and possible side reactions for the EPD of graphene-related materials (GRM) [366].

on the FTO surface, deposition occurred within 1 min. Following this, the electrode underwent drying at room temperature and was subsequently placed in a 60 °C oven for an hour. The color of the electrode transitioned from light brown to dark brown, indicating the pre-reduction of GO nanosheets [391].

Electrophoretic deposition utilizes an electric field to drive ions toward a conductive surface, enabling the creation of diverse MOFs with unique material characteristics. However, a drawback of this technique is the potential occurrence of defects during synthesis, impacting the MOFs' surface charge. Electrophoresis addresses these issues by guiding particles toward oppositely charged electrodes, depositing MOFs with negative charges on positively charged electrodes. Changing the electrode polarity results in deposition on the opposite electrode, affirming the role of charged surfaces in the deposition process. Although not directly applied to MOF synthesis, this approach underscores the impact of electric fields on the ionic components of the solution [392,393].

Utilizing the electrophoretic deposition method, thin films of HKUST-1 and ZIF-8 were effectively coated onto porous stainless steel. Both anodic and cathodic EPD methods were experimented with, revealing that cathodic EPD led to the deterioration of MOF crystals. This issue was circumvented by opting for anodic deposition. The EPD process for MOF crystals on porous stainless steel was conducted at 100 V for 2 min, and the resulting substrate was subsequently air-dried at room temperature [394].

An interesting technique for crafting MOF films is highlighted in the literature—electrophoretic deposition. This method enables the swift and straightforward fabrication of MOF films, eliminating the need for a specific substrate in the process [393,395–397]. A composite film of a metal−organic framework (MOF) functionalized with carbon quantum dots (C-QDs) has been synthesized using electrophoretic deposition. During the process of electrophoretic deposition, the composite can be deposited onto the positive electrode. The uncoordinated carboxylic groups within the structure can produce negative charges, enhancing the deposition of the film onto the positive electrode throughout the EPD process [398]. Using an electrophoretic deposition approach involves depositing colloidal particles into a material through the influence of a DC electric field in a stable suspension. This method facilitates the creation of films containing MOF@QDs [399].

The electrophoretic deposition technique proves to be a highly effective approach for creating functional thin films on a variety of substrates for diverse applications [400]. This method enables the organized assembly of charged particles from a colloidal suspension in a brief timeframe. For instance, the EPD process was executed in a single-cell EPD device using AC power in an organic solvent like acetonitrile to fabricate covalent organic framework (COF) films on the nonporous ITO glass electrode [401,402].

Nanosheets of covalent organic framework (COF) containing ethidium bromide units were synthesized through interfacial crystallization. Subsequently, these COF nanosheets were introduced into the synthesis process of zeolitic imidazolate framework-8 (ZIF-8) for electrically driven co-deposition. ZIF-8 nuclei were formed by coordinating Zn2+ ions with deprotonated ligands either in the cathode or solution. Positively charged COF nanosheets and ZIF-8 nuclei in solution were electrophoresed onto the cathode via EPD [403].

Electrophoretic deposition is a colloidal process where particles densely and compactly pack directly from a suspension under the influence of an electric field. This method offers advantages such as simplicity, rapid deposition rates, cost-effectiveness, uniformity, and the absence of substrate shape requirements. As a result, it finds widespread use in achieving the deposition of various metal oxides [338,404–407].

A comprehensive examination of factors affecting the electrophoretic deposition of metal oxides delves into both suspension-related parameters (zeta potential, solvent, particle concentration, and electrolyte conductivity) and operational variables (voltage, deposition time, and substrate conductivity). Under a constant applied voltage, the deposition rate diminishes over time due to the formation of an ion depletion zone near the deposition electrode. There exists a critical voltage threshold below which no deposition occurs, linked to the electrochemical reaction inherent in the deposition process. However, the evolution of bubbles associated with this reaction hinders the deposition of charged particles, leading to a decrease in deposition efficiency with increasing current density or applied voltage. In efforts to enhance deposit quality by mitigating bubble formation, researchers have explored various strategies, including the introduction of alternative half-reactions with sacrificial reactants, the use of porous substrates, and the application of modulated electric fields. Among these methods, modulated electric fields show particular promise for the formation of dense green bodies [404].

Investigating the electrophoretic deposition of copper and manganese oxides, followed by in-situ reactive sintering, as a potential method for Cu–Mn spinel coating in SOFC interconnects. The study explores the impact of electrophoretic deposition parameters—such as time, voltage, solid concentration, and dispersant content—on the quantity and quality of the deposition. Employing a DC power supply, electrophoretic deposition was executed on the AISI-430 surface to apply a green layer consisting of CuO and MnO_2. To maintain a 10 × 10 mm^2 active area during EPD, other exposed parts were covered with rubber bands. Adjusting the electrode distance to 10 mm and covering the rear sides facilitated a confined parallel electric field within a 10 × 10 × 10 mm^3 cubic volume between the electrodes. The study investigated various parameters, including deposition time (0–10 min), applied voltage (0–100 V), suspension solid concentration (0–20 g/L), and dispersant to solid w/w ratio (0–1:4), assessing their impact on the extent and quality of deposited layers, considering weight gain and physical appearance at both macro and microscopic scales. The electrophoretic deposition of Mn and Cu oxides on AISI-430, followed by an in-situ reactive sintering process for spinel synthesis, demonstrated promising results in preventing oxidation, spallation, and chromium poisoning [408]. Manufacturing processes such as electrophoretic deposition (EPD) have been documented as successful techniques for the fabrication of electrodes in nickel oxide capacitors. Among these methods, the EPD technique has emerged as a dependable approach for creating thin films of functional materials on diverse substrates [409–413].

Phthalocyanines with metal-based reduction processes have been identified as promising cathode electrocatalysts for enhancing the reaction kinetics of the HER, thanks to their higher activities [177]. By using electrophoretic deposition, the cathode electrocatalyst can be fabricated via electrophoretic deposition [414–416]. Despite the numerous organic species explored for device applications, metallo phthalocyanines (MPc) are noted for their poor solubility in commonly used solvents. Trifluoroacetic acid (TFAA), employed as a solution-processible technique, has been utilized to protonate the meso-bridging aza nitrogen atoms of MPc. This protonation facilitates the directed movement of molecules toward the negative electrode under a direct current (DC) electric field [417]. Building on this approach, an organic thin film composed of CuPc molecules was successfully fabricated using electrophoretic deposition [418]. Within a brief duration, a compact blue film of CuPc was generated on an ITO cathode by applying a voltage of 100 V cm^{-1} (104 V m^{-1}) between two plates. This process utilized a DCE solution comprising 1 mM CuPc and 3 M TFAA [416]. The template-free production of one-dimensional structures of CuPc molecules through electrophoretic deposition (EPD) holds promise as a method for achieving controlled organization of such molecular materials into highly ordered, low-dimensional assemblies. This approach offers the advantage of employing less intricate processing procedures [414]. Nanowires of erbium bis phthalocyanine ($HErPc_2$) were produced using the electrophoretic deposition method. This involved utilizing a mixed solution of chloroform and trifluoroacetic acid containing protonated $HErPc_2$. Aggregation of $HErPc_2$ molecules occurs on the ITO electrode as a result of their increased electrophoretic mobility in the solution [419]. The electrophoretic deposition (EPD) technique is investigated as a viable alternative method for preparing films of phthalocyanines, including nanowires and microcrystallites of $GdPc_2$. The morphology of the nano/microcrystallites is significantly influenced by factors such as deposition time, concentration in the mixed solvents, and applied voltage [420].

4.1.3. Stability and scalability of electrodes prepared by electrophoretic deposition (EPD)

EPD is a promising technique for electrode fabrication due to its versatility and control over deposition parameters. However, stability and scalability remain crucial aspects for real-world applications. Electrodes for energy storage are classically prepared in various ways in industry, such as dip coating or slurry casting. In these methods, the electrode materials are dispersed/dissolved in a solvent to form a viscous slurry, and a film is obtained after evaporation of the coating and solvent. Despite this, it is not easy to optimize thickness control or film assembly efficiency. Moreover, traditional techniques have significant disadvantages due to the lack of control over the effective distribution of nanoparticles in viscous slurry pastes, which leads to flocculation. This applies mainly to chemical and thermal methods, which are susceptible to mixing active materials with undesirable impurities, and therefore the electrode performance is reduced. Alternatively, the reported methods, such as physical and/or chemical vapor deposition (CVD), have high processing costs [421]. Given the above-mentioned approaches for producing effective electrode materials, it is imperative to find versatile and cost-effective processes that do not compromise electrode performance. In this regard, electrophoretic deposition (EPD) has several advantages, including scalability, easy and economical installation, as well as the ability to prepare electrode materials on relatively small-time scales. When the stability of the electrodes prepared with EPD is considered in terms of material properties, EPD allows the deposition of various materials, including metals, metal oxides and composites, providing good adhesion and uniform coatings. This strong adhesion is very important for the long-term stability of the electrodes. Looking at its chemical and electrochemical stability, the electrodes prepared by EPD generally exhibit good chemical stability in various pH environments, which makes them suitable for both acidic and alkaline electrolytes [422]. For example, nickel and metal oxide coatings have excellent resistance to corrosion in alkaline solutions [309]. In terms of mechanical stability, EPD, for coatings with controlled porosity and morphology, which improve the mechanical stability of the electrodes, the electrode material needs to adhere well to the substrate. Surface pretreatment and optimization of the suspension can improve adhesion [423]. EPD technology is industrially scalable and includes simple processing methodologies for manufacturing at the appropriate scale. EPD is a relatively simple process with minimal equipment requirements, potentially leading to lower production costs compared to complex techniques. Since EPD is a low-cost coating process, it allows uniform deposition on complex substrates with irregular morphology, making it suitable for large-scale electrode production. In addition, high-quality film coatings with the required application-specific morphology can be designed via EPD, the versatility of the process is emphasized by the fact that film thicknesses can vary from the nanoscale to the millimetre scale, creating a direct effect [422]. Despite the advantages mentioned above, EPD is mainly related to colloidal concentration, composition, electric field effects, pH, electrophoretic mobility, etc. it is affected by the time dependence of important EPD processing parameters such as, which seriously affects the quality of the deposited surface films/coatings. For example, scaling up requires precise control over deposition parameters such as electric field strength, deposition time, and suspension concentration. Moreover, while EPD stands out for small-scale production, cost-effectiveness for large-scale production becomes a cause for concern. Optimizing factors such as the reuse of the suspension and minimizing material waste is very important in terms of keeping costs manageable. Therefore, the focus should be on optimizing the interaction of complex factors affecting EPD for large-scale processes. Developing robust models that predict deposition behavior based on material properties and process parameters can significantly help optimize EPD for large-scale production [349]. Despite these stability and scalability concerns, EPD has disruptive potential in the large-scale production of high-performance electrodes for various energy storage and conversion technologies, such as batteries, supercapacitors, and fuel cells. With continued research focused on overcoming scalability barriers, EPD can close the gap between laboratory success and real-world production, paving the way for a more efficient and sustainable future [422]. Electrophoretic Deposition (EPD) offers a promising approach to produce electrodes for water splitting applications, with potential advantages in both economic and environmental sustainability compared to traditional methods. From the point of view of economic sustainability; it allows targeted accumulation by minimizing material waste compared to techniques such as slurry casting that require excess material. It also provides precise control over the deposition thickness, minimizing material waste compared to spraying or rough coating methods. In terms of environmental sustainability, while traditional methods are usually based on hard organic solvents, EPD can reduce the environmental impact by using water-based suspensions. Moreover, the low operating temperatures and the absence of toxic gas emissions make EPD a more environmentally friendly technology. It reduces the environmental impact by producing fewer emissions and dangerous by-products [422,424]. However, the use of aqueous media has interesting consequences from an environmental and economic point of view. The main problem with water as a suspension medium is that it is subjected to electrolysis at a low voltage value (+1.23 V), which causes the formation of hydrogen and oxygen bubbles on the surface of the electrodes and affects the uniformity of the deposited films. To avoid this problem, some strategies can be used, such as using pulsed direct current (DC) or alternating current (AC) methods [363]. EPD also offers a potentially more sustainable and cost-effective approach to the Manufacture of water separation electrodes compared to conventional methods, paving the way for the development of cleaner and more efficient hydrogen production. It offers a combination of economic and environmental benefits, making it a standout approach for sustainable water separation electrode production. Moreover, the precise control over the deposition parameters in the EPD allows the electrode properties to be adapted for optimal water separation performance. Research should also focus on further optimizing EPD processes to minimize energy consumption and waste production. Developing scalable and cost-effective EPD setups for large-scale electrode manufacturing is crucial for real-world applications. Research into the use of low-cost, earth-abundant materials in EPD suspensions can further improve economic and environmental sustainability. Overall, EPD research for water separation electrodes shows significant promise. By addressing scalability and cost challenges, EPD can become a key technology for sustainable hydrogen production and contribute to a cleaner energy future [422,425].

5. Conclusion

In this review, hydrogen production with water splitting and the electrolyzer cells used in electrolysis were summarized. Water electrolysis studies in the literature show that many different electrolyzers can be used to produce hydrogen from water. Each electrolyzer technology has significant advantages and disadvantages compared to the others. Depending on the intended use, the amount of hydrogen to be produced, and the available energy source, different electrolyzers stand out. It is seen that the most important component that determines the performance of electrolyzers is the electrocatalysts and membranes used in the electrodes. In the literature, a large number of electrocatalysts have been tested for both HER and OER processes. It is seen that the most important parameter determining the performance of the electrocatalysts is the activity and stability of the electrocatalyst. However, the process of coating the electrocatalyst on the electrode surface also has a significant effect on the performance of electrocatalysts. Among the many different electrode modifications, electrophoretic coating stands out especially due to its ability to form films in desired thickness without the use of binders and the need for high temperature and pressure under atmospheric conditions. Thus, electrophoretic deposition, which has attracted great attention, especially in the manufacture of advanced materials in recent years, has been discussed in detail in this review. Electrophoretic deposition garners interest for its simplicity and ability to effectively deposit high-quality layers at ambient temperature, making it a versatile technique. Low cost-effectiveness, relatively short processing time, easy modification, and simple equipment use allow the deposition of layers with controlled microstructures at the nanoscale. Moreover, electrophoretic deposition eliminates the need for binders to produce thin films and coatings and stands out as a user-friendly technique with the ability to efficiently deposit high-quality layers. In addition to these advantages, a disadvantage arises due to the electrolysis of water at high voltages, which leads to the formation of H_2 and O_2 in EPD electrodes. The production and capture of H_2 gas at the cathode and O_2 gas at the anode pose difficulties in terms of the uniformity of the deposited layers. However, the process needs a reasonable selection of solvent media, so that a significant surface charge size is developed on the dust surface in the suspension to ensure the stability of the suspension and at the same time facilitate high electrophoretic mobility.

Although the mechanisms are still completely unclear, the possibility of deposition on non-conducting substrates opens a whole new range of applications including gas separation sensors, thermal barrier coatings, etc. However, it requires further research in fundamental understanding of the mechanisms by appropriate modeling of the process, and charge development in non-aqueous suspension for proper control of suspension stability and deposition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

Rabia Turan: Writing – original draft, Investigation. **Elif Bilgen:** Visualization, Methodology, Investigation. **Atıf Koca:** Writing – review & editing, Supervision, Conceptualization.

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