



# Influence of Electrochemical Activation on the Alkaline Oxygen Evolution Performance of NiCo Tungstates

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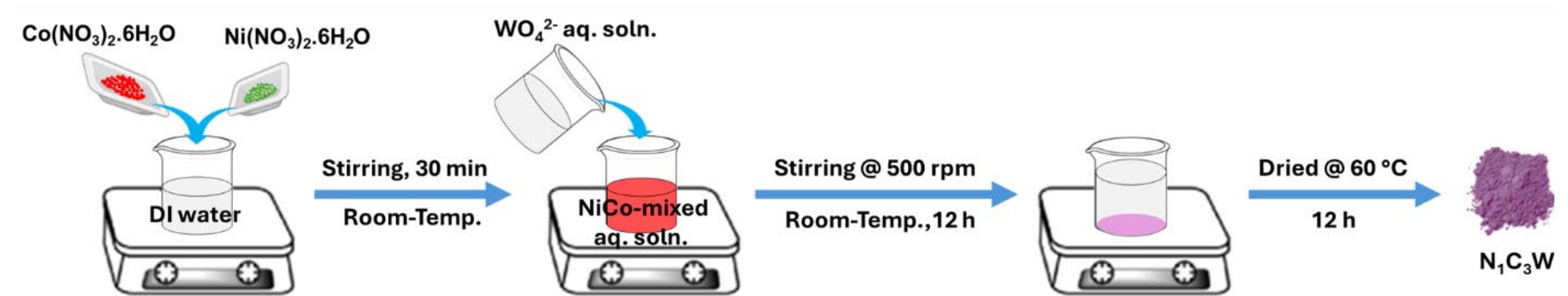
## Introduction

To catalyze the OER, choice of non-precious electrocatalysts is preferred over the precious platinum group metal (PGM)-based catalysts owing to their relatively higher abundance and cost-effectiveness. In lab scale research, the preliminary evaluation of OER electrocatalysts is usually performed using thin-film rotating disk electrodes (TF-RDEs) in a three-electrode system. In this process, the very first step involves multiple cyclic voltammetry (CV) cycles for better wettability of the catalyst layer to the electrolyte, which is considered as electrochemical activation (EA). Incomplete activation of the catalyst layer would lead to inconsistent OER performance of the electrocatalysts. This is much more pronounced in non-precious electrocatalysts compared to the precious ones in alkaline media. Despite its importance, EA is overlooked most of the time and rarely studied.

In this study, the critical role of EA is systematically investigated for nickel cobalt tungstate (NCW) in alkaline OER and compared to its monometallic counterpart, cobalt tungstate (CW) by using *in situ* electrochemical impedance spectroscopy (EIS).

## Experimental

### Co-precipitation synthesis of NCW:



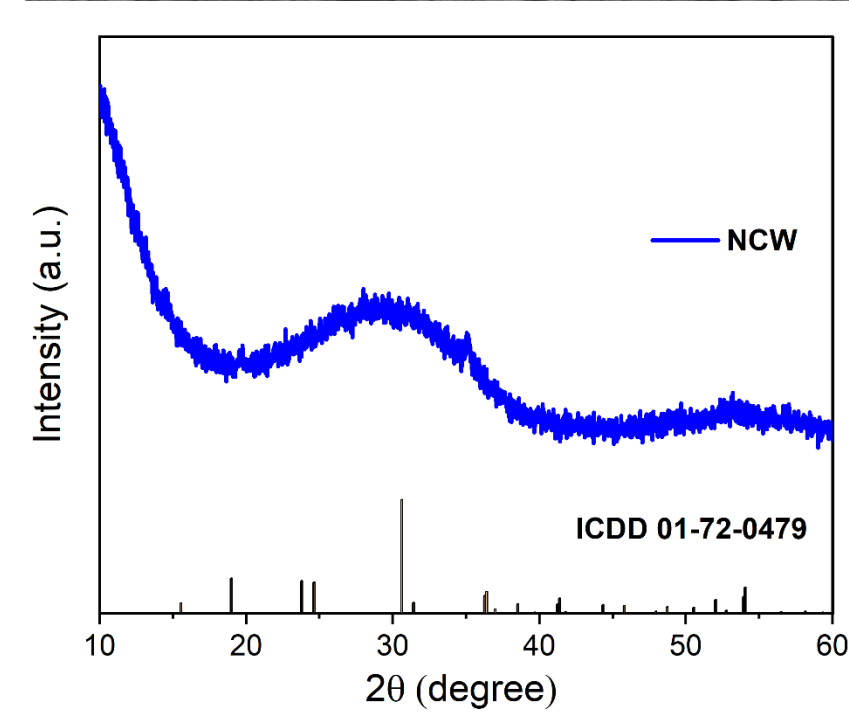
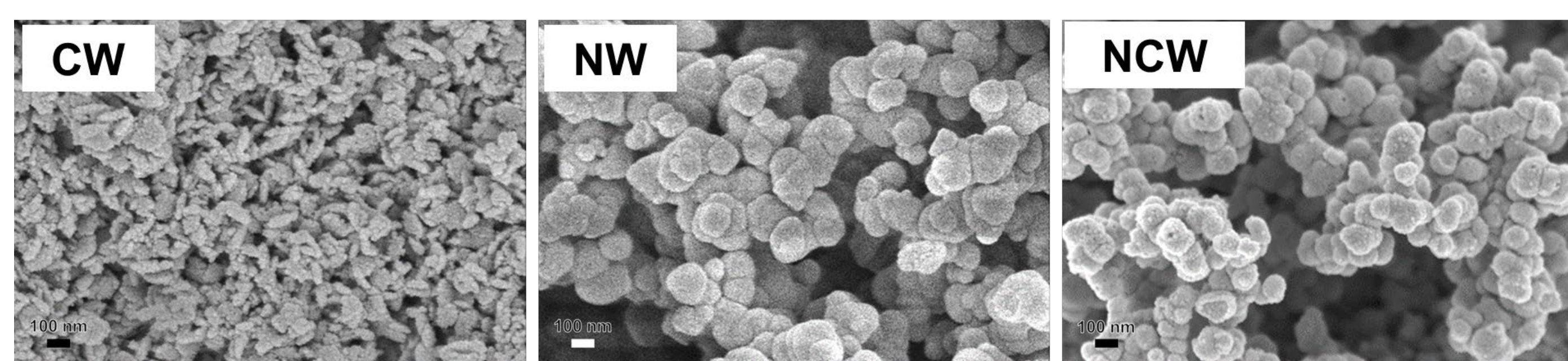
Amount of product obtained in a single batch was >1.2 g.

Electrochemical testing (Three-electrode set-up):

- Autolab PGSTAT128N: CV, LSV, and EIS techniques
- N<sub>2</sub>-saturated 1.0 M KOH, thin-film coated-RDE (5 mm diameter) with loading of ~0.24 mg/cm<sup>2</sup>, Hg/HgO reference electrode, Pt mesh counter electrode.

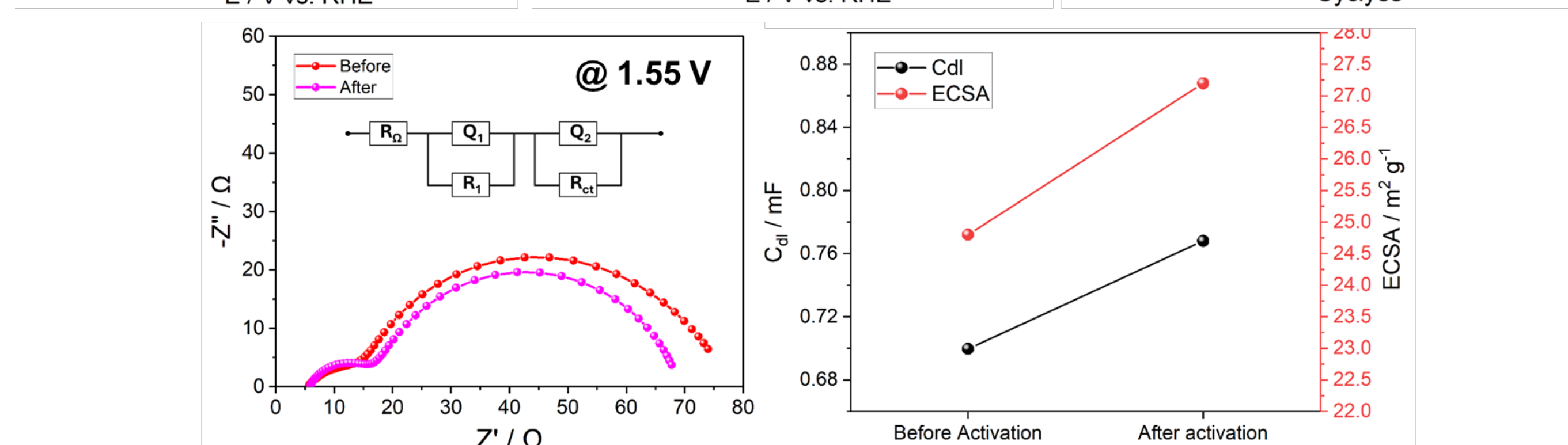
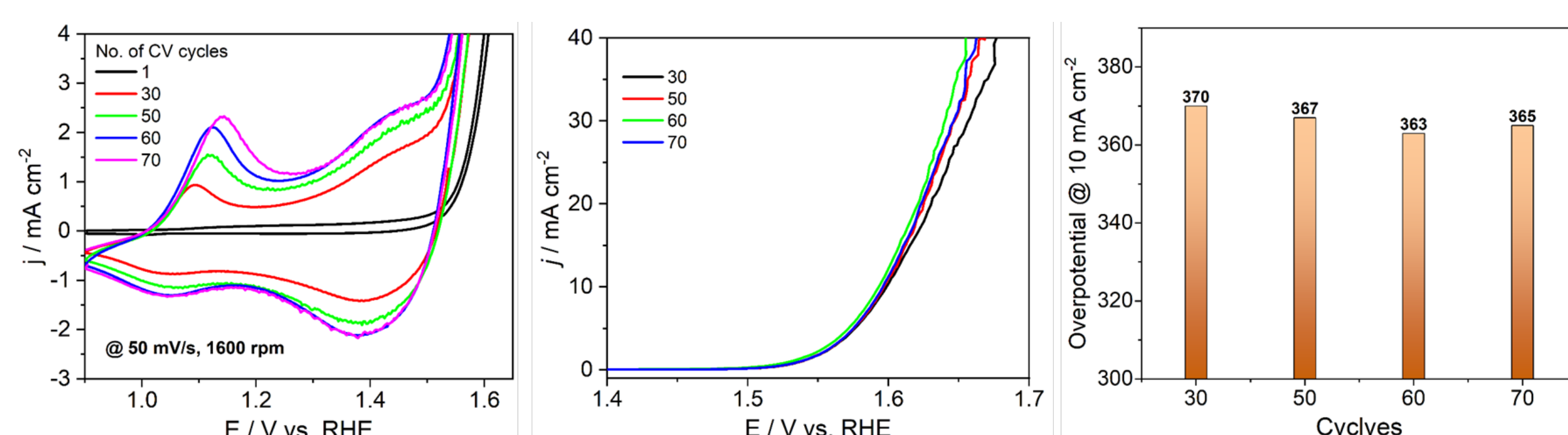
## Results and Discussion

### FESEM and XRD



- Ni incorporation significantly influenced the morphology.
- The XRD of NCW shows semicrystalline structure.

### Electrochemical Activation of CW



- Electrochemical activation of CW was carried out by successive CV cycling.
- The CV curves suggested change in the EDL structure.
- The overpotential derived from the LSV curves indicated that at least 60 CV cycles were needed to activate CW before considering its OER activity.
- The EDL capacitance ( $C_{dl}$ ) and corresponding ECSA of CW were estimated using *in situ* EIS at 1.55 V.

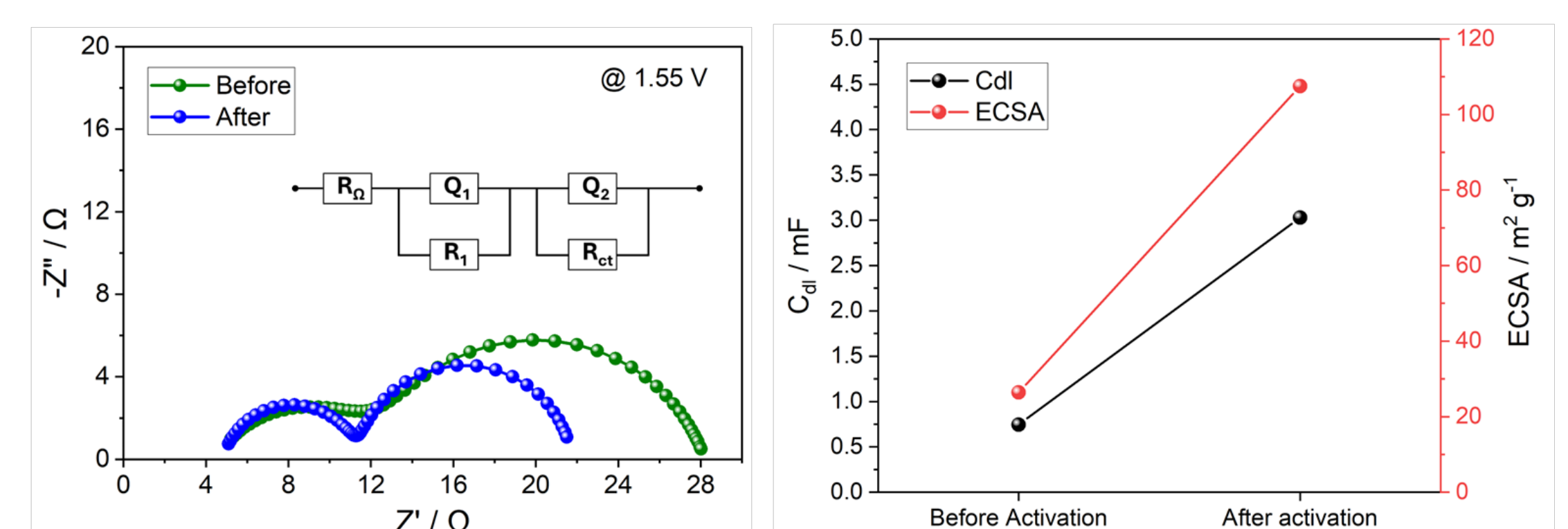
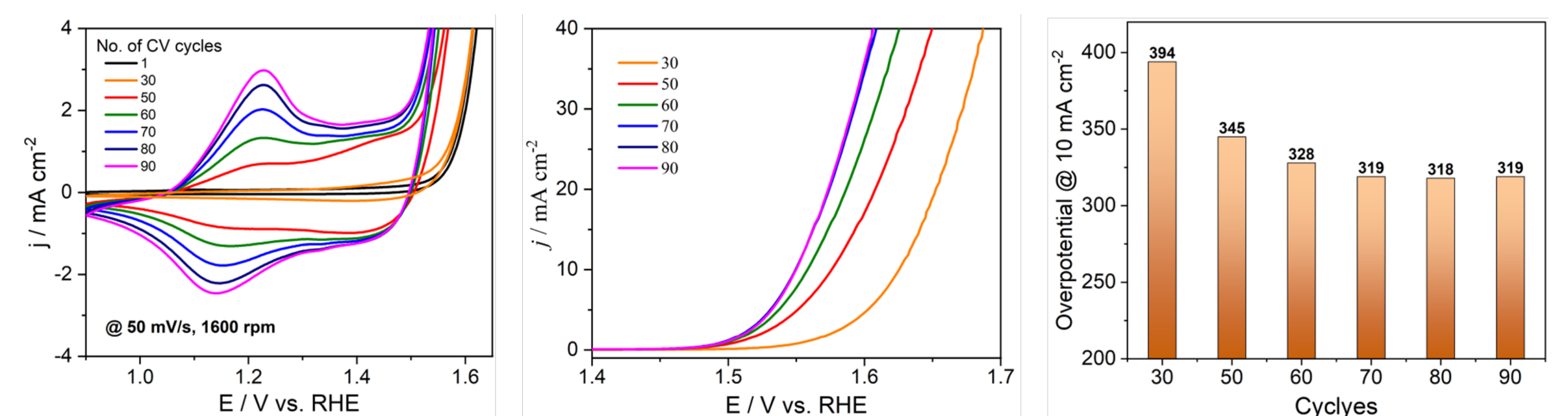
## Conclusions

- The change in the  $C_{dl}$  values indicate EDL reconstruction during the EA and increased ECSA suggests participation of greater number of accessible active sites in the activated electrodes.
- Electrochemical activation is a crucial step to assess the true alkaline OER performance of an anode catalyst in water splitting.

## Reference

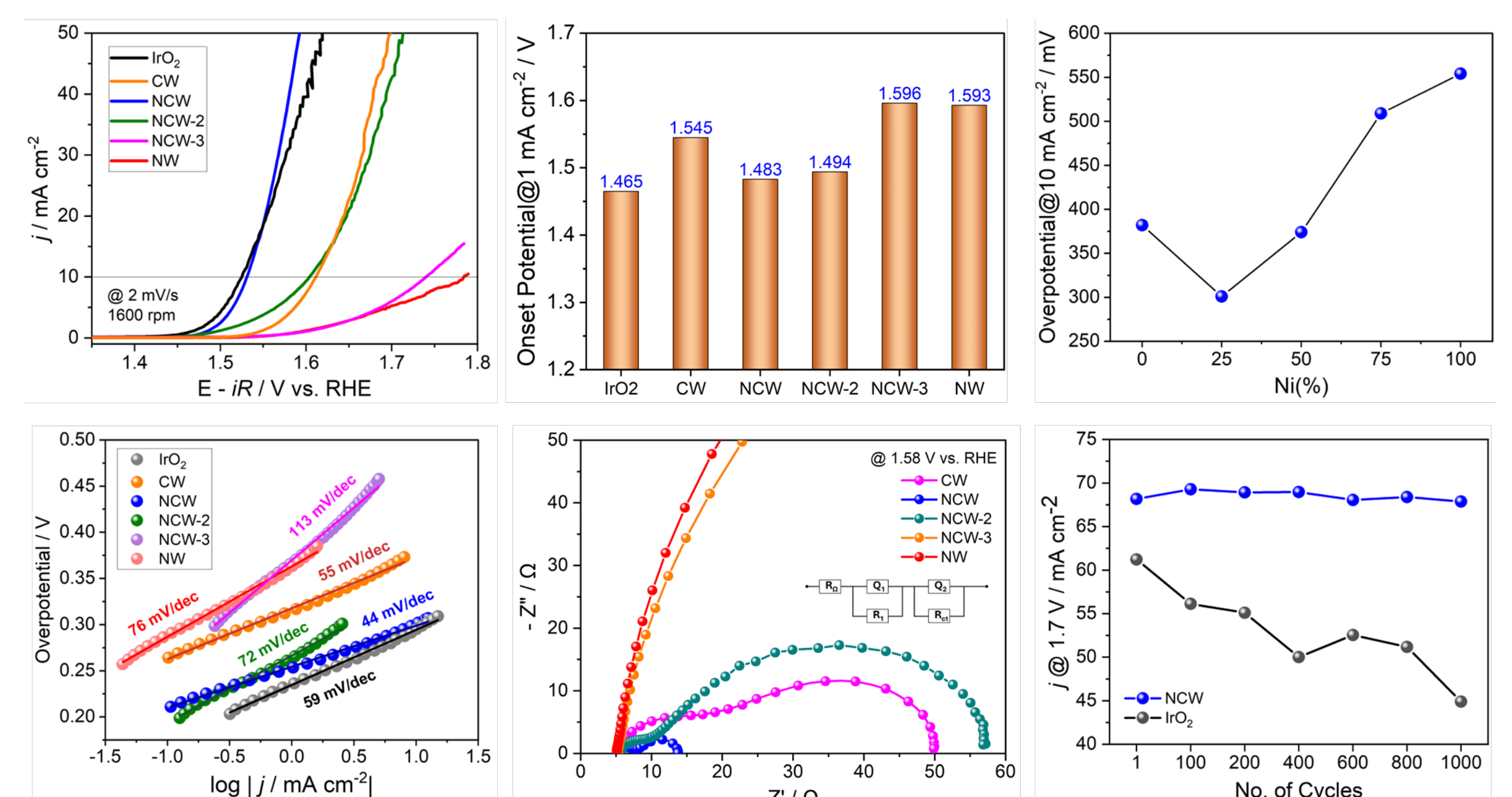
- Karmakar, A.; Chuang, P. Y. A. *ACS Appl. Energy Mater.* **2025** (Nov 24, 2025).

### Electrochemical Activation of NCW



- CV showed significant change until 80 cycles.
- From LSV curves and derived overpotential values indicated activation of NCW after 70 CV cycles.
- The calculated  $C_{dl}$  and ECSA values for NCW significantly increased after EA.

### Alkaline OER activity of NCW samples based on varying Ni-content



- NCW with 25% Ni-content showed the best alkaline OER than other samples.
- NCW outperformed commercial IrO<sub>2</sub> considering high current application and accelerated stability.

## Acknowledgement

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