

**Unveiling the Origins of Activity Gap between Rotating Disk
Electrodes and Membrane Electrode Assemblies: Pt Seed-Mediated
Iridium-Doped Octahedral Platinum Nickel Catalysts for Proton
Exchange Membrane Fuel Cells**

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Experimental part

Chemicals and materials

Platinclorid (IV) hexahydrat [$\text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$], Platinum (II) acetylacetonate [$\text{Pt}(\text{acac})_2$](Pt 48.0% min), Nickel (II) acetylacetonate [$\text{Ni}(\text{acac})_2$](95%), Iridium (III) acetylacetonate [$\text{Ir}(\text{acac})_3$], Vulcan® XC 72R, Ketjen Black (KB300JD), benzoic acid(BA, $\geq 99.5\%$), N,N-dimethylformamide (DMF, 99.8%). For washing, ethanol (VWR, absolute, $\geq 99.8\%$), acetone (Sigma-Aldrich, $\geq 99.5\%$) and ultrapure water (Milli-Q, 18.2 M Ω)

Synthesis of oh-PtNi/C

$\text{Pt}(\text{acac})_2$ (48.0 mg, 122.0 μmol , 1.31 equiv. relative to $\text{Ni}(\text{acac})_2$), $\text{Ni}(\text{acac})_2$ (24.0 mg, 93.4 μmol , 1.00 equiv.) and benzoic acid (360 mg, 2.95 mmol) were weighted into an autoclave glass liner with a magnetic stirring bar. A suspension of carbon (80 mg) in DMF (40 mL) was filled in a centrifuge tube and prepared by strong ultrasonication (Branson Sonifier® SLPe, for 15 min). The suspension was added to the autoclave glass liner and the mixture was put in an ice bath under ultrasonic treatment for 30 min. Afterwards, it was moved into an autoclave (100 ml/100 bar Model I Roth) which sealed with a polytetrafluoroethylene (PTFE) ring. The mixture was heated to 160 °C and reacted for 12 hours while stirring. After cooling down, the autoclave was opened and $\text{Pt}(\text{acac})_2$ (12.0 mg, 30.5 μmol), $\text{Ni}(\text{acac})_2$ (6.0 mg, 23.3 μmol) were weighted and mixed in 5 ml of DMF and dissolved by sonication for 5 min. After dissolution, it was transferred in to the glass liner and ultrasonicated (sonication bath) for 40 min. Afterwards, it was transferred to the autoclave and the mixture was heated to 180 °C for 48 hours. The autoclave was cooled down to room temperature and the product was washed and dried in the vacuum freezer dryer overnight.

The washing was carried out by washing with acetone/ethanol, acetone/ethanol, ethanol/water and water. To separate the phases, the liquid mixture was centrifuged with centrifuger under 7600 rpm.

Scaled-up seed-mediated synthesis of oh-PtNiIr/C (shown in Figure S4)

Synthesis of the Pt Seed

Platinclorid(IV) hexahydrat [$\text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$] (58.0 mg) was weighed into a centrifuge tube, to dissolve the Platinum precursor, 1 mL mili-Q water was added. In the meanwhile, the carbon support (200 mg) was humidified with 3 mL isopropanol and 2 mL mili-Q water, then the carbon suspension was transferred to a horn sonifer to better distribute and mix with the precursor solution prepared above. The solution was added dropwise into the carbon suspension. After addition the mixture was sonifered in an ice bath for 15 min to get better distributed. The tube contained the carbon and platinum precursor was immediately moved into a liquid nitrogen bath when the sonifering process finished and the freezing lasted for 30 min. This step makes sure that the precursor was well distribution onto the carbon support. Then the frozen mixture was transported into a freezer dryer with overnight drying.

After drying process, the powder was granulated with an agate mortar and then transferred into a furnace tube. The furnace tube was mounted into a vertical furnace with a shaker, which enabled the dispersion while heating up. The reaction took place in 4% H_2 /Argon atmosphere under 200 °C for 2 hours. When the reduction was done, the powder was used as carbon with Pt seed for the further synthesis.

Pt(acac)₂ (288.0 mg, 732.0 μmol, 1.31 equiv. relative to Ni(acac)₂), Ni(acac)₂ (144.0 mg, 560.4 μmol, 1.00 equiv.) and benzoic acid (2160.0 mg, 17.7 mmol) were weighted into an autoclave glass liner with a magnetic stir bar. A suspension of carbon (80 mg) in DMF (40 mL) was filled in a centrifuge tube and prepared by strong ultrasonication (Branson Sonifier® SLPe, for 15 min). The suspension was added to the autoclave glass liner and the mixture was put in an ice bath under ultrasonic for 30 min. Afterwards, it was moved into an autoclave (100 ml/100 bar Model I Roth) which sealed with a polytetrafluoroethylene (PTFE) ring. The mixture was heated to 160 °C and reacted for 12 hours while stirring. After the cooling down, the autoclave was opened and Pt(acac)₂ (12.0 mg, 30.5 μmol), Ni(acac)₂ (6.0 mg, 23.3 μmol) and Ir(acac)₃ (8.8 mg, 18.0 μmol) were weighted and mixed in 5 ml of DMF and sonicated for 5 min to dissolve. After dissolution, it was transferred in to the glass liner and ultrasonicated (sonication bath) for 40 min. Afterwards, it was transferred to the autoclave and the mixture was heated to 180 °C for 48 hours. The autoclave was cooled down to room temperature and the product was washed and dried in the vacuum freezer dryer overnight.

Synthesis of the Ir seed

59.3 mg IrCl₆·xH₂O were dissolved in 1mL ultrapure water and slowly added to 200 mg carbon support distributed in 6 mL isopropanol. The slurry was homogenized via a horn sonifier (Branson, 6 W) for 15 min in a cooling bath. The suspension was immediately placed in liquid nitrogen for rapid freezing. After 20 min, the completely frozen product was placed in the freeze dryer overnight. The resulting dry product was grounded to powder and then placed in a fluidized bed tube furnace and reduced by flowing hydrogen gas under 200 °C (5 K min⁻¹) for 2 h to become the final Ir seeds.

XRD

X-ray diffraction patterns were collected on a Bruker D8 Advance (Siemens KFL Cu 2K X-ray tube and Lynx Eye Detektor) diffractometer in Bragg Brentano geometry using a Cu Kα1. The patterns were recorded between 20-90° with a step size of 0.04°.

ICP-OES

An inductively coupled plasma optical emission spectroscopy (ICP-OES) analysis was used to determine the elemental composition of the synthesized particles using a 715-ES-ICP analysis system (Varian). The selected wavelengths for the concentration determination were 203.604; 212.863; 214.424; 217.468; 265.945; 306.471 nm for Pt. For Ni they were 216.555; 221.648; 222.486; 227.021; 230.078; 230.299 nm. For Ir were 204.419; 205.116; 212.618; 224.268 nm.

TEM

Transmission electron microscopy (TEM) images were recorded on a FEI Tecnai G2 20 S-TWIN with a LaB6 cathode operating with 200 kV acceleration voltage and a resolution limit of 0.24 nm. Samples were dispersed in isopropanol and drop casted on a Cu grid (400 mesh).

RDE

Ink preparation

To prepare an ink 2.5 mg of catalyst with 20 Pt wt% were added into 1.99 mL water, 5 μL Nafion™ (5 wt%) and 0.5 mL isopropanol (≥ 99.5 %) and horn ultrasonicated (Branson Sonifier 150) for 30 minutes. For the catalysts with 30 Pt wt%, around 1.7 mg powder was added into the identical ink solvent

recipe and dispersed the same way. 10 μL of the ink were placed on a glassy carbon (GC) rotating disk electrode (diameter $\varnothing=5$ mm), which results in a Pt loading of 10 $\mu\text{g cm}^{-2}$. The film was dried at 50 $^{\circ}\text{C}$ for 10 minutes.

Electrochemical characterization

For electrochemical characterizations, a conventional three electrode cell with a Pt mesh as counter electrode (Pt furled mesh Pt 5x5 cm^2), a reference electrode (MMS Hg/Hg₂SO₄ with the calibrated potential $E = -0.725 V_{\text{RHE}}$) and a glassy carbon working electrode was used. The working electrode was always immersed into the electrolyte under potential control at 0.05 V_{RHE} . A 0.1 M HClO₄ was used for electrochemical measurements (diluted from 70 % conc. HClO₄, 99.999 % trace metal bases, Sigma Aldrich with milli-Q water). All measurements were performed with a BioLogic Science Instruments potentiostats SP-150 and SP-200. The degree of purity for the gases used was 99.998 % for oxygen, 99.999 % for nitrogen, and 99.999 % for hydrogen (used for the reference electrode calibration).

Activation step and H_{upd}-ECSA evaluation

GC working electrode potential was lowered at 0.05 V_{RHE} while rotating at 1600 rpm and the rotation was stopped when in position without bubbles on the electrode surface.

The electrochemical activation was performed via potential cycling between 0.05 and 0.925 V_{RHE} with a scan rate of 100 mV s^{-1} 50 times under nitrogen atmosphere. the next 3 cycles were applied within the same potential range at 20 mV s^{-1} . The third cycle of the 20 mV s^{-1} scan was used to determine the electrochemical catalytic surface area (ECSA) from hydrogen under potential deposition (H_{upd}).

H_{upd}-ECSA Evaluation: the current of the cyclic voltammogram (CV) was background subtracted. The values were calculated by integrating cathodic sweeping between around 0.05 and 0.4 V. The integration range was between the cathodic currents corresponding to the capacitive background current and the current before the hydrogen evolution reaction onset (HER). The measured QH value was normalized with respect to the theoretical value of $Q_{\text{Htheo}} = 210 \mu\text{C cm}^{-2}$, which assumes a one electron transfer between one H atom and one Pt atom, forming a hydrogen monolayer and normalized by the scan rate.

Catalytic activity for oxygen reduction reaction

The catalytic activity of the catalysts was measured by linear sweep voltammetry. The measurement of the background current was conducted in a potential range between 0.05 and 1.0 V_{RHE} with a scan rate of 20 mV s^{-1} and a rotation speed of 1600 rpm in nitrogen-saturated electrolyte (at least 15 min bubbling). The scan was repeated once. Then the working electrode was elevated above the liquid and oxygen was bubbling through the electrolyte for at least 15 min. Then the test was performed in oxygen saturated electrolyte in the same potential range with a scan rate of 20 mV s^{-1} for 3 times and a scan rate of 5 mV s^{-1} once under a rotation speed of 1600 rpm. The kinetic currents were calculated using the Koutecky-Levich equation,

$$1/j = 1/j_k + 1/j_d$$

where j was measured at 0.9 V_{RHE} and j_d was determined in the diffusion-limited current area and j_k is the calculated kinetic current density. Both currents obtained from the test were IR corrected firstly, where the resistance R was determined by potential electrochemical impedance spectroscopy at 0.5 V_{RHE} , and then subtracted with the background current, which was the current from the cathodic voltammetry sweeping in N₂ saturated electrolyte at 0.4 V_{RHE} . The mass activity was calculated based

on the kinetic current normalized by Pt weight loading and corresponding specific activity was normalized by the H_{upd} -ECSA.

Stability test

The stability test was conducted after the electrochemical activation, impedance spectrometry and activity measurement. Stability measurements have been performed via potential cycling between 0.6 and 0.925 V_{RHE} with a scan rate of 100 mV s^{-1} and in N_2 saturated electrolyte for 10k cycles without working electrode rotating. In the meanwhile, the N_2 was flown above the electrolyte level. After the stability test protocol, CV was applied to obtain the H_{upd} -ECSA in the catalyst state “after the stability test”: 3 times at 100 mV s^{-1} and 3 times at 20 mV s^{-1} . Then, impedance spectroscopy and the ORR test procedure were also implemented.

CO stripping

CO stripping experiments were performed following ORR activity test. For a sample tested with the stability protocol test, the CO stripping was performed following the ORR activity test after the stability test, not the ORR activity test before the stability test. This is because the surface composition may alter after the CO stripping, due to the interaction of surface Pt or Ni atoms with CO. Thus, the CO stripping is the last electrochemical test step in the RDE.

For CO stripping, 0.05 V_{RHE} was applied and the rotation was set to 400 rpm after bubbling N_2 for minimum 15 min. Then CO was bubbled for another 5 min at the same rotation speed. The gas was switched back to N_2 and the electrolyte was bubbled for another 10 min to remove CO in the electrolyte. Then the rotation was turned off after the removal of bubbles on the electrode surface. Then 3 cyclic voltammetry cycles were recorded at 20 mV s^{-1} between 0.05 V_{RHE} and 1 V_{RHE} , while venting N_2 above the electrolyte. The oxidation of surface-adsorbed CO (assumed as one monolayer) occurred during the first scanning CV, the corresponding oxidation was represented by the CO stripping peak. The IR corrected current of the first positive scan was deducted by one of the second positive scan, which was considered as the background current. Then the integral of the peak in the range where the current was positive was calculated and translated based on an assumption of one layer of CO, which occupied a charge of 420 $\mu\text{C cm}^{-2}$.

HT RDE test

The tests were performed using on V30 AND PT/C catalysts. The configuration of the experimental cell consisted of a jacket RDE cell, with 80 °C water circulating into and out of the jacket to keep the electrolyte heated. The working electrode preparation and ink recipe was the same as room temperature RDE to stay consistent. The electrolyte used was HClO_4 0.1 M. The Reference electrode was a RHE electrode. Other openings were covered by PARAFILM® or plugs. The RDE protocol and analysis were the same as adopted before in this study.

MEA fabrication

The membrane electrode assemblies (MEAs) used in this work consist of five layers. Nafion™ 1100EW (equivalent weight in g polymer/mol H^+) was used to fabricate thin-layer electrodes. The cathode catalyst layers were prepared at an ionomer/carbon weight ratio of ca. 0.8/1 ~ 0.9/1 and metal loadings of ca 0.10 and 0.20 $\text{mg}_{\text{Pt}} \text{cm}^{-2}$. The anode catalyst layer was kept constant at an ionomer/carbon weight ratio of ca. 1.5/1 and a metal loading of 0.1 $\text{mg}_{\text{Pt}} \text{cm}^{-2}$. The membrane used was a perfluorosulfonic acid type, fabricated at JMFC with a thickness of ca. 20 μm . Catalyst layers

were produced on a PTFE substrate and transferred via a decal method onto the membrane. Single cells (50 cm² active area) were assembled by sandwiching the catalyst coated membranes between the GDLs and applying an average compression onto the active area.

MEA operating conditions

The fuel cell station was built in-house at JMFC. Pure oxygen and synthetic air were used as cathode reactants and pure H₂ as the anode reactant (all gases of 99.9% purity). Stoichiometric flow rates of anode ($s = 2$) and cathode ($s = 9.5$ for O₂ and $s = 2$ for air) reactants were used at current densities >0.2 A cm⁻² and constant flows (corresponding to 0.2 A cm⁻² flows) at <0.2 A cm⁻². Reactant humidification was achieved by water-bubblers, the temperatures of which were calibrated to yield the desired relative humidity (RH) values. Humidity and cell pressure were measured at the inlet for both electrodes. Cell resistances as a function of current density (i.e., the sum of the proton-conduction resistance in the membrane and the various electronic resistances, bulk, and contact resistances) were determined using an AC perturbation of 1 kHz at three different current densities of 25, 50 and 100 mA cm⁻² and also using a current interrupt method. For each data point, the cell voltage was stabilized for 4 min where the current was measured. Multiple-path serpentine flow fields (two and three parallel channels for the anode and cathode, respectively) machined into sealed graphite blocks were used for testing.

The MEAs were conditioned by the application of a constant current density of 500 mA cm⁻² under H₂/Air at 50 kPa gauge, 100% RH and 80 °C. The cell voltage was monitored until a stable value was observed. The conditioning step lasted 2h unless specified otherwise. Afterwards, the cathode catalyst layer was exposed to a series of cathode starvation steps followed by 2h current hold at 500 mA cm⁻² until a stable voltage was observed. The cathode starvation step (purging of the cathode compartment with pure nitrogen) reduces the cathode voltage to below 0.1 V and it is intended to provide an electrochemical cleaning step for the cathode catalyst before measuring its activity under H₂/O₂. After the starvation steps the MEA was ready for testing by H₂/O₂ polarization curves for MA quantification (50 kPa gauge, 100% RH and 80 °C). The polarization curves were recorded from low (i.e., 0.05 A cm⁻²) to high current (i.e., 2 A cm⁻²) ascending direction and backwards, descending direction. The lower current density limit was determined by maintaining the stoichiometry, at even lower currents there would be a risk of working under over-stoichiometric conditions with the used setup. The current density was maintained for 3 minutes at each step and the MA value was obtained from the ascending polarization curve at 0.9 V by extrapolation, after correction for the cell ohmic resistance. For this study, H₂ crossover current densities were measured using the procedure described by Kocha et al.⁵. In this test the hydrogen that permeates through the membrane to the cathode is oxidized by the application of a voltage (typically 250-300 mV are sufficient, and the latest above 400 mV one is in the mass transport limit) and the resulting current measured. Therefore, the cell was operated under H₂/N₂ and the gas crossover measurements were done at each of the operating conditions (i.e., temperature and H₂-partial pressure). The catalyst mass activity was not corrected on the basis of H₂-crossover current densities, i_{eff} (i.e., $i_{\text{eff}} = i + i_x$). Under the conditions used in this work i_x was on the order of 2–5 A cm⁻². Performance curves in 50 cm² single cells were done with a three-way switch polarization curve in H₂ / (O₂, Helox and Air) at 100 kPa gauge-inlet and 80 °C. The cell current density was held for 10 min at each point and the cell voltage was averaged on the last minute. Cell performance in H₂/Air is plotted in the descending direction, from low currents to high currents. The cell resistance was calculated from the intercept at high frequency and the values obtained were 60±2 mOhm cm at 100 kPa gauge-inlet and 80 °C. Performance in H₂/Air was not iR corrected.

The ECSA was measured with the CO stripping method using the cell in half cell mode where the anode electrode acts as a pseudo reference electrode. The cathode voltage was controlled at 0.125 V at 80

°C, 100% RH and 50 kPa gauge whilst purging with 1% CO in N₂ at 300 ml min⁻¹ for 15 min. Afterwards the cathode was purged with N₂ at the same flow rate for 2 h to ensure that CO is removed from the bubblers and the catalyst layer pores. The adsorbed CO is oxidized electrochemically by scanning the cathode voltage from 0.125 V to 0.85 V and back to 0.05 V, at 20 mV s⁻¹ for three cycles. The area under the CO oxidation peak is integrated by subtracting the third scan from the first scan and using a 420 μC cm⁻² constant for a CO monolayer on Pt.

Floating electrode technique

The floating electrodes consisted of four components which are a polycarbonate track-etched membrane (PCTE membrane, Sterlitech, 0.4 μm pore size, 47 mm), a gold layer deposited on the PCTE by sputter coating (Quorum Q150T), a catalyst layer and a hydrophobic coating. After the sputter coating, the gold-coated PCTE membrane was exterminated to floating electrodes with 1 x 3 cm size followed by isopropanol and water washing each for 16h at 130 °C and 150 °C, respectively, in a Soxhlet extractor to remove the residual contaminant. After the washing process, the clean floating electrodes were dried and stored for further usage. The deposition of the catalyst layer with 2 mm diameter on a washed floating electrode was achieved by vacuum filtration. A homemade polychlorotrifluoroethylene (PCTFE) adapter was used as a mask with 2 mm diameter hole. 450 μL of the diluted ink above was pipetted in the PCTFE adapter and a PTFE air filter with 0.2 μL pore size sealed the vacuum filtration. The catalyst layer on the gold side was formed by vacuum filtration for ~10 min. Afterwards, the back side of the catalyst-deposited floating electrode was brushed with 7.5 μL of 0.25 wt.% Teflon AF2400 solution and then dried under vacuum at 70 °C overnight.

The FET measurements were performed in 1.6 M HClO₄ at room temperature by adapting a three-electrode half-cell. A mercury-mercurous sulfate electrode and a Pt-mesh were used as the reference electrode and the counter electrode, respectively. Before measuring the ORR activity, the floating electrode was conditioned by scanning CVs from -0.1 V_{RHE} to 1.0 V_{RHE} with a scan rate of 50 mV/s in H₂ and O₂ alternately for 8~10 loops, until the current density of hydrogen evolution/oxidation reaction (HER/HOR) and ORR overlapped and no more increased.^[2] Then CVs from 0.05 V_{RHE} to 1.0 V_{RHE} were measured in N₂ with a scan rate of 100 mV/s for determining the actual Pt-loading on the floating electrode by adapting the following equation:

$$L_{Pt, FET} = \frac{Q_{measured, FET}}{Q_{Hupd} \cdot ECSA_{RDE} \cdot S_{FET}}$$

Where,

$L_{Pt, FET}$ is the Pt loading on the floating electrode

$Q_{measured, FET}$ is the measured accumulative charge in the Hupd region with an unit of μC

Q_{Hupd} is the theoretical value for Hupd charge with 210 μC/cm_{Pt}²

$ECSA_{RDE}$ is the electrochemical surface area of catalysts measured in RDE with an unit of m_{Pt}²/g_{Pt}

S_{FET} is the catalyst layer surface area of the floating electrode with 0.0314 cm²

Afterwards, the ORR polarization curves were collected by scanning the potential from 0.1 V_{RHE} to 1.0 V_{RHE} with a scan rate of 10 mV/s in O₂.

Table S1: Compositional atomic % and Pt weight % data of non-seed-mediated oh-PtNi(Ir) obtained from ICP-OES

| | Composition | Pt wt% |
|---------------|---|--------|
| Oh-PtNi(Ir)/C | Pt _{67.8} Ni _{30.8} Ir _{1.4} | 21.2 |

Pt, PDF no.03-065-2868

Ni, PDF no.03-065-2865

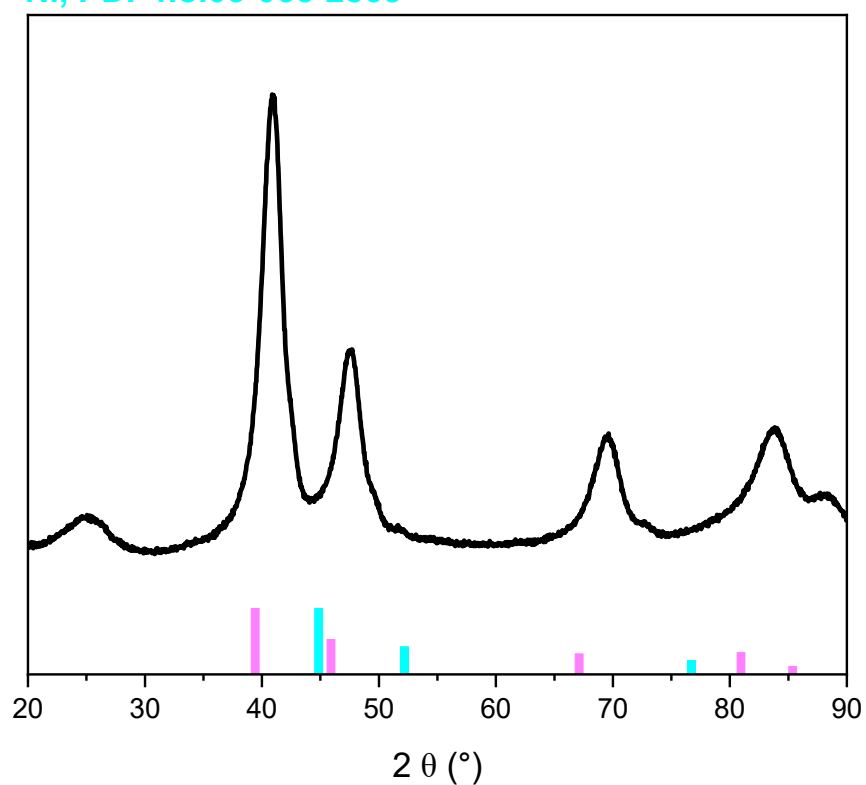


Figure S1: XRD patterns of non-seed mediated oh-PtNi(Ir).

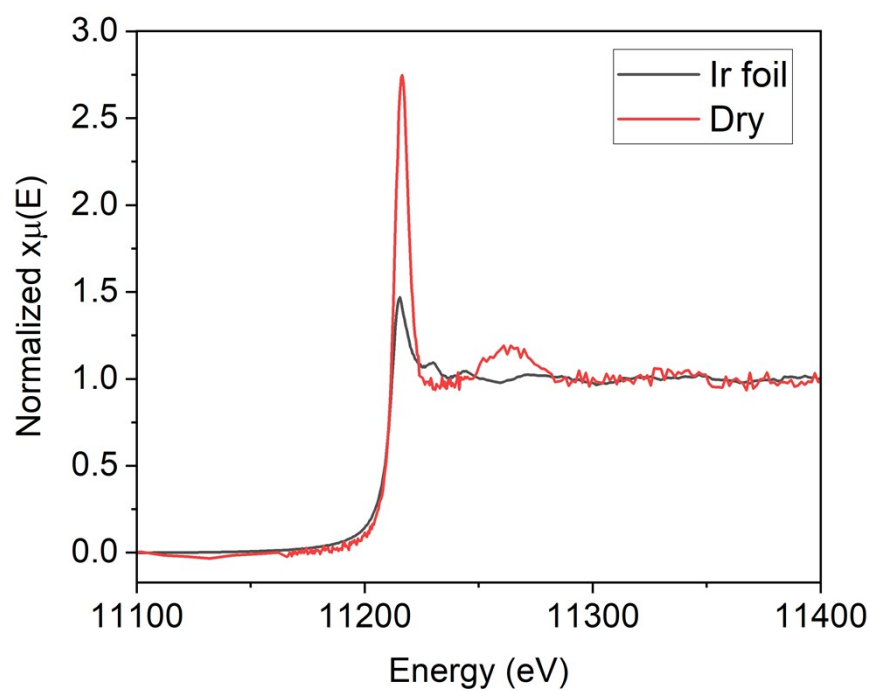


Figure S2: Electronic structure of Ir-doped PtNi octahedra. XAS of Ir-doped PtNi octahedra (red line) at the Ir L-edge compared to Ir foil (black).

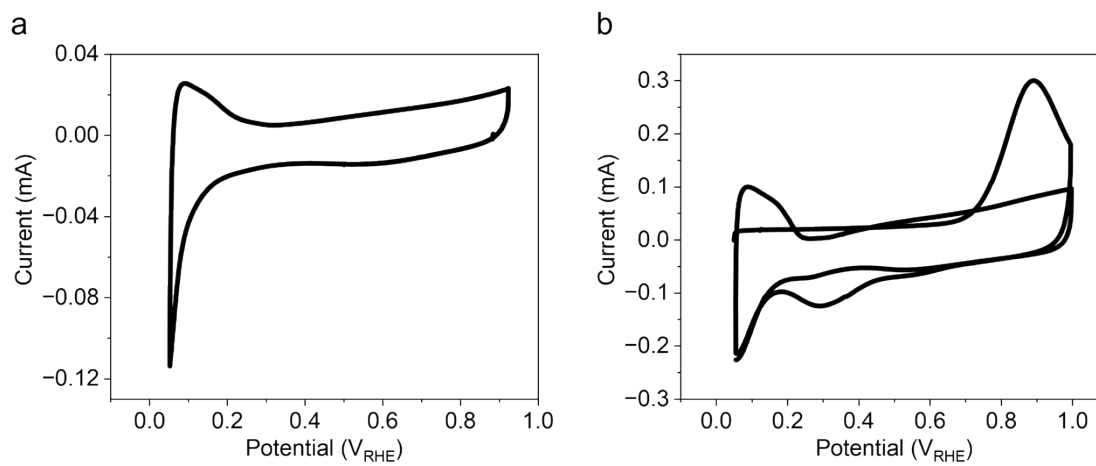


Figure S3: a) CV and b) CO stripping curve of Ir/C catalyst. Loading of Ir was 8.7 wt%.

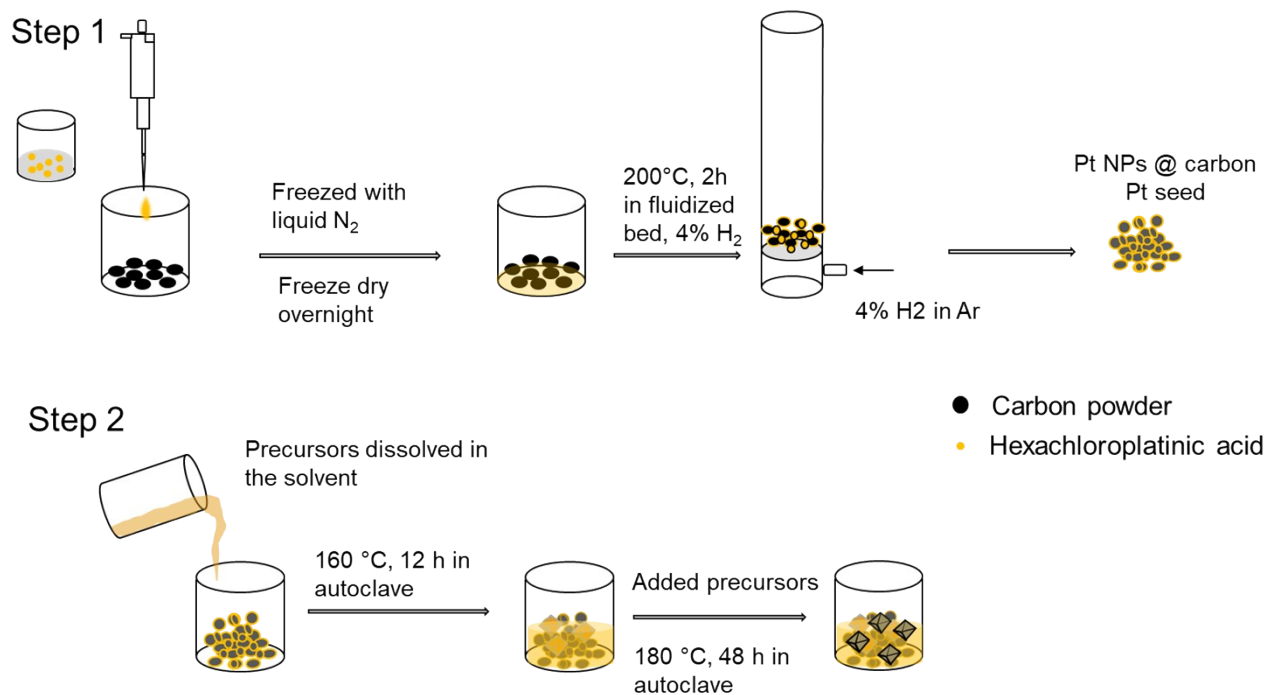


Figure S4: Schematic illustration of seed-mediated synthesis PtNi(Ir).

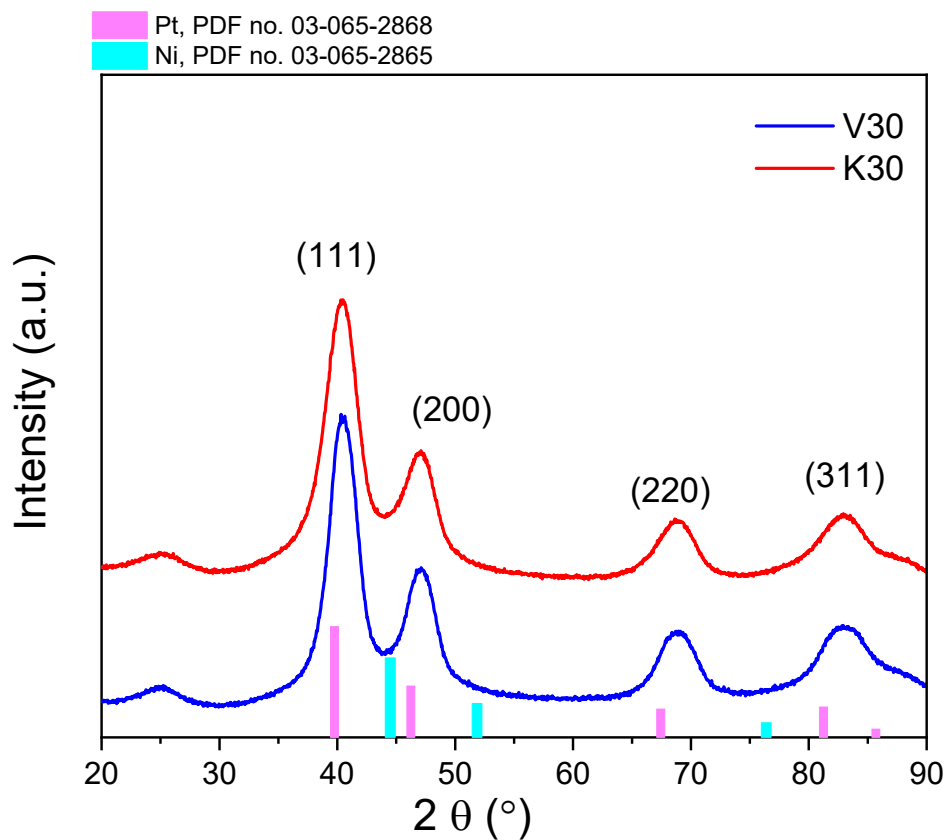


Figure S5: XRD patterns of V30 and K30.

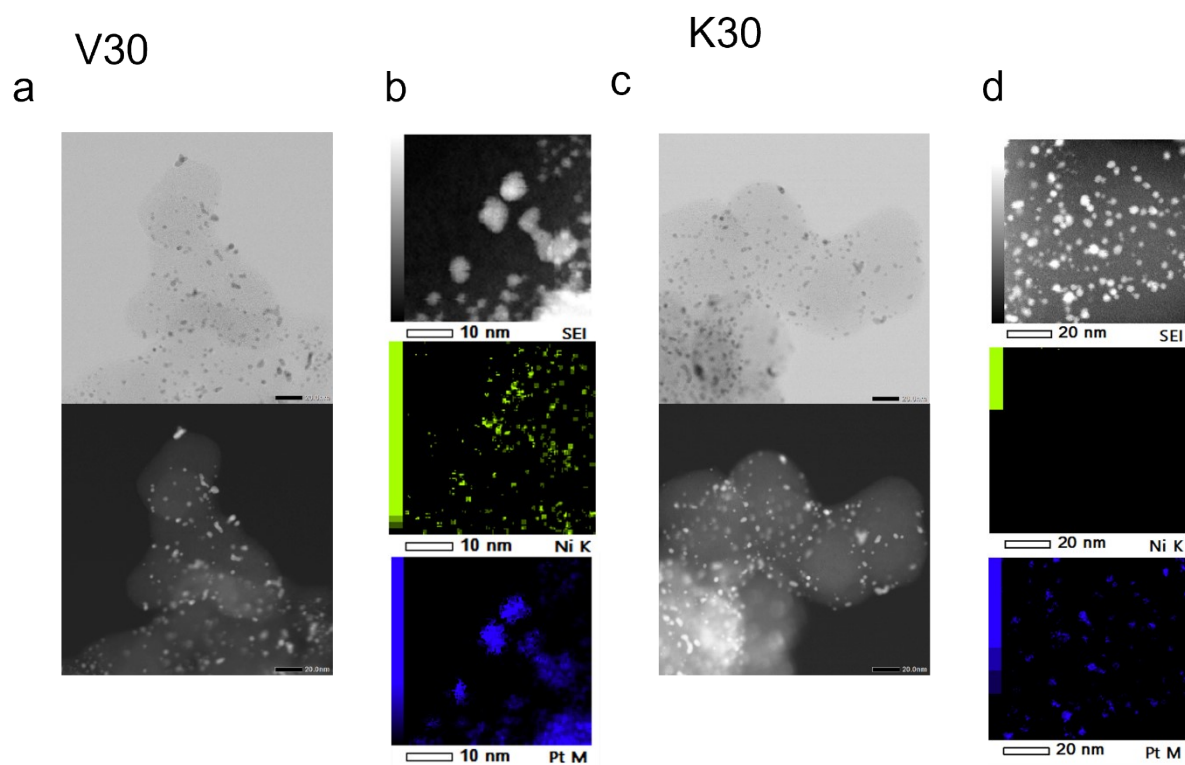


Figure S6: Elemental mapping TEM after MEA test. a) TEM of V30, b) elemental mapping of V30, c) TEM of K30, d) elemental mapping of K30.

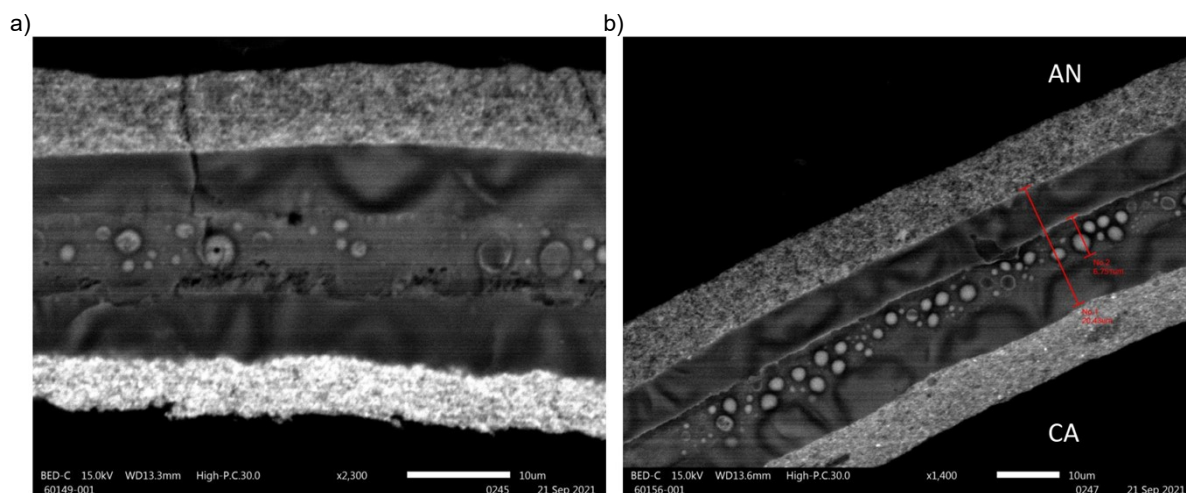


Figure S7: Cross-section SEM images for post-tested MEAs. a) V30 and b) K30. Layers on top were anode side and lower layers for cathode side. V30 catalyst layer thickness was 4.5 μm and for K30 was 8 μm .

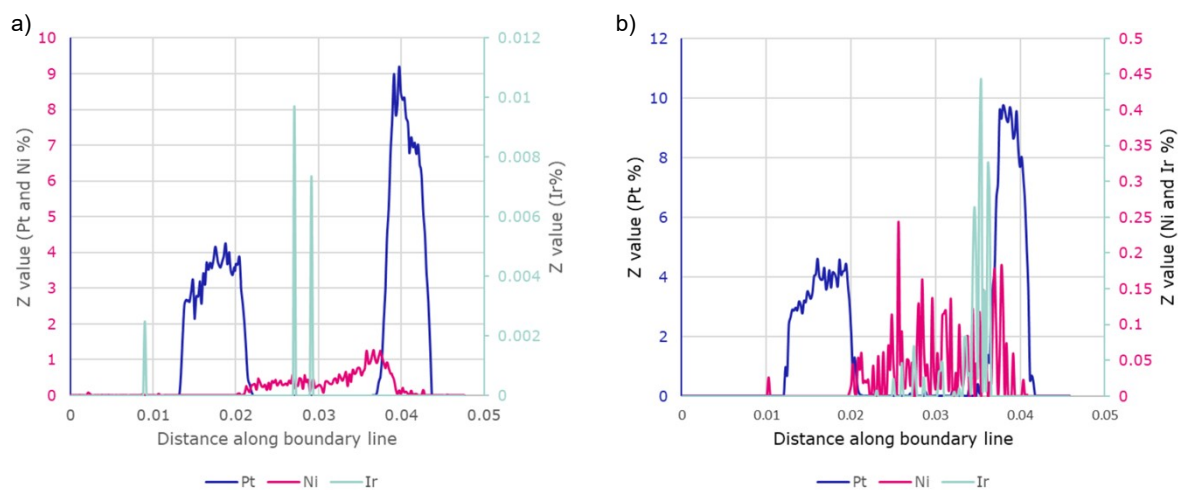


Figure S8: Electron probe microanalysis (EPMA) of cross section linescan data for post-tested MEAs. a) V30 and b) K30

Table S2: composition of V30 and K30 as prepared and after making into MEA ink according to ICO-OES

| | V30 As synthesized | V30 MEA ink | K30 As synthesized | K30 MEA ink |
|-------------|---|---|---|---|
| Composition | 30.1 Ptwt% $\text{Pt}_{68.2}\text{Ni}_{31.2}\text{Ir}_{0.5}$ | 19.7 Ptwt% $\text{Pt}_{70}\text{Ni}_{29.8}\text{Ir}_{0.2}$ | 25.6 Ptwt% $\text{Pt}_{72.8}\text{Ni}_{26.8}\text{Ir}_{0.4}$ | 17.5 Ptwt% $\text{Pt}_{74}\text{Ni}_{25.7}\text{Ir}_{0.3}$ |

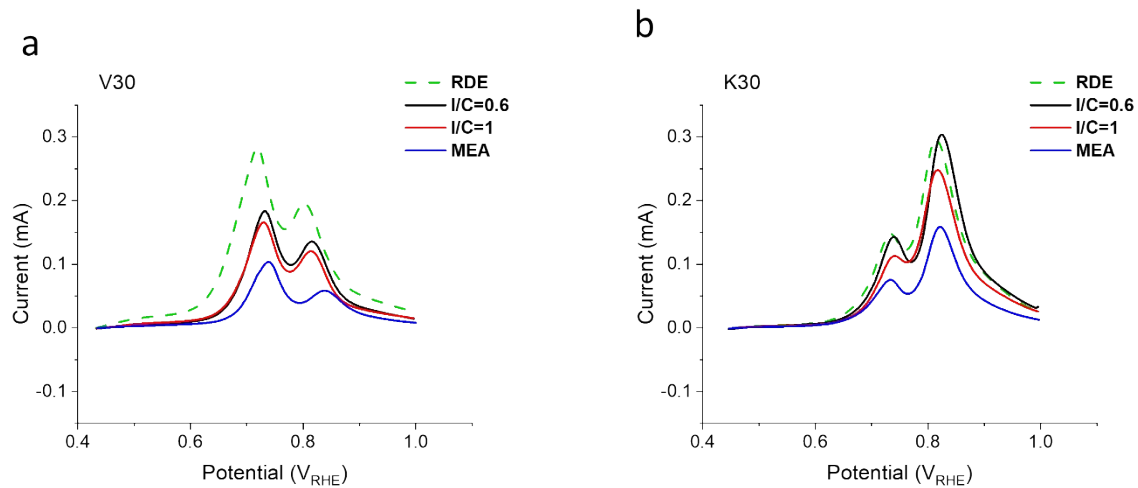


Figure S9: CO stripping curves of sample V30 and K30 with different ink recipes

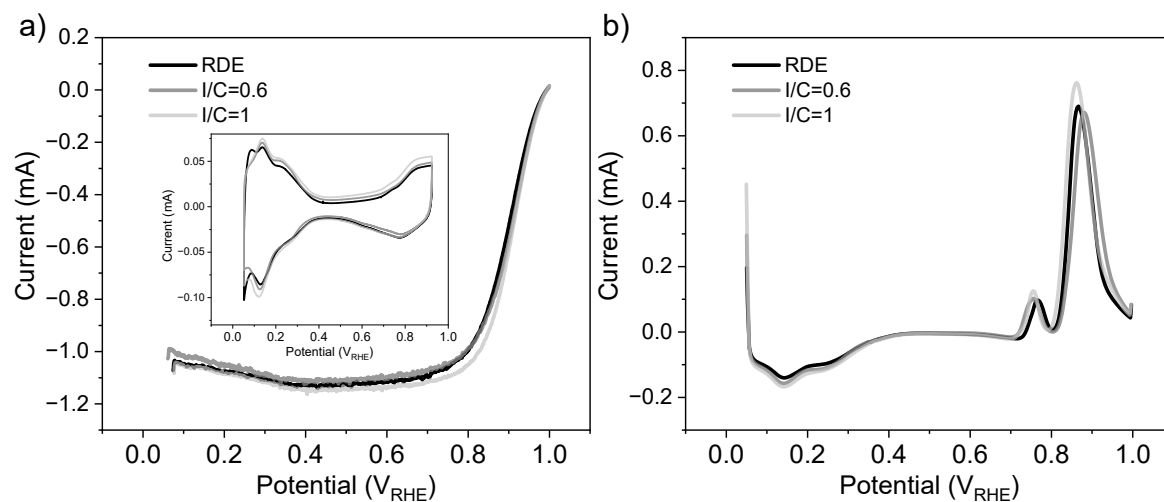


Figure S10: RDE data of Pt catalyst under room temperature. a) LSV and CV in the inset. b) CO stripping curves

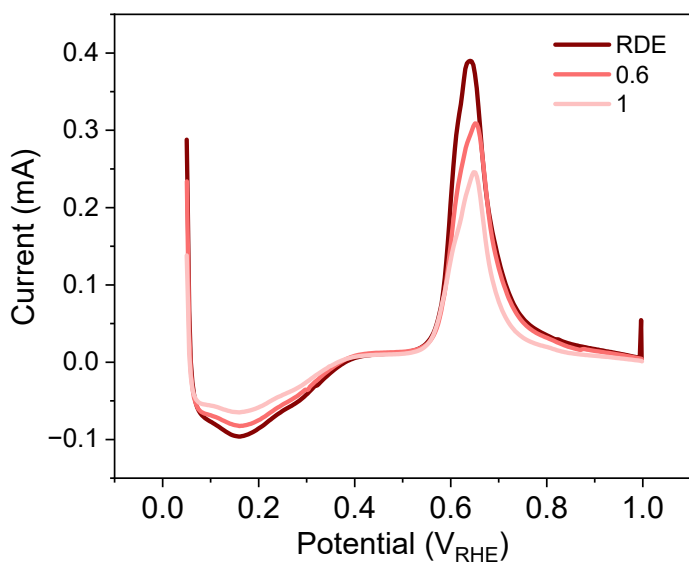


Figure S11: CO stripping curves of sample V30 with different ink recipes under high temperature

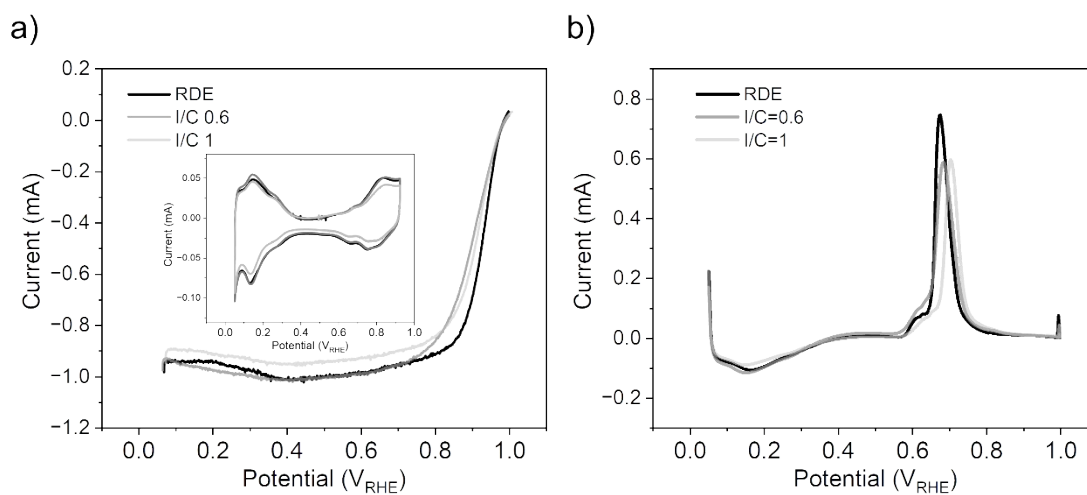


Figure S12: RDE data of Pt catalyst under high temperature. a) LSV and CV in the inset. b) CO stripping curves

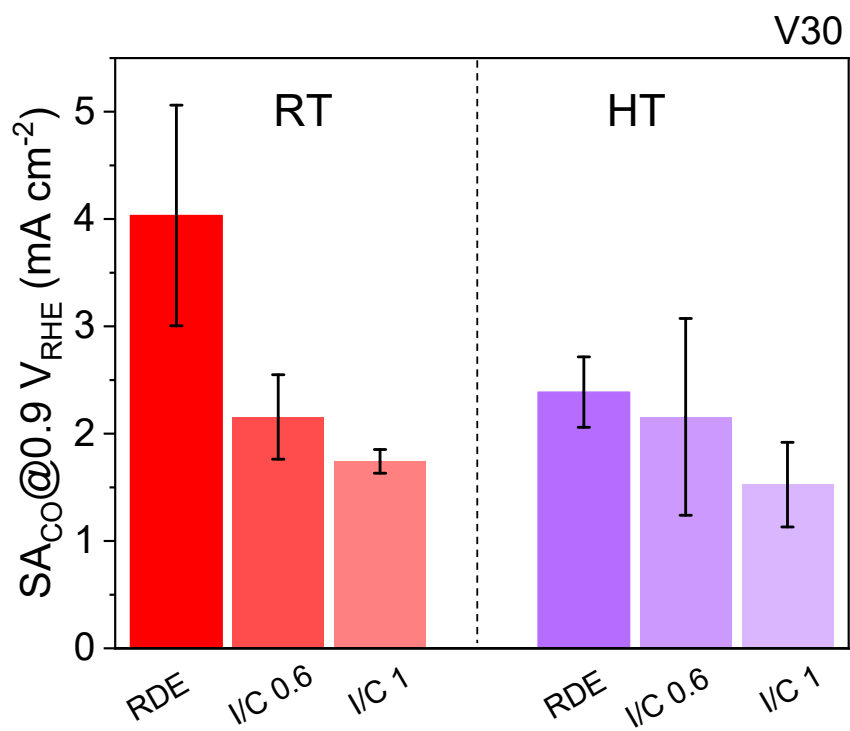


Figure S13: Specific activity of V30 under RT and HT