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Electrochemical characterisation at laboratory scale of non-noble porous metal-based electrodes for hydrogen generation in acidic medium

CCMC will prepare and attach the official title page.

Contents

Foreword

This CEN Workshop Agreement (CWA 18302:2025) has been developed in accordance with the CEN-CENELEC Guide 29 “CEN/CENELEC Workshop Agreements – A rapid way to standardization” and with the relevant provisions of CEN/CENELEC Internal Regulations - Part 2. It was approved by the Workshop CEN “Electrochemical characterization at laboratory scale of non-noble porous metal-based electrodes for hydrogen generation in acidic medium”, the secretariat of which is held by “Spanish Association for Standardization, UNE” consisting of representatives of interested parties on 2025-10-29, the constitution of which was supported by CEN following the public call for participation made on 2025-04-14. However, this CEN Workshop Agreement does not necessarily include all relevant stakeholders.

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Introduction

Europe has set an ambitious target to achieve net-zero greenhouse gas emissions by 2050. As part of this transition, the role of hydrogen as a clean energy carrier is expected to grow significantly. One of the most viable methods for sustainable hydrogen production is water electrolysis. However, current acidic water electrolysis processes rely heavily on scarce and expensive platinum-group metals (PGMs), such as platinum (Pt), which poses economic and supply-chain challenges.

To address this issue, researchers are actively exploring non-noble metal alternatives that can function as efficient and durable electrocatalysts for the Hydrogen Evolution Reaction (HER). Transition metal alloys, particularly those based on nickel (Ni) and other iron-group (Fe-group) metals, have demonstrated promising catalytic activity, as predicted by the volcano plot.[1] This enhanced performance arises from their ability to balance hydrogen adsorption and desorption energies effectively. Nevertheless, catalytic activity alone is insufficient for real-world applications. The long-term durability of these materials under aggressive electrochemical conditions must also be assessed. Although numerous materials have been reported in the scientific literature as promising HER catalysts, only a small fraction proves viable for practical implementation. One of the major challenges in this field is the lack of standardized testing protocols, which makes it difficult to compare data from different research groups. Variations in experimental conditions can significantly influence reported catalytic activity and stability, particularly for non-noble materials in acidic media.

Additionally, testing PGM-based and non-noble metal-based HER catalysts requires different protocol considerations. PGM-based electrodes benefit from inherent stability, allowing for direct electrochemical evaluation. In contrast, non-noble metal-based catalysts susceptible to corrosion and dissolution in acidic media, require careful electrode preparation and stabilization strategies. Thus, defining an appropriate testing protocol depends on ensuring both accurate performance assessment and material stability under real electrochemical conditions.

Although harmonized EU protocols exist for testing final products (i.e. water electrolyzer cells [2]), there remains a critical gap at the laboratory scale. The rapid evaluation and direct comparison of emerging materials is severely hampered by the lack of dedicated, standardized lab testing protocols. Without these protocols, research laboratories struggle to benchmark and validate the performance of new materials consistently, leading to unreliable early-stage data and delays in decision-making. This gap not only slows the innovation cycle but also undermines confidence in the preliminary results, hindering the efficient transition of promising materials from the lab to large-scale production.

By providing clear guidelines, this document seeks to facilitate the reliable screening and fast evaluation of newly developed electrocatalysts, ensuring reproducibility and comparability across different research laboratories, while setting ground for further in-depth analysis of the underlying mechanisms, if required. This document has been developed in the frame of the project NICKEFFECT (Grant Agreement No. 101058076).

1 Scope

This CEN Workshop Agreement (CWA) aims to establish recommendations for the electrochemical characterization of non-noble, porous metal-based electrodes for hydrogen generation in acidic environments at the laboratory scale.

This document provides recommendations on the following key aspects:

- Electrochemical cell for catalyst testing.
- Definition of parameters for assessing catalytic activity and evaluating the durability of the catalysts.
- Analysis and representation of the electrochemical data obtained.

Regarding the types of non-noble materials covered by this methodology, there is no restriction on specific metal compositions. Any porous metal-based electrode with potential HER activity in acidic media falls within the scope of this document.

The document excludes the analysis of the HER mechanisms taking place on the surface of the electrode, however the data collected using the proposed protocol allow further in-depth analysis, if required. Additionally, the interpretation of the obtained electrochemical data remains out of scope of this document, as it often requires a complex approach including other physical and chemical characterization techniques.

The potential users of this document are:

— Researchers developing new non-noble metal-based catalysts for hydrogen evolution.

— Laboratories and experimental facilities conducting electrochemical testing of electrocatalysts.

— Developers of electrochemical cells and testing methodologies for HER materials.

— Research centres focused on complementary technologies for water electrolysis and hydrogen production.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purpose of this document, the following terms, definitions and abbreviations apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at http://www.iso.org/obp/

— IEC Electropedia: available at http://www.electropedia.org/

3.1

Hydrogen Evolution Reaction (HER)

Electrochemical process in which hydrogen gas (H_2) is generated by the reduction of protons (H^+) or water molecules at the electrode surface in an aqueous electrolyte under applied potential (or current)

3.2

non-noble catalyst

Catalyst composed of transition metals (e.g., nickel (Ni), iron (Fe), cobalt (Co), and their alloys) that does not belong to the platinum-group metals (PGMs) yet exhibits catalytic activity for the hydrogen evolution reaction (HER). These catalysts are explored as cost-effective and sustainable alternatives to noble metals like platinum (Pt)

3.3

electrocatalyst

Material that increases the rate of an electrochemical reaction by lowering the activation energy without being consumed in the process. In the context of HER, electrocatalysts improve the efficiency of hydrogen production by facilitating proton reduction at the electrode surface

3.4

porous electrode

Electrode characterized by a high surface area due to its intentional porous structure, which enhances the number of active catalytic sites available for electrochemical reactions

3.5

electrocatalyst durability

Ability of an electrocatalyst or electrode to maintain its performance over time under operating conditions without significant degradation. In HER testing, durability is assessed through long-term stability experiments, including chronopotentiometry and accelerated degradation tests

3.6

overpotential

Extra potential required beyond the thermodynamic equilibrium potential (0 V vs. reversible hydrogen electrode, RHE) to drive the HER at a specified current density. It accounts for kinetic, mass transport, and ohmic losses in the system and is a key indicator of catalyst efficiency. Lower overpotential values indicate a more efficient electrocatalyst

3.7

onset potential

Potential at which the HER begins to occur, typically defined as the voltage at which a small but measurable current density is detected. A more negative onset potential indicates that the catalyst requires greater energy input to initiate hydrogen production

3.8

ohmic drop

Parameter referred to the voltage loss across the electrolyte and the internal resistance of the porous electrode itself during an electrochemical reaction. This voltage loss is due to the resistance of the conducting materials (such as the electrode and electrolyte)

NOTE 1 to entry When the Ohmic drop is determined from impedance measurements (i.e., from the cut of the spectrum with the real axis in the Nyquist diagram) it also includes contact resistances and wire leads.

3.9

Open Circuit Potential (OCP)

Electrical potential of an electrochemical system (such as a metal in an electrolyte) when no external current flows through it. In open circuit conditions, the system is at equilibrium, meaning that the anodic and cathodic reactions naturally balance each other, and there is no net current

Note 1 to entry OCP (Open Circuit Potential) and OCV (Open Circuit Voltage) are often used interchangeably, but they have slightly different contexts depending on the field of study. OCP refers to the potential of the electrode, whereas OCV apparently relates to voltage of the entire electrochemical cell. OCV is used in cell analysis when no current flows through the cell and no potential difference is applied to the cell.

4 Description of the methodology for laboratory testing

4.1 Electrochemical Cell for Catalyst Testing

To ensure reliable and reproducible testing of electrocatalysts, an H-type electrochemical cell shall be used. A double jacketed cell shall be employed for temperature control when the tests are run at elevated temperatures. The cell consists of two compartments separated by a proton exchange membrane (e.g. Nafion212 membrane) to prevent crossover effects. The selection of electrodes shall include:

— Working electrode (WE): A porous non-noble metal-based electrode under investigation. It is recommended to employ the substrates comparable with characteristics of real electrolyzer electrodes.

— Counter electrode (CE): Typically a platinum wire or mesh with high surface area to ensure sufficient current flow.

NOTE 1 In the case the H-type cell cannot be used, Pt electrode is placed in a glass jacket to avoid the electrolyte poisoning, or Pt surface contamination with oxides of leached metals, see [3]. This applies to any material used as counter electrode.

NOTE 2 Other materials may be used as counter electrodes provided that they offer high electrical conductivity, have a surface area at least ten times that of the working electrode, and are inert to the experimental conditions. In this regard, carbon-based electrodes could be used as an alternative to platinum (e.g. carbon felt, graphite rod or graphite plate). Their use is, however, not encouraged for long-term stability tests in the conditions that are considered in this document, as carbon is not stable at the anode’s oxidising conditions in acidic media and will gradually decompose, affecting the obtained results because of changes in the total surface area and leading to accumulation of carbon particles in the anodic compartment.

— Reference electrode (RE): A standard hydrogen electrode (SHE) or a saturated calomel electrode (SCE) shall be used to provide a stable reference potential. The Luggin capilar, properly placed, shall be used to minimize the Ohmic drop due to the solution resistance.

— Electrolyte: For simulating the acidic conditions an aqueous solution of 0,5 M H_2SO_4 shall be employed.

— Gas management: All electrochemical measurements shall be conducted under a nitrogen (N_2) atmosphere to eliminate interference from dissolved oxygen. The gas management setup should include:

  - Cell Gas Purging: Continuous or pre-experimental purging of the electrolyte with high-purity N_2 to remove dissolved oxygen. Recommended flow 60 ml/min.
  - Holder Gas Environment: The working electrode holder should also be purged with N_2 to ensure minimal atmospheric contamination. Recommended flow 20 ml/min.

The size of the cell should be big enough to guarantee proper fitting of the electrodes and gas management system, and to ensure that the WE's active area remains fully covered with electrolyte throughout the experiment. As a reference, a volume of 250 ml per compartment and a WE size of 1 cm^2 are recommended. Significantly larger volumes of electrolyte will require longer deaerating periods in Step 1 to ensure that the electrolyte is properly purged, which implies that the WE stays exposed to the acidic environment for a longer time before starting the experiment, thus leading to surface modification prior to the experiment.

4.2 Definition of parameters for assessing the electrode performance

The sequence of the tests described below shall be applied to analyse the performance of electrodes. The testing is based on a certain combination of Linear Sweep Voltammetry (LSV) and Electrochemical Impedance Spectroscopy (EIS) that allows to obtain both the parameters required for assessment of activity, dynamic and constant load durability.

4.2.1 Step 1. Electrode stabilization

In this step the porous electrode under study is introduced in the cell. Being non-noble composition, long exposure at OCP conditions can lead to surface modification (e.g. dissolution, oxidation, cracking, etc.). Therefore, the conditioning shall be realized as follows:

- Temperature: 20 ± 1 °C, 65 ± 1 °C
- 5 min deaerating the cell at OCP conditions
- 5 min OCP recording

NOTE 1 The deaeration time may vary depending on the volume of electrolyte. If the configuration of the cell permits introducing the electrode after deaeration, gas purging time can be increased to 20 min.

4.2.2 Step 2. Electrochemical Impedance Spectroscopy (EIS)

After the porous electrode is stabilized, EIS shall be performed to assess the initial state of the surface. In this experiment, a small alternating voltage is applied over a range of frequencies, and the resulting current response is measured to determine the system's impedance. The impedance data is then analyzed using an equivalent electric circuit model, which provides insights into interfacial properties such as charge transfer resistance and double-layer capacitance. Additionally, fitting the EIS data allows for the determination of the Ohmic resistance, which is used to correct the LSV curves by accounting for voltage losses in the electrolyte. The EIS conditions should be defined as follows:

- Frequency range: 10 kHz to 1 Hz
- AC amplitude: 5 mV
- Applied potential: At OCP

NOTE 1 For more general guidelines on Electrochemical Impedance Spectroscopy testing see ISO 16773-1:2016 [4].

4.2.3 Step 3. Linear Sweep Voltammetry (LSV) — slow

This step is used to obtain data on catalytic activity of the porous electrodes and determine the onset potential and overpotential at a given current density. The testing conditions shall be the following:

- Potential range: 0 V to -0,4 V vs. RHE (Reversible Hydrogen Electrode)

- Scan rate: 1 mV/s
- Repetitions: 3

NOTE 1 The data obtained under these conditions can be also used for the kinetic study of the reaction (e.g. determination of Tafel slope).

NOTE 2 The potential range is recommended to minimize potential issues related to intensive H_2 bubbling which can cause electrode’s surface blockage and severe surface modification. For electrodes with low HER activity the range may be extended up to a maximum of -0,8 V vs RHE. The potential range selected in this step should also be applied to steps 4, 6 and 9.

4.2.4 Step 4. Linear Sweep Voltammetry (LSV) — fast

A series of LSV curves is recorded to stress the functionality of the porous electrode, and thus allow the assessment of the electrocatalyst durability under dynamic conditions. During the LSV test, the electrode potential is swept linearly while measuring the resulting current. By monitoring changes in current response and potential shifts over successive scans, this approach provides insights into electrode stability and potential degradation mechanisms. For this, the following LSV conditions shall be applied:

- Potential range: 0 V to -0,4 V vs. RHE (Reversible Hydrogen Electrode)
- Scan rate: 50 mV/s
- Repetitions: 200

4.2.5 Step 5. Electrochemical Impedance Spectroscopy (EIS)

An EIS spectrum shall be taken after the fast LSVs in order to capture the surface condition after the accelerated dynamic stress. The following EIS conditions should be applied:

- Frequency range: 10 kHz to 1 Hz
- AC amplitude: 5 mV
- Applied potential: At OCP

4.2.6 Step 6. Linear Sweep Voltammetry (LSV) — slow

This step is used to obtain data on catalytic activity of the porous electrodes after the accelerated stress testing and used to determine how the onset potential and overpotential at a given current density have changed after the stress test. The testing conditions shall be the following:

- Potential range: 0 V to -0,4 V vs. RHE (Reversible Hydrogen Electrode)
- Scan rate: 1 mV/s
- Repetitions: 3

NOTE 1 The data obtained under these conditions can be also used for the kinetic study of the reaction (e.g. determination of Tafel slope) and its evolution after the dynamic load test.

4.2.7 Step 7. Chronopotentiometry

This step is used to assess the electrocatalyst durability under constant load conditions. A constant current density of -10 mA/cm^2 (calculated considering the geometric area of the electrode) shall be applied for 168 h (7 days) monitoring the response in potential. It also recommended to take an aliquot of the liquid electrolyte medium each 24 h for measuring the concentration of leached metal ions to get the information needed for the evaluation of the metal leaching into the solution. The degradation rate should be calculated by fitting the obtained E vs. t curve.

NOTE 1 A constant potential corresponding to -10 mA/cm^2 registered in LSV (Step 3) can be applied instead of current density.

4.2.8 Step 8. Electrochemical Impedance Spectroscopy (EIS)

An EIS spectrum shall be taken after the chronopotentiometry test in order to capture the surface condition after the constant load application. The following EIS conditions should be applied:

- Frequency range: 10 kHz to 1 Hz
- AC amplitude: 5 mV
- Applied potential: At OCP

4.2.9 Step 9. Linear Sweep Voltammetry (LSV) — slow

This step is used to obtain data on catalytic activity of the porous electrodes after the chronopotentiometry test and used to determine how the onset potential and overpotential at a given current density have changed after the durability test. The testing conditions shall be the following:

- Potential range: 0 V to -0,4 V vs. RHE (Reversible Hydrogen Electrode)
- Scan rate: 1 mV/s
- Repetitions: 3

4.3 Data analysis and representation

After electrochemical measurements are performed, rigorous data analysis is essential for extracting accurate performance metrics of the electrocatalysts. The following subclauses describe the key parameters and the exact methods for their determination.

4.3.1 Ohmic drop

All the LSV curves shall be corrected for Ohmic drop before extracting the electrochemical parameters. The Ohmic drop is determined from the data obtained in Step 2 (4.2.2), and a 90 % correction is applied during manual data analysis. For this the next procedure shall be followed:

- Determine the solution resistance (R_s) using the high-frequency intercept in Electrochemical Impedance Spectroscopy (EIS)

- Calculate the corrected potential using the following equation (1):

$$V_{\mathrm{corrected}} = V_{\mathrm{measured}} - I \times 0{,}9 * R_{\mathrm{s}} \quad (1)$$

where

V_{measured} is the potential swept in LSV,

I is the corresponding current in LSVs,

R_{s} is the solution resistance determined from EIS.

In the optimized cell configuration, the Ohmic drop should be minimal.

NOTE 1 For further considerations regarding the ohmic drop correction see [5].

NOTE 2 It is recommended to represent the LSV curves after Ohmic drop correction along with the LSV curves without the Ohmic drop correction, to avoid masking the poor conductivity of the electrocatalyst electrode.

4.3.2 Current density calculation

Normalization of current, i.e. current density calculation based on the registered current, is a critical aspect of standardizing catalytic activity testing protocols that allows to compare the performance of different electrodes. Several normalization methods exist, each with its own advantages and limitations. These include:

- Geometric area normalization, where the recorded current is divided by the electrode's projected area.

- Electrochemically active surface area (ECSA) normalization, which accounts for the actual active sites available for reaction, typically measured using techniques like double-layer capacitance determination.

- Mass activity normalization, where current is divided by the mass of the deposited catalyst rather than the electrode area.

For porous electrodes, as a starting point, the geometric area normalization shall be performed. This is because, for practical applications in electrolyzers, the electrode's lateral size is what ultimately determines the device's performance, while the catalyst mass influences cost considerations. In the case of non-noble catalysts, the required loading is often higher than that of platinum-based electrodes. Porosity can be introduced without altering the electrode’s macroscopic dimensions, effectively increasing the active surface area and enhancing catalytic performance. While ECSA measurements can provide a more detailed assessment of active sites what is vital for understanding the intrinsic activity of the catalysts, normalizing by geometric area remains a practical and reproducible baseline for early-stage characterization. Thus, although advanced normalization techniques can offer deeper insights, geometric area normalization provides a straightforward and standardized approach for the preliminary evaluation of porous non-noble electrocatalysts.

4.3.3 Onset potential

The onset potential shall be determined at the threshold current density of -0,1 mA/cm^2, where the current density is normalized by geometric area of the electrode.

NOTE 1 The onset potential is inherently sensitive to the surface catalyst loading and the specific surface area of the catalytic material and must be reported along with the sample characteristics [6].

4.3.4 Overpotential for HER

After ohmic drop correction, the potential at which a current density of -10 mA/cm^2 is reached shall be determined using LSV data. The values from both Step 3 (4.2.3) and Step 6 (4.2.6) shall be reported.

NOTE 1 To ensure that electrochemical measurements on non-noble-metal hydrogen evolution reaction (HER) catalysts in an acidic medium can be reliably extrapolated to their performance in a single-cell electrolyzer, the testing protocol should be designed to mimic real operating conditions as closely as possible. For this, reporting overpotential at industrially relevant current densities, e.g., -200 mA/cm^2 to -500 mA/cm^2 can be recommended.

Annex A
(informative)

Application case

A.1 Evaluation of performance and durability of porous Ni-P electrocatalysts for hydrogen evolution reaction in acidic medium

The Ni-P layers have been deposited by electroless method onto the polyurethane foam (PUF) substrates to render a porous electrode that can efficiently catalyse the hydrogen evolution reaction when polarized negatively in a liquid acidic medium. The porosity in this electrode is governed by the porous nature of the substrate. Two alloy compositions have been targeted: Low P content alloy with 2 wt%P, herein denoted as Ni-LP, and high P content alloy with 12 wt.%P, herein denoted as Ni-HP. Heat treatment was also used to increase the activity of obtained electrodes.

A.1.1 Electrochemical set-up

The electrochemical tests have been performed employing a three-electrode cell with separated cathodic and anodic compartments, designed at CIDETEC, as shown in Figure A.1. The two compartments have been separated by a proton exchange Nafion membrane that guaranteed that no reaction products formed on the anode during the measurements will be incorporated onto the surface of the working electrode. The hydrogen reference electrode (RHE) was placed next to the cathode (analysed sample). Pt mesh was acting as an anode. The WE is 1 cm^2 (projected area) and each compartment has a volume of 250 ml.

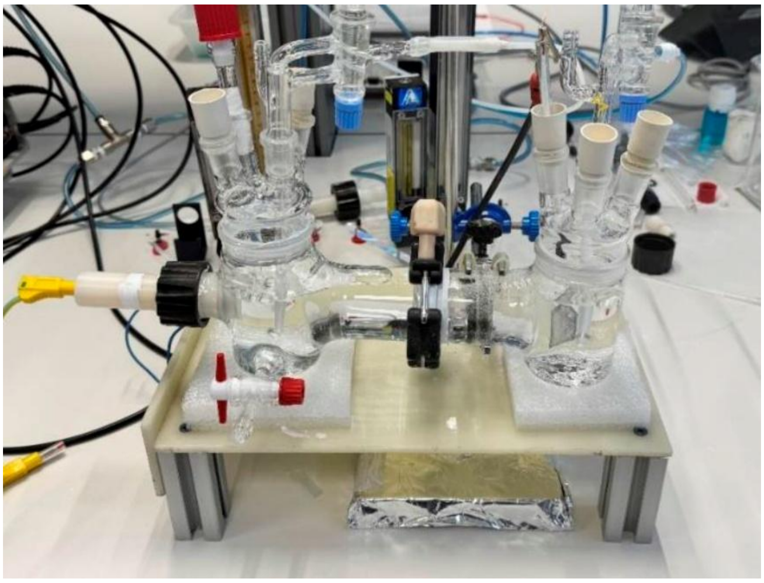
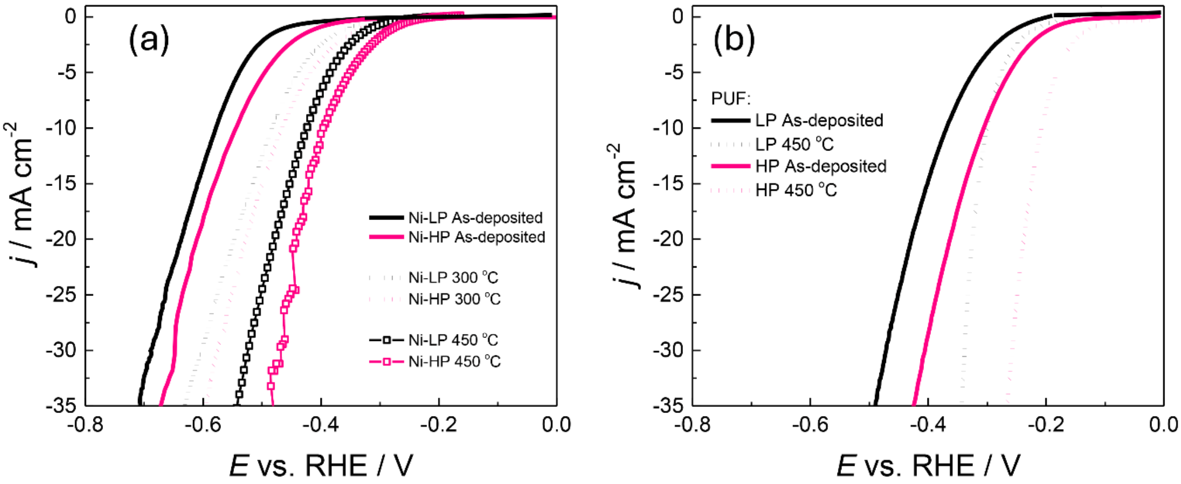


Figure A.1 — The two-compartment electrochemical cell, designed at CIDETEC, employed for performance testing and long-term stability tests

A.1.2 Evaluation of performance

Figure A.2(a) shows the LSV curves corresponding to Ni-LP and Ni-HP before and after heat treatment when deposited onto slat steel substrate (used for sake of comparison). It is important to mention that the curves were normalized to lateral area of the sample, considering both sides of PUFs. Additionally, all the curves were corrected for ohmic drop since its contribution was found to be essential to make accurate and meaningful comparisons. In their initial, as-prepared state, both compositions exhibit similar behaviour. However, the Ni-HP compositions demonstrate a slightly enhanced performance, a trend that remains consistent even when evaluating the effects of post-heat treatment. Additionally, it is notable that the heat treatment leads to a significant shift in the LSV curves, indicating an overall improved catalytic performance of the electrodes. Notably, the material displaying the highest catalytic activity on a flat substrate is the Ni-HP alloy heat-treated at 450°C. These results align with existing literature data, which support the observation on improved catalytic activity for Ni phosphides in hydrogen generation processes. Figure A.2(b) shows the LSVs obtained on the surface of porous Ni-P electrodes. The effect of heat treatment is the same, that is, higher temperature of heat treatment results in a higher activity of the electrodes.



Key

(a) LSV curves recorded on flat Ni-P samples before and after heat treatment at different temperatures

(b) LSV curves recorded on macroporous Ni-P before and after heat treatment at 450°C

The curves shown in (a) and (b) are corrected for IR and normalized by lateral sample area.

Figure A.2 — LSV curves recorded on Ni-P alloys in 0,5 M H_2SO_4

A.1.3 Evaluation of durability

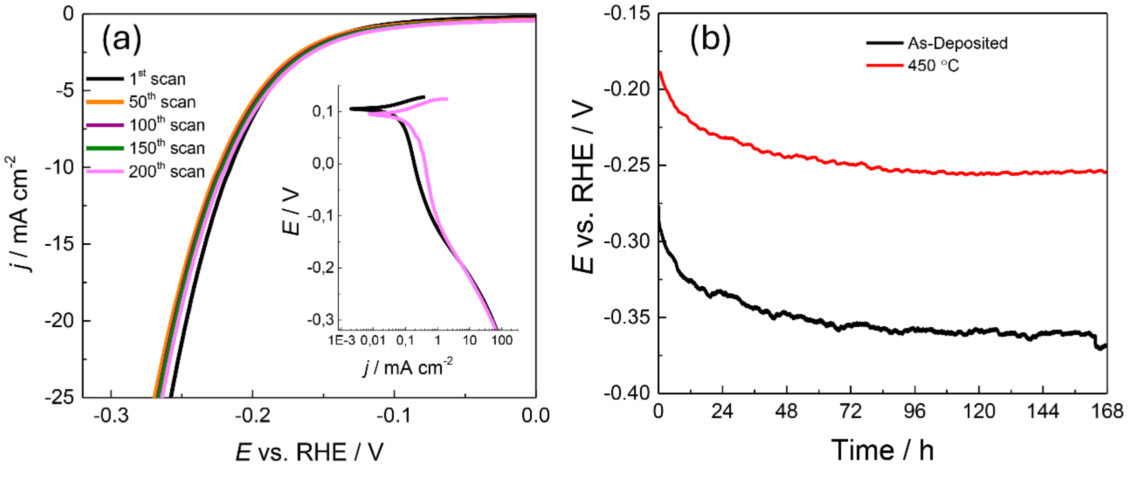
As Ni-HP alloys demonstrated a higher activity towards HER they were selected to assess the durability of Ni-P electrodes, two types of tests were performed:

(i) dynamic stability tests by running 200 LSVs at high sweep rate and monitoring the changes in overpotential and Tafel slopes, and

(ii) constant load testing, where a constant current of -10 mA/cm^2 was applied to the electrode and the response in potential was monitored for 168 h (7 days).

Ensuring that the products generated on the anode do not influence the cathode's performance is a critical aspect of these assessments. It has been demonstrated that Pt is not a suitable counter for HER testing due to anodic electrochemical dissolution-deposition process which leads to an apparent improvement in activity and stability of tested catalysts. Therefore, all stability measurements were carried out in a two-compartment cell, where the cathodic and anodic compartments were separated by an ion-conducting membrane. FigureA.3(a) illustrates the Ni-HP electrodes have excellent stability under dynamic conditions, as depicted by highly reproducible LSV curves (yet, small changes in Tafel slope up to 200 cycles can be expected). Furthermore, Figure A.3(b) illustrates the results of 168 h chronopotentiometry test on Ni-HP samples deposited onto PUF substrate (highest HER activity), in which a constant current density of -10 mA/cm^2 was applied. It is expected that the Ni-HP deposits possess enhanced corrosion resistance in acidic medium due to the development of a dense phosphate and hypophosphate passive film on the surface of the as-deposited films. However, the phase transformation resulting from the heat treatment carried out to enhance catalytic activity, significantly affects the material's corrosion stability. Remarkably, both as-deposited and heat treated electrodes show an excellent stability in 0,5M H_2SO_4 over 168 h under negative polarization. It is evident that some potential variation of about 50 mV occurred within the first 72 h of testing, while the potential tends to further stabilize over time. The fitting of the linear segment of the curve (corresponding to 72 h - 168 h) indicates that the changes in potential are in order of $3 \cdot 10^{-5}$ V/h for the Ni-HP/PUF-450°C electrode under tested conditions.

The comparison of the EIS data obtained at OCP conditions in an initial sample state and after completing 168 h demonstrates an increase in the radius of the semicircle in Nyquist plot, Figure A.3. The obtained results indicate that although the potential was maintained well over the test duration, the surface undergo changes. The nature of the changes is to be further understood employing post-characterization techniques such as, for instance scanning electron microscopy for observing the surface of the electrode after the test or inductively coupled plasma optical emission spectroscopy for measuring the concentration of Ni ions leached into the solution during the test.



Key

(a) Selected LSVs from cycling the Ni-HP/PUF-450°C electrode at 50 mV/s. The curves are normalized by lateral area of the sample and corrected for ohmic drop. The inset shows the 1st and the 200th cycle curves in semilogarithmic scale.

(b) Chronopotentiometry test of the Ni-HP/PUF-450°C electrode polarized at - 10 mA/cm^2. The data for the as-deposited Ni-HP/PUF are also shown for sake of comparison.

Figure A.3 — Stability of Ni-HP/PUF-450°C electrode catalysts in 0,5M H_2SO_4

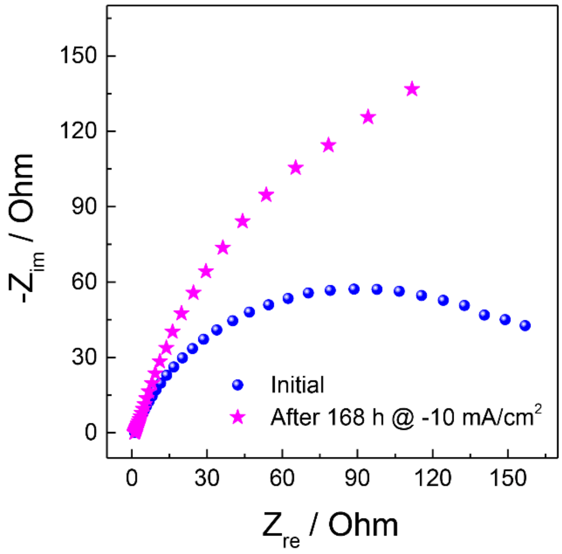


Figure A.4 — Nyquist plot recorded at OCP on the surface of Ni-HP/PUF-450°C electrode before and after negative current application

A.1.4 Summary of the extracted parameters

In the Table A.1 it is included a summary of the extracted parameters. The obtained results on activity and the durability indicate that the most promising electrode is the Ni-HP/PUF-450°C electrode which exhibits the onset potential of 100 mV and the overpotential for HER of 205 mV at -10 mA/cm^2 in acidic medium.

Table A.1 — Onset potential and variation in overpotential extracted from the LSV curves recorded on various Ni-P electrodes

Electrode	Onset potential (mV)	Overpotential at -10 mA/cm^2 (mV)	
		Initial	After 200 cycles
Ni-LP	319	580	—
Ni-LP_300°C	270	483	—
Ni-LP_450°C	240	420	—
Ni-HP	270	540	536
Ni-HP_300°C	270	460	455
Ni-LP_450°C	210	390	402
PUF_Ni-LP	205	370	—
PUF_Ni-LP_450°C	150	300	—
PUF_Ni-HP	97	305	307
PUF_Ni-HP_450°C	100	205	210

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