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Best practices in using foam-type electrodes for electrocatalytic performance benchmark

Open-pore foam-type electrodes are three-dimensional (3D) materials (typically, Ni, Cu, Fe, and C, images shown in **Figure S1A**) with a wide porosity range (70 ~ 98%) where their porous cellular structures are interconnected so fluid and gas can pass through.[1] The unique properties of conductive foams, such as high specific surface area and structural rigidity, make them suitable and popular supports on which the active materials are coated[2] or grown.[3-5] For example, nearly 8% of the water splitting electrocatalysts reported in 2019 (**Figure S1B**) involve foam-type electrodes, claiming outstanding electrocatalytic performances and superiority over the others (**Figure S2**). Electrocatalysts comparable to commercial noble metal catalysts are often found amid the headlines of various journals.[2, 5-6]

Two indicators, overpotential at given current density (often 10 or 100 mA cm^{-2}, denoted as η_{10} or η_{100}) and Tafel slope (potential required to achieve every current density increment of 10 mA cm^{-2}), are usually employed for catalytic activity evaluation and comparison. However, we believe some oversimplified and overlooked quantitative and experimental issues in using foam-type electrodes have made the electrocatalyst benchmarking physically groundless and often incommensurable, eventually obstructing the understanding and rational design of electrocatalysts. In this Viewpoint, several problems of using foam-type electrodes as electrocatalyst support are discussed, proving some conventional approaches, such as current density normalization, surface area evaluation, and experimental setup, are problematic. (**Figure 1**)

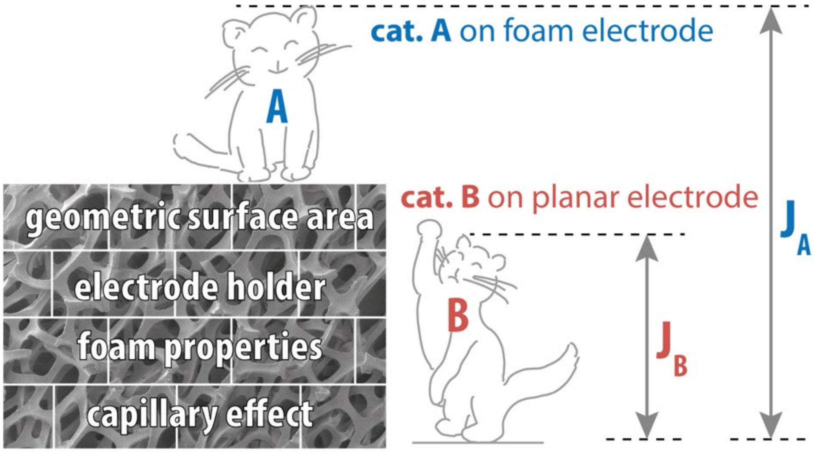
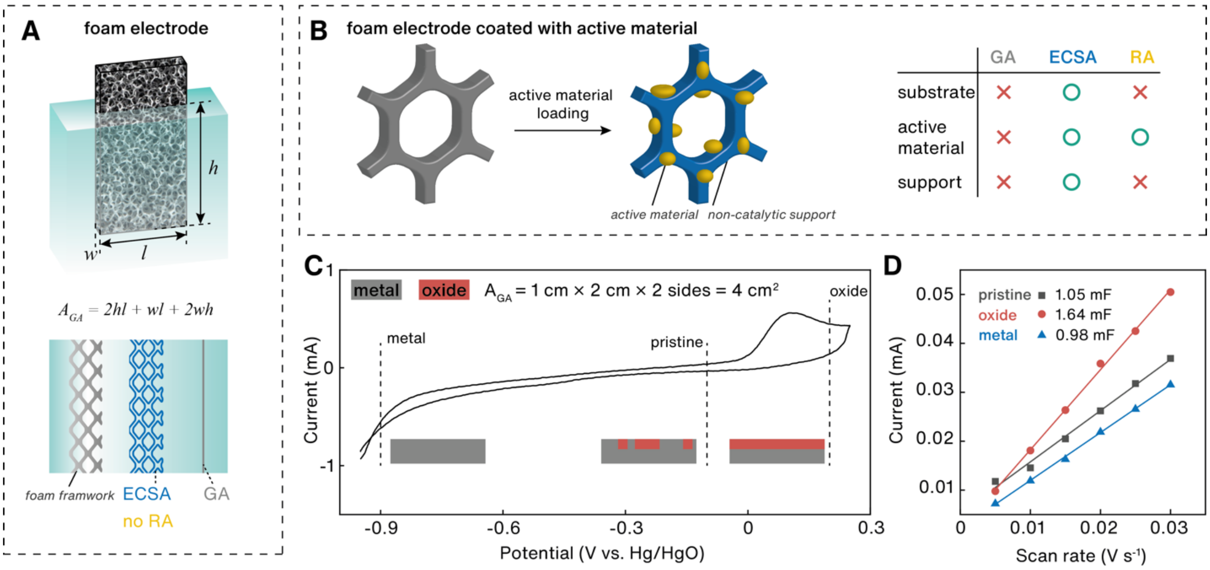


Figure 1 The comparisons overpotential and Tafel slope make the catalysts supported by foam-type electrodes (cat. A) look "superior" to those supported on planar electrodes (cat. B). The four main reasons are discussed in this Viewpoint.

Surface area matters. In most reports, the surface area value used for current density (denoted as J) calculation is not defined,[6-7] which directly impacts overpotential determination. The ideal normalization of current should be performed with the **real active surface** (RA, denoted as A_{real}): the number or the area of the active sites that are responsible for Faradic current transfer during electrocatalysis (or atomic sites that transfer electrons for electrocatalysis). In an ideal situation where non-Faradic current can be ignored, the normalization of current at any given overpotential to A_{real} would reflect the charge (Q) input/output per site/unit area every second.

However, the determination of the real active surface area is challenging for many systems because it often depends on the applied potential, electrolyte, and the nature of the reaction involved, making it somewhat too complicated to estimate. Instead, two alternatives, **geometric area** (GA, denoted as A_{GA}) and **electrochemically active surface area** (ECSA, denoted as A_{ECSA}), are often used.



	GA	ECSA	RA
substrate	×	○	×
active material	×	○	○
support	×	○	×

Figure 2 Illustrations of concepts related to the GA, ECSA, and the RA of (**A**) bare foam-type electrode and (**B**) catalyst-supported foam-type electrode. (**C**) CV plot of Ni foam in 1.0 M KOH. (**D**) Correlation between charging current ($\Delta I/2$) and scan rate.

The use of GA for current normalization (denoted as J_{GA}) could be traced back to the period when smooth planar electrodes were widely used in the electrochemical community, and it could be calculated based on the dimension of the surface contacting the electrolyte[8]: $A_{GA} = 2hl + wl + 2wh$ (**Figure 2A**). In most cases, the thickness of the planar electrode is much smaller than its length and height, thus $A_{GA} \approx 2hl$. ECSA is generally evaluated based on the double-layer capacitance value (C_{DL}) of the electrode. All surface species that are capable of forming a double-layer structure would contribute to the ECSA.[9] However, the surface sites involved in the non-Faradaic charging/discharging processes do not necessarily participate in the Faradaic processes. Specifically, for a *non-catalytic* foam electrode (such as carbon foam): $A_{ECSA} >> A_{GA}$, $A_{real} = 0$. (**Figure 2A**) For a foam electrode fully coated with a *partially catalytic* rough layer (**Figure 2B**): $A_{ECSA} = A_{ECSA\ of\ non\text{-}catalytic\ sites} + A_{ECSA\ of\ catalytic\ sites}$, and $A_{ECSA\ of\ catalytic\ sites} = A_{real}$, $A_{ECSA} > A_{GA}$, $A_{real} >> A_{GA}$.

Normalizing the Faradaic current with A_{ECSA} (denoted as J_{ECSA}) will usually produce a smaller value than the real current density (denoted as J_{real}). Most problematically, normalizing with A_{GA} will significantly overestimate the electrocatalytic rate ($J_{GA} >> J_{real} > J_{ECSA}$). In other words, a lower overpotential of $\eta_{10 \text{ or } 100}$ is undoubtedly expected.

Most reports avoided describing the normalization of current: many just presented the "electrochemical performance" before a few lines about the ECSA, leaving the readers to guess how the current density is calculated.[10-12] Meanwhile, some reports compared the current density normalized by GA and ECSA to study the intrinsic activity of the catalyst,[13-16] and found that the J_{ECSA} was much smaller than J_{GA} at the same overpotential (**Figure S3**). All the systems of high current density values in **Figure S2** report only J_{GA} despite repeated criticism by electrochemists and journal editors of such a method.[8, 17] Although J_{GA} may be practically sound for the industry, the normalizing of current with ECSA or RA (if the active sites are known) is essential for meaningful comparisons among various electrocatalysts.

Questionable "capacitance" method for ECSA evaluation. In the typical "capacitance" method, cyclic voltammetry (CV) is performed in a small potential window (normally ±50 mV around the open circuit potential (OCP)) in which non-Faradaic processes occur. Half of the current difference ($\Delta I/2$) between the anodic current (I_a) and the cathodic current (I_c) at the centered potential is obtained and plotted against the scan rate (v) to fit a straight line with the slope value of C_{DL}. Assuming the C_{DL} is contributed by two components (A and B with a proportion of x and $1-x$) on the surface with specific capacitance values of C_{SA} and C_{SB} (the capacitance of smooth planar surface of the material per unit area) with the total contribution of C_A and C_B, then:

$$C_{DL} = C_A + C_B = A_{ECSA-A} \times C_{SA} + A_{ECSA-B} \times C_{SB}$$

$$A_{ECSA-A} = x \times A_{ECSA}$$

and

$$A_{ECSA} = A_{ECSA-A} + A_{ECSA-B} = \frac{C_{DL}}{x \times C_{SA} + (1 - x) \times C_{SB}}$$

Therefore, the correct evaluation of the ECSA using the "capacitance" method requires the proportion of the surface species and their specific capacitance. Pre-treating the electrocatalyst at or near the reaction potential to convert the surface species can improve ECSA evaluation accuracy. For example, when bare Ni foam is used for oxygen evolution reaction (OER), all metallic Ni and oxide (Ni^{2+}) species form their oxide/hydroxide (Ni^{3+}) before oxygen production (**Figure 2C**). A chronoamperometric pre-treatment at the oxidation potential can reduce the population of metallic sites to a negligible level to show a C_{DL} of 1.64 mF (A_{GA} = 4 cm^2), comparing to 1.05 mF at its pristine form. (**Figures 2D** and **S4**) Similarly, for hydrogen evolution reaction (HER), the metallic Ni surface shows the capacitance of 0.98 mF. If a specific capacitance of 19 μF cm^{-2} of metallic Ni is adopted,[18] the ECSA of Ni foam (A_{GA} = 4 cm^2) for HER is 51.58 cm^2. As to OER, the presence of various species on the surface of Ni foam, including $Ni(OH)_x$, NiO_x, and NiOOH, makes it difficult to obtain a certain specific capacitance value for ECSA calculation.

Compared to the bare Ni foam-type electrode, most reported electrocatalysts are much more complicated, especially for composite electrocatalysts whose surface nature (component species and population) is unknown under the reaction conditions. The use of a universal specific capacitance of 40 μF cm^{-2}, regardless of material or reaction, has been suggested,[19] and adopted in many publications.[20-21] However, for metal electrodes, typical specific capacitance varies from 22 to 130 μF cm^{-2} in KOH solution.[9] An assumption of 60 μF cm^{-2} for all metal oxides has been suggested too,[22] but more fundamental studies are required to get each type of materials' specific capacitance.

As a compromised solution, we believe all electrocatalysts should be treated at or near the reaction potential before conducting capacitance measurement. Also, one should be aware that the calculated ECSA depends on the specific capacitance value chosen. Full details of the potential sequence and the calculation process should always be given.

Not all foam-type electrodes are the same. Clear descriptions of the foam-type electrodes are often missing in the literature, and frequently only the names of the manufacturer were given. Depending on the fabrication method, the thickness, density, porosity, and purity of the electrode material may vary.[23] For instance, the thickness of commercially available Ni foams, the most frequently used substrate, ranges from 1.0 mm[20] to 1.5 mm[24] in the literature. With such a large variety of thickness, the ECSA of bare Ni foam will be completely different even for the same GA, same as different porosity.

The purity of metal foam is another factor that is often neglected. It typically ranges from 99% to 99.99% for Ni foam, and Fe, Cu, S, and C are the typical impurity contents.[25] As shown by Nenad M. Markovic and co-workers, even trace amounts of Fe leaking into the electrolyte can dramatically change the electrocatalytic performance of the pristine catalyst.[26]

For a better demonstration of the varying properties of the metal foam employed by different researchers, we compared the electrocatalytic performance data of bare Ni foam for both HER and OER in **Figure S5**. In some cases, the performance of bare Ni foam is even better than the electrocatalysts reported by others (**Figure S2**). Such incompatible results are most likely due to Ni foam's different physical properties, especially after performance normalization with the GA. Other than that, we believed that it could also be attributed to the experimental setup.

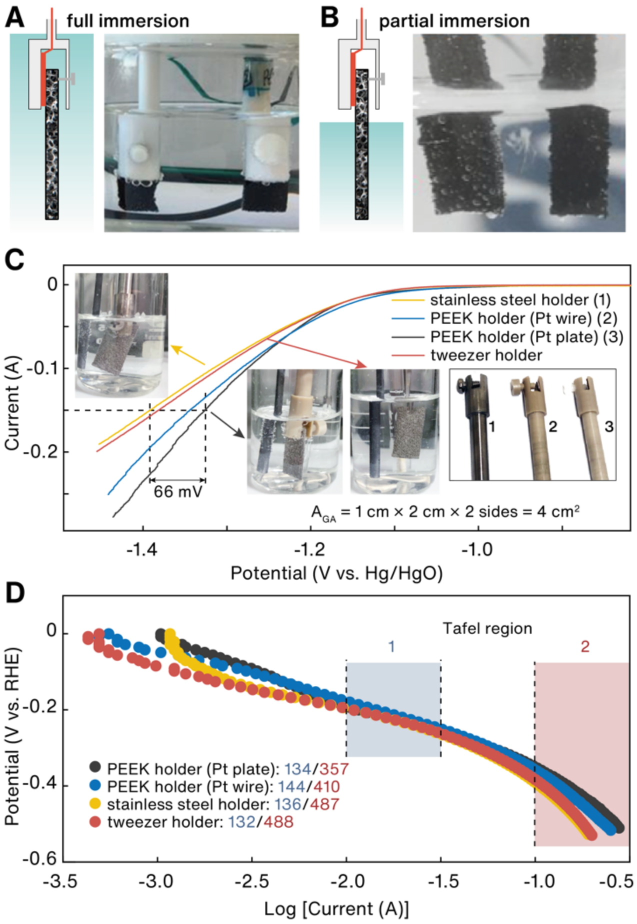


Figure 3 Illustration of the (**A**) full and (**B**) partial immersion. Images in **A** and **B** are reprinted from ref. 14 and 27. (**C**) HER polarization curves of the bare Ni foam electrode attached to different electrode holders in 1.0 M KOH. Insets show images of the experimental setup and electrode holders. (**D**) The corresponding Tafel plots. The Tafel slopes for the two Tafel regions are given after the holder names.

Full or partial immersion, that is the question. Nearly all foam-type electrodes mentioned in the literature were fixed by an electrode holder and dipped into the electrolyte. The foam electrode could be either fully (**Figure 3A**)[14] or partially immersed into a solution (**Figure 3B**).[21, 27] In a typical setup, one end of the foam-type electrode is firmly pressed against a conductor to assure low resistance. Three commonly used electrode holders are shown in the inset of **Figure 3C**. For full immersion, since Fe (stainless steel holder) and Pt (polyether ether ketone (PEEK) electrode with a Pt wire/plate) are in contact with the electrolyte, they can also contribute to the overall current signal.

We compared the HER activities of the bare Ni foam-type electrode fixed by different electrode holders in 1.0 M KOH aqueous electrolyte (**Figure 3C**). The use of an electrode holder with a Pt plate conductor can reduce the overpotential by 66 mV at a current of 0.15 A (current density of 37.5 mA $cm^{-2}{}_{GA}$) compared with using a stainless steel holder. This is understandable since Pt is known as one of the best HER electrocatalysts[28] while stainless steel isn't.[29] Two Tafel regions were chosen (**Figure 3D**) to represent the low and high current density regions. There is little difference among the Tafel slopes in the low current region (from 132 to 144 mV dec^{-1}). However, in the high current region, using the PEEK holder with a Pt plate can produce a much lower Tafel slope (357 mV dec^{-1}) than using a stainless steel holder (487 mV dec^{-1}).

It is expected that when evaluating electrocatalysts with much higher HER activity than Ni foam, the impact of Pt would be intensified at high current density. For the full immersion setup, using a stainless steel holder for HER evaluation is therefore preferred. However, possible contamination by a trace amount of Fe from the stainless steel needs to be noted.

Capillary action. Partial immersion can prevent unwanted disruption from the electrode holder but introduces another problem, the capillary effect. For an open-pore structure, the electrolyte, driven by the interaction between the liquid molecule and the foam walls, can rise to the structure above the solution level (**Figure 4A**). For instance, DI water can wet the Cu foam with a thickness of 1.5 mm by 5 cm above the water level within 1000 s (**Figure 4B**).[30] The capillary action provides extra active sites for electrocatalysis that are not counted when GA is used, resulting in an overestimation of current density. A few researchers have already noticed such effects and tried to prevent it by filling the pores with epoxy glue.[31]

To demonstrate the capillary effect, we tested the HER performance of three bare Ni foam-type electrodes of different lengths (1, 2, and 3 cm) and had them dipped in 0.1 M KOH electrolyte. For the same depth of electrode immersion, two scenarios, “dry” and “wet”, were compared. The “wet” electrodes were prepared by dipping the foam electrodes 4 cm into the electrolyte and pulling them out to the desired depth, while the “dry” ones were immersed to the required depth directly (shown in the insets of **Figure 4C**). The current response is significantly different between the “dry” and “wet” electrodes for the same dipping depth. As expected, the “wet” electrodes can always provide extra Ni sites for HER above the solution level.

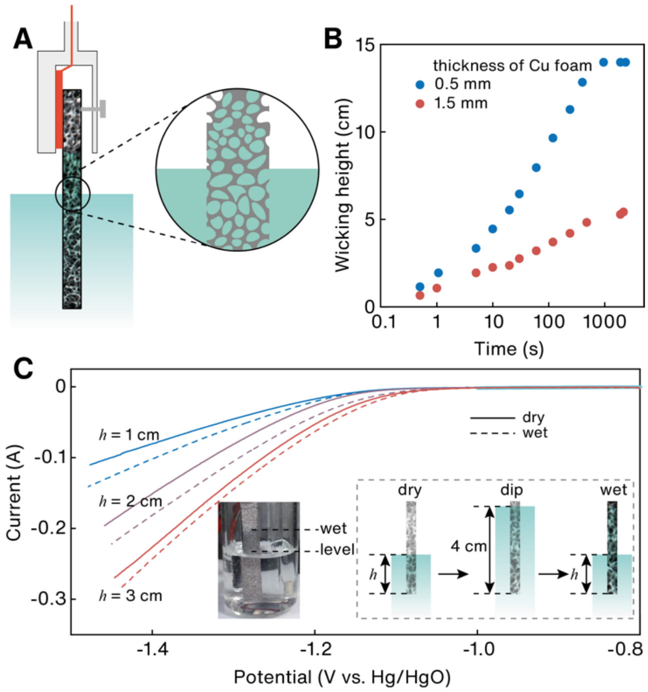


Figure 4 (**A**) Illustration of the pore filling of the foam-type electrode by electrolyte due to capillary action. (**B**) Rate of deionized (DI) water rise in a copper foam with different thicknesses. Data replotted from ref. 30. (**C**) HER polarization curves of the “dry” (solid line) and “wet” (dotted line) Ni foam electrode in 1.0 M KOH. Inset shows the sequence of preparing “dry” and “wet” Ni foam electrode and the image of a “wet” electrode.

When the surface of the foam-type electrode is coated with a layer of electrocatalyst, the impact of the capillary effect varies according to the surface properties of the active material. For example, water travels better in the pores of a metal oxide surface than a metal surface.[30] When a partial immersion setup is used, it is critical to realize and minimize such experimental inconsistencies due to the capillary effect for accurate performance evaluation.

To conclude, despite the high electrocatalytic performance reported, the current fashion of using foam-type electrodes as the substrate for electrocatalyst is rather problematic. The underestimation of surface area produces overestimated data, and the impact of the different properties of the foams is generally ignored. For meaningful benchmarking, it is preferred to use the ECSA. However, the "capacitance" method, widely adopted in literature, has made a few assumptions to simplify the calculation. One of them is that the surface of the catalyst is smooth with a universal specific capacitance at all potential ranges and different pH values, which is hardly true. Moreover, the ECSA is not necessarily the real surface area of the active sites. The most meaningful approach for electrocatalyst benchmarking is the use of turnover frequency (TOF) whenever possible.[32-33] However, its calculation requires a deeper understanding of the role of surface species and the identification of active sites, both of which are equally challenging.

The errors introduced by experimental setups are also troublesome. Both full and partial immersion of the foam-type electrode into the electrolyte can bring significant interferences/errors, either from the electrode holder or as a result of capillary action. There is no absolute better choice, but one should bear in mind both the pros and cons of the different setups when planning an electrochemical experiment.

To better benchmark various foam-supported electrocatalysts, we believe that the electrocatalytic results of the same electrocatalysts coated on planar inactive support under the identical conditions need to be presented together, as demonstrated by a few researchers.[31, 34] Such comparisons can minimize the errors caused by both using the geometric area of the foam support and experimental setup. The evaluation of the ECSA of the electrocatalyst should also be based on the results acquired from the planar support.

Finally, we would like to add that other porous supports, such as carbon cloth and carbon fiber paper, share the problems similar to those of the foam-type electrodes we have discussed. The electrochemical community needs to pay more attention to the possible overestimation of performance resulting from the careless use of foam-type supports to guide the insight to rational catalyst design.

Weiran Zheng[†]

Mengjie Liu[†]

Lawrence Yoon Suk Lee*[,†,‡]

[†]Department of Applied Biology and Chemical Technology and the State Key Laboratory of Chemical Biology and Drug Discovery, The Hong Kong Polytechnic University, Hung Hom, Kowloon, Hong Kong SAR, China.

[‡]Research Institute for Smart Energy, The Hong Kong Polytechnic University, Hung Hom, Kowloon, Hong Kong SAR, China.

ASSOCIATED CONTENT

Supporting information

The supporting information is available free of charge at: https://

> Experimental details, summary of data from the literature for both HER and OER using foam-supported electrocatalysts and bare Ni metal foam; Photos of different foams; SEM image of Ni foam; ECSA testing of Ni foam.

AUTHOR INFORMATION

Corresponding Author

***Email:** lawrence.ys.lee@polyu.edu.hk

ORCID

Weiran Zheng: 0000-0002-9915-6982

Lawrence Yoon Suk Lee: 0000-0002-6119-4780

Notes

Views expressed in this Viewpoint are those of the authors and not necessarily the views of the ACS.

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