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Viewpoint

Metal Foam Morphology Affects the Run to Run Reproducibility of OER Using Nickel Catalysts

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ABSTRACT: Nickel (Ni) foam-based electrodes are excellent catalysts for the oxygen evolution reaction; however, we found that the random pore-size distribution of Ni foams contributes to a significant variability in electrochemically active surface area, compromising experimental reproducibility. We provide insights into quantifying this critical material property, verified by four electrochemical laboratories.

Oxygen evolution reaction (OER) is a common anodic reaction in many electrochemical applications (e.g., H_2 production, CO_2/N_2 reduction).[1,2] Noble metal catalysts based on iridium and ruthenium are widely acknowledged to display superior OER performance under acidic conditions.[3] The scarcity and cost of these materials motivate a shift toward nonprecious, Earth-abundant alternatives.[4] Nickel (Ni) has been reported as a promising alternative for OER in alkaline conditions. Ni is frequently used in the form of a porous foam electrode, with increased surface area and porosity relative to a foil form. Ni foam can be deployed without further treatment[5] or serve as a support for the deposition of more active OER catalysts, e.g., layered double hydroxide (LDH).[6] Considering Ni foam as both a catalyst and a substrate, accurate electrochemical characterization of Ni foams remains challenging, and considerable attention has been devoted to establishing experimental guidelines to enable **cross-study** comparisons. A Viewpoint by Zheng et al. and other contributions discussed the importance of surface-area characterization, ensuring efficient mixing and mass transfer through cell design, electrolyte purity, and the choice of counter and reference electrodes.[7−11] These guidelines provide excellent support for designing experiments in which key OER performance metrics, such as overpotential, can be measured without interference from cell configurations or electrical connections. *However, does following the established guidelines guarantee that the same result will be obtained when repeating the same experiment?* We noted that the reproducibility of results within a **single study** (i.e., run to run reproducibility) has not been studied at the same level of detail as the general OER benchmarking. The ability to reproduce results is critical for adopting catalyst synthesis methods beyond a single lab and ultimately for commercial applications, where performance with acceptable variation needs to be achieved using different catalyst batches, suggesting an urgent need to establish the guidelines for reproducibility assessment for Ni foam catalysts. To address this gap, we report our insights from triplicate OER experiments conducted under varying conditions and across four laboratories worldwide.

QUANTIFYING REPRODUCIBILITY

To establish a framework for reproducibility assessment, we sought to evaluate the standard deviation (SD) of recorded potentials across triplicate chronopotentiometry OER experiments, shown below.

Standard deviation, averaged over 10 min (data points collected every 0.1 s)

$$\sigma = \sqrt{\frac{1}{n-1}\sum_{j}^{n}(x_j(t_i) - \mu(t_i))^2} \tag{1}$$

where $x_j(t_i)$ potential at time t_i in trial j, $\mu(t_i)$ = mean potential across the three trials at time t_i, and n = number of trials = 3.

$$\overline{\sigma} = \frac{1}{T}\sum_{i=1}^{T}\sigma(t_i) \tag{2}$$

where T = the total number of time points (data collected every 0.01 s for 10 min).

For a perfectly reproducible experiment, the SD would be zero, since we would expect to read the same potential for the same catalyst and experimental conditions (i.e., the lines in Figure 1b and 1c showing raw chronopotentiometry data should overlap). We prepared fresh electrolytes and electrodes for all experiments, except for those involving the use of expensive platinum (Pt) foil. We carefully followed protocols from OER benchmarking papers regarding electrode holder choice, electrode spacing, cleaning, and other relevant factors.[7] We did not consider *iR* correction since it would be the same across triplicates, and therefore would not impact reproducibility (however, it is important to practice careful *iR* compensation and report methods used when discussing catalyst performance[12]). Additionally, tests at Northeastern

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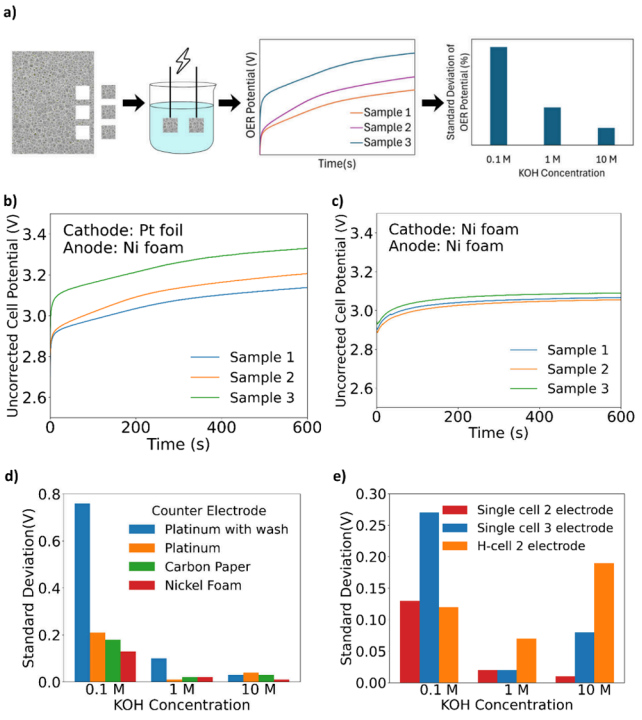


Figure 1. Insights into the reproducibility of OER experiments using Ni foam. (a) Experimental workflow used for characterizations. Triplicate chronopotentiometric results at 100 mA cm^{-2} for a nickel foam working electrode when using a (b) platinum counter electrode in 1 M KOH, and (c) nickel foam counter electrode in 1 M KOH. Decreased standard deviation is seen in c) using a nickel foam counter electrode. Comparisons of the average standard deviation across 10 min of (d) all counter electrodes tested and (e) different cell configurations using nickel foam working and counter electrode in 0.1, 1, and 10 M KOH. Standard deviation calculations are shown in eq 1.

University used stamps (1.8 × 1.8 cm) to cut the electrodes, thereby eliminating errors arising from geometric area differences (area was calculated considering both sides of the foam immersed in the electrolyte, without accounting for the foam thickness). As commercial water electrolysis must operate at high enough current densities to reduce electrolyzer cost,[11,13] we performed all tests at 100 mA cm^{-2} geometric area.

The goal of these experiments is to quantify the variability in recorded potentials across nominally identical Ni foam electrodes by using the standard deviation from triplicate measurements. In the following discussion, we will evaluate how identical pieces of nickel foam can lead to vastly different results, despite maintaining material and geometric consistency, due to their inconsistent surface morphologies within the same sheet. Importantly, this work does not aim to make claims about Ni activity, but rather to establish the expected variability associated with Ni foam electrodes under common OER conditions.

REVISITING THE BEST REACTOR CONFIGURATION

While the choice of the counter electrode has been thoroughly discussed in the literature in the context of cross-study comparisons,[7−11] we revisited these design considerations with a focus on single-study reproducibility. The choice of the counter electrode is particularly important for the experiments performed in a single cell, where the cathode and anode are not separated by a membrane or a porous frit. Due to its simplicity, single cells are frequently used[14] and are also practical to deploy in high-throughput, machine-learning guided screening studies, which are gaining interest.[15] To understand how the counter electrode affects reproducibility, we conducted a simple two-electrode electrolysis using untreated Ni foam as an OER catalyst, and platinum (Pt) with and without an acid wash before electrolysis, as well as carbon paper as a counter electrode, in 0.1, 1, and 10 M potassium hydroxide (KOH) electrolyte.

Concentrations between 0.1 and 1 M KOH are common in OER electrocatalysis reports and 10 M was chosen to illustrate an extreme case in which high currents can be achieved for industrial applications.[16] Reproducibility is particularly important when considering industrial applicability because the performance of electrodes must be predictable to ensure safe and consistent operation. In agreement with previous cross-study reports, these common counter electrodes significantly affected reproducibility in a single-cell experiment. Pt can dissolve in both acidic and alkaline conditions,[17−19] particularly with an acid wash,[20] and carbon paper showed visible sedimentation. Therefore, we investigated whether reproducibility could be improved by using the same catalyst for both the cathode and anode, hypothesizing that this would reduce contamination observed while using Pt or carbon paper. Using Ni foam as both the cathode and the anode allowed us to significantly reduce the SD compared to other counter electrodes (Figure 1d).

Despite the usefulness of 2-electrode single-cell configurations, 3-electrode single-cell configurations, and h-cells are critical for various electrochemical measurements and should be evaluated for single-study reproducibility. Reference electrodes are required for accurate overpotential measurements, whereas h-cells enable separation of the cathode and anode compartments, which is necessary for practical electrochemical applications. When comparing these configurations, we unexpectedly found that the simple 2-electrode single-cell setup was the most reproducible, likely due to its reduced complexity (Figure 1e). Based on these results, the nickel foam counter electrode single-cell setup served as the basis for subsequent comparisons.

IMPORTANCE OF THE ELECTROLYTE CONCENTRATION

Interestingly, our experiments revealed that reproducibility strongly depends on electrolyte concentration. The SD in 0.1 M KOH, even with the optimized counter electrode and reactor configuration, is as high as 130 mV, decreasing to 20 mV in 1 M KOH and 7 mV in 10 M KOH. For context, it is common to encounter reports on new materials that cite a 50−100 mV reduction in overpotential as proof of an improved catalyst. Hence, it is critical to test the catalysts at higher electrolyte concentrations (min. 1 M KOH), which helps decrease the SD.

There are a few important factors that can explain the influence of electrolyte concentration on reproducibility. The operating potential in 0.1 M KOH is significantly higher than that of 1 and 10 M, with potentials fluctuating around 8 V compared to 4 and 3.4 V. Activity is expected to increase in higher concentrations due to increased hydroxide activity and conductivity,[21] but this also has an impact on reproducibility that is rarely discussed. The high potential in 0.1 M KOH

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could indicate unstable reaction conditions, leading to greater fluctuations between trials. Within each concentration, the same batch of KOH was used to minimize any variability introduced by the electrolyte. However, due to increased activity, hydroxide supply, and conductivity, it is reasonable to expect differences between the studied KOH concentrations. Higher concentrations of KOH provide a greater driving force for ionic transport than lower concentrations, which are more limited in transport and kinetics. Low driving forces for the ionic transport may make differences in nickel foam morphology and other environmental variations more pronounced in 0.1 M KOH.

Furthermore, the impurities present in the electrolyte need to be taken into consideration. Trace Fe contamination is known to influence and improve the performance of Ni-based OER electrodes[22,23] and therefore it is important to report and account for Fe impurities when assessing catalyst performance. Fe purification protocols are recommended to ensure catalyst performance cannot be attributed to impurities; however, these protocols can vary in terms of reproducibility and cause interferences with electrochemical tests.[24] Since this study focuses on reproducibility and does not strive to make claims on Ni activity, we did not purify our KOH. We used KOH with less than 0.001% of Fe[25] and used the same fresh batch of KOH, glassware, and DI water for each concentration. Therefore, impurities in the bulk are consistent within each electrolyte condition.

Since impurities increase with concentration, at higher KOH concentration, there is a higher absolute concentration of impurities. In 10 M KOH, this may result in more rapid and consistent Fe incorporation into the NiOOH layer, whereas in 0.1 and 1 M KOH the Fe incorporation is more variable and sensitive to small kinetic, transport, and geometric differences. This may contribute to increased reproducibility at higher concentrations. Similar logic follows for cation effects (K^+). There is emerging work to explain the influence of cations on alkaline OER mechanisms.[26] However, at low concentrations, these effects are again more sensitive to environmental variations and can contribute to reduced reproducibility. Based on these findings, it is important that catalysts be tested in a range of KOH concentrations, not just at 0.1 M KOH. In dilute KOH, decreased reproducibility is likely due to increased sensitivity to slight variations in the experimental setup and reaction environment between trials. To ensure that the results are not due to natural deviations from the use of low-concentration solutions, catalyst superiority must be confirmed across multiple KOH concentrations, especially at or above 1 M KOH.

SURFACE AREA CONSIDERATIONS

Seeking further opportunities to minimize SD in OER experiments, we investigated the role of surface definition in single-study reproducibility. We sought to use a surface-area metric to normalize the applied current density during chronopotentiometry and to account for the potential structural differences among seemingly identical pieces of nickel foam. Despite cutting each piece of nickel foam with a consistent 1.8 cm × 1.8 cm stamp, we expected differences in pore size and distribution among the samples due to manufacturing processes[27,28] (Figure 2a). Electrochemically active surface area (ECSA) is a widely used metric in the community, despite criticisms of its accuracy.[7,8,29−33] Capacitance-derived ECSA measures double-layer charging currents

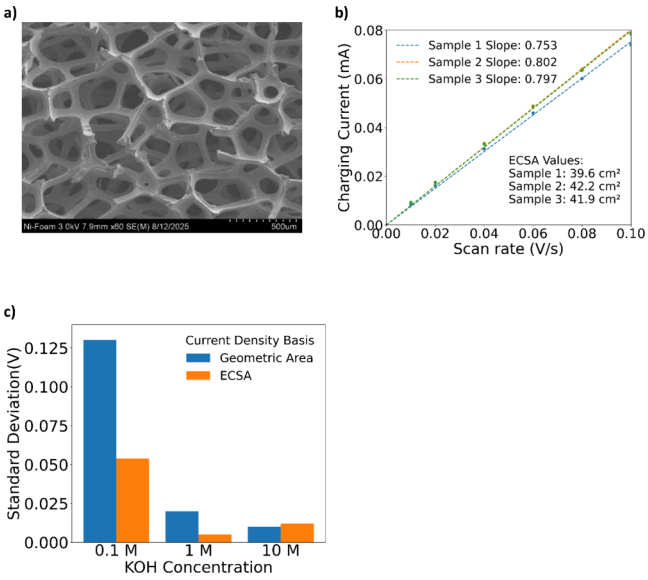


Figure 2. Differences in nickel foam morphology shown through (a) scanning electron microscopy (SEM) image of bare nickel foam, showing various pore sizes at a 500-μm scale using a Hitachi S-4800 SEM, (b) ECSA measured in 1 M KOH for different pieces of Ni foam (all 2.42 cm^2 geometric area) cut from the same Ni sheet using a stamp, highlighting the randomness of Ni foam pores distribution, and (c) comparison of standard deviation conducted at 100 mA cm^{-2} based on geometric area of nickel foam electrode vs using ECSA corrected current density, at comparable applied current.

in a nonfaradaic potential region, where the current scales linearly with scan rate, thereby identifying the electrochemically accessible area. Recent work has identified drawbacks of this method, particularly regarding pseudocapacitance and specific capacitance. What appears as nonfaradaic on a cyclic voltammetry graph may include contributions from pseudocapacitance and ion penetration into the bulk in addition to the desired double layer charging.[34−39] This can lead to an overestimation in the ECSA. Additionally, the value of specific capacitance selected to convert from double layer capacitance to ECSA can be very complicated and a source of error.[8] In OER, there are various species on nickel foam ($Ni(OH)_x$, NiO_x, NiOOH). To accurately use specific capacitance, the exact composition on the surface must be known, and the specific capacitance of each species must be used proportionally.[7]

Other methods, like the use of BET[39] and geometric modeling[40] can gather more precise geometric areas, but fail to consider reaction conditions and electrolyte wetting, which is an important consideration when using porous electrodes.[29,34] Because of this, we decided to use ECSA, keeping in mind its limitations and emphasizing that our measurements are not to be taken as true surface areas, rather as comparative metrics between samples. As highlighted in a recent Viewpoint by Mygind et al., ECSA measurements are highly dependent on methods and reaction conditions, so careful definition of protocol and measurement methods is essential for any comparison to be made.[41] Therefore, it is better suited as a metric to compare within an experiment, where conditions are kept constant. Our ECSA results show no faradaic peaks, linear scan rate vs current plot, stable behavior after several cycles, and no change in shape over several cycles. Measurements were conducted under identical conditions (scan rates,

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electrolyte composition, pretreatment conditions, potential window). Therefore, the values serve as a consistent comparative metric within this study. We found that pieces of nickel foam exhibited measurably different electrochemical surface responses under identical conditions, with differences exceeding 2 cm^2 (ca. 4%, Figure 2b, Table S3).

To assess the importance of these differences, we repeated our electrolysis experiment applying an ECSA-corrected current to achieve a consistent current density across all samples. This significantly reduced SD to 50 mV in 0.1 M KOH and 5 mV in 1 M KOH, demonstrating the impact of varying surface areas (Figure 2d). By decreasing the standard deviation in 0.1 M KOH, we demonstrate that reproducibility concerns at low concentrations are not solely due to high potential ranges, but also to surface area differences. The decrease in standard deviation validates the impact of the use of ECSA as a surface metric within studies. We caution that this approach may not be applicable between studies due to the limitations of ECSA measurements explained above. This approach is also complicated by more complex catalysts, which exhibit pseudocapacitances and greater intricacies in specific capacitance values. However, it is important to note that differences in the electrochemical surface responses of substrates among triplicates within a single study can affect results, and surface area metrics should be considered prior to catalyst deposition.

QUANTIFYING VARIABILITY IN FOAM MORPHOLOGY

While measuring the ECSA for each piece of Ni foam and correcting the results provides excellent reproducibility, we recognize it might not be easily adopted by the field, nor applicable for more complex catalysts, considering the complications associated with specific capacitance.[7] Therefore, we sought a simplified approach to quantify the anticipated error arising from structural differences in foam electrodes. In all experiments, we observed that more consistent results were obtained at higher KOH concentrations, with the SD decreasing from 130 mV at 0.1 M to 7 mV at 10 M for the Ni−Ni system. To account for differences in full cell potential across concentrations, we introduced an additional reproducibility metric: the relative standard deviation (RSD), which expresses the standard deviation as a percentage of the full-cell potential, normalizing for differences in absolute potential, shown below.

Relative Standard Deviation (RSD), averaged over 10 min (data points collected every 0.1s)

$$\mathrm{RSD}(t_i) = \left| \frac{\sigma(t_i)}{\mu(t_i)} \right| \times 100\% \tag{3}$$

While this adjustment reduced the apparent variation in concentrations, the overall trend persisted (Figure 3c), suggesting that a correlation may exist that expresses the maximum RSD expected at a given KOH concentration.

MULTILAB VALIDATION

To confirm our hypothesis, researchers from three electrochemical laboratories − in Australia, The Netherlands, and Singapore − repeated our chronopotentiometry experiments in 0.1, 1, and 10 M KOH, using the Ni−Ni system (Figure 3a). Each lab followed the same experimental guidelines (Supporting Information), except that only the Northeastern lab used

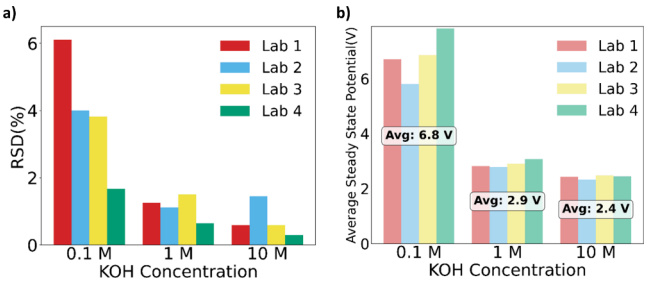


Figure 3. Comparison of results across laboratories: proposed correlation between the relative standard deviation and electrolyte concentration for OER experiments under 100 mA cm^{-2}, validated by four different laboratories. (a) Relative standard deviation of triplicate OER trials using nickel foam working and counter electrode, and (b) average steady state potential of the same OER trials in 0.1, 1, and 10 M KOH, with the averages between the 4 laboratories displayed.

stamps to cut the electrodes. Notably, all laboratories used different Ni foam suppliers and potentiostats. The results showed the same correlation between RSD and KOH concentration trends and confirmed differences in ECSA measured across different pieces of Ni foam (Table S4, Figure S10). Based on these multiple lab results, the expected deviation in the reported potential at 100 mA cm^{-2} (geometric area, 2.42 cm^2 electrode size) is ∼ 6% of the full cell potential in 0.1 M KOH and ∼ 2% in 1 and 10 M KOH. While this correlation is expected to apply to a specific size of the catalyst, electrolyte, and experimental setup, we anticipate that if the full cell potential and electrolyte concentration are reported, one can roughly assess the expected range of the deviation in the recorded potential. Attaching a similar correlation to reports on new catalysts could also be a simple, resource-efficient way to provide insights into reproducibility and the effects of pore-size variability in the Ni foam used.

In addition to considering the variation in morphology, it is notable that the average steady-state potential values across laboratories differ significantly—up to 2 V in 0.1 M KOH and 0.2 V in 10 M KOH, highlighting the benefit of testing OER under high KOH concentrations (Figure 3b), as well as the need to provide that the average steady-state potential for the bare Ni foam in any studies that deploy it as electrode or electrode support.

CONCLUSIONS AND BROADER IMPACTS

Our work shows that differences between nominally identical pieces of bare Ni foam electrodes can substantially affect the reproducibility of electrochemical measurements. Across four independent laboratories, differences in nickel foam morphology contribute to this variability, as shown by ECSA surface area metrics. Preliminary investigations into the applicability of this study to electrodeposited catalysts, other metal foam materials, and different-sized electrodes prove that the significance of these findings spans further than just nickel foam. The same reproducibility challenges were observed when triplicate electrolysis experiments were conducted using nickel−iron deposited on nickel foam, bare copper foam, and a nickel−iron electrode with half the surface area (Figure 4).

To enable accurate and reproducible catalyst characterization, we urge the electrochemical community to recognize the importance of variability among nominally identical pieces of Ni foam and to quantify it before performing subsequent catalyst deposition on Ni foam or using it in OER experiments.

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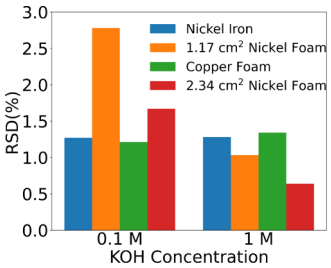


Figure 4. Insights into the applicability of this study by conducting triplicate chronopotentiometry trials at 100 mA cm^{-2} using a nickel–iron electrodeposited catalyst, nickel foam electrodes of 1.17 cm^2 and 2.34 cm^2, and copper foam.

ECSA measurements of multiple pieces of foam can be a helpful comparative approach to understand this variability. In the absence of ECSA data for the Ni foam, we propose that surface variability can also be assessed by comparing results from multiple repetitions of the OER experiment (repetitions of, e.g., chronopotentiometry for the bare Ni foam), if other sources of experimental error are eliminated.

If the data described above are not provided, a rough estimate of the influence of Ni foam variability can be made for the known electrolyte concentration and steady-state potential. As confirmed by four independent laboratories, using Ni foam from different suppliers, the relative standard deviation was up to 6% of full cell potential in 0.1 M KOH, and 2% in 1 and 10 M KOH, establishing a benchmark for anticipated variability. However, these values were measured using a protocol designed to maximize reproducibility and will be affected by the choice of reactor, electrode configuration, and other parameters.

Reviewing the experimental design from a reproducibility perspective should become a routine step in any report on Ni-foam catalysts. Ni foam morphology not only influences the electrochemically active surface area, but also mass transport and impurity incorporation, which collectively affect experimental reproducibility. The extent to which this phenomenon manifests will be directly related to the electrolyte concentration; the choice of this concentration must always be carefully reviewed from a reproducibility standpoint. The use of low concentration electrolytes, such as e.g.,0.1 M KOH might not suffice in making OER performance claims due to the limitations in reproducibility. Instead, we recommend testing the catalysts over a range of concentrations, including those above 1 M KOH. Including this additional consideration in the experimental design and reporting will support the pursuit of next-generation, scalable, and reproducible OER catalysts, which are urgently needed for diverse electrochemical applications.

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■ ASSOCIATED CONTENT

Supporting Information

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Potential and ECSA comparison and equipment (XLSX)
Experimental methods and materials, additional SEM images, chronopotentiometry data, ECSA measurements, and reproducibility statistics (PDF)

■ AUTHOR INFORMATION

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Notes

The authors declare no competing financial interest.

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