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Benchmarking performance: A round-robin testing for liquid alkaline electrolysis

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ABSTRACT

Liquid alkaline water electrolysis has gained considerable interest in recent years due to its promising role in an energy sector based on renewable energy sources. Its main advantage is the low investment cost of industrial alkaline water electrolyzers compared to other electrolysis technologies. A challenge remains in developing cost-efficient materials, stable in corrosive electrolytes, and offering competitive cell performance. Although there are many publications in liquid alkaline electrolysis, there is insufficient standardization of experimental conditions and procedures, reference materials, and hardware. As a result, comparability and reproducibility suffer, significantly slowing down research progress. This manuscript presents the initial efforts towards the development of such reference hardware and procedures within the framework of Task 30 Electrolysis in the Technology Collaboration Programme on Advanced Fuel Cells (AFC TCP) of the International Energy Agency (IEA). For this purpose, a homogenized setup including the electrolysis cell, functional materials, experimental conditions, and a test protocol was developed. The protocol and hardware were tested simultaneously at eleven different institutions in Europe and North America. To evaluate the success of this approach, polarization and run-in data were collected and analyzed for comparison, and performance differences were calculated. Significant disparities between the laboratories were observed and some key influence factors were identified: iron content in the electrolyte resulted to be a main source of deviation between experiments, along with temperature control and the conditioning of the cells. The results suggest that additional attention to detailed experimental conditions should be paid to obtain meaningful performance data in future research.

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1. Introduction

Hydrogen is considered a valuable fuel and chemical feedstock due to its high energy density, storage capacity, its role in the production of hydrocarbons, and its utilization in various industrial applications [1–3]. It is considered a crucial component in the shift towards a carbon-neutral society. The production of hydrogen through electrolysis with renewable electricity does not result in any carbon emissions. However, a significant challenge for the widespread use of green hydrogen is the high cost of production compared to extracting fossil hydrocarbons [4–6]. As a result, many companies, governments, and research institutions are conducting or funding research to improve water electrolysis technologies.

Low-temperature liquid alkaline water electrolysis (LAWE) has been an established technology for producing hydrogen via water electrolysis since the beginning of the last century and is used on a large scale [7–9]. The main advantage of LAWE compared to the other water electrolysis technology that has reached industrial viability, the proton exchange membrane water electrolysis (PEMWE), is the lack of necessary rare and expensive materials. Due to this, the investment costs of LAWE are generally lower than those of PEMWE plants and the scale-up of stack production will face fewer material bottlenecks [4,10]. At the same time, LAWE suffers from several drawbacks: it typically operates at lower current densities and many materials are not stable long-term in the concentrated alkaline electrolyte (e.g. KOH, NaOH). For this reason, there is significant research activity in different aspects such as cell components, electrode materials, geometry, and operation [11]. There are many different approaches to increasing cell efficiency, such as the application of highly active catalyst layers [12–16], nano-structuring to increase surface areas [17,18], reduction of the separator resistance between the two half cells [19,20], or improved cell designs [21,22]. One of the primary challenges for the translation of this research into tangible advancements arises from the diverse range of factors that can influence the overall cell performance, including electrode materials, electrolyte concentration, operating conditions, and system configurations [23–26]. As a result, experimental results obtained from different research groups may vary widely, making direct comparisons difficult. An example of this is the development of electrodes, which are usually measured for short periods at low KOH concentrations and room temperatures without verifying the performance and stability at more realistic operating conditions [27,28].

The lack of standardized testing protocols exacerbates the challenge of evaluating and comparing test results further and leads to discrepancies in reported performance metrics, hindering the ability to draw meaningful conclusions and identify optimal materials, systems, and operating conditions. In other industrial electrochemical systems, such as car batteries, there are internationally agreed testing norms that can be used as a baseline for experimental research [29]. In Alkaline water electrolysis research, while there are efforts to define some baseline operating conditions [30,31] and the standardization of catalyst testing [32,33], there are no universally agreed reference test cells, materials, or procedures. This study aims to create a simple and efficient round-robin experiment and assess its feasibility across different institutions. The round-robin method involves each participating institution conducting the same experiments in its laboratory, allowing for the practical evaluation of a standardization protocol. One advantage of conducting a round-robin at multiple, dispersed institutions is that it includes typical differences in laboratory setups, the impact of different experimenters, and often overlooked location factors, such as elevation, on the results. Variations in results can be attributed to specific inconsistencies between setups or in the execution of the tests, providing insight into the shortcomings and successes of the comparative experiments. A round-robin standardization initiative was also carried out for PEMWE, resulting in the successful development of a detailed benchmarking guideline for other researchers and the identification of previously underreported influences on the performance of PEMWE cells [34,35]. While the results presented here indicate that the protocol used in this study could be further improved, the authors are confident that these findings are already significantly valuable for advancing experimental methodology within the community, and can serve as an accessible benchmark for other research institutions to compare against.

2. Methodology

Eleven institutions in seven countries participated in this study, demonstrating both the interest of researchers in this endeavor and providing a robust data foundation for identifying the successes and challenges of the protocol. To conduct the experiments, we defined a universal cell design based on previous benchmarking efforts published by Karacan et al. in Ref. [26]. We chose to conduct a full-flow cell test under industrial conditions to ensure the transferability of results to industrial applications. Cell hardware, cell components, and testing protocol were harmonized, whereas balance of plant hardware, which is more difficult to adapt, was different from lab to lab.

Commonly used materials can be determined from other publications in the field of LAWE: nickel or platinum are the most common state-of-the-art electrode materials [23,36,37] and the commercially available Zirfon™ by Belgian manufacturer Agfa is the standard of separators in published research [19,38,39]. Concentrations of KOH and temperatures were selected to align with large scale technical applications [24]. All participants adhered to standardized electrode and separator materials, minimum setup requirements, and test procedures. The cells were then characterized and compared based on the measured polarization curves and conditioning behavior. The measurements were performed at all institutions during a timeframe of nine months. Results were collected and analyzed at Clausthal University of Technology (TUC) and the National Renewable Energy Laboratory (NREL). Based on the results, an effort was made to connect the deviations between laboratories to differences in setups and procedures.

3. Experimental setup

3.1. Hardware

A first benchmarking attempt was conducted and published at Forschungszentrum Jülich (FZJ) using an alkaline single cell designed in-house [26]. Therefore, it was decided to use the same cell for this study to build on the existing operational experience. FZJ provided complete sets of the 5 cm^2 cell hardware to each participating laboratory, this ensured that all laboratories could conduct testing promptly using identical cell designs. This included the backplates, nickel flow fields, PTFE sealing gaskets, centering pins and isolation foil. The cell hardware had a typical research and development single-cell design with thick endplates for compression using 8 bolts that were tightened crosswise in steps of 3, 5 and 10 Nm. The anode and cathode utilized nickel monopolar plates (MPPs) with a meandering flow field as seen in Fig. 1a. The drawings for the cell designs will be made public by FZJ.

Pure nickel foam from Alantum Europe GmbH with a thickness of 1.6 mm and an average pore size of 450 μm was used as anodic and cathodic electrode material [40–42]. The foam was compressed by 25%, which was controlled by the thickness of the PTFE gaskets of 1.2 mm per half-cell and 0.4 mm around the separator and which also sealed the cell. The commercial material Zirfon™ Perl UTP 500 by Agfa was used as the separator between the two half cells [43]. To ensure that there were no influences from the production batch or treatment before the experiments, the separator (courtesy of TUC) and foam (courtesy of Fraunhofer Institute of Manufacturing Technology and Advanced Materials (IFAM)) were sent out from the same source to all participants with detailed storage and handling instructions. In contrast to the preceding study conducted at FZJ [26], no pretreatment of the foam was performed.

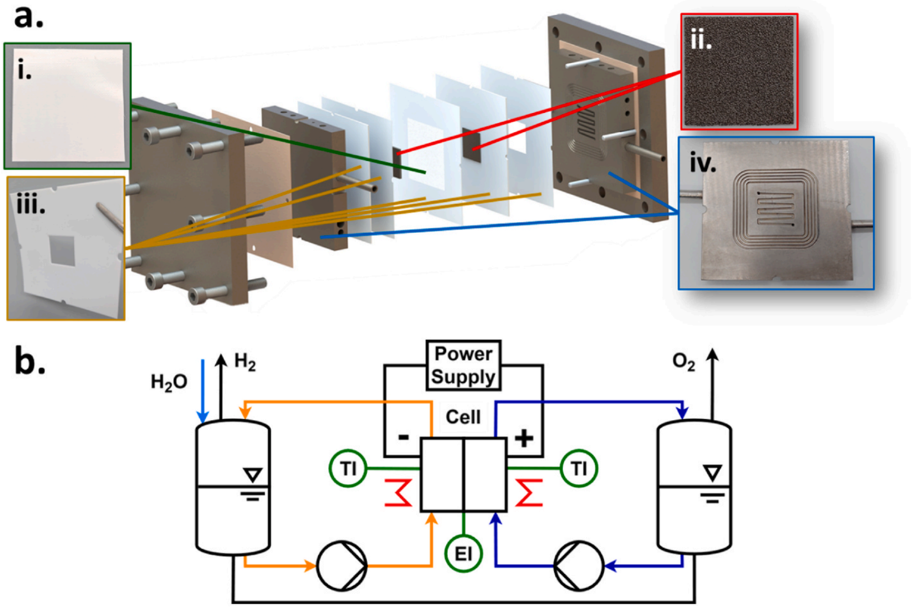

Figure 1. a.: Schematic of the test cell designed by FZJ. Highlighted components are i. separator, ii. electrode foams, iii. PTFE gaskets and iv. nickel flow fields. b.: Schematic diagram of the minimum test station requirements set for the round-robin tests, including separate tanks with a balancing line, pumps, and heating.

3.2. Station requirements and operating procedures

The experimentally relevant test conditions, such as assembly torque, electrolyte flow, temperature, and test protocols were decided upon, written down and distributed to the institutions to avoid possible misinterpretation. The measurements were performed at each institution using either commercial or built-in-house test stations. Minimal test station requirements were defined for the round-robin experiments to establish straightforward guidelines that could be consistently adhered to by all participants while controlling the most significant influences on cell performance, a diagram of which is presented in Fig. 1b. It includes two KOH tanks with a balancing line, pumps, temperature control, an electrical power supply, and a cell potential measurement. Since most participants used already existing setups for the round robin, differences between the setups remained: the type of pumps, power supply, sensors, electrolyte source, amount of electrolyte, water refill, and position of heating were individual for all participants. The legends in the figures will only identify the laboratories as Lab 1 through Lab 11 to improve readability.

The operating conditions utilized during the round-robin experiments are summarized in Table 1, and were chosen to reflect technical scale and industrial systems, ensuring that laboratory results are as transferable as possible [24]. As such, the experiments were conducted at a temperature of 80 °C and ambient pressures at each lab location. Some setups did not conform to the defined conditions completely, which is reflected in the actual condition variability given in Table 1. The cells were heated by either setting temperature points for the KOH reservoirs, the cell inlet, and/or the cell body, with variations between institutions. After a cell heat-up and equilibration time of 30 min, a galvanostatic step-wise polarization curve was recorded in defined steps from 1 mA cm^{-2} to 2000 mA cm^{-2} and back to 1 mA cm^{-2} with 1 min of holding time for each step. To evaluate the polarization curves, the values at the end of the holding time are compared. Next, conditioning with a constant cell voltage of 2 V for 24 h was conducted. In addition to ensuring stable cell conditions for the evaluation, conditioning has been proven advantageous in reducing the variability of cell performance during repeated experiments [25,26,34]. After the conditioning, another polarization curve was performed in the same manner as described previously. For the evaluation of results, this second polarization curve is taken as a measure of cell performance and compared between experiments.

4. Results & discussion

The following section presents the results obtained from the cell hardware that was distributed to the eleven different laboratories. Fig. 2a presents the averaged performance curves measured at 80 °C after the activation procedure at the eleven participating laboratories. Significant deviations are observed in the experiments, with current densities at 2 V ranging from 110 to 800 mA cm^{-2}, and cell voltages at 1 A cm^{-2} mA between 2.08 V and 2.69 V, corresponding to 610 mV of maximum deviation.

Fig. 2b presents the average results and maximum deviations for labs that conducted multiple tests. The repeatability within a single lab was found to be significantly better than between different labs, with absolute ranges in deviations between experiments spanning from 51 mV (Lab 7) to 150 mV (Lab 9) at 1 A cm^{-2}. This indicates that the majority of discrepancies can be attributed to differences in setups, electrolyte

Table 1
Operating conditions with planned and actual maximum deviations in setups during round-robin tests.

Operating condition	Chosen value
Temperature	80 ± 2 °C (actual + 2/ - 10 °C)
Pressure	Ambient (≈1 bar(a))
Electrolyte concentration	30 ± 2 wt% KOH in H_2O
Liquid flow per half cell	50 ± 5 ml min^{-1} (actual ± 10 ml min^{-1})
Voltage limit during cell operation	3 V
Polarization steps	1; 2; 3; 5; 10; 15; 20; 30; 50; 70; 100; 150; 200; 250; 300; 400; 500; 600; 800; 1000; 1500; 2000 mA cm^{-2}

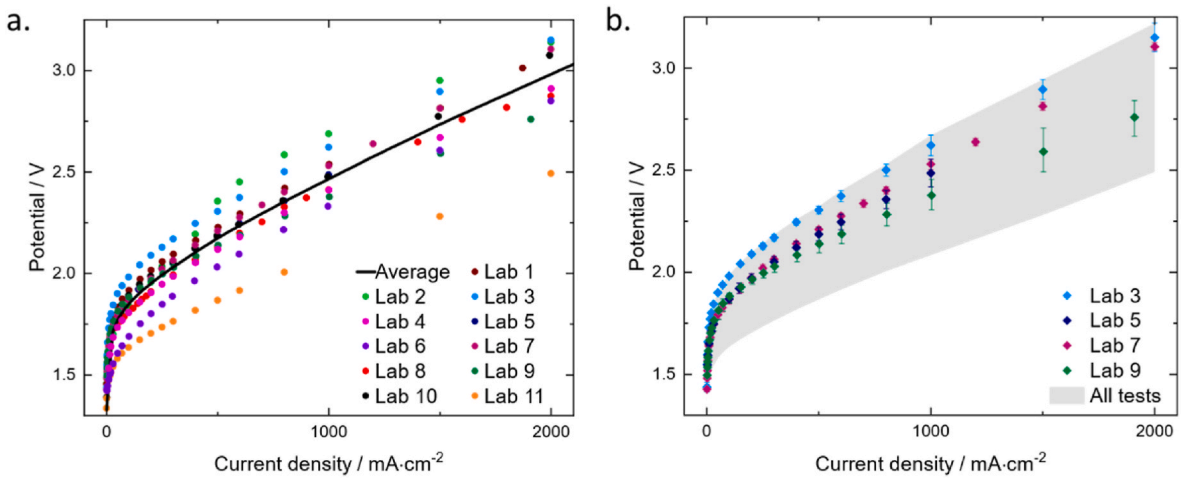

Figure 2. Performance comparison of experiment sets at 80 °C after the conditioning step. a.: Comparison of the polarization curves of all participating laboratories and the total average. For labs that conducted multiple experiments, only their average is depicted. b.: Average polarization measurements of labs which conducted numerous experiments with maximum deviations.

concentrations and purities, and potentially the handling of materials and cells. Nevertheless, even within individual research institutions, there are still notable discrepancies in the results of laboratory cell testing. It is consequently necessary to determine whether these discrepancies are due to unstable experimental conditions, such as fluctuating temperatures, inconsistent flow rates, or variable electrolyte concentrations, or if they are related to the assembly of the cell itself.

4.1. *Influence of iron and conditioning*

The intra-lab deviations indicate that there are performance-relevant parameters that were not considered during the initial standardization effort. Several factors have been considered as the main reasons for these variations. The first and most significant factor to emerge from the analysis is the availability of iron species in the electrolyte with nickel electrodes. When KOH and steel are in direct contact, the leaching of iron species into the electrolyte will occur. Trace quantities of iron lead to the formation of active surface species on Ni, such as NiFeOOH, which are highly active in the oxygen evolution reaction [44–48]. The test stations were not harmonized in this round-robin, and several institutions used stainless steel components as part of their system. Fig. 3a shows the current density measured during the conditioning of the cell at 2 V, comparing the systems using stainless steel contact to those without. The experiments for which iron leaching was prevented demonstrate the degradation of the cell potential during conditioning, the mechanisms of which were discussed in Ref. [26]. Contrarily, for cells where iron leaching was possible, their performance improved significantly during the conditioning period [49,50]. An additional comparison of the average polarization curves for laboratories with and without steel contact is provided in Fig. S1. To confirm and compare the formation of iron-containing species on electrodes, energy-dispersive X-ray spectroscopy (EDX) analysis of post-electrolysis anode electrodes was taken at Lab 11, which had iron present in the electrolyte from the use of stainless-steel parts, and at Lab 9, for which the balance of plant was completely free of steel parts. Iron could be reliably detected at the electrode surface at Lab 11, with an average of 0.46 wt% Fe at multiple measurement spots, while Fe was below the detection limit at Lab 9. As iron species are also a typical impurity in commercially available KOH, the handling of iron species must be considered carefully during experiments, as even small amounts will influence the results. Table 2 gives an overview of maximum iron and other impurities that were reported by the KOH manufacturers used across the labs. When iron is not present, the cell performance tends to decrease but eventually stabilizes, indicating that the conditioning process is sufficient in achieving a

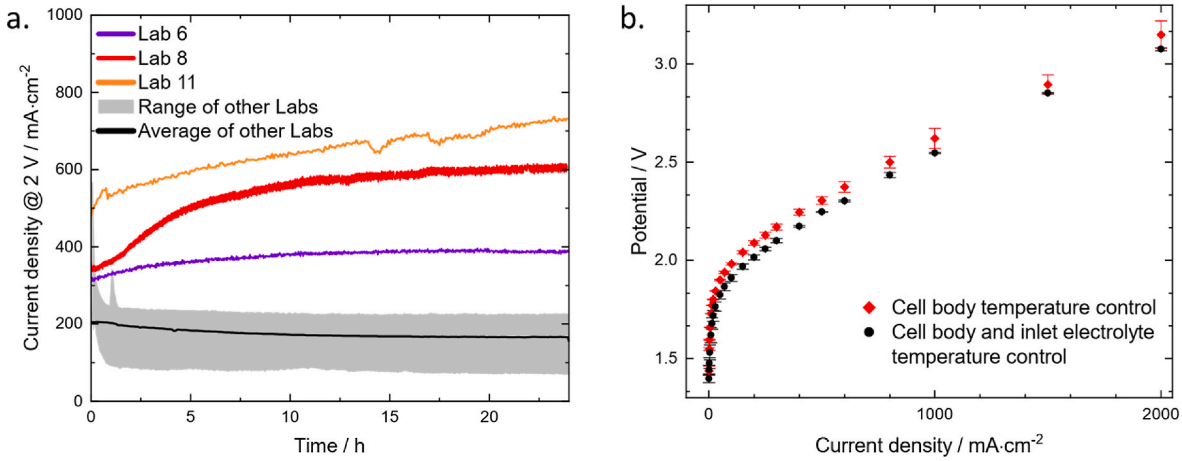

Figure 3. a.: Average current density and maximum deviations measured during the 24-h conditioning at constant 2 V cell potential. Labs 6, 8 and 11, the three labs with stainless steel components in their system, are shown separately. Lab 11 used stainless steel tanks in their setup, whereas Lab 6 and Lab 8 used stainless steel immersion heaters in the tanks. b.: Effect of temperature control of only the cell body compared to temperature control of the cell body and inlet electrolyte flow on the polarization curve of cells after experiments.

Table 2
Maximum reported impurities across all KOH sources used in the different labs. ppm is given in relation to total weight.

KOH wt.%	K_2CO_3 wt.%	Na wt.%	Cl ppm	Fe ppm	Si ppm	S ppm	N ppm	P ppm	Al ppm	Ca ppm	Pb ppm	Heavy metals ppm
≥ 85.0	≤ 2.0	≤ 0.5	≤ 100	≤ 10	≤ 50	≤ 30	≤ 10	≤ 5	≤ 10	≤ 50	≤ 5	≤ 10

consistent performance. This supports the importance of a controlled conditioning phase to ensure reliable and repeatable cell performance.

4.2. *Influence of temperature control*

The inconsistency in heating methods and temperature control across institutions was identified as the second major factor influencing the deviation in cell performance. In this study, we used different heating methods and positions for temperature control, which presented a challenge in achieving uniform temperatures across the tests and labs. For example, in some laboratories, the electrolyte in the tanks was heated to the desired temperature whereas the cell body temperature or both electrolyte and cell were controlled in others. Table S1 listing the detailed heating method and temperature measurement position of each laboratory is included in the supplementary information. While only heating the KOH tank can stabilize the electrolyte temperature, it does not address temperature gradients within the cell itself, which can lead to varying performance. The rate of heat loss from the cell is particularly significant when a solid metal cell body is employed, as has been the case in this study. On the other hand, heating only the cell body disregards the electrolyte inlet temperature. Fig. 3b illustrates the performance differences when only the cell or only the electrolyte is heated, highlighting the importance of ensuring uniform and precise temperature control for consistent and reliable cell performance across different experimental setups. In the case where only the cell body was heated to 80 °C, the inlet and outlet electrolyte temperatures remained around 70 °C. When the electrolyte inlet is heated to 80 °C as well, the average measured cell potential is reduced by 75 mV at 1 A cm^{-2}. The initial approaches and round-robin testing in PEMWE also showed that the cell temperature affects the cell's performance by 3.5 mV K^{-1} [34]. In the case of LAWE, the conductivity of KOH is reduced by roughly 1 % per 1 °C in 32 wt% KOH and electrolyte at 80 °C [51], in addition to a reduction in electrode activity. Slight temperature offsets and differential temperature from inlet to outlet may thus account for up to a third of the observed changes. Moreover, the heating mechanisms and their precision in maintaining a uniform temperature across the cell vary significantly between institutions, introducing additional variability. Due to these reasons, the definition of just a uniform temperature was not enough in this approach and future benchmarking will have to provide more detailed instructions on this topic.

4.3. *Other factors*

The last factor to consider is the variation in flow and electrolyte concentration. The variations in flow rates, high or low, may cause concentration change, uneven distribution of the electrolyte and influence bubble detachment and discharge speed as well as overall cell performance [52,53]. Additionally, the concentration of the electrolyte and its control varies between institutions, affecting the ionic conductivity and the overall electrochemical reaction dynamics [54,55]. The differences in flow and concentration can occur due to variations in the preparation and handling of the electrolyte solutions, the use of different types of pumps, and the specific requirements of test stations. Additionally, these variations can be attributed to the accuracy of measurement and control equipment used by different institutions. The only way to eliminate these remaining differences is by defining completely harmonized test setups.

5. Conclusions

In this work, a round-robin test for liquid alkaline water electrolysis was conducted with eleven participants from seven different countries to establish guidelines for experimental testing in this field. A standardized material and testing protocol was developed, covering electrolysis cell construction, functional materials, testing conditions, and procedures. Despite these efforts, significant quantitative variations were observed among the participants' results, though similar cell behavior was demonstrated during the experiments. The study found that systems using pure nickel electrodes, a common reference material in LAWE testing, are highly sensitive to iron impurities in the electrolyte. While iron impurities improved cell performance, they negatively impacted result comparability. Given that most KOH sources contain small quantities of iron, pre-treatment of materials or the use of a different material system is necessary for sufficient reproducibility [56]. Furthermore, the study demonstrated that simply defining the test temperature is insufficient for achieving comparable cell performance. The impact of local temperature distribution on cell performance is significant, with the location of heating and temperature measurement having a discernible effect on results. Therefore, precise control and documentation of these factors are crucial in similar studies.

This work underscores the necessity of standardization efforts in LAWE testing, more rigorous and standardized guidelines for experimental control and documentation are essential to ensure that research efforts in this field are reliable, reproducible, and productive.

Outlook

As this work has shown, additional efforts in standardization and investigation of influences are necessary. The group part of IEA TCP Task 30 will focus on improving the reproducibility and thus the capability for comparison between laboratories. An outcome of this will be a second instance of the presented round-robin with significant changes to reduce the impact of iron impurities, temperature differences, and setup deviations from the defined standard. It is expected that these adaptations will significantly reduce the deviations between laboratories. At a minimum, the authors target repeatability of experiments within 50 mV at 1 A cm^{-2} as a goal for these investigations.

CRediT authorship contribution statement

Simon Appelhaus: Writing – review & editing, Writing – original draft, Visualization, Validation, Resources, Project administration, Methodology, Investigation, Formal analysis, Data curation. **Lukas Ritz:** Writing – review & editing, Resources, Methodology, Investigation, Data curation, Conceptualization. **Sharon-Virginia Pape:** Writing – review & editing, Resources, Methodology, Investigation, Data curation, Conceptualization. **Felix Lohmann-Richters:** Methodology, Conceptualization. **Mikkel Rykaer Kraglund:** Writing – review & editing, Methodology, Investigation, Data curation, Conceptualization. **Jens Oluf Jensen:** Supervision, Conceptualization. **Francesco Massari:** Writing – review & editing, Supervision, Methodology, Funding acquisition, Conceptualization. **Mehrdad Boroomandnia:** Methodology, Investigation, Data curation. **Maurizio Romanò:** Methodology, Investigation. **Justin Albers:** Resources, Investigation, Data curation, Conceptualization. **Clemens Kubeil:** Supervision, Resources, Data curation, Conceptualization. **Christian Bernäcker:** Writing – review & editing, Supervision, Resources, Conceptualization. **Michelle Sophie**

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Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Martin Mueller reports financial support was provided by International Energy Agency. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.ijhydene.2024.11.288.

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