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Review Article

Upgrading the detection of electrocatalyst degradation during the oxygen evolution reaction

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**Abstract**

Electrocatalysts for the oxygen evolution reaction (OER) are an important component for the transition from fossil to sustainable energy. Commercialization of cost-effective earth-abundant electrocatalysts is in large parts hindered by their degradation. In this short review, I identify common processes leading to a decrease in electrocatalyst activity, followed by an introduction of staple methods to determine degradation electrochemically and by additional physical characterization, which has the potential to remove ambiguities of purely electrochemical studies. I conclude by a summary of the key challenges for an accurate determination of degradation processes and highlight interesting directions to advance the understanding of degradation processes on electrocatalysts in materials-centered model studies.

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Keywords

Electrocatalysis, Oxygen evolution reaction, Corrosion, Electrocatalyst degradation, Mechanisms, Best practices.

Abbreviations

OER, Oxygen evolution reaction.

Introduction

Electrocatalysts for the oxygen evolution reaction (OER) are an important component for the transition from fossil to sustainable energy. The OER is the most popular anodic reaction that is coupled to the cathodic production of H₂ and other valuable products [1].

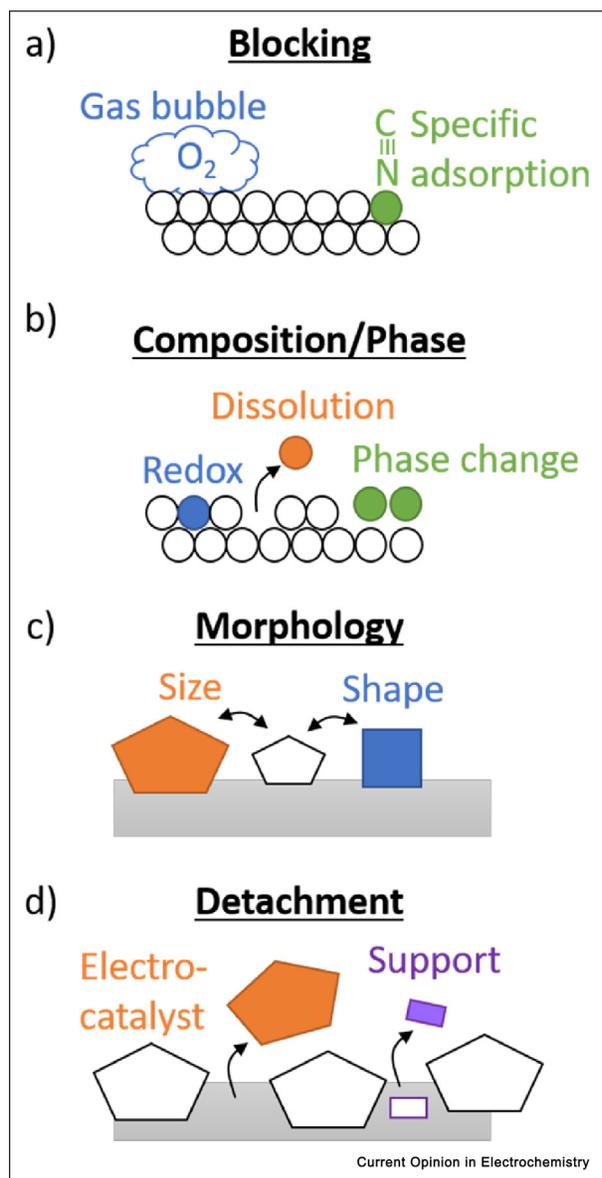
Degradation processes are a key aspect that hinders the commercialization of OER electrocatalysts, particularly when made from earth-abundant elements. The mechanisms of electrocatalyst degradation have been reviewed before in detail on the one hand for atomistic processes on electrocatalyst materials, often with model character [2–4], and on the other hand for membrane electrode assemblies (MEA) [3,4] used in devices. There are important discrepancies in degradation between model and applied studies as discussed in the studies by Cherevko et al., Ehelebe et al., Knöppel et al. [5–7], for example, faster degradation in model systems. In devices, degradation processes on either electrode may affect the other electrode, for example, dissolution of the anode may alter the composition of the cathode [8] or the properties of other parts of an electrolyzer cell that could, for example, lead to a short-circuit [9].

In this short review, I comment on the detection of electrocatalyst degradation during the OER with a focus on materials-centered model studies of the anode. Firstly, I identify common processes leading to a decrease in electrocatalyst activity on various length scales, where I point out that only a subset constitutes electrocatalyst degradation. The misassignment is in part due to the widespread determination of degradation solely by indirect electrochemical methods, which I argue is insufficient. This is followed by a brief discussion of suitable complementary direct methods to corroborate and identify the degradation process(es). I conclude by a summary of the key challenges for an accurate determination of degradation processes and highlight interesting directions to advance the understanding of degradation processes on electrocatalysts.

What processes degrade the apparent electrocatalytic activity?

Several processes may lead to an actual or apparent degradation of the electrocatalyst (Figure 1). Thus, it is important to identify the origins of electrocatalyst degradation to device appropriate measures for quantification of the loss or in the best-case mitigation thereof. Spöri et al. [4] categorized catalyst stability in material stability and operational stability, which is also used herein from the viewpoint of degradation. Material degradation relates to irreversible materials changes of

Figure 1



Processes leading to an apparent degradation of electrocatalyst activity and/or selectivity: (a) blocking of active sites, (b) changes of composition and/or phase, (c) changes of morphology, and (d) detachment of the electrocatalyst and/or support.

the electrocatalyst and its support. In contrast, operational degradation relates to reversible changes existing in situ on the solid–liquid interface due to the electrolyte composition and the OER.

Possible active sites on the surface of the electrocatalyst may be blocked over time by ions, that is, without degrading the electrocatalyst material. The electrolyte cations may interact with reaction intermediates [10,11] or alter the properties of the double layer such that the OER activity decreases in the presence of large cations

[12]. Cations such as Na⁺ are released from typical laboratory glass [13], which may create a reduction of activity over time due to interactions with reaction intermediates and the double layer, that is, operational degradation. On the other hand, materials degradation is also possible, for example, when a dissolution–redeposition equilibrium results in depletion of active metal sites. The anions may also interact with reaction intermediates [14] where PO₄²⁻ lowered the activity on RuO₂ through stabilization of surface hydroxide [15]. Yet, the impact on the OER activity is often negligible, for example for SO₄²⁻ and Cl⁻ anions [16–19]. In general, the effect of intentionally or unintentionally added electrolyte ions on the OER is not well studied and the distinction between operational and materials degradation rarely made explicitly.

Gas bubbles on the surface may also prevent the reactants of the OER (hydroxide or water [20]) from reaching active sites as part of operational degradation. The impact of gas bubbles on OER electrocatalysts and its mitigation have recently been reviewed by Zeradjain et al. [21] Gas bubbles can be introduced by gas purging, immersing electrodes or be produced in situ by the OER where the bubble release depends, e.g., on the electrocatalyst morphology [22]. El-Sayed et al. [23] pointed out that microbubbles result in reduced currents over time, which may be mistaken for materials degradation. Rotation and bath sonication was insufficient to fully prevent the operational degradation [24]. In summary, blockage of active sites is expected to occur frequently due to non-optimal experimental designs and discussions lack a distinction between operational and materials degradation. Based on insights from this distinction, improved experiments can mitigate or even avoid the reduction of the measured currents due to blocking processes.

Electrocatalysts frequently undergo changes of their composition and structure on the atomic scale during the OER (Figure 1b). The loss of the active site to the electrolyte is an obvious example leading to irreversible materials degradation by loss of the active site at the electrocatalyst anode (and potentially poisoning of other device components). Yet, neighboring atoms or redox-inert cations may also dissolve and thereby degrade the properties of the active sites remaining in the solid phase. Note that dissolution of catalytically inert elements may have no effect until their number is sufficiently low to trigger other materials changes such as a phase change (see below). Furthermore, the active sites of the electrocatalyst can undergo reversible or irreversible redox reactions that render them inactive, for example, the oxidation of Mn³⁺ to Mn⁴⁺ at potentials lower than the onset of the OER [25,26]. Reversible redox changes should be considered as operational degradation while irreversible changes are part of materials degradation, for example, those leading to drastic

reduction of the conductivity of the electrocatalyst material [27]. Dissolution and/or metal redox reactions may also lead to phase changes of the electrocatalyst material [3] that can result in materials degradation. These phase changes are perhaps best studied for NiOOH where the formation of the so-called over-charged γ -NiOOH phase constitutes materials degradation [28,29].

Dissolution and phase changes are commonly summarized as corrosion, which the IUPAC defines as “an irreversible interfacial reaction of a material (metal, ceramic, polymer) with its environment, which results in consumption of the material or in dissolution into the material of a component of the environment.” [30] Electrocatalyst degradation is most frequently attributed to corrosion, which can be understood and modeled atomistically [2]. Note that the atoms of an electrocatalyst material are not immobile under typical electrochemical conditions and their location may oscillate over short time scales, while these changes average out on longer time scales. We are just uncovering these effects with high relevance for degradation through new methods such as environmental transmission electron microscopy (ETEM) studies [31,32].

Electrocatalyst degradation may also occur on larger scales even in materials-centered studies with model character, namely when the morphology changes (Figure 1c) or when macroscopic parts of the electrode detach (Figure 1d) [4], both of which constitutes materials degradation.

Morphology changes include the size and shape of the electrocatalyst particles. Both is expected to alter the distribution of surface facets, where less degradation of the (100) surface of Co spinels as compared to the (111) surface has been attributed to the lower surface energy of the former [33]. It may be possible to normalize for the change in particle size in a well-designed model system, which increases the surface area for unchanged mass loading (i.e., negligible dissolution and detachment) but the needed measurements for surface normalization are rarely performed in situ with sufficient frequency to correct for the effect as the degradation processes may uncover previously inaccessible areas over time [34].

The effect of detachment (or erosion) is similar to dissolution in the sense that active sites are irreversibly lost (Figure 1d). The electrocatalyst particles could detach as one piece or parts may detach. Another possibility is the detachment of the support material which may impact the function of the support such as electric connection of the electrocatalyst particles. Additionally, the detachment of the support is coupled to loss of electrocatalyst material that may be dispersed on the support or mechanically fixed to it. Non-optimized

electrocatalyst electrodes may also detach as a single film from their support, that is, delaminate [4].

The function of the support may also be affected by corrosion [35], which strongly impacts the observed evolution of the activity of the composite electrode [34]. It is well known that carbon supports corrode at potentials lower than the onset of the OER by releasing CO₂ [36,37] or by forming insulating surface oxides [34,38]. In summary, degradation of the composite electrode, that is, electrocatalyst, support and potential additives, may happen frequently. Yet, degradation of the support and additives are rarely discussed and the relevant length scale is understudied.

In addition to changes in morphology and detachment, degradation studies of more applied systems, for example, MEAs, need to consider further aspects in a wide range of length scales, namely the mass transport rate of the dissolved (or detached) material, dissolution-redeposition equilibria, the effect of the binder such as Nafion, the effect of the electrolyte pH on the components of the MEA, differences in electrode architecture (e.g., size of 0.1–1 cm² in academic research and up to 100 cm² for MEA and complexity) and operating conditions [6,7]. An in-depth discussion of the significance of these differences between materials-centered and applied research is beyond the scope of this review and the reader is referred to the studies by Cherevko et al., Ehelebe et al., Knöppel et al. [5–7].

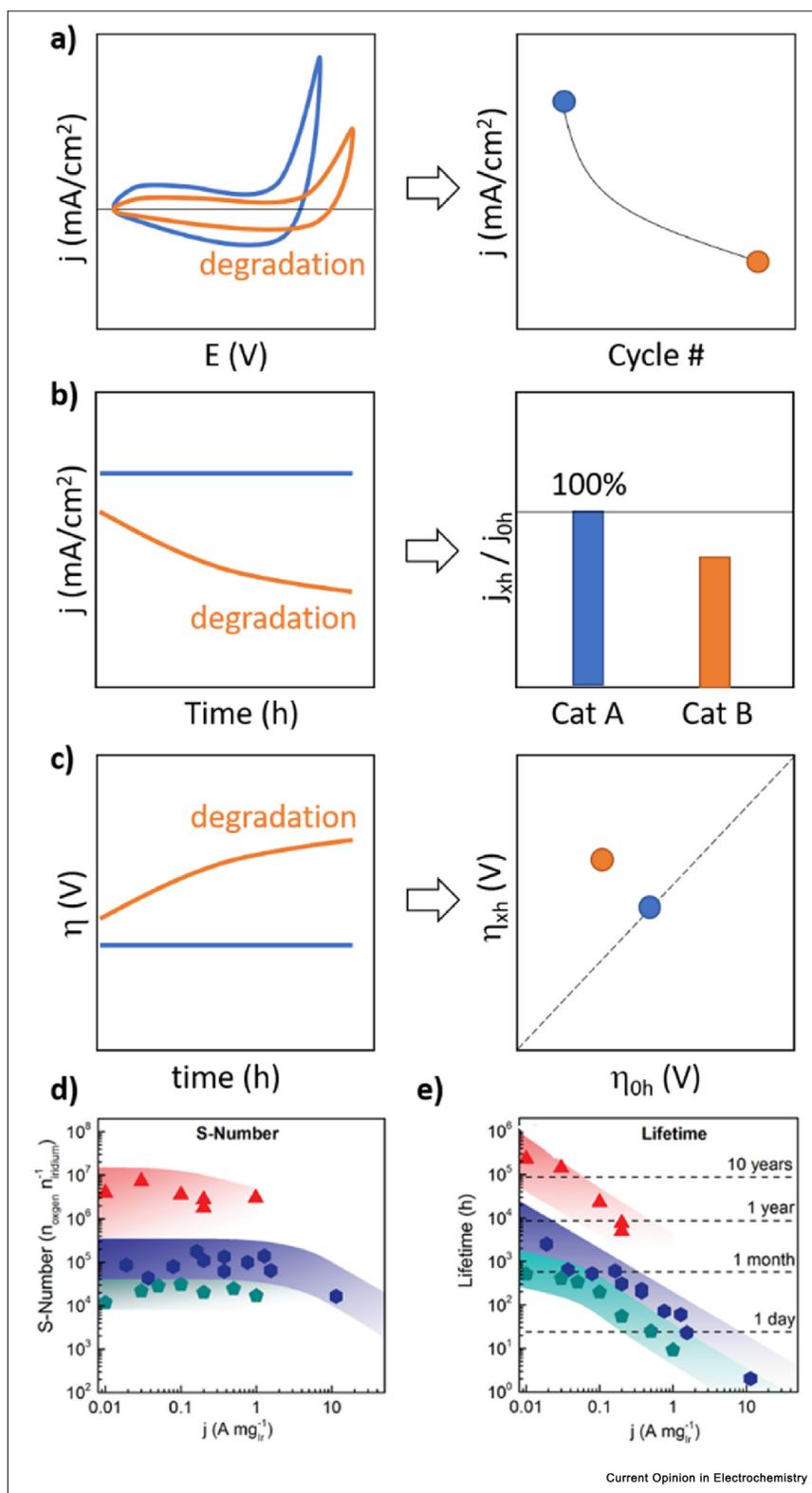
In conclusion, multiple processes are relevant to fully understand the observed reduction in currents over time and to associate an appropriate electrocatalyst degradation process. Often, multiple processes occur simultaneously and/or sequentially, which imposes high demands on experimental design to isolate the effects of a single processes. I have also highlighted that a comprehensive picture of electrocatalyst degradation requires investigations on a wide range of lengths scales from the atomic to the macroscopic scale.

How is electrocatalyst degradation detected?

In most studies, electrocatalyst degradation is detected as the by-product of another electrochemical experiment such as a protocol to determine activity or repeated applications of the activity protocol rather than an experiment designed to decouple the processes illustrated in Figure 1. Examples for a degradation-specific protocol are the accelerated degradation testing (AST) protocols proposed by Spöri et al. [4,39] Popular methods for degradation studies are cyclic voltammetry (CV), chronoamperometry (CA) or chronopotentiometry (CP).

In CV (Figure 2a), the potential is cycled between appropriate limits where the upper limit needs to

Figure 2



Possible ways to pre-screen for electrocatalyst degradation by electrochemical methods and exemplary quantitative representation: (a) Cyclic voltammetry and trends of current at a reference potential with cycling; (b) Chronoamperometry at a selected overpotential and bar plot of relative changes in current density during the measurement; (c) Chronopotentiometry at a selected current density and plot of the overpotential at the end of the measurement (η_{xh}) relative to that at the beginning (η_{0h}). (d) The S-number [46] for various Ir oxides and (e) estimated catalyst lifetimes. Reprinted by permission from Springer Nature, Nature Catalysis, Geiger et al. [46], Copyright 2018.

include the current rise due to the OER. Often the cycling is performed faster as compared to activity studies (e.g., 100 mV/s vs. 10 mV/s) and up to several 10,000's cycles are recorded [4]. Often, new processes become apparent not only during the first 100 cycles [24,40] but also during much later cycles [41]. The current density at one or more reference potentials is then plotted against cycling, ideally after correction for capacitive and ohmic currents [42]. Possible degradation is seen as a reduction of the currents with the cycle number, either in absolute values or relative to the initial (or another relevant) current density.

In CA (Figure 2b), the electrode is held at a selected reference potential, for example, 0.27 V overpotential (1.5 V vs. reversible hydrogen electrode, RHE) [4], and the current density is recorded. Possible degradation is seen as a reduction of the current with time. Usually, durations of a few hours are reported (e.g. the study by Melder et al. [43]), which is short for a degradation test (see next paragraph). Absolute or relative changes of the current can be calculated from the data. Bar charts are often found to compare electrocatalysts.

In CP (Figure 2c), the electrode is held at a selected reference current density such as 10 mA/cm² [44] and the measured potential is recorded. CP is used more frequently than CA in electrochemical degradation studies. Possible degradation is seen as an increase in (over)potential with time. Two hours are seen frequently as used in the important benchmarking work of McCrory et al. [44] However, 2 h are only a first step for screening and should be complemented by longer experiments, where tests for longer than a week (e.g. 1000 h – 6 weeks [45]) are unfortunately still rare. For this data, bar graphs are common and also a plot of the overpotential at the start of the experiments vs. the end is often found. In the latter plot, degradation is indicated by data above a line with unit slope.

The degree of current reduction or (over)potential increase can be quantified in all of these experiments, which is unfortunately rarely done. I need to point out that the duration of the experiment is only a measure of the electrocatalyst degradation when the experiment is stopped by complete deactivation of the electrocatalyst (i.e., end of lifetime (EOL) studies). In all other cases, absolute or relative changes of the current density or (over)potential should be used to compare degradation of the composite electrode and ideally of the electrocatalysts in a specified time interval.

The activity–stability factor (ASF) [47] and stability number (S-number) [46] go one step further in the quantification of degradation. The latter S-number also allows estimating the lifetime of the electrocatalyst (Figure 2d–e). The S-number indicates the ratio of evolved O₂ to dissolved metal(s), that is, how much O₂

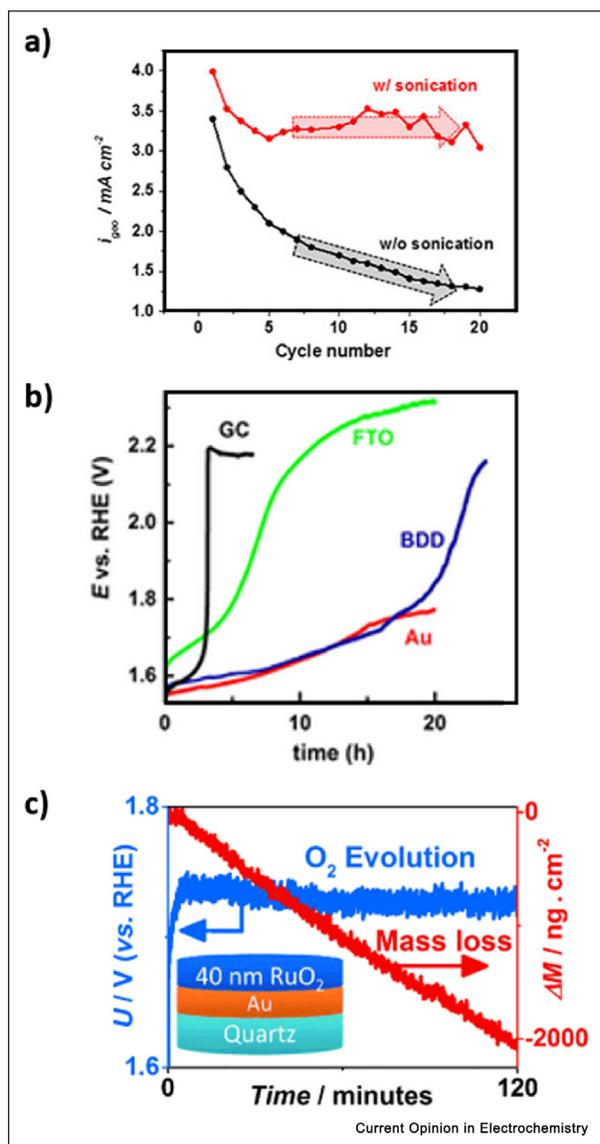
must be evolved to dissolve one active metal site. It is similar to the turn-over number (TON) in homogenous (electro)catalysis, which equals the ratio of evolved moles of O₂ per mole of active metal site before becoming inactive. A quantification of the dissolved metals(s) is required for the calculation of the S-number, which is not readily available so that it is less commonly reported as compared to the purely electrochemical quantifications. The discussion of the S-number supports the need of complementing electrochemical data with further investigations to unambiguously identify electrocatalyst degradation and then quantify it.

To further illustrate the ambiguity of electrochemical data, I highlight the results of three key studies in Figure 3. Firstly, the aforementioned studies of El-Sayed et al. [23,24] show an apparent decrease in current density in a CV experiment using a rotating disk electrode (RDE). Sonicating the electrolyte to remove bubbles (or waiting at open-circuit; not shown) partially mitigates the current loss (Figure 3a). Secondly, Geiger et al. [34] studied Ir black deposited on glassy carbon (GC), gold (Au), boron doped diamond (BDD) and fluorine-doped tin oxide (FTO) substrate electrodes. Only the ubiquitous GC substrate showed a severe increase in (over)potential of about 0.8 V after less than 5 h, which did not correlate with dissolution of the catalyst but rather passivation of GC [38]. Thirdly, Frydendal et al. [48] studied RuO₂ and MnO₂ using an electrochemical quartz microbalance. While there was no change in (over)potential that would indicate degradation, the mass loss clearly indicated loss of the electrocatalyst material.

Therefore, a change in (over)potential or current density does not necessarily indicate electrocatalyst degradation, it could be due to operational degradation such as bubble formation or materials degradation of the used support electrode. Furthermore, electrocatalyst degradation is also possible in the absence of detectable changes in current density or (over)potential. Thus, the three selected studies substantiate the need to complement electrochemical studies of degradation with gravimetric, microscopic, spectroscopic or other suitable methods.

Examples of direct and indirect methods to investigate the processes in Figure 1 are summarized in Table 1. Materials degradation in Figure 1 can be resolved by ex situ or post-mortem experiments, while operational degradation may require in situ or operando experiments (definition in the study Risch et al. [49]). Direct methods are used to measure the existence of some species involved in the process. Examples are the detection of adsorbates by their fluorescence or atomic force microscopy (AFM). The adsorbed species could be further investigated by vibrational spectroscopy such as attenuated total reflection Fourier-transform infrared spectroscopy (ATR-FTIR) [50] to fingerprint the adsorbate and

Figure 3



Three important insights for understanding the degradation of electrocatalysts for the oxygen evolution reaction: (a) a reduction in current density may not be due to irreversible materials changes but rather due to bubbles blocking the electrocatalyst surface. Reproduced with permission from Hartig-Weiss et al. [24], Copyright 2020 American Chemical Society. (b) The choice of the supporting electrode may have strong impact on electrocatalyst degradation (GG = glassy carbon, FTO = fluorine-doped tin oxide, BDD = boron-doped diamond, Au = gold). Reproduced from the study by Geiger et al. [34], Copyright 2017, Wiley and Sons. (c) The absence of changes in electrocatalytic studies does not always indicate the absence of electrocatalyst degradation. Reproduced from the study by Frydendal et al. [48], Copyright 2014, Wiley and Sons.

to quantify the interaction with the electrocatalyst surface. Other direct methods include the determination of a change in the redox state by X-ray photoelectron spectroscopy (XPS) or X-ray absorption spectroscopy (XAS) with the caveat that some redox changes may only be observed in situ or operando [49], for example, metastable

Table 1

Processes leading to a change in current density and (over)potential and suggested exemplary methods to directly or indirectly study the process.

Process	Selected methods and approaches
Gas bubble	Direct: spatially resolved O ₂ fluorescence [52] Indirect: Electrochemical protocol with resting times and sonication [24]
Specific adsorption	Direct: in situ AFM ^a [53] Indirect: concentration variation of expected adsorbing ion [54]
Dissolution	Direct: ICP-MS ^b on electrolyte [46]; EDS/EDX ^c on degraded material [55] Indirect: low loading studies showing complete dissolution of the electrocatalyst [32]
Redox	Direct: (in situ) XAS ^d [26], (in situ) XPS ^e [56], titration [57] Indirect: Replacing a redox-active ion by a redox-inactive ion during synthesis [58]
Phase change	Direct: XRD ^f [41], analysis of the (in situ) EXAFS ^g [41], Indirect: Comparing redox peaks in a CV before and after an expected phase change [49]
Morphology	Direct: AFM ^a [51], TEM ^h [59], SEM ⁱ [41]
Detachment	Direct: IL-TEM ^j [56]

^a Atomic force microscopy

^b Inductively coupled plasma mass spectrometry

^c Energy-dispersive X-ray spectroscopy

^d X-ray absorption spectroscopy

^e X-ray photoelectron spectroscopy

^f X-ray diffraction

^g extended X-ray absorption fine structure

^h Transmission electron microscopy

ⁱ Scanning electron microscopy

^j Identical location TEM

phases [51]. Indirect methods vary a key parameter affecting the process such as the concentration of a species that could specifically adsorb, replacing a redox-active ion with an inactive one, comparing the redox peaks in a CV or using a low electrocatalyst loading so that complete dissolution occurs in a reasonably short time frame. Table 1 is by no means comprehensive but should serve as a starting point to identify suitable methods to identify and/or study the processes responsible for changes in current density or (over)potential to understand degradation of the composite electrode of interest. Further discussion of detection methods and characterization examples may also be found in the studies by Li et al., Chen et al., Spöri et al. [2–4].

Conclusion and outlook

In the last decade, more weight has been put on aspects of electrocatalyst degradation in studies of the OER; virtually all studies with a focus on electrocatalyst

materials published nowadays contain data to discuss electrocatalyst degradation. Including these discussions is an auspicious direction for the field to identify promising electrocatalyst materials for device tests with the caveat that there are large discrepancies between model and applied studies [6,7]. The sorest spots hindering a better understanding degradation processes are.

- Degradation may happen on very different length scales. While there is much attention on atomistic processes, which are certainly very important, the processes on larger length scales such as blocking by bubble formation and particle/support detachment are clearly understudied relative to their importance for use in devices.
- Changes of the electrocatalyst material need to be clearly distinguished from those due to blockage of surface sites or due to support degradation to device appropriate strategies for remediation of the degradation process. Many electrocatalysts were likely discarded prematurely due to inappropriate electrolyte composition, support electrodes or experimental apparatuses.
- The identification of the suspected process of degradation can likely be achieved in a well-designed post-mortem investigation with the caveat that the degradation of the material may occur before electrocatalysis [49], for example, a reaction with an additive during sample preparation [60]. Understanding the mechanism of degradation needs an *operando* or *in situ* experiment, particularly those with long duration [49]. Mechanistic insight is a key prerequisite for the knowledge-guided mitigation of electrocatalyst degradation, for example, by optimizing the composition [61].
- The elucidation of degradation has higher demands on experimental design as compared to activity (benchmarking) studies simply due to the needed long duration, during which important parameters, such as temperature and electrolyte concentration, need to be held constant and which mandates highly stable support electrodes. Optimizing the experimental design is particularly important for another positive recent direction of the field, namely the investigations of “industry-relevant” conditions of high currents, high electrolyte concentration and temperature above room temperature.
- There is no common protocol (or industry standard) used to quantify the degree of electrocatalyst degradation, which is a shortcoming shared with activity benchmarking studies. This reduces the comparability among studies. Most likely, it is not practical to search for a universal protocol for more fundamental research of the electrocatalyst due to the high diversity of the studied materials. I advocate using a specific protocol for degradation studies on similar types of electrocatalysts, for example, the one in the study by Spöri et al. [4] for ink-casted rotating disks

and the one in the study by Weber et al. [32] for thin films. Using a protocol tailored to electrocatalyst degradation is needed as I argued above that these studies have specific experimental demands differing from activity benchmarking studies.

- There are also no minimum requirements for reporting electrochemical degradation data. I plead to thrive for a quantitative report that can be compared to other works. For this, one needs to report both absolute current density and (over)potential differences as well as relative changes in current density for clearly specified conditions during multiple defined durations, for example, 2 h (reference data in the studies by McCrory et al. [44,62,63]), 24 h, 1 week, 1 month, etc. Meaningful degradation studies need to be performed at the same current density or (over)potential used for activity studies. For solar fuels devices 10 mA/cm²_{geo} [44] or 400 mV overpotential [64]. For electrolyzers, Spöri et al. [4] proposed 270 mV overpotential and 10 A/g [65] for material testing and 2 A/cm² for polymer electrolyte membrane (PEM) electrolyzers, while I note that 0.5 A/cm² are typical for alkaline electrolyzers. The field urgently needs quantitative data (even with expected high scatter due to different protocols) to identify the most promising materials families and/or morphologies to focus on for the next generation of durable electrocatalysts, particularly those made from non-critical and abundant materials.
- I have focused on degradation of structural stability in this review. Yet, there is also recent work on dynamic stability [66], where the degradation processes are reversed under specific conditions, which leads to self-repair or even self-healing. Dynamic stability likely only affects changes in atomic composition and structure, where it is an attractive alternative to structural stability that would lead to no detectable degradation by the methods described herein.

In this short review, I summarized some existing shortcomings in the determination of electrocatalyst degradation during the OER and commented which aspects should be urgently upgraded. I wish that my thoughts stipulate discussion in the electrocatalyst community how to make electrocatalyst characterization, in particular of degradation, more reproducible and comparable. At the same time, I hope that this review provides clear guidance to newcomers from other fields how to report (ideally the lack of) degradation of new electrode materials.

Declaration of competing interest

The author declares that he has no known competing financial interests or personal relationships that could have appeared to influence the work reported in this article.

Data availability

No data were used for the research described in the article.

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