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Enhancing Reproducibility and Uniformity of RDEs: Spray-Coating vs Drop-Casting for IrO_2 OER Catalyst

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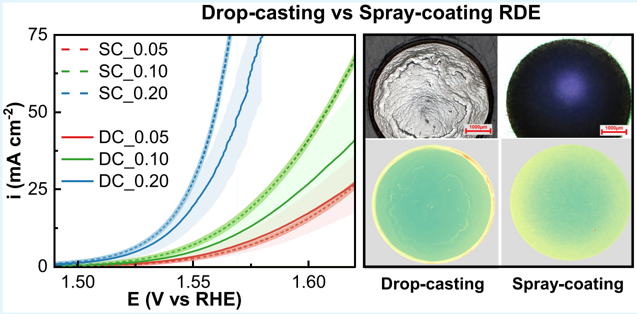
ABSTRACT: The thin-film rotating disk electrode (TF-RDE) technique is widely used for catalyst screening in oxygen evolution reaction (OER) studies due to its ability to separate mass transport from redox kinetics and its low catalyst requirement. However, the manual drop-casting method frequently yields unsatisfactory reproducibility, primarily due to the "coffee-ring" effect, caused by nonuniform catalyst distribution, leading to inconsistent measurements. This study proposes an automated spray-coating method for depositing commercial iridium oxide (IrO_2) nanoparticles on RDEs as a reliable alternative. Spray-coating ensures uniform catalyst layers, significantly improving the reproducibility in electrochemical measurements. A comparison of the morphology and the electrochemical properties of the catalyst layers prepared by both methods identifies the sources of the performance variations. Requiring minimal catalyst loading (0.10 mg cm^{-2}), spray-coating establishes a standardized approach for OER catalyst evaluation, offering improved consistency and accuracy in electrochemical characterization.



KEYWORDS: *rotating disk electrode (RDE), oxygen evolution reaction (OER), spray-coating, drop-casting, coffee-ring effect, reproducibility, iridium oxide (IrO_2)*

1. INTRODUCTION

The oxygen evolution reaction (OER) is an electrochemical process that occurs at the anode of an electrolyzer cell, where the sluggish kinetics critically governs the overall efficiency of water splitting by limiting the achievable rate of hydrogen production at a given cell voltage. The development of highly active electrocatalysts for the OER remains a significant challenge, as the inherently slow reaction kinetics of this process are a primary factor limiting the overall efficiency and hydrogen production rate in both alkaline water electrolyzers (AELs) and anion exchange membrane electrolyzers (AEMs).[1−3]

The rotating disk electrode (RDE) configuration has been widely employed over the past few decades for half-cell testing in the evaluation of the OER electrocatalysts. This method can actively control the thickness of the diffusion layer by varying the rotation speed. This enables the separation of mass transport from redox kinetics. As a result, the OER activity can be directly correlated with the intrinsic kinetic activity of the catalyst at the working electrode. Compared to gas diffusion electrode (GDE) configurations in membrane electrode assemblies (MEAs), the RDE configuration requires less preparatory effort and time while allowing precise measurements of OER catalyst activity without interference from external factors.[4] Among RDE techniques, the thin-film RDE (TF-RDE) method has been extensively established as a reliable approach for catalyst screening.[5−7]

In practice, achieving an ideal thin-film catalyst layer on RDEs is challenging due to issues such as a nonuniform surface distribution, "coffee-ring" effects, and catalyst layer aggregation.[8,9] These physical inconsistencies lead to overlapping OER activity signals, large errors in linear sweep voltammetry (LSV) results, and misinterpretation of the OER activity data. Uniformity of catalyst films prepared using TF-RDE has been identified as a critical factor in ensuring reproducibility and precise OER activity measurements.[10,11] Several researchers have proposed modifications to TF-RDE methodologies to fabricate more uniform thin-film surfaces.[12−16] These include rotating the RDE during ink deposition or adjusting catalyst ink formulations.[17] However, these modifications are still based on the fundamental drop-casting technique in which the electrocatalyst ink is manually dropped onto the conducting area. Despite these efforts, the "coffee-ring" phenomenon continues to occur, making it challenging to consistently

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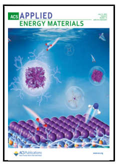


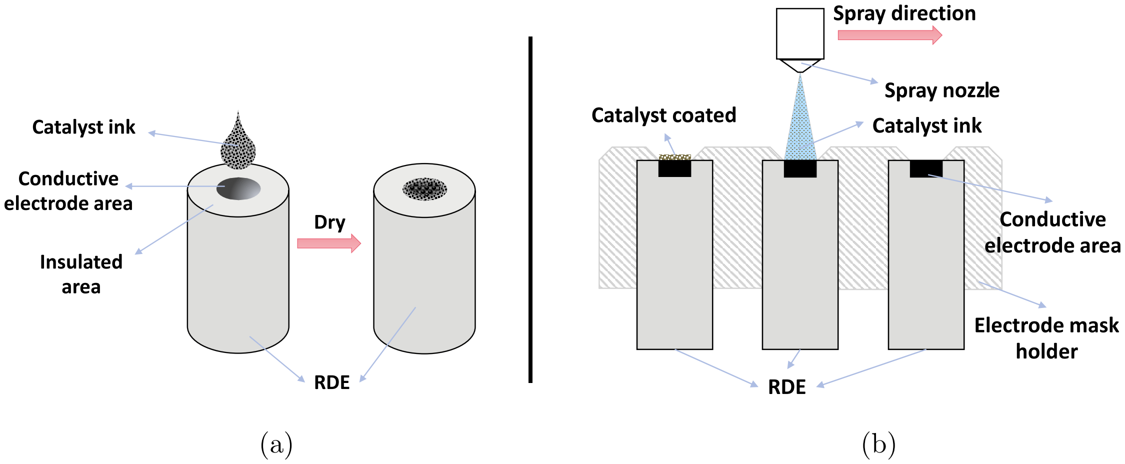


Figure 1. Schematic of (a) drop-casting (DC) and (b) spray-coating (SC) method of thin-film catalyst load on RDE.

optimize layering conditions.[18] This reliance on manual preparation results in variability that depends on individual skill and significantly increases the time required for the catalyst loading process. Inaba et al. introduced spray-coating as a method for preparing highly uniform, thin-film catalyst layers on RDEs for accurate determination of oxygen reduction reaction (ORR) catalysts and their mass activities based on electrochemical surface area (ECSA). Spray-coating has also been widely used for scaling up electrode fabrication in various research groups, including depositing catalyst layers on gas diffusion layers (GDLs) or MEAs in electrolysis cells.[19]

In this study, the primary objective is to examine the correlation between the reproducibility and uniformity of catalyst films on RDEs that contain glassy carbon as a conductive material. Our objective is to contribute to the standardization of TF-RDE preparation with the aim of achieving more accurate OER activity measurements by minimizing standard deviations associated with manual drop-casting. A comparative analysis of surface topography resulting from both deposition techniques, drop-casting and spray-coating, is conducted to validate the hypothesis that uniform catalyst distribution directly improves electrochemical reproducibility. We report further advancements in using spray-coating techniques to improve control over the physical roughness of IrO_2, which is one of the standard catalysts for OER,[20] thin films for the OER. The enhanced physical precision achieved through spray-coating leads to improved measurement reliability by reducing standard deviations and enhancing resolution in OER activity measurements for IrO_2 catalysts. Notably, spray-coating can simulate uniform surface conditions similar to those in MEAs while consuming less time and fewer resources, making electrocatalyst screening more efficient and realistic. Additionally, spray-coating enables thinner catalyst layers, providing more detailed insights into the mass activity. Furthermore, the specific double layer capacitance (C_{dl}) values at identical IrO_2 mass loadings provide insights about the active surface areas achieved with each technique, highlighting how spray-coating improves active surface areas through enhanced uniformity while addressing limitations inherent to conventional methods.

2. METHODOLOGY AND EXPERIMENTS

2.1. Preparation of the Rotating Disk Electrode (RDE).

2.1.1. Ink Preparation and Formulation. The rotating disk electrodes cited in this work were made with mirror-polished glassy carbon disks as conductive electrode materials. The ink formulation was controlled with 1 wt % of solid content, commercially available IrO_2 catalysts (Iridium(IV) oxide, Premion, 99.99% (metal base), Ir 84.5%), and 5% of Nafion solution (Nafion D-521 Dispersion, 5 w/w% in water and 1-Propanol) from Thermo-Scientific Chemicals, Germany, and controlled in a liquid matrix.[19] The Nafion content is 10% by weight of the catalyst amount, which is dispersed in a liquid content of 1:3 volume ratio between Milli-Q water and isopropanol. The catalysts were stirred vigorously for 30 min, then sonicated in a cooling bath for 30 min and completely dispersed using an Ultraturrax (IKA) for 10 min. In both processes, the catalyst ink was always stirred, while the catalyst is applied to the RDEs.

2.1.2. Thin-Film Catalyst-Loaded RDEs. The different procedures to prepare catalyst-loaded RDEs (5 mm diameter, glassy carbon rotating disk electrode, Pine Research, USA) are depicted in Figure 1. The RDE preparation by the conventional drop-casting (DC) method is controlled by a fixed amount of catalyst colloid in the catalyst ink. A droplet is manually loaded on the glassy carbon (GC) conduction area of the RDE by using a micropipet (see Figure 1a). The spray-coating (SC) technique uses an automatic spray-coating machine and software (ExactaCoat from Sono-Tex) with an injection rate of 0.3 mL min^{-1}. The machine applies the coating layer by layer at room temperature with nitrogen gas (N_2) as the carrier gas (see Figure 1b). The distance between the ultrasonic nozzle (120 kHz) and the substrate is fixed at 70 mm. To be able to control the position of the catalyst ink on the RDE during the spraying, a self-designed RDE holder has been made as shown in Figure S1, which works as a cover mask on the RDE to control the ink droplet precisely onto only the glassy carbon area of the RDEs. All of the prepared RDEs from both techniques are dried for 24 h under an ambient air condition. The catalyst mass loading of both techniques is adjusted at 0.05, 0.1, and 0.2 mg cm^{-2}, confirmed by weighing the final mass after drying by a microscale (Ultramicrobalance, XP6U, Mettler Toledo, Germany). The same batch of IrO_2 catalyst ink was used for both techniques of this study.

2.2. Surface Characterization. One of the most straightforward, nondestructive, and time-efficient methods for physically observing the IrO_2 catalyst layer on RDEs at the microscale is optical microscopy combined with laser scanning.

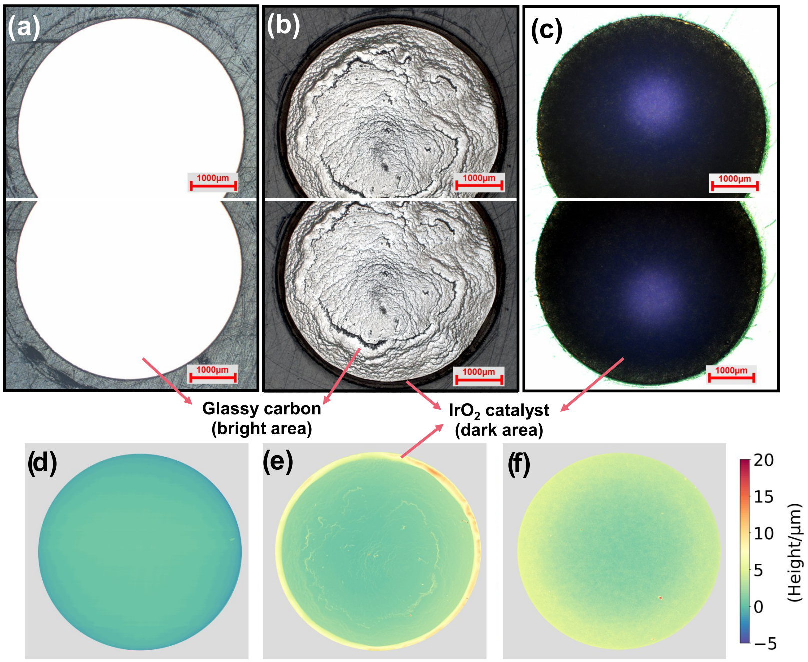


Figure 2. Optical microscopic images of RDEs with different catalyst loading techniques: (a) a mirror-polished glassy carbon (GC); (b) DC; and (c) SC. The dark area refers to the IrO_2 catalysts covered on the GC, whereas the bright area refers to the uncovered IrO_2 catalyst on the GC. Contour plot of the surface through laser scanning of (d) a mirror-polished, (e) DC, and (f) SC with a 0.10 mg cm^{-2} IrO_2 catalyst.

The 2-D top view images are taken with an optical microscope (Polarized light microscope, ZEISS Axio Imager 2, Germany) at 2.5× magnification (EC Epiplan-NEOFLUAR) and the surface roughness was analyzed on the basis of 3-D images taken with a laser scanner (CT-300 by cyberTECHNOLOGIES at ZEA-1, Forschungszentrum Jülich, Germany). Both techniques provide catalyst loading distribution information.

2.3. Electrochemical Characterization. Every catalyst-loaded RDE has been placed in a PTFE electrochemical cell with a volume of 175 mL. The RDE is operated with a rotation speed of 1600 rpm as the working electrode (WE) in a three-electrode system setup by using a Pt-mesh as the counter electrode (CE) and a reversible hydrogen electrode (RHE) as the reference electrode (RE) in a 1 M KOH solution as the electrolyte.[21,22] All electrochemical measurements are performed at a controlled room temperature of 21 °C ± 1 °C, using an electrochemical workstation (VSP potentiostat and EC-Lab software from Biologic). All RDEs are activated prior to any electrochemical measurement by performing cyclic voltammetry (CV) at a scan rate of 100 mV s^{-1} in the potential range of 1.0−1.7 V vs RHE for 5 cycles under argon saturation of the electrolyte to activate and remove impurities from the catalyst surface. Oxygen evolution reaction (OER) activity is measured by linear sweep voltammetry (LSV) at a scan rate of 5 mV s^{-1} in a potential range of 1.0−1.7 V vs RHE under oxygen saturation of the electrolyte. The uncompensated resistance (R_u) of each electrode is calculated from internal resistance values by an impedance measurement at high frequencies, 0.1 MHz and electrode potential of 1.8 V vs RHE. Postcorrection of the *iR* drop was applied manually by subtracting the product of current and R_u from recorded potentials. While this method assumes constant R_u, dynamic resistance change in nonuniform films may introduce residual ohmic losses. Scan rates were kept at 5 mV s^{-1} to mitigate these effects.

As the same IrO_2 was used for all experiments in this study, the double layer capacitance (C_{dl}) value is an important parameter to compare the active surface area of IrO_2 between the DC and SC techniques. The C_{dl} value can be determined by CV measurements at different scan rates. Scans with a rate of 10, 20, 40, 60, 80, and 100 mV s^{-1} are performed in the potential region of 0.9−1.0 V vs RHE in a 1 M KOH electrolyte (the same condition as the OER activity measurements), where the electrode exhibits solely capacitive behavior without redox reactions.

The purpose of the electrochemical measurements is to compare the results of electrochemical properties obtained by using two different preparation techniques for the catalyst layer on the RDE, namely, DC and SC. In the following, we will focus on the OER activity, the standard deviation of the performance, and the electrochemically active surface, using the same IrO_2 catalyst and mass loading.

3. RESULTS AND DISCUSSION

3.1. Surface Characterization. The surface characteristics of the different catalyst loading techniques can be visualized, especially under magnification. The optical microscopic images show the difference between a clean, mirror-polished glassy carbon area on an RDE (Figure 2a), IrO_2 catalysts loaded by DC (Figure 2b), and SC (Figure 2c). Due to the limitation of our optical microscope, it was not possible to capture the entire GC-RDE surface in a single image. Therefore, the surface of each electrode is fully captured by an upper and lower microscopic image, demonstrating uniformity of the IrO_2 catalyst.

The DC process resulted in an uneven catalyst distribution, where bright areas correspond to regions of the GC surface not covered by IrO_2, while darker areas indicate zones with a higher IrO_2 catalyst density. This suggests localized agglomeration of the catalyst. Notably, at the interface between the GC and PTFE regions of the RDEs, the IrO_2 catalyst forms a densely packed circular arrangement in a phenomenon known as the coffee-ring effect. While the catalyst applied by SC (Figure 2c) exhibits a uniform and even distribution, fully covering the GC area of the RDE (evident from the homogeneous dark regions), the contour plot of the surface analysis by means of laser scanning supports the above discussion. First, a clean mirror-polished surface of the RDE has an average surface height of less than 0.40 μm (Figure 2d). The distribution of the IrO_2 catalyst on the RDE (Figure 2e) results in an agglomerate and nonuniform catalyst layer, which is due to the "coffee-ring" effect by DC. Figure 2f, on the other hand, shows that the IrO_2 catalyst deposited by SC exhibits a smooth surface with a uniform thickness. Figure 2e and 2f are presented to clearly show the uniformity of the catalyst distribution for each loading technique alongside the corresponding optical microscopy images.

In contrast, Figure 3a and 3b displays the same surfaces as Figure 2e and 2f but includes the laser scanning measurement parameters where the inner dashed line circle refers to P1 and the outer thick line circle refers to P2. This surface analysis reported in Table S1 shows the values for quantifying the surface roughness and height variation compared to both techniques when analyzing the different areas (P1,P2 and the difference between both points). With an IrO_2 catalyst mass loading of 0.10 mg cm^{-2}, the DC method yields an average from P1 and P2 of an arithmetic mean height (Sa), a measure of the surface roughness defined as the absolute height deviation from a mean plane, of 2.39 μm, while, the SC method yields 3.63 μm. This lower average Sa value of DC signals an uneven catalyst distribution, where the catalyst is minimally present in the center and clumps strongly at the edges of GC, forming a characteristic "coffee-ring" pattern. This evaluation is supported by the difference of Sa values between P1 and P2 of DC and SC that are 0.75 and 0.59 μm, respectively, indicating a more uniform IrO_2 catalyst distribution and consistent thickness on SC.

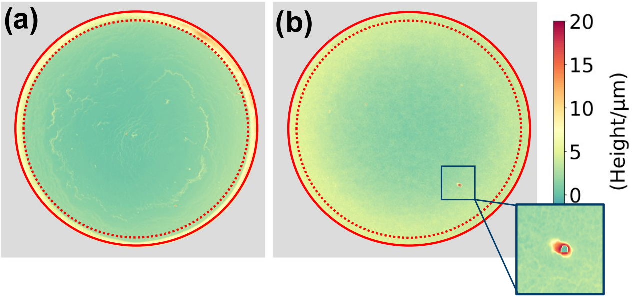


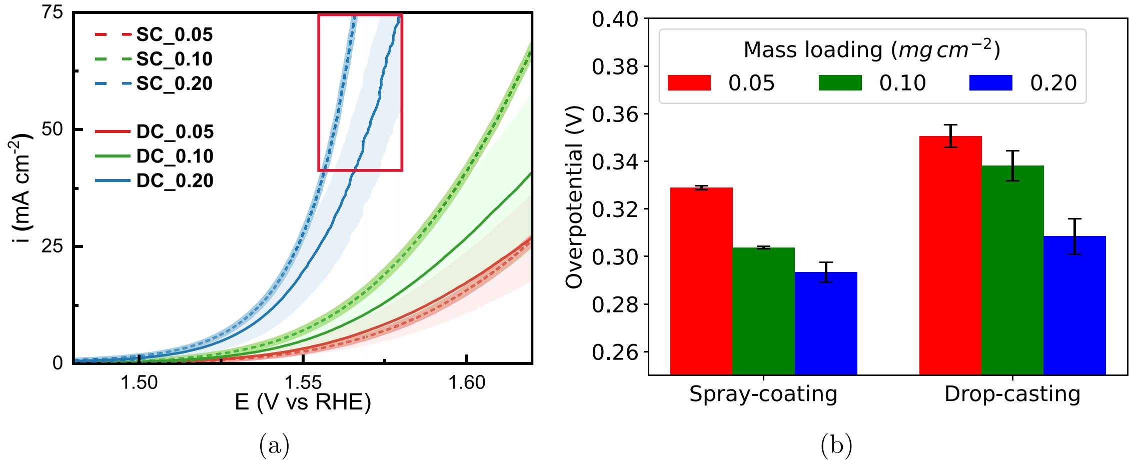
Figure 3. Contour plot of laser scanning with different TF-RDE preparations: (a) DC and (b) SC at 0.10 mg cm^{-2} IrO_2 catalyst. The dashed inner circle line refers to the area P1 and the thick outer circle line refers to the area P2 of the surface analysis parameters.

Further precision of the SC technique is shown by its maximum peak height (Sp) of 19.99 μm and maximum valley depth (Sv) of 14.44 μm, which are consistent across P1 and P2. This highest peak value is obviously from the scatter spot on the SC surface, as shown in the inset image of Figure 3b. These values are also lower than those of the DC method (Sp: 22.08 μm, Sv: 12.58 μm, with a difference of ~2 μm between P1 and P2). These results demonstrate the superior uniformity and surface smoothness achieved with the SC technique compared to DC.

3.2. Electrochemical Measurement.

The OER activity of the commercially available IrO_2 electrocatalyst is measured by linear sweep voltammetry (LSV), as shown in the legend of Figure 4a. For the same IrO_2 catalyst mass, the SC method demonstrates lower onset potential and higher current density compared to DC films at both 0.10 and 0.20 mg cm^{-2}. This enhancement is attributed to the uniform morphology of SC, which optimizes hydroxide ion (OH^-) diffusion pathways and promotes the detachment of smaller oxygen (O_2) bubbles, thereby reducing active site blockage and local ionic resistance. In contrast, nonuniform surface films of DC trap larger bubbles and create regions of inefficient OH^- transport, increasing resistive losses and lowering apparent activity. Figures S10 and S11 document the SEM images of the discrepancy in morphology and its impact on the OER activity from both techniques.

At higher IrO_2 loading, DC is increasing the "coffee-ring" dimension as compared in Figure S7, leading to an increase in ionic resistivity by creating localized regions of the agglomerated catalyst. This agglomerated catalyst hinders OH^- diffusion due to an extended, tortuous pathway, while trapped O_2 bubbles obstruct the active site and elevate ohmic resistance. This combination of effects manifests as erratic



(a) (b)

Figure 4. (a) Average LSV of the filled area of standard deviation and (b) comparison of overpotential at the current density of 10 mA cm^{-2} of IrO_2 loading at 0.05, 0.10, and 0.20 mg cm^{-2} between DC and SC techniques.

fluctuations in DC 0.20 mg cm^{-2} film at current density 50−70 mA cm^{-2} (see the red frame in Figure 4a), consistent with intermittent bubble detachment in heterogeneous catalyst layers. These fluctuations arise from dynamic changes in uncompensated resistance R_u due to irregular bubble evolution, which static postcorrection cannot fully resolve. Meanwhile, the uniform film of SC 0.20 mg cm^{-2} ensures a shorter OH^- diffusion path and smaller, rapidly detaching bubbles, stabilizing the ionic pathway and current output. At low IrO_2 loading, 0.05 mg cm^{-2}, both methods show comparable OER performance. Here, minimal catalyst thickness reduces the coffee-ring phenomena in DC films (Figure S7), as thinner layers limit the agglomerate formation. This mitigates OH^- transport barriers and bubble adhesion, narrowing performance differences between the two techniques.

Furthermore, the filled area of the standard deviation of the DC and SC LSV curves in Figure 4a indicates that the DC method consistently exhibits a significantly higher standard deviation at every mass load compared to the SC technique. In contrast, the standard deviation of all SC samples remains relatively constant across different IrO_2 loadings. This discrepancy occurs because of the fact that the manual DC process is more prone to error, especially at low catalyst loadings, whereas the automatic and precise SC process ensures more uniform catalyst deposition. These results indicate that the SC technique provides higher precision in TF-RDE preparation, leading to greater reproducibility of the OER results compared to DC. The standard deviation (error bars) of the average LSV results can interfere with the interpretation of the OER activity, as can be seen from the overlapping data of DC 0.05 and 0.10 mg cm^{-2} in Figure 4a. This highlights the importance of considering both the LSV polarization curve and the error bars from repeated experimental measurements when comparing OER activities. Reducing the error bar through improved catalyst production techniques, such as transitioning from DC to SC, is crucial for ensuring more reliable and consistent data.

The comparison of the overpotential from both techniques was evaluated at the current density of 10 mA cm^{-2} with the error bar in Figure 4b. The increase in mass load reduces the overpotential for both techniques. However, for every load of SC, the overpotential and error bar are lower than for the same mass load for DC. This supports the improved accuracy of SC over that of the DC. The Tafel analysis derived from the *iR*-corrected LSVs (Figure S12) shows similar slopes for both techniques, but at 10 mA cm^{-2}, there are notable differences in the overpotential, even though this current density remains within the kinetic region.[23] Figure S13 presents the corresponding Nyquist plot, which illustrates the overall effect of the charge transfer resistance (R_{ct}) of both techniques. As the mass loading increases, the semicircles decrease in size. However, at the same mass loading, the DC semicircle is larger than the SC semicircle. This indicates that SC is more efficient and has better kinetics in the OER process.

For the mass loading of IrO_2, the absolute double layer capacitance (C_{dl}) between SC and DC is extracted from the plot of current density versus applied scan rates (Figure 5). The results show that the SC technique yields nearly twice the absolute C_{dl} value compared to the DC method, indicating a higher active surface area for the SC. This observation is in line with our surface characterization data from laser scanning and optical microscopy, which revealed that the SC technique produces a more uniform arrangement of the IrO_2 catalyst on the GC surface than DC.

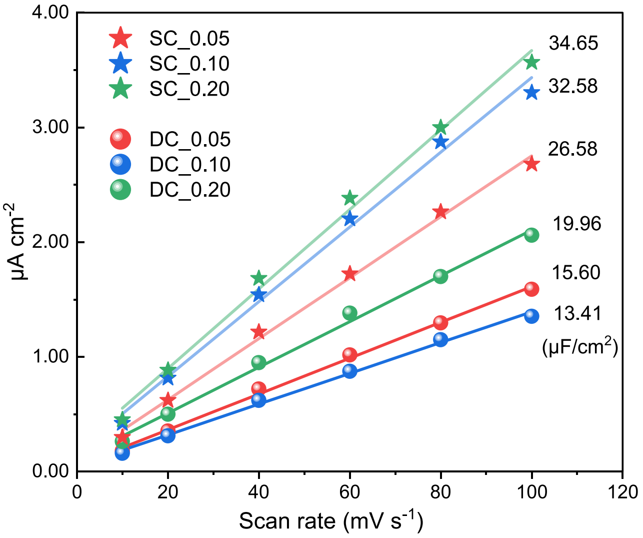


Figure 5. Double layer capacitance (C_{dl}) values of SC and DC of 0.05, 0.10, and 0.20 mg cm^{-2} from CV measurements at a scan rate of 10, 20, 40, 60, 80, and 100 mV s^{-1} in the potential range of 0.9−1.0 V vs RHE in 1 M KOH.

For both techniques, an increase in the IrO_2 catalyst mass loading leads to an increase in the C_{dl} value. However, an exception is observed with DC films at 0.10 mg cm^{-2}, which display the lowest C_{dl} value despite having a higher nominal loading. This unexpected result highlights the critical influence of film morphology and catalyst distribution, especially at low loadings. For DC films at these ultralow loadings, the catalyst tends to form sparse, agglomerated islands rather than continuous layers, as seen in optical microscopy (Figure S7). Doubling the nominal load can actually prevent clustering, creating disconnected regions that hinder ionic access and reduce the effective active surface area. Moreover, at such low loading, the measured C_{dl} is strongly affected by the GC substrate, which can also dominate the capacitance signal, and by local heterogeneity, it is the catalyst layer that distorts the CV measurements. The difference in C_{dl} between 0.05 and 0.10 mg cm^{-2} of DC films is very small (on the order of 2 μF cm^{-2}), and in combination with large standard deviations and overlapping average values of the LSV result (Figure 4a), underscores the unpredictability and limited reliability of C_{dl} measurements for nonuniform DC films. These observations emphasize that, particularly for DC films at low loadings, the nominal catalyst mass does not reliably translate to increased active surface area or improved OER performance. Instead, film morphology and catalyst distribution play a far more decisive role in determining both C_{dl} and apparent electrocatalytic activity. This finding highlights the challenges of using C_{dl} as a metric for ECSA in discontinuous catalyst systems and underscores the need for careful morphological characterization when interpreting the results of the OER at low loadings.

4. LIMITATIONS

This work has several limitations. The SC technique, while providing uniform catalyst layers, currently results in significant catalyst loss due to overspray and requires a larger volume of ink compared to DC. However, the SC protocol is still under development, and future improvements, such as optimizing the nozzle or redesigning the holder, could help reduce this material loss. In contrast, the more material-efficient DC method faces challenges at low mass loadings, where the nonuniform catalyst distribution and the coffee-ring effect can compromise the accuracy and reproducibility of the electrochemical measurements. Additionally, the use of static postcorrection for *iR* compensation may not fully capture dynamic resistance changes, particularly in nonuniform DC films, potentially affecting the reliability of the results. Overall, both techniques have trade-offs that impact catalyst utilization and data accuracy, highlighting the need for further protocol optimization.

5. CONCLUSIONS

This study aims to optimize the first step to standardize RDE test protocols through the spray-coating method, which improves the electrochemical performance of thin-film IrO_2 catalysts, especially in the OER region. By eliminating the coffee-ring effect and evenly distributing the catalysts on the GC-RDE surface, spray-coating minimizes mass transport limitations and improves the data reliability. We identified an optimal IrO_2 mass loading of 0.10 mg cm^{-2} for this method, which ensures high performance while reducing the standard deviations of the LSV results. Furthermore, we investigated the causes of error and variations in RDE systems and illustrated the critical relationship among the deposition method, the layer morphology, and the electrochemical performance. These results highlight the importance of accurate TF-RDE preparation and the need for a standardized approach that enhances reproducibility and accuracy in electrochemical studies and provides a solid foundation for the evaluation of OER catalysts.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsaem.5c00762.

> Additional optical microscopy, contour plot, parameter details from laser scanning, SEM images, Tafel and Nyquist plot (PDF)

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Notes

The authors declare no competing financial interest.

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