

Electrochemical assessment of binder free $\text{Co}_x\text{Ni}_{1-x}\text{S}_y$ electrocatalysts supported on carbon nanofibers for oxygen evolution reaction in alkaline media.

by

Sauda Kiggundu Namiiro

A THESIS

submitted to

Oregon State University

Honors College

in partial fulfillment of

the requirements for the

degree of

Honors Baccalaureate of Science in Chemical Engineering

(Honors Scholar)

Presented June 2, 2023

Commencement June 17 2023

AN ABSTRACT OF THE THESIS OF

Sauda Kiggundu Namiiro for the degree of Honors Baccalaureate of Science in Chemical Engineering presented on June 2, 2023. Title: Electrochemical assessment of binder free $\text{Co}_x\text{Ni}_{1-x}\text{S}_y$ electrocatalysts supported on carbon nanofibers for use in oxygen evolution reaction in alkaline media.

Abstract approved: _____

Zhenxing Feng

Water splitting is a green method being researched to produce green hydrogen in order to combat greenhouse gases emitted by the most popular energy source; fossil fuels. Water splitting involves two half reactions: hydrogen evolution reaction (HER) and oxygen evolution reaction (OER). Of these two, OER is the bottleneck water splitting's development because of its sluggish kinetics and large energy demand. Noble metal catalysts such as IrO_2 and RuO_2 have been noted as the best in OER, however they are rare and expensive, thus there's need for cheaper and more abundant metal catalysts such as transition metal-based ones. Conventional electrocatalysts are used in powder form and are attached to electrodes using polymer binders. The objective of this study is to evaluate an alternative test method to the ink-based one by utilizing silver paste to attach bimetallic sulfide electrocatalysts embedded in carbon nanofibers, onto electrodes. This method was compared to the ink-based one and similar electrochemical trends were observed such as the high activity being exhibited by $\text{Co}_{0.83}\text{Ni}_{0.17}\text{S}$, which had the smallest nickel composition. However, the ink-based method had smaller onsite overpotentials despite the smaller Tafel slopes reported by the method in this study. We conclude our findings by providing the pros and cons of the two methods.

Keywords: Hydrogen Evolution Reaction (HER), Oxygen Evolution Reaction (OER), polymer binders, bimetallic sulfide electrocatalysts, carbon nanofibers

Corresponding e-mail address: saudahkiggundu6@gmail.com

©Copyright by Sauda Kiggundu Namiro

June 2, 2023

Electrochemical assessment of binder free $\text{Co}_x\text{Ni}_{1-x}\text{S}_y$ electrocatalysts supported on carbon nanofibers for oxygen evolution reaction in alkaline media.

by

Sauda Kiggundu Namiiro

A THESIS

submitted to

Oregon State University

Honors College

in partial fulfillment of

the requirements for the

degree of

Honors Baccalaureate of Science in Chemical Engineering

(Honors Scholar)

Presented June 2, 2023

Commencement June 17 2023

Honors Baccalaureate of Science in Chemical Engineering project of Sauda Kiggundu Namiiro
presented on June 2, 2023.

APPROVED:

Zhenxing Feng, Mentor, representing School of Chemical, Biological and Environmental Engineering

Konstantinos Goulas, Committee Member, representing School of Chemical, Biological and
Environmental Engineering

Yang Dongqi, Committee Member, representing School of Chemical, Biological and Environmental
Engineering

Toni Doolen, Dean, Oregon State University Honors College

I understand that my project will become part of the permanent collection of Oregon State University,
Honors College. My signature below authorizes release of my project to any reader upon request.

Sauda Kiggundu Namiiro, Author

ACKNOWLEDGEMENTS

I would like to thank Dr. Zhenxing Feng, Yang Dongqi, Saowaluk Soonthornkit, Dr. Marcos Lucero, Brian Muhich, Alvin Chang and Dr. Maouyu Wang for their guidance and support while conducting this research. Additionally, I would like to thank our collaborators Professor Ling Fei and Zhizou He of University of Louisiana at Lafayette, for providing us with the catalyst coated carbon nanofibers used in this study. Furthermore, I would like to thank Dr. Konstantinos Goulas and Yang Dongqi for being part of my committee.

This work was financially supported by the National Science Foundation under Grant No. CBET-1949870, CBET-2016192, CBET-2151049, and Oregon State University Accelerator Innovation Development (AID) Fund.

TABLE OF CONTENTS

A: Introduction.....	1
B: Background.....	3
C: Materials and Methods.....	7
D: Results and Discussions.....	10
E: Conclusion.....	15
F: References.....	16
G: Appendix.....	21

A: INTRODUCTION

Fossil fuels are a dominant energy source worldwide and these have led to emissions of large amounts of carbon dioxide into the atmosphere. As a result, researchers are looking into renewable energy sources that are environmentally friendly such as wind power, solar and hydrogen.^{2,13,14} Hydrogen is an emerging energy source with its primary source of production being steam methane reformation. Steam methane reformation is a currently most used method because of its relatively low cost, however it comes with a price of carbon dioxide emissions and therefore researchers are diving into green hydrogen sources. One source of green hydrogen being targeted is water splitting as water is an abundant resource on earth and thus, can be obtained cheaply. Electrochemically water splitting process involves two half reactions: Hydrogen Evolution Reaction (HER) and Oxygen Evolution Reaction (OER). However, OER is slowing down the overall water splitting process due to its sluggish kinetics, which makes the process energy intensive. Precious metals such as iridium and platinum are the best OER and HER catalysts respectively so far because of their high catalytic activity and stability, but their high cost makes the hydrogen produced from electrolysis very expensive.^{5,6}

Consequently, researchers are exploring the use of catalysts, preferably transition metal catalysts as they are more abundant, cheaper than their precious metal counterparts and they could exhibit high catalytic activity.^{6,8} For OER in particular, first row transition metals such as nickel, cobalt and iron have shown high OER activity and thus their compounds such as sulfides are being explored.^{5,6} In this study of OER, bimetallic sulfide catalysts ($\text{Co}_x\text{Ni}_{1-x}\text{S}_y$) depicted from nickel sulfide (Ni_3S_2) and cobalt sulfide (Co_9S_8) catalysts were analyzed while varying the atom compositions of nickel and cobalt. During the OER, Co exhibits irreversible surface reconstruction

into more oxidative species that enhance OER activity.²⁶ This study analyzes the impact of various Ni substitutions on Co₉S₈'s activity. Conventional electrochemical tests use catalyst powders combined with polymer binders such as Nafion 117, deionized water and isopropyl alcohol to make inks, however the polymer binders are believed to block the catalysts' active sites and thus limit their OER activity.^{14,22,23} Following this, an alternative thin film method of binder free catalysts supported on carbon nanofibers was tested and compared to the ink-based method in efforts to eradicate the use of binders.

Initial experiments with the thin film method were conducted in the same conditions as ink-based, with oxygen gas being used for saturation. However, later on it was discovered that similar experiments with catalyst coated nanofibers had been conducted with inert gases such as nitrogen and an inert atmosphere was more favorable for observing catalytic activity since oxygen tends to undergo unwanted hydrolysis reactions with water and the catalysts in question, which can alter the electrochemical results.^{6,10,11} Therefore, the results provided in this thesis for the thin film method were conducted using nitrogen gas for saturation. The catalysts were attached to the working electrode using silver paste that cured at room temperature in 5 hours. Since the catalysts are sensitive to air, the curing was performed in an inert environment using nitrogen gas.

B: BACKGROUND

B1: Water splitting:

With increased population growth and fossil fuel use, scientists have predicted the need for renewable energy sources to meet the high energy demand in the future.^{2,14} The major fossil fuels used include coal, natural gas and crude oil. However, the use of fossil fuels as an energy source has resulted in increased carbon dioxide emissions. Renewable energy sources being considered include solar, wind and hydrogen. Among these, hydrogen has become popular because of its high gravimetric energy density of $142 \frac{MJ}{kg}$ and potential to be recycled.¹⁴ Currently, the primary source of hydrogen is steam methane reformation, which results in large emissions of carbon dioxide gas. Therefore, researchers are looking into alternative green hydrogen production methods and one method being considered is water splitting.

Green hydrogen can be produced via water splitting using an electrolyzer as shown in figure 1a) below. Water splitting involves two half reactions: Hydrogen Evolution Reaction (HER) and Oxygen Evolution Reaction (OER). In the electrolyzer, hydrogen gas is evolved at the cathode whereas oxygen gas is evolved at the anode.^{11,16} The electrolyte used can be either acidic or alkaline and the half reactions pertaining to both media are shown in Figure 1b).

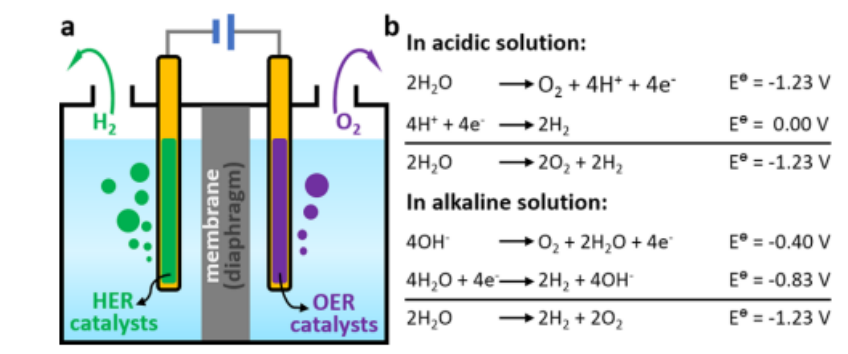


Figure 1: a) Water splitting using an electrolyzer. b) Water splitting half and full reactions for acidic and alkaline solutions used as electrolytes in the electrolyzer.

Despite water splitting being a source of green hydrogen, its half reactions are energy intensive and conventionally this process requires operation at potentials higher than its standard equilibrium potential (1.23V).¹⁶ Consequently, researchers are exploring the use of electrocatalysts in order to overcome this thermodynamic barrier. Noble metal catalysts have shown high catalytic activities for both half reactions with Pt and Ni electrodes for HER in acidic and basic media respectively while IrO_2 and RuO_2 are notable catalysts for OER in both acidic and alkali media.^{3,11,16} However, these noble metals are expensive and rare to find, thus researchers are investigating low-cost catalysts for instance transition metal-based sulfides, oxides, nitrides and chalcogenides as substitutes.

OER comprises a discrete four electron process, which makes it more energy intensive than HER and thus it's hindering the development of water splitting.^{4,11,16} The equations pertaining to acidic and alkaline electrolytes are shown in Figure 2 as a series of 4 reactions denoting the 4-electron process. This 4-electron process causes OER to have very sluggish kinetics and thus catalysts are required to reduce the activation energy of the intermediate species.

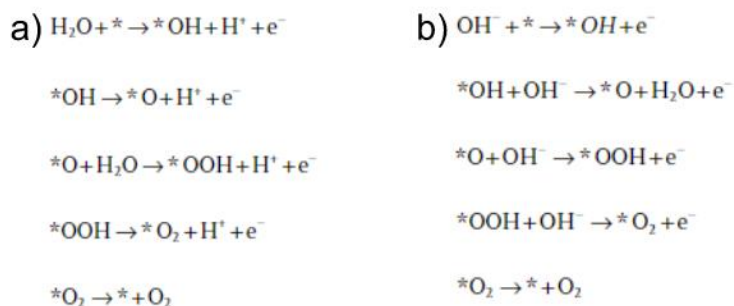


Figure 2: a) OER equations in acidic electrolyte. b) OER equations in alkaline or neutral electrolyte.

Most OER catalysts are prepared by doping different transition metal and nonmetal (sulfur, nitrogen) atoms in order to combine their intrinsic activities and yield a better composite activity.^{4,16,21} Low cost electrocatalysts being studied involve first row transition metals including nickel and cobalt as these have shown promising OER activity, high stability and cheaper costs than noble metal-based ones.

B2: Self Supported Electrocatalysts

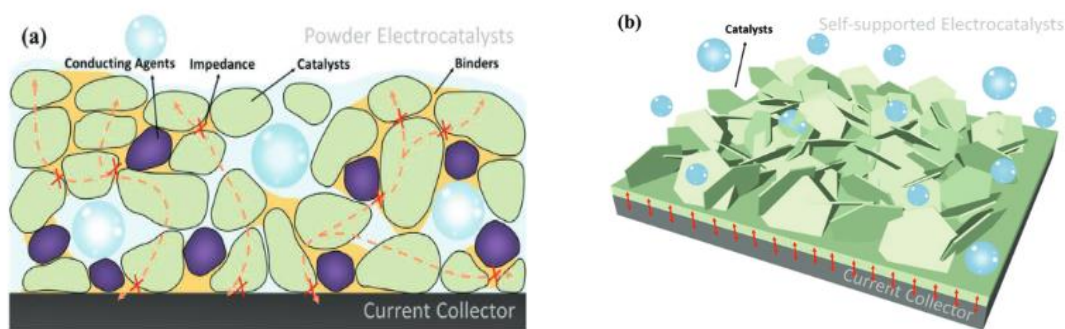


Figure 3: a) Electrode with binder supported powder electro catalysts. b) Electrode with self-supported electro catalysts.

Polymer binders are used to immobilize the powdered catalysts such that they can be evaluated electrochemically while in a stationary mode. Commonly used binders include Nafion, sodium dodecyl sulfate, Triton X-100, polytetrafluoroethylene and so forth.^{14,22} Among these,

Nafion is the most popular one and has been proven to be the most feasible for electrochemical measurements.^{22,23} On the contrary, from Figure 3a), it can be seen that polymer binders block the active sites of catalysts, resulting in shifts in their onset potentials and lower currents, thus reducing their activity.^{14,22,23} Additionally, they are highly hydrophobic and thus can hinder electrolyte interaction with the catalyst's active sites.^{14,25} Therefore, there is a need for self-supported electrodes such that their active sites are exposed during the reaction as shown in Figure 3b).

Additional advantages of self-supported electrocatalysts include the capacity of increase in catalyst loading resulting in abundance of active sites, reduction of mechanical shedding of catalysts due to strong adhesion of the catalysts with the electrode, simpler and cheaper fabrication process due to reduced costs associated with binders and other additives.¹⁴ This notion of self-supported electrocatalysts is the motive behind this study using catalyst embedded nanofibers whose back is attached to the electrode and the top is exposed to the electrolyte. For this study, silver paste was used as the mode of attachment because of silver's high electrical conductivity ($6.30 \times 10^7 S m^{-1}$ at 20 °C) compared to other metals, comparatively low cost and high stability in alkaline media.²⁴

C: MATERIALS AND METHODS

C1: Materials

$\text{Co}_x\text{Ni}_{1-x}\text{S}_y$ carbon nanofibers provided by Professor Ling Fei and her team from University of Louisiana at Lafayette. These nanofibers were prepared using electrospinning and subsequently annealed. Silver epoxy conductive paste (8331D) used for room temperature curing was purchased from MG chemicals. Rotating Disk Electrode (RDE, model no. AFMSRCE) purchased from Pine Research Instrumentation Inc. (Durham, NC), was used as the working electrode in the electrochemical cell. Rotating electrode speed control machine purchased from Pine Research Instrumentation Inc. (Durham, NC) was used to adjust the rotation speed of the working electrode. Potentiostat (Model no. 760E) purchased from CH Instruments Inc. (Austin, TX) was used to supply voltage and perform the cyclic voltammetry analysis.

C2: Methods

C2a) Sample preparation:

Silver paste was prepared by mixing the two syringe contents (A and B) in a 1:1 ratio and a dime sized amount of the mixture was deposited on the glassy carbon disk. The plain and catalyst coated carbon nanofibers were punched into circular sheets that had the same diameter as the glassy carbon disk (GC, 0.196 cm^2 geometric area) of the working electrode. The circular sheets were then weighed to ensure that they were less than 0.27 mg, which guaranteed absence of attached loose sheets. They were then placed on the silver paste and slightly tapped to spread the paste and ensure good electrical contact. Acetone was used to rinse off paste in case it spread to the Teflon casing enclosing the disk and the assembly was repeated. The samples were then placed under a nitrogen purge set up to cure the silver paste at room temperature for a minimum of 5 hours in an oxygen free environment. This inert atmosphere prevented reactions of the catalysts with air since

they are sensitive to oxygen. After 4 hours, potassium hydroxide solution (0.1 M) was prepared and the nitrogen gas saturation was done for an hour via a benchtop purge system.

C2b) Electrochemical measurements:

After curing, the 3-electrode system was set up comprising a platinum wire as the counter electrode, a calomel (Hg/HgO) electrode as the reference electrode and a working electrode (GC) with the attached nanofiber samples. Nitrogen gas was purged at a reduced flow rate and the samples were precycled using cyclic voltammetry (CV) at a scan rate of 100 mV/s in a sweeping range of 1-2.0 (V vs RHE) with the working electrode rotating at 1600 rpm. After precycling, CV analysis was performed at 10 mV/s with the same rotation speed and sweeping range as the precycling.

The current obtained from CV measurements was normalized by the geometric disk area (0.196 cm^2) in order to get current density ($\frac{\text{mA}}{\text{cm}^2}$). The voltage (V vs Hg/HgO) obtained from CV measurements was converted to V vs RHE using the following equation:

$$E(V \text{ vs RHE}) = E_{\text{Hg/HgO}} + (0.059 * pH) + E^0_{\text{Hg/HgO}},$$

where E is the voltage from CV measurements, the Ph of the electrolyte (KOH) was 13 and the equilibrium potential ($E^0_{\text{Hg/HgO}}$) of the reference electrode (Hg/HgO) was 0.098 V. The catalytic activity was assessed using the onsite overpotential (overpotential at $10 \frac{\text{mA}}{\text{cm}^2}$) and Tafel slopes. The Tafel slopes were calculated using low currents ($< 5 \frac{\text{mA}}{\text{cm}^2}$) as this is where the Tafel slopes of the different nanofiber mass loadings converged to the fundamental kinetic Tafel slopes.¹² The equation used to calculate the Tafel slopes was

$\eta = a + b \log(j)$, where η is the overpotential calculated by subtracting 1.23 V from the measured voltages, a is the intercept ($-b \log(j_0)$), where j_0 is the exchange current density), b is the Tafel slope and j is current density.

Current density was converted into its logarithm values and they were plotted against the overpotential in order to generate a linear plot that yielded its slope as the Tafel slope. A linear regression was performed on the linear region used to determine the Tafel slope.

D. RESULTS AND DISCUSSION

The OER catalytic activity was assessed with a three-electrode system using cyclic voltammetry at a scan rate of 10 mV/s and sweeping range of 1-2 V vs RHE, with the working electrode rotating at 1600 rpm. After performing the current normalization to the geometric disk area and iR-correction, plots of current density versus iR-corrected voltage were generated as shown in Figure 4. Figures 4a) and b) represent the minimized Linear Sweep Voltammetry (LSV) and maximized CV curves for the ink-based method whereas figures 4c) and 4d) represent the minimized and maximized CV curves for the thin film method. Comparing the maximized CV curves of both methods, both methods showed pronounced oxidative peaks for all the compositions. However, with the thin film method, there were no asymmetric redox peaks corresponding to the Ni^{3+}/Ni^{4+} redox reactions as seen in the ink-based method, but Ni_3S_2 had only an oxidative peak which indicated that the reduction reaction (Ni^{4+}/Ni^{3+}) didn't occur in the thin film method. Comparing the oxidative peaks of Ni_3S_2 and Co_9S_8 , Ni_3S_2 had a smaller peak, but it was within the same voltage range (around 1.25 V vs RHE) as Co_9S_8 . In the ink-based method, the Ni_3S_2 peak was at around 1.4 V vs RHE, whereas the Co_9S_8 was at around 1.1 V vs RHE, which was close to where the thin film Co_9S_8 peak was located. Because the thin film method had only oxidative peaks, therefore both cobalt and nickel underwent irreversible oxidation into more catalytically active species. Ni_3S_2 acted as a precursor for the more oxidative species (nickel oxyhydroxide), which explained the increase in catalytic activity.^{17,19,20}

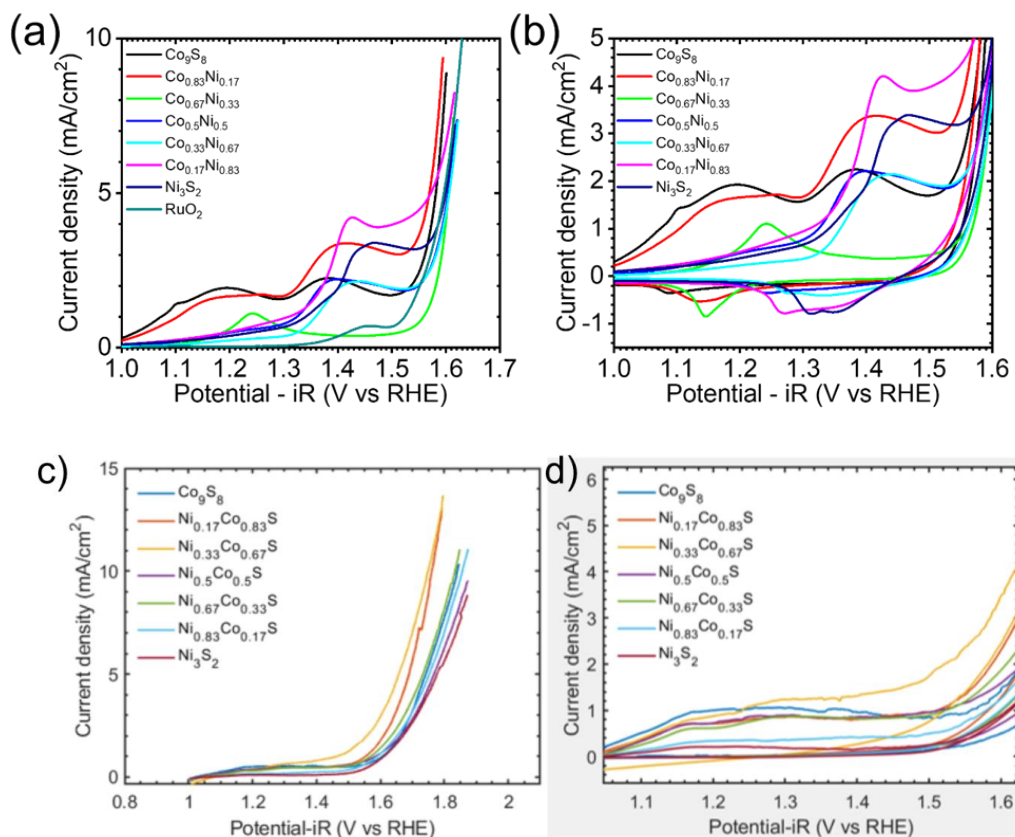


Figure 4: Voltammetry curves for the 1st cycle of catalysts prepared by ink-based method and thin film method. The x- axis represents the iR corrected voltage whereas the y- axis represents the current normalized to the geometric disk area. a) Minimized view of Linear Sweep Voltammetry measurements for ink-based method. b) Maximized view of cyclic voltammetry measurements for ink-based method. c) Minimized view of Linear Sweep Voltammetry measurements for the thin film method. d) Maximized view of Cyclic Voltammetry measurements for the thin film method.

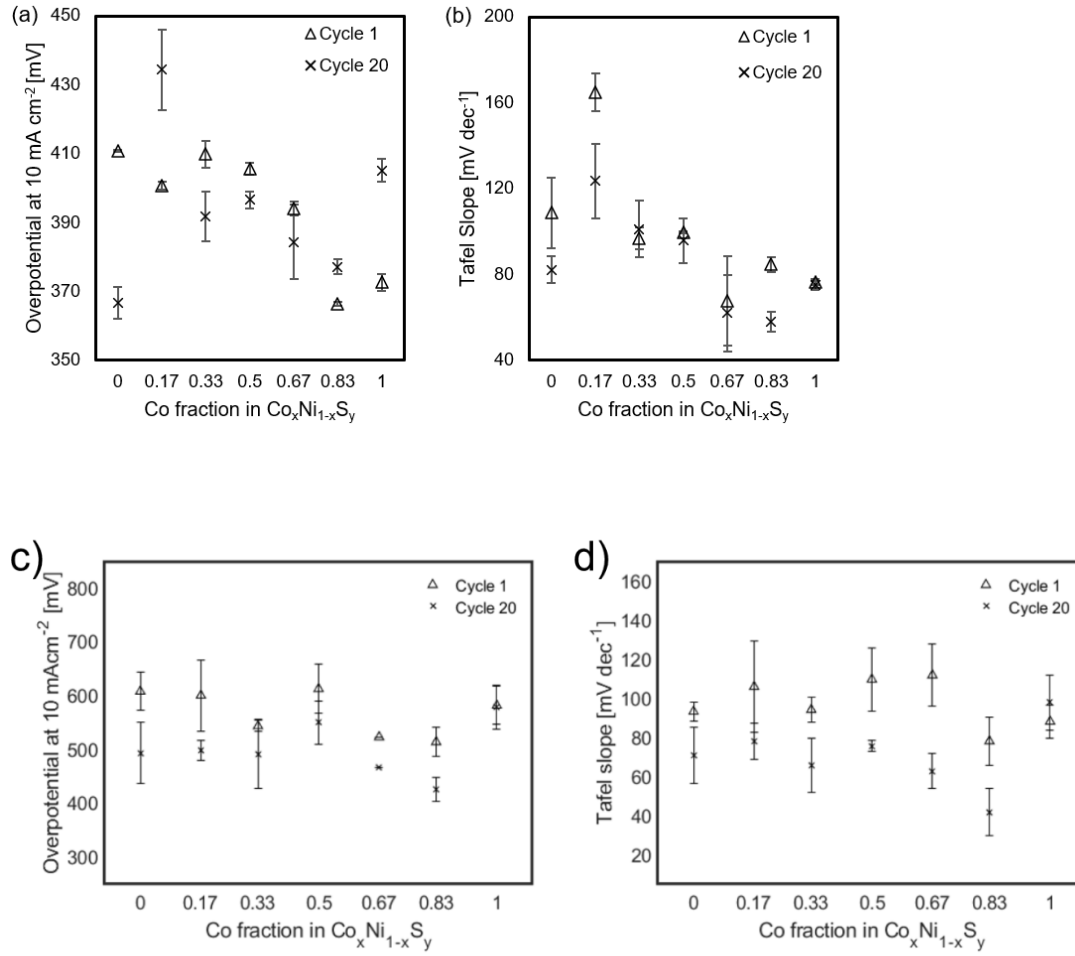


Figure 5: Onsite overpotential and Tafel slope comparison for the 1st and 20th cycles of catalysts prepared by ink-based method and thin film method, with error bars indicating one standard deviation. a) Onsite overpotential for 1st and 20th cycles of the ink-based method (extrapolated with Tafel slope). b) Tafel slopes for 1st and 20th cycles of the ink-based method. c) Onsite overpotential for 1st and 20th cycles of the thin film method. d) Tafel slopes for 1st and 20th cycles of the thin film method.

The catalytic activity of all compositions with both methods was evaluated using the overpotential at $10 \frac{\text{mA}}{\text{cm}^2}$ and Tafel slopes of the first and 20th cycles. Comparing figures 5a) and c), catalysts with high cobalt composition ($x > 0.67$) had smaller overpotentials compared to those with low cobalt compositions for the 1st and 20th cycles of both methods. Of these compositions, $\text{Co}_{0.83}\text{Ni}_{0.17}\text{S}$ had the smallest overpotential and thus best activity, for both the ink-based²⁶ and thin film methods, whereas $\text{Co}_{0.17}\text{Ni}_{0.83}\text{S}$ had the worst catalytic activity as it had the largest

overpotentials in both methods. However, the ink-based method ²⁶ had slightly smaller onsite overpotentials for $Co_{0.83}Ni_{0.17}S$ of around 365 mV and 378 mV for the 1st and 20th cycles respectively, than the thin film method which had onsite overpotentials of around 514 mV and 426 mV for the 1st and 20th cycles respectively. Despite having larger over potentials, the thin film method had a smaller overpotential for the 20th cycle, which indicates an increase in catalytic activity and stability compared to the ink-based that had the reversed trend. Comparing the 1st cycle of the catalyst with even composition ($Co_{0.5}Ni_{0.5}S$) with the pure sulfides (Ni_3S_2 and Co_9S_8), the pure sulfides had smaller onsite over potentials (608 and 578 mV respectively) than the even one (613 mV), which showed that the catalytic activity was worse when cobalt and nickel had even compositions.

Comparing the Tafel plots of the first and 20th cycles of both methods (Figures 5b and 5d), a similar trend as the onsite overpotential was observed, as catalysts with high Co composition ($x > 0.67$) had smaller Tafel slopes compared to those with low Co compositions for the 1st and 20th cycles of both methods. It can also be seen that $Co_{0.83}Ni_{0.17}S$ had the smallest Tafel slopes and thus the best catalytic activity, whereas $Co_{0.17}Ni_{0.83}S$ had the worst catalytic activity as it had the largest Tafel slopes for both the ink-based and thin film methods. With the ink-based method²⁶, the Tafel slopes for $Co_{0.83}Ni_{0.17}S$ were $57.8 \frac{mV}{dec}$ for cycle 1 and increased to $84.6 \frac{mV}{dec}$ for cycle 20, while those for the thin film method were $78.1 \frac{mV}{dec}$ for cycle 1 and decreased to $41.8 \frac{mV}{dec}$ for cycle 20. Additionally, with the thin film method, all the compositions had smaller tafel slopes and onsite over potentials for the 20th cycle than the 1st cycle. This trend of the last cycle (20th) having smaller Tafel slopes showed that catalytic activity of these compositions increased since the current was increasing faster with changes in potential. Furthermore, comparing the 1st cycle of

the catalyst with even composition ($Co_{0.5}Ni_{0.5}S$) with the pure sulfides (Ni_3S_2 and Co_9S_8), the pure sulfides had smaller Tafel slopes (93.2 and $88.2 \frac{mV}{dec}$ respectively) than the even one ($109.7 \frac{mV}{dec}$), which showed that the catalytic activity was worse when cobalt and nickel had even compositions. This trend of the even composition having worse activity than the pure metallic sulfides was also observed in the ink-based method. Discrepancies in the thin film method's results were attributed to the differences in mass loadings of the catalysts, changes in ohmic resistances, changes in internal hydroxide gradients, changes in ambient temperature and so forth.^{13,18,21}

E. CONCLUSION

In conclusion, a thin film method for testing nickel substituted cobalt sulfide catalysts coated on carbon nanofibers was assessed and compared with the ink-based method which uses polymer binders. The carbon nanofibers were attached to the working electrode by means of a conductive silver epoxy paste. A three-electrode setup was used to conduct the electrolysis and cyclic voltammetry was used to measure the current incurred for the range of voltages (1-2 V vs RHE) applied. From the results obtained, both methods had similar trends in the irreversible restructuring, onsite overpotentials and Tafel slopes. However, for the cyclic voltammetry curves of the thin film method, there were no asymmetric redox peaks corresponding to the Ni^{3+}/Ni^{4+} redox reaction for all compositions, but rather only an oxidative peak attributed to cobalt and nickel's irreversible restructuring to more oxidative species. For the onsite overpotential and Tafel slope analyses, $Co_{0.83}Ni_{0.17}S$ and $Co_{0.17}Ni_{0.83}S$ had the best and worst catalytic activities respectively for both methods. Despite the similar trends, the thin film method had larger overpotentials than the ink-based method. Nonetheless, the thin film method had smaller Tafel slopes and a shorter sample preparation time of 5 hours as opposed to the 24-hour drying duration of the ink-based method. Based on the results, the thin film method had overall similar trends with the ink-based method, with minor differences. However, the ink-based method was concluded to be better in assessing the intrinsic activity because it reflects detailed redox reactions such as more pronounced oxidation peaks and more realistic overpotentials at the standard reference current density of $10 \frac{mA}{cm^2}$. Overall, the thin film method showed promising results and therefore it is recommended to perform electrochemical tests with other conductive pastes and nanofibers with equivalent masses in order to assess which would yield closer or better results than the ink-based method or the current thin film method.

F. REFERENCES

1. Alegre, C.; Modica, E.; Di Blasi, A.; Di Blasi, O.; Busacca, C.; Ferraro, M.; Aricò, A. S.; Antonucci, V.; Baglio, V. NiCo-Loaded Carbon Nanofibers Obtained by Electrospinning: Bifunctional Behavior as Air Electrodes. *Renewable Energy* 2018, 125, 250–259. <https://doi.org/10.1016/j.renene.2018.02.089>.
2. Al-Qahtani, A.; Parkinson, B.; Hellgardt, K.; Shah, N.; Guillen-Gosalbez, G. Uncovering the True Cost of Hydrogen Production Routes Using Life Cycle Monetisation. *Applied Energy* 2021, 281, 115958. <https://doi.org/10.1016/j.apenergy.2020.115958>.
3. Badruzzaman, A.; Yuda, A.; Ashok, A.; Kumar, A. Recent Advances in Cobalt Based Heterogeneous Catalysts for Oxygen Evolution Reaction. *Inorganica Chimica Acta* 2020, 511, 119854. <https://doi.org/10.1016/j.ica.2020.119854>.
4. Ede, Sivasankara Rao, and Zhiping Luo. “Tuning the Intrinsic Catalytic Activities of Oxygen-Evolution Catalysts by Doping: A Comprehensive Review.” *Journal of Materials Chemistry A*. The Royal Society of Chemistry, August 13, 2021. <https://pubs.rsc.org/en/content/articlelanding/2021/ta/d1ta04032d>.
5. Hyun, S.; Shanmugam, S. Hierarchical Nickel–Cobalt Dichalcogenide Nanostructure as an Efficient Electrocatalyst for Oxygen Evolution Reaction and a Zn–Air Battery. *ACS Omega* 2018, 3 (8), 8621–8630. <https://doi.org/10.1021/acsomega.8b01375>.
6. Kim, K.; Kang, T.; Kim, M.; Kim, J. Exploring the Intrinsic Active Sites and Multi Oxygen Evolution Reaction Step via Unique Hollow Structures of Nitrogen and Sulfur Co-Doped Amorphous Cobalt and Nickel Oxides. *Chemical Engineering Journal* 2021, 426, 130820. <https://doi.org/10.1016/j.cej.2021.130820>.

7. Li, A.; Sun, Y.; Yao, T.; Han, H. Earth-Abundant Transition-Metal-Based Electrocatalysts for Water Electrolysis to Produce Renewable Hydrogen. *Chemistry – A European Journal* 2018, 24 (69), 18334–18355. <https://doi.org/10.1002/chem.201803749>.
8. Locke, E.; Jiang, S.; Beaumont, S. K. Catalysis of the Oxygen Evolution Reaction by 4–10 Nm Cobalt Nanoparticles. *Topics in Catalysis* 2018, 61 (9-11), 977–985. <https://doi.org/10.1007/s11244-018-0923-4>.
9. 8331 – Silver Conductive Epoxy Adhesive. mgchemicals.com. <https://mgchemicals.com/products/adhesives/electrically-conductive-adhesives/silver-conductive-epoxy> (accessed 2022-06-20).
10. *Conducting Electrochemical Experiments in an Inert Atmosphere a Practical Guide for Air-Free Electrochemical Techniques*. <https://www.pineresearch.com/shop/wp-content/uploads/sites/2/2018/01/DRA10072-Conducting-Electrochemical-Experiments-in-an-Inert-Atmosphere-REV001.pdf>. (accessed 2023-03-01).
11. Suryanto, B. H. R.; Wang, Y.; Hocking, R. K.; Adamson, W.; Zhao, C. Overall Electrochemical Splitting of Water at the Heterogeneous Interface of Nickel and Iron Oxide. *Nature Communications* 2019, 10 (1). <https://doi.org/10.1038/s41467-019-13415-8>.
12. Van der Heijden, O.; Park, S.; Eggebeen, J. J. J.; Koper, M. T. M. Non-Kinetic Effects Convolute Activity and Tafel Analysis for the Alkaline Oxygen Evolution Reaction on NiFeOOH Electrocatalysts. *Angewandte Chemie International Edition* 2023, 62 (7). <https://doi.org/10.1002/anie.202216477>.
13. Da Silva Veras, T.; Mozer, T. S.; da Costa Rubim Messeder dos Santos, D.; da Silva César, A. Hydrogen: Trends, Production and Characterization of the Main Process Worldwide.

- International Journal of Hydrogen Energy* 2017, 42 (4), 2018–2033.
<https://doi.org/10.1016/j.ijhydene.2016.08.219>.
- 14.** Yang, H.; Driess, M.; Menezes, P. W. Self-Supported Electrocatalysts for Practical Water Electrolysis. *Advanced Energy Materials* 2021, 11 (39), 2102074.
<https://doi.org/10.1002/aenm.202102074>.
- 15.** Yang, J.; Fujigaya, T.; Nakashima, N. Decorating Unoxidized-Carbon Nanotubes with Homogeneous Ni-Co Spinel Nanocrystals Show Superior Performance for Oxygen Evolution/Reduction Reactions. *Scientific Reports* 2017, 7 (1), 45384.
<https://doi.org/10.1038/srep45384>.
- 16.** You, B.; Sun, Y. Innovative Strategies for Electrocatalytic Water Splitting. *Accounts of Chemical Research* 2018, 51 (7), 1571–1580.
<https://doi.org/10.1021/acs.accounts.8b00002>.
- 17.** Mabayoje, O.; Shoola, A.; Wygant, B. R.; Mullins, C. B. The Role of Anions in Metal Chalcogenide Oxygen Evolution Catalysis: Electrodeposited Thin Films of Nickel Sulfide as “Pre-Catalysts.” *ACS Energy Letters* 2016, 1 (1), 195–201.
<https://doi.org/10.1021/acsenergylett.6b00084>.
- 18.** Anantharaj, S.; Kundu, S. Do the Evaluation Parameters Reflect Intrinsic Activity of Electrocatalysts in Electrochemical Water Splitting? *ACS Energy Letters* **2019**, 4 (6), 1260–1264. <https://doi.org/10.1021/acsenergylett.9b00686>.
- 19.** He, R.; Huang, X.; Feng, L. Recent Progress in Transition-Metal Sulfide Catalyst Regulation for Improved Oxygen Evolution Reaction. *Energy & Fuels* **2022**, 36 (13), 6675–6694. <https://doi.org/10.1021/acs.energyfuels.2c01429>.

20. Zhang, J.; Li, Y.; Zhu, T.; Wang, Y.; Cui, J.; Wu, J.; Xu, H.; Shu, X.; Qin, Y.; Zheng, H.; Ajayan, P. M.; Zhang, Y.; Wu, Y. 3D Coral-like Ni₃S₂ on Ni Foam as a Bifunctional Electrocatalyst for Overall Water Splitting. *ACS Applied Materials & Interfaces* **2018**, *10* (37), 31330–31339. <https://doi.org/10.1021/acsami.8b09361>.
21. Yu, M.; Budiyo, E.; Tüysüz, H. Principles of Water Electrolysis and Recent Progress in Cobalt-, Nickel-, and Iron-Based Oxides for the Oxygen Evolution Reaction. *Angewandte Chemie* **2021**, *134* (1). <https://doi.org/10.1002/ange.202103824>.
22. Asad, M.; Shah, A.; Iftikhar, F. J.; Nimal, R.; Nisar, J.; Zia, M. A. Development of a Binder-Free Tetra-Metallic Oxide Electrocatalyst for Efficient Oxygen Evolution Reaction. *Sustainable Chemistry* **2022**, *3* (3), 286–299. <https://doi.org/10.3390/suschem3030018>.
23. Praveen Narangoda; Neugebauer, S.; Schloegl, R.; Mechler, A. The Influence of the Binder on the Investigation of Electrocatalysts for Oxygen Evolution Reaction in Alkaline Water Electrolysis. *Meeting abstracts* **2018**, *MA2018-02* (52), 1767–1767. <https://doi.org/10.1149/ma2018-02/52/1767>.
24. Yu, M.; Moon, G.-W.; Castillo, R. G.; DeBeer, S.; Weidenthaler, C.; Harun Tüysüz. Dual Role of Silver Moieties Coupled with Ordered Mesoporous Cobalt Oxide towards Electrocatalytic Oxygen Evolution Reaction. *Angewandte Chemie* **2020**, *59* (38), 16544–16552. <https://doi.org/10.1002/anie.202003801>.
25. Xie, X.; Du, L.; Yan, L.; Park, S.; Qiu, Y.; Sokolowski, J.; Wang, W.; Shao, Y. Oxygen Evolution Reaction in Alkaline Environment: Material Challenges and Solutions. *Advanced Functional Materials* **2022**, *32* (21), 2110036. <https://doi.org/10.1002/adfm.202110036>.

- 26.** Muhich, B. Enhanced Oxygen Evolution Reaction Activity in Ni Substituted CoS_x Electrocatalysts from Reaction-Induced Restructuring. *Oregon State University Scholars Archive* **2022**. <https://ir.library.oregonstate.edu/?locale=en>

G. APPENDIX

G1a): Matlab script for a function that reads data from text files (with text and data) and stores them into current and voltage arrays.

```

1 %This script generates a code that reads text files for of
2 %one sample and stores the data into voltage and current arrays.
3 function [Ann,Vn_n,In_n,n_n] = OER_rep(f)
4 %constants
5 pH=13; %pH of electrolyte
6 E0=0.098; %V equilibrium potential of Hg/HgO reference electrode
7 %importing data
8 Ann=importdata(f);
9 %voltage array
10 Vn_n=Ann.data(:,1)+E0+(0.059*pH); %voltage conversion from V to V vs RHE
11
12 %current array
13 In_n=Ann.data(:,2); %current
14
15 %length of voltage array
16 n_n=length(Vn_n);
17 end

```

G1a): Matlab script for a function that reads data from text files (with deleted text and only data) and stores them into current and voltage arrays.

```

1 %This script generates a code that reads text files (with only values) for
2 %one sample and stores the data into voltage and current arrays.
3 function [Ann,Vn_n,In_n,n_n] = OER_rep1(f)
4 %constants
5 pH=13; %pH of electrolyte
6 E0=0.098; %V equilibrium potential of Hg/HgO reference electrode
7 %importing data
8 Ann=importdata(f);
9 %voltage array
10 Vn_n=Ann(:,1)+E0+(0.059*pH); %voltage conversion from V to V vs RHE
11
12 %current array
13 In_n=Ann(:,2); %current
14
15 %length of voltage array
16 n_n=length(Vn_n);
17 % end
18

```

G2: Matlab script for a function that splits CV data into 1st and 20th cycles while performing the current normalization and voltage iR-correction.

```

1 %This script develops a code for a function that processes OER data with
2 %inputs being resistance, current and voltage arrays. It splits the data
3 %into the 1st and 20th cycles
4
5 function [In_1,Vn_1,In_20,Vn_20]=OER_cycle(In,Vn,Rn)
6 %constants
7 %pH=13; %pH
8 %E0=0.098; %V equilibrium potential of Hg/Hg0 reference electrode
9 C=1000/0.196; % current conversion constant to mA/cm^2 based on geometric
10 % surface area of RDE disk
11
12 %preallocating arrays prior to for loop use
13 In_1=zeros(2000,1);Vn_1=zeros(2000,1);In_20=zeros(2000,1);Vn_20=zeros(2000,1);
14
15 %performing for loop to split arrays into 1st and 20th cycles
16 for i=1:2000
17     In_1(i,1)=In(i+135)*-C; %1st cycle current
18     Vn_1(i,1)=Vn(i+135)-((In(i+135))*Rn); %1st cycle voltage
19     In_20(i,1)=In(i+38135)*-C ; %20th cycle current
20     Vn_20(i,1)=Vn(i+38135)-((In(i+38135))*Rn); %20th cycle voltage
21 end
22 end

```

G2: Matlab script for a function that converts CV data into LSV data by means of averaging.

```

1 %This script generates a code that converts one cyclic voltammetry cycle
2 %into a linear sweep voltammetry curve.
3
4 function [nn_n,Cn_1st_n,Cn_last_n,En_1st_n,En_last_n,Cn_n,En_n] = OER_cycle_avg(In_n,Vn_n)
5 %current
6 nn_n = ceil(numel(In_n)/2); %amount of half the data
7 Cn_1st_n = In_n(1:nn_n);%First half of data
8 Cn_last_n = flip(In_n(nn_n:end)); %Last half of data, flipped to match trend of first half
9 %voltage
10 En_1st_n = Vn_n(1:nn_n);%First half of data
11 En_last_n = flip(Vn_n(nn_n:end)); %Last half of data
12 %Averaging forward and backward sweeps to form lsv curves
13 %preallocating arrays prior to for loop use
14 Cn_n=zeros(nn_n,1);En_n=zeros(nn_n,1);
15
16 %performing for loop to split arrays into 1st and 20th cycles
17 for k=1:nn_n
18     Cn_n(k,1)=(Cn_1st_n(k)+Cn_last_n(k))/2;
19     En_n(k,1)=(En_1st_n(k)+En_last_n(k))/2;
20 end
21 end

```

G4: Matlab script for function that performs the Tafel slope calculation.

```

1 %This script develops a function that performs the tafel slope analysis on
2 %CV data for OER of 20 cycles
3 function [x,E_n,C_n,pn_n,an_n]=tafel_slope(y,z,En_n,Cn_n)
4 %Cycle analysis of LSV curves in tafel region
5 x=y; %starting value in tafel region
6 E_n=En_n(x:z); %voltage to be changed into overpotential for tafel plots
7 C_n=Cn_n(x:z); %current to be changed into logj for tafel plots
8
9 %Using linear regression to obtain the tafel slope and intercept
10 pn_n = polyfit(log(abs(C_n)),((E_n-1.23)),1);
11
12 %tafel equation
13 an_n=(pn_n(1).*log(abs(C_n)))+pn_n(2);% overpotential(y-value) of tafel eqn y=mx+b, after linearizing
14
15 end

```

G5: Matlab script for function that performs the averaging of currents from 3 replicates of 1 composition.

```

1 %This script merges 3 CV replicates into 1 CV curve by averaging them
2
3 function [JCn_n] = OER_rep_avg(Cn1_n,Cn2_n,Cn3_n)
4
5 %Averaging CV curves for 3 replicates of 1 composition
6 %performing for loop to split arrays into 1st and 20th cycles
7 %1st cycle
8     JCn_n=(Cn1_n+Cn2_n+Cn3_n)./3; %current
9
10 end

```

G6: Matlab script for processing collected data.

```

%This script generates a code that analyses data from bimetallic
% (cobalt and nickel) sulfide electrocatalysts attached to carbon
% nanofibers. These catalysts enhance oxygen evolution reaction via surface
% reconstruction into more oxidative species. Nitrogen gas was used for
% both curing and saturation

%preamble
clear all; close all; clc;

%constants
pH=13; %pH of electrolyte (KOH)
E0=0.098; %V equilibrium potential of Hg/HgO reference electrode
C=-1000/0.196; % current conversion constant to mA/cm^2 based on geometric
               % surface area of RDE disk

%-----
%Catalyst nanofiber voltage and current arrays
%-----
%Co9S8
%Reading data from downloaded file
%Replicate 1
R01=59.1; %electrolyte resistance in ohms
filename01='co9s8 rep 1.txt'; %filename for text file
[A01,V01,I01,n_01] = OER_rep(filename01);
%Replicate 2
R02=61.5; %electrolyte resistance in ohms
filename02='co9s8 rep2.txt'; %filename for text file
[A02,V02,I02,n_02] = OER_rep(filename02);
%Replicate 3
R03=56.1; %electrolyte resistance in ohms
filename03='cos rep3.txt'; %filename for text file
[A03,V03,I03,n_03] = OER_rep1(filename03);
%-----
%Ni.17Co.67S

```

```

%Reading data from downloaded file
%Replicate 1
R171=61.1; %electrolyte resistance in ohms
filename171='ni.17 rep1.txt'; %filename for text file
[A171,V171,I171,n_171] = OER_rep1(filename171);
%Replicate 2
R172=193.5; %electrolyte resistance in ohms
filename172='ni.17 rep2.txt'; %filename for text file
[A172,V172,I172,n_172] = OER_rep(filename172);
%Replicate 3
R173=61.1; %electrolyte resistance in ohms
filename173='ni.17 rep 3.txt'; %filename for text file
[A173,V173,I173,n_173] = OER_rep1(filename173);
%-----
%Ni.33Co.67S
%Reading data from downloaded file
%Replicate 1
R331=65; %electrolyte resistance in ohms
filename331='ni.33co.67s sample1 normal.txt'; %filename for text file
[A331,V331,I331,n_331] = OER_rep(filename331);

%Replicate 2
R332=63.6; %electrolyte resistance in ohms
filename332='ni.33co.67 sample 2 normal.txt'; %filename for text file
[A332,V332,I332,n_332] = OER_rep(filename332);

%Replicate 3
R333=70.6; %electrolyte resistance in ohms
filename333='ni.33 3rd replicate.txt'; %filename for text file
[A333,V333,I333,n_333] = OER_rep(filename333);
%-----
%Ni.5Co.5S
%Reading data from downloaded file
%Replicate 1
R51=95.5; %electrolyte resistance in ohms
filename51='ni.5s rep1.txt'; %filename for text file
[A51,V51,I51,n_51] = OER_rep(filename51);

%Replicate 2
R52=58.9; %electrolyte resistance in ohms
filename52='ni.5 rep2.txt'; %filename for text file
[A52,V52,I52,n_52] = OER_rep(filename52);

%Replicate 3
R53=78.1; %electrolyte resistance in ohms
filename53='ni.5 rep3.txt'; %filename for text file
[A53,V53,I53,n_553] = OER_rep(filename53);
%-----
%Ni.67Co.33S
%Reading data from downloaded file
%Replicate 1
R671=59.5; %electrolyte resistance in ohms
filename671='ni.67 rep1.txt'; %filename for text file
[A671,V671,I671,n_671] = OER_rep1(filename671);

```

```

%Replicate 2
R672=89.6; %electrolyte resistance in ohms
filename672='ni.67 rep2.txt'; %filename for text file
[A672,V672,I672,n_672] = OER_rep(filename672);

%Replicate 3
R673=141; %electrolyte resistance in ohms
filename673='ni.67 rep3.txt'; %filename for text file
[A673,V673,I673,n_673] = OER_rep1(filename673);
%-----
%Ni.83Co.17S
%Reading data from downloaded file
%Replicate 1
R831=56.6;%electrolyte resistance in ohms
filename831='ni.83 rep5.txt';%filename for text file
[A831,V831,I831,n_831] = OER_rep1(filename831);
%Replicate 2
R832=58.3; %electrolyte resistance in ohms
filename832='ni.83 rep3.txt'; %filename for text file
[A832,V832,I832,n_832] = OER_rep1(filename832);
%Replicate 3
R833=56.6; %electrolyte resistance in ohms
filename833='ni.83 rep4.txt'; %filename for text file
[A833,V833,I833,n_833] = OER_rep1(filename833);
%-----
%Ni3S2
%Reading data from downloaded file
%Replicate 1
R31=61.1; %electrolyte resistance in ohms
filename31='nis rep1.txt'; %filename for text file
[A31,V31,I31,n_31] = OER_rep(filename31);
%Replicate 2
R32=62.2; %electrolyte resistance in ohms
filename32='nis rep2 (1).txt'; %filename for text file
[A32,V32,I32,n_32] = OER_rep(filename32);
%Replicate 3
R33=59.9; %electrolyte resistance in ohms
filename33='nis rep3.txt'; %filename for text file
[A33,V33,I33,n_33] = OER_rep(filename33);
%-----
%-----

%Cycle separation into 1st and 20th for all NiCoS samples
%-----
%-----
%Co9S8
[I01_1,V01_1,I01_20,V01_20]=OER_cycle(I01,V01,R01); %Replicate 1 1st and 20th cycle voltage and
currents
[I02_1,V02_1,I02_20,V02_20]=OER_cycle(I02,V02,R02); %Replicate 2 1st and 20th cycle voltage and
currents
[I03_1,V03_1,I03_20,V03_20]=OER_cycle(I03,V03,R03); %Replicate 3 1st and 20th cycle voltage and
currents

```

```

%Ni.17Co.83S
[I171_1,V171_1,I171_20,V171_20]=OER_cycle(I171,V171,R171); %Replicate 1 1st and 20th cycle
voltage and currents
[I172_1,V172_1,I172_20,V172_20]=OER_cycle(I172,V172,R172); %Replicate 2 1st and 20th cycle
voltage and currents
[I173_1,V173_1,I173_20,V173_20]=OER_cycle(I173,V173,R173); %Replicate 3 1st and 20th cycle
voltage and currents
%Ni.33Co.67S
[I331_1,V331_1,I331_20,V331_20]=OER_cycle(I331,V331,R331); %Replicate 1 1st and 20th cycle
voltage and currents
[I332_1,V332_1,I332_20,V332_20]=OER_cycle(I332,V332,R332); %Replicate 2 1st and 20th cycle
voltage and currents
[I333_1,V333_1,I333_20,V333_20]=OER_cycle(I333,V333,R333); %Replicate 3 1st and 20th cycle
voltage and currents

%Ni.5Co.5S
[I51_1,V51_1,I51_20,V51_20]=OER_cycle(I51,V51,R51); %Replicate 1 1st and 20th cycle voltage and
currents
[I52_1,V52_1,I52_20,V52_20]=OER_cycle(I52,V52,R52); %Replicate 2 1st and 20th cycle voltage and
currents
[I53_1,V53_1,I53_20,V53_20]=OER_cycle(I53,V53,R53); %Replicate 3 1st and 20th cycle voltage and
currents

%Ni.67Co.33S
[I671_1,V671_1,I671_20,V671_20]=OER_cycle(I671,V671,R671); %Replicate 1 1st and 20th cycle
voltage and currents
[I672_1,V672_1,I672_20,V672_20]=OER_cycle(I672,V672,R672); %Replicate 2 1st and 20th cycle
voltage and currents
[I673_1,V673_1,I673_20,V673_20]=OER_cycle(I673,V673,R673); %Replicate 3 1st and 20th cycle
voltage and currents

%Ni.83Co.17S
[I831_1,V831_1,I831_20,V831_20]=OER_cycle(I831,V831,R831); %Replicate 1 1st and 20th cycle
voltage and currents
[I832_1,V832_1,I832_20,V832_20]=OER_cycle(I832,V832,R832); %Replicate 2 1st and 20th cycle
voltage and currents
[I833_1,V833_1,I833_20,V833_20]=OER_cycle(I833,V833,R833); %Replicate 3 1st and 20th cycle
voltage and currents

%Ni 3S2
[I31_1,V31_1,I31_20,V31_20]=OER_cycle(I31,V31,R31); %Replicate 1 1st and 20th cycle voltage and
currents
[I32_1,V32_1,I32_20,V32_20]=OER_cycle(I32,V32,R32); %Replicate 2 1st and 20th cycle voltage and
currents
[I33_1,V33_1,I33_20,V33_20]=OER_cycle(I33,V33,R33); %Replicate 3 1st and 20th cycle voltage and
currents

%-----
%-----
%-----
%-----
%AVERAGING 1st CYCLE DATA (LSV)
%Co9S8
[n01_1,C01_1st_1,C01_last_1,E01_1st_1,E01_last_1,C01_1,E01_1] = OER_cycle_avg(I01_1,V01_1);

```

```

%Replicate 1 1st cycle averaged
[n02_1,C02_1st_1,C02_last_1,E02_1st_1,E02_last_1,C02_1,E02_1] = OER_cycle_avg(I02_1,V02_1);
%Replicate 2 1st cycle averaged
[n03_1,C03_1st_1,C03_last_1,E03_1st_1,E03_last_1,C03_1,E03_1] = OER_cycle_avg(I03_1,V03_1);
%Replicate 3 1st cycle averaged
%-----
%-----

%Ni.17Co.83S
[n171_1,C171_1st_1,C171_last_1,E171_1st_1,E171_last_1,C171_1,E171_1] =
OER_cycle_avg(I171_1,V171_1); %Replicate 1 1st cycle averaged
[n172_1,C172_1st_1,C172_last_1,E172_1st_1,E172_last_1,C172_1,E172_1] =
OER_cycle_avg(I172_1,V172_1); %Replicate 2 1st cycle averaged
[n173_1,C173_1st_1,C173_last_1,E173_1st_1,E173_last_1,C173_1,E173_1] =
OER_cycle_avg(I173_1,V173_1); %Replicate 3 1st cycle averaged
%-----
%-----

%Ni.33Co.67S
[n331_1,C331_1st_1,C331_last_1,E331_1st_1,E331_last_1,C331_1,E331_1] =
OER_cycle_avg(I331_1,V331_1); %Replicate 1 1st cycle averaged
[n332_1,C332_1st_1,C332_last_1,E332_1st_1,E332_last_1,C332_1,E332_1] =
OER_cycle_avg(I332_1,V332_1); %Replicate 2 1st cycle averaged
[n333_1,C333_1st_1,C333_last_1,E333_1st_1,E333_last_1,C333_1,E333_1] =
OER_cycle_avg(I333_1,V333_1); %Replicate 3 1st cycle averaged
%-----
%-----

%Ni.5Co.5S
[n51_1,C51_1st_1,C51_last_1,E51_1st_1,E51_last_1,C51_1,E51_1] = OER_cycle_avg(I51_1,V51_1);
%Replicate 1 1st cycle averaged
[n52_1,C52_1st_1,C52_last_1,E52_1st_1,E52_last_1,C52_1,E52_1] = OER_cycle_avg(I52_1,V52_1);
%Replicate 2 1st cycle averaged
[n53_1,C53_1st_1,C53_last_1,E53_1st_1,E53_last_1,C53_1,E53_1] = OER_cycle_avg(I53_1,V53_1);
%Replicate 3 1st cycle averaged
%-----
%-----

%Ni.67Co.33S
[n671_1,C671_1st_1,C671_last_1,E671_1st_1,E671_last_1,C671_1,E671_1] =
OER_cycle_avg(I671_1,V671_1); %Replicate 1 1st cycle averaged
[n672_1,C672_1st_1,C672_last_1,E672_1st_1,E672_last_1,C672_1,E672_1] =
OER_cycle_avg(I672_1,V672_1); %Replicate 2 1st cycle averaged
[n673_1,C673_1st_1,C673_last_1,E673_1st_1,E673_last_1,C673_1,E673_1] =
OER_cycle_avg(I673_1,V673_1); %Replicate 3 1st cycle averaged
%-----
%-----

%Ni.83Co.17S
[n831_1,C831_1st_1,C831_last_1,E831_1st_1,E831_last_1,C831_1,E831_1] =
OER_cycle_avg(I831_1,V831_1); %Replicate 1 1st cycle averaged
[n832_1,C832_1st_1,C832_last_1,E832_1st_1,E832_last_1,C832_1,E832_1] =
OER_cycle_avg(I832_1,V832_1); %Replicate 2 1st cycle averaged
[n833_1,C833_1st_1,C833_last_1,E833_1st_1,E833_last_1,C833_1,E833_1] =
OER_cycle_avg(I833_1,V833_1); %Replicate 3 1st cycle averaged
%-----
%-----

%Ni3S2
[n31_1,C31_1st_1,C31_last_1,E31_1st_1,E31_last_1,C31_1,E31_1] = OER_cycle_avg(I31_1,V31_1);

```

```

%Replicate 1 1st cycle averaged
[n32_1,C32_1st_1,C32_last_1,E32_1st_1,E32_last_1,C32_1,E32_1] = OER_cycle_avg(I32_1,V32_1);
%Replicate 2 1st cycle averaged
[n33_1,C33_1st_1,C33_last_1,E33_1st_1,E33_last_1,C33_1,E33_1] = OER_cycle_avg(I33_1,V33_1);
%Replicate 3 1st cycle averaged

```

```

%-----
%-----
%-----

```

%AVERAGING 20th CYCLE DATA (LSV)

%Co9S8

```

[n01_20,C01_1st_20,C01_last_20,E01_1st_20,E01_last_20,C01_20,E01_20] =
OER_cycle_avg(I01_20,V01_20); %Replicate 1 1st cycle averaged
[n02_20,C02_1st_20,C02_last_20,E02_1st_20,E02_last_20,C02_20,E02_20] =
OER_cycle_avg(I02_20,V02_20); %Replicate 2 1st cycle averaged
[n03_20,C03_1st_20,C03_last_20,E03_1st_20,E03_last_20,C03_20,E03_20] =
OER_cycle_avg(I03_20,V03_20); %Replicate 3 1st cycle averaged

```

```

%-----
%-----

```

%Ni.17Co.83S

```

[n171_20,C171_1st_20,C171_last_20,E171_1st_20,E171_last_20,C171_20,E171_20] =
OER_cycle_avg(I171_20,V171_20); %Replicate 1 20th cycle averaged
[n172_20,C172_1st_20,C172_last_20,E172_1st_20,E172_last_20,C172_20,E172_20] =
OER_cycle_avg(I172_20,V172_20); %Replicate 2 20th cycle averaged
[n173_20,C173_1st_20,C173_last_20,E173_1st_20,E173_last_20,C173_20,E173_20] =
OER_cycle_avg(I173_20,V173_20); %Replicate 3 20th cycle averaged

```

```

%-----
%-----

```

%Ni.33Co.67S

```

[n331_20,C331_1st_20,C331_last_20,E331_1st_20,E331_last_20,C331_20,E331_20] =
OER_cycle_avg(I331_20,V331_20); %Replicate 1 20th cycle averaged
[n332_20,C332_1st_20,C332_last_20,E332_1st_20,E332_last_20,C332_20,E332_20] =
OER_cycle_avg(I332_20,V332_20); %Replicate 2 20th cycle averaged
[n333_20,C333_1st_20,C333_last_20,E333_1st_20,E333_last_20,C333_20,E333_20] =
OER_cycle_avg(I333_20,V333_20); %Replicate 3 20th cycle averaged

```

```

%-----
%-----

```

%Ni.5Co.5S

```

[n51_20,C51_1st_20,C51_last_20,E51_1st_20,E51_last_20,C51_20,E51_20] =
OER_cycle_avg(I51_20,V51_20); %Replicate 1 20th cycle averaged
[n52_20,C52_1st_20,C52_last_20,E52_1st_20,E52_last_20,C52_20,E52_20] =
OER_cycle_avg(I52_20,V52_20); %Replicate 2 20th cycle averaged
[n53_20,C53_1st_20,C53_last_20,E53_1st_20,E53_last_20,C53_20,E53_20] =
OER_cycle_avg(I53_20,V53_20); %Replicate 3 20th cycle averaged

```

```

%-----
%-----

```

%Ni.67Co.33S

```

[n671_20,C671_1st_20,C671_last_20,E671_1st_20,E671_last_20,C671_20,E671_20] =
OER_cycle_avg(I671_20,V671_20); %Replicate 1 20th cycle averaged
[n672_20,C672_1st_20,C672_last_20,E672_1st_20,E672_last_20,C672_20,E672_20] =
OER_cycle_avg(I672_20,V672_20); %Replicate 2 20th cycle averaged
[n673_20,C673_1st_20,C673_last_20,E673_1st_20,E673_last_20,C673_20,E673_20] =
OER_cycle_avg(I673_20,V673_20); %Replicate 3 20th cycle averaged

```

```

%-----

```

```

-----
%Ni.83Co.17S
[n831_20,c831_1st_20,c831_last_20,E831_1st_20,E831_last_20,c831_20,E831_20] =
OER_cycle_avg(I831_20,V831_20); %Replicate 1 20th cycle averaged
[n832_20,c832_1st_20,c832_last_20,E832_1st_20,E832_last_20,c832_20,E832_20] =
OER_cycle_avg(I832_20,V832_20); %Replicate 2 20th cycle averaged
[n833_20,c833_1st_20,c833_last_20,E833_1st_20,E833_last_20,c833_20,E833_20] =
OER_cycle_avg(I833_20,V833_20); %Replicate 3 20th cycle averaged
%-----
-----
%Ni3S2
[n31_20,c31_1st_20,c31_last_20,E31_1st_20,E31_last_20,c31_20,E31_20] =
OER_cycle_avg(I31_20,V31_20); %Replicate 1 20th cycle averaged
[n32_20,c32_1st_20,c32_last_20,E32_1st_20,E32_last_20,c32_20,E32_20] =
OER_cycle_avg(I32_20,V32_20); %Replicate 2 20th cycle averaged
[n33_20,c33_1st_20,c33_last_20,E33_1st_20,E33_last_20,c33_20,E33_20] =
OER_cycle_avg(I33_20,V33_20); %Replicate 3 20th cycle average
%-----
-----

%Plots of 1st and 20th cycles
%Co9S8
figure(1)
tiledlayout(1,2)
%top plot
ax01=nexttile;
plot(V01_1,I01_1,LineWidth=1.5)
hold on
plot(V02_1,I02_1,LineWidth=1.5)
plot(V03_1,I03_1,LineWidth=1.5)
plot(V01_20,I01_20,LineWidth=1.5)
plot(V02_20,I02_20,LineWidth=1.5)
plot(V03_20,I03_20,LineWidth=1.5)
xlim([0.8 2.0])
title('Co_9S_8')
legend('rep1:1','rep2:1','rep3:1','rep1:20','rep2:20','rep3:20',Location='northwest')
lgd = legend;
lgd.FontSize = 12;
xlabel('E-iR (V vs RHE)','FontSize', 12)
ylabel('j [mA/cm^2]','FontSize', 12)
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

%bottom plot
%LSV plots
ax02=nexttile;
plot(E01_1,C01_1,LineWidth=1.5)

```

```

hold on
plot(E02_1,C02_1,LineWidth=1.5)
plot(E03_1,C03_1,LineWidth=1.5)
plot(E01_20,C01_20,LineWidth=1.5)
plot(E02_20,C02_20,LineWidth=1.5)
plot(E03_20,C03_20,LineWidth=1.5)
xlabel('E-iR (V vs RHE)','FontSize', 12)
ylabel('j [mA/cm^2]','FontSize', 12)
legend('rep1:1','rep2:1','rep3:1','rep1:20','rep2:20','rep3:20',Location='northwest')
lgd = legend;
lgd.FontSize = 12;
xlim([0.8 2.0])
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

%Ni.17Co.83S
figure(2)
tiledlayout(1,2)
%top plot
ax171=nexttile;
plot(V171_1,I171_1,LineWidth=1.5)
hold on
plot(V172_1,I172_1,LineWidth=1.5)
plot(V173_1,I173_1,LineWidth=1.5)
plot(V171_20,I171_20,LineWidth=1.5)
plot(V172_20,I172_20,LineWidth=1.5)
plot(V173_20,I173_20,LineWidth=1.5)
xlim([0.8 2.0])
title('Ni_0._1_7Co_0._8_3S')
legend('rep1:1','rep2:1','rep3:1','rep1:20','rep2:20','rep3:20',Location='northwest')
lgd = legend;
lgd.FontSize = 12;
xlabel('E-iR (V vs RHE)','FontSize', 12)
ylabel('j [mA/cm^2]','FontSize', 12)
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

%bottom plot
%LSV plots

```

```

ax172=nexttile;
plot(E171_1,C171_1,LineWidth=1.5)
hold on
plot(E172_1,C172_1,LineWidth=1.5)
plot(E173_1,C173_1,LineWidth=1.5)
plot(E171_20,C171_20,LineWidth=1.5)
plot(E172_20,C172_20,LineWidth=1.5)
plot(E173_20,C173_20,LineWidth=1.5)
xlabel('E-iR (V vs RHE)','FontSize', 12)
ylabel('j [mA/cm^2]','FontSize', 12)
legend('rep1:1','rep2:1','rep3:1','rep1:20','rep2:20','rep3:20',Location='northwest')
lgd = legend;
lgd.FontSize = 12;
xlim([0.8 2.0])
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

%Ni.33Co.67S
figure(3)
tiledlayout(1,2)
%top plot
ax331=nexttile;
plot(V331_1,I331_1,LineWidth=1.5)
hold on
plot(V332_1,I332_1,LineWidth=1.5)
plot(V333_1,I333_1,LineWidth=1.5)
plot(V331_20,I331_20,LineWidth=1.5)
plot(V332_20,I332_20,LineWidth=1.5)
plot(V333_20,I333_20,LineWidth=1.5)
xlim([0.8 2.0])
title('Ni_0._3_3Co_0._6_7S')
xlabel('E-iR (V vs RHE)','FontSize', 12)
legend('rep1:1','rep2:1','rep3:1','rep1:20','rep2:20','rep3:20',Location='northwest')
lgd = legend;
lgd.FontSize = 12;
ylabel('j [mA/cm^2]','FontSize', 12)
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

%bottom plot

```

```

%LSV plots
ax332=nexttile;
plot(E331_1,C331_1,LineWidth=1.5)
hold on
plot(E332_1,C332_1,LineWidth=1.5)
plot(E333_1,C333_1,LineWidth=1.5)
plot(E331_20,C331_20,LineWidth=1.5)
plot(E332_20,C332_20,LineWidth=1.5)
plot(E333_20,C333_20,LineWidth=1.5)
xlabel('E-iR (V vs RHE)','FontSize', 12)
ylabel('j [mA/cm^2]','FontSize', 12)
legend('rep1:1','rep2:1','rep3:1','rep1:20','rep2:20','rep3:20',Location='northwest')
lgd = legend;
lgd.FontSize = 12;
xlim([0.8 2.0])
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.LineWidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

%plots for Ni0.5Co0.5S
figure(4)
tiledlayout(1,2)
%top plot
ax51=nexttile;
plot(V51_1,I51_1,LineWidth=1.5)
hold on
plot(V52_1,I52_1,LineWidth=1.5)
plot(V53_1,I53_1,LineWidth=1.5)
plot(V51_20,I51_20,LineWidth=1.5)
plot(V52_20,I52_20,LineWidth=1.5)
plot(V53_20,I53_20,LineWidth=1.5)
title('Ni0.5Co0.5S')
legend('rep1:1','rep2:1','rep3:1','rep1:20','rep2:20','rep3:20',Location='northwest')
lgd = legend;
lgd.FontSize = 12;
ylabel('j [mA/cm^2]','FontSize', 12)
xlabel('E-iR (V vs RHE)','FontSize', 12)
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.LineWidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

%bottom plot

```

```

%LSV
ax5=nexttile;
plot(E51_1,C51_1,Linewidth=1.5)
hold on
plot(E52_1,C52_1,Linewidth=1.5)
plot(E53_1,C53_1,Linewidth=1.5)
plot(E51_20,C51_20,Linewidth=1.5)
plot(E52_20,C52_20,Linewidth=1.5)
plot(E53_20,C53_20,Linewidth=1.5)
xlabel('E-iR (V vs RHE)','FontSize', 12)
ylabel('j [mA/cm^2]','FontSize', 12)
legend('rep1:1','rep2:1','rep3:1','rep1:20','rep2:20','rep3:20',Location='northwest')
lgd = legend;
lgd.FontSize = 12;
xlim([0.8 2.0])
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

%plots for Ni0.67Co0.33S
figure(5)
tiledlayout(1,2)
%top plot
ax671=nexttile;
plot(V671_1,I671_1,Linewidth=1.5)
hold on
plot(V672_1,I672_1,Linewidth=1.5)
plot(V673_1,I673_1,Linewidth=1.5)
plot(V671_20,I671_20,Linewidth=1.5)
plot(V672_20,I672_20,Linewidth=1.5)
plot(V673_20,I673_20,Linewidth=1.5)
ylabel('j [mA/cm^2]','FontSize', 12)
xlabel('E-iR (V vs RHE)','FontSize', 12)
title('Ni0.67Co0.33S')
legend('rep1:1','rep2:1','rep3:1','rep1:20','rep2:20','rep3:20',Location='northwest')
lgd = legend;
lgd.FontSize = 12;
xlim([0.8 2.0])
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

```

```

%bottom plot
%LSV plots
ax672=nexttile;
plot(E671_1,c671_1,linewidth=1.5)
hold on
plot(E672_1,c672_1,linewidth=1.5)
plot(E673_1,c673_1,linewidth=1.5)
plot(E671_20,c671_20,linewidth=1.5)
plot(E672_20,c672_20,linewidth=1.5)
plot(E673_20,c673_20,linewidth=1.5)
xlabel('E-iR (V vs RHE)','FontSize', 12)
ylabel('j [mA/cm^2]','FontSize', 12)
legend('rep1:1','rep2:1','rep3:1','rep1:20','rep2:20','rep3:20',Location='northwest')
lgd = legend;
lgd.FontSize = 12;
xlim([0.8 2.0])
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

%plots for Ni.83Co.17S
figure(6)
tiledlayout(1,2)
%top plot
ax831=nexttile;
plot(v831_1,i831_1,linewidth=1.5)
hold on
plot(v832_1,i832_1,linewidth=1.5)
plot(v833_1,i833_1,linewidth=1.5)
plot(v831_20,i831_20,linewidth=1.5)
plot(v832_20,i832_20,linewidth=1.5)
plot(v833_20,i833_20,linewidth=1.5)
ylabel('j [mA/cm^2]','FontSize', 12)
xlabel('E-iR (V vs RHE)','FontSize', 12)
title('Ni_0._.8_3Co_0._.1_7S')
legend('rep1:1','rep2:1','rep3:1','rep1:20','rep2:20','rep3:20',Location='northwest')
lgd = legend;
lgd.FontSize = 12;
xlim([0.8 2.0])
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

```

```

%bottom plot
%LSV plots
ax832=nexttile;
plot(E831_1,c831_1,linewidth=1.5)
hold on
plot(E832_1,c832_1,linewidth=1.5)
plot(E833_1,c833_1,linewidth=1.5)
plot(E831_20,c831_20,linewidth=1.5)
plot(E832_20,c832_20,linewidth=1.5)
plot(E833_20,c833_20,linewidth=1.5)
xlabel('E-iR (V vs RHE)','FontSize', 12)
ylabel('j [mA/cm^2]','FontSize', 12)
legend('rep1:1','rep2:1','rep3:1','rep1:20','rep2:20','rep3:20',Location='northwest')
lgd = legend;
lgd.FontSize = 12;
xlim([0.8 2.0])
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

%plots for Ni3S2
figure(7)
tiledlayout(1,2)
%top plot
ax31=nexttile;
plot(v31_1,i31_1,linewidth=1.5)
hold on
plot(v32_1,i32_1,linewidth=1.5)
plot(v33_1,i33_1,linewidth=1.5)
plot(v31_20,i31_20,linewidth=1.5)
plot(v32_20,i32_20,linewidth=1.5)
plot(v33_20,i33_20,linewidth=1.5)
ylabel('j [mA/cm^2]','FontSize', 12)
xlabel('E-iR (V vs RHE)','FontSize', 12)
title('Ni_3S_2')
legend('rep1:1','rep2:1','rep3:1','rep1:20','rep2:20','rep3:20',Location='northwest')
lgd = legend;
lgd.FontSize = 12;
xlim([0.8 2.0])
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;

```

```

leg.Box = 'off';

%bottom plot
%LSV plots
ax32=nexttile;
plot(E31_1,C31_1,LineWidth=1.5)
hold on
plot(E32_1,C32_1,LineWidth=1.5)
plot(E33_1,C33_1,LineWidth=1.5)
plot(E31_20,C31_20,LineWidth=1.5)
plot(E32_20,C32_20,LineWidth=1.5)
plot(E33_20,C33_20,LineWidth=1.5)
xlabel('E-iR (V vs RHE)','FontSize', 12)
ylabel('j [mA/cm^2]','FontSize', 12)
legend('rep1:1','rep2:1','rep3:1','rep1:20','rep2:20','rep3:20',Location='northwest')
lgd = legend;
lgd.FontSize = 12;
xlim([0.8 2.0])
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.LineWidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

%TAFEL SLOPE DETERMINATION
%1st CYCLE
%Co9S8
%Replicate 1
x01_1=590; %starting value in tafel region
y01_1=780; %starting value in tafel region
[x01_01,E01t_1,C01t_1,p01_1,a01_1]=tafel_slope(x01_1,y01_1,E01_1,C01_1); %p0_1 gives tafel slope
(p0_1(1)) and intercept (p0_1(2))
%Replicate 2
x02_1=590; %starting value in tafel region
y02_1=820; %ending value in tafel region
[x02_01,E02t_1,C02t_1,p02_1,a02_1]=tafel_slope(x02_1,y02_1,E02_1,C02_1); %p0_1 gives tafel slope
(p0_1(1)) and intercept (p0_1(2))
%Replicate 3
x03_1=590; %starting value in tafel region
y03_1=820; %ending value in tafel region
[x03_01,E03t_1,C03t_1,p03_1,a03_1]=tafel_slope(x03_1,y03_1,E03_1,C03_1) ;
%-----
%-----
%Ni.17Co.83S
%Replicate 1
x171_1=550; %starting value in tafel region
y171_1=660; %ending value in tafel region 680
[x171_01,E171t_1,C171t_1,p171_1,a171_1]=tafel_slope(x171_1,y171_1,E171_1,C171_1); %p0_1 gives
tafel slope (p0_1(1)) and intercept (p0_1(2))
%Replicate 2

```

```

x172_1=590;% %starting value in tafel region
y172_1=770;% %ending value in tafel region
[x172_01,E172t_1,C172t_1,p172_1,a172_1]=tafel_slope(x172_1,y172_1,E172_1,C172_1); %p0_1 gives
tafel slope (p0_1(1)) and intercept (p0_1(2))
%Replicate 3
x173_1=550; %starting value in tafel region
y173_1=710; %ending value in tafel region 750
[x173_01,E173t_1,C173t_1,p173_1,a173_1]=tafel_slope(x173_1,y173_1,E173_1,C173_1);
%-----
%Ni.33Co.67S
%Replicate 1
x331_1=620; %starting value in tafel region
y331_1=685; %ending value in tafel region
[x331_01,E331t_1,C331t_1,p331_1,a331_1]=tafel_slope(x331_1,y331_1,E331_1,C331_1); %p0_1 gives
tafel slope (p0_1(1)) and intercept (p0_1(2))
%Replicate 2
x332_1=600; %starting value in tafel region
y332_1=700; %ending value in tafel region
[x332_01,E332t_1,C332t_1,p332_1,a332_1]=tafel_slope(x332_1,y332_1,E332_1,C332_1);
%Replicate 3
x333_1=670; %starting value in tafel region
y333_1=760; %ending value in tafel region
[x333_01,E333t_1,C333t_1,p333_1,a333_1]=tafel_slope(x333_1,y333_1,E333_1,C333_1);
%-----
%Ni.5Co.5S
%Replicate 1
x51_1=550; %starting value in tafel region
y51_1=890; %ending value in tafel region
[x51_01,E51t_1,C51t_1,p51_1,a51_1]=tafel_slope(x51_1,y51_1,E51_1,C51_1); %p0_1 gives tafel slope
(p0_1(1)) and intercept (p0_1(2))
%Replicate 2
x52_1=570; %starting value in tafel region
y52_1=750; %ending value in tafel region
[x52_01,E52t_1,C52t_1,p52_1,a52_1]=tafel_slope(x52_1,y52_1,E52_1,C52_1);
%Replicate 3
x53_1=550; %starting value in tafel region
y53_1=870; %ending value in tafel region
[x53_01,E53t_1,C53t_1,p53_1,a53_1]=tafel_slope(x53_1,y53_1,E53_1,C53_1);
%-----
%Ni.67Co.33S
%Replicate 1
x671_1=580; %starting value in tafel region
y671_1=700; %ending value in tafel region
[x671_01,E671t_1,C671t_1,p671_1,a671_1]=tafel_slope(x671_1,y671_1,E671_1,C671_1); %p0_1 gives
tafel slope (p0_1(1)) and intercept (p0_1(2))
%Replicate 2
x672_1=700; %starting value in tafel region
y672_1=810; %ending value in tafel region
[x672_01,E672t_1,C672t_1,p672_1,a672_1]=tafel_slope(x672_1,y672_1,E672_1,C672_1);
%Replicate 3
x673_1=580; %starting value in tafel region

```

```

y673_1=850; %ending value in tafel region
[x673_01,E673t_1,C673t_1,p673_1,a673_1]=tafel_slope(x673_1,y673_1,E673_1,C673_1);
%-----
%Ni.83Co.17S
%Replicate 1
x831_1=620; %starting value in tafel region
y831_1=750; %ending value in tafel region
[x831_01,E831t_1,C831t_1,p831_1,a831_1]=tafel_slope(x831_1,y831_1,E831_1,C831_1) ;%p0_1 gives
tafel slope (p0_1(1)) and intercept (p0_1(2))
%Replicate 2
x832_1=620; %starting value in tafel region
y832_1=740; %ending value in tafel region
[x832_01,E832t_1,C832t_1,p832_1,a832_1]=tafel_slope(x832_1,y832_1,E832_1,C832_1);
%Replicate 3
x833_1=670; %starting value in tafel region
y833_1=790; %ending value in tafel region
[x833_01,E833t_1,C833t_1,p833_1,a833_1]=tafel_slope(x833_1,y833_1,E833_1,C833_1);
%-----
%Ni.3S2
%Replicate 1
x31_1=580; %starting value in tafel region
y31_1=800; %ending value in tafel region
[x31_01,E31t_1,C31t_1,p31_1,a31_1]=tafel_slope(x31_1,y31_1,E31_1,C31_1) ;%p0_1 gives tafel slope
(p0_1(1)) and intercept (p0_1(2))
%Replicate 2
x32_1=580; %starting value in tafel region
y32_1=800; %ending value in tafel region
[x32_01,E32t_1,C32t_1,p32_1,a32_1]=tafel_slope(x32_1,y32_1,E32_1,C32_1);
%Replicate 3
x33_1=580; %starting value in tafel region
y33_1=800; %ending value in tafel region
[x33_01,E33t_1,C33t_1,p33_1,a33_1]=tafel_slope(x33_1,y33_1,E33_1,C33_1);

%displaying the tafel slopes for all nanofibers
fprintf(['The 1st cycle tafel slopes (mV/dec) for compositions:Co_9S_8 rep ' ...
'1 %0.1f,Co_9S_8 rep 2 %0.1f, Co_9S_8 rep 3 %0.1f\n Ni.17 rep 1 %0.1f, ' ...
'Ni.17 rep 2 %0.1f,Ni.17 rep 3 %0.1f, \nNi.33 rep 1 %0.1f, Ni.33 rep 2 ' ...
'%0.1f, Ni.33 rep 3 %0.1f \n, Ni.5 rep1 %0.1f, Ni.5 rep 2 ' ...
'%0.1f, Ni.5 rep 3 %0.1f,\n Ni.67 rep 1 %0.1f, Ni.67 rep 2 %0.1f, ' ...
'Ni.67 rep 3 %0.1f \n, Ni.83 rep 1 %0.1f ,Ni.83 rep 2 %0.1f, Ni.83 rep 3' ...
' %0.1f\n,Ni3 rep 1 %0.1f, Ni3 rep 2 %0.1f, Ni3 rep 2 %0.1f\n'], ...
p01_1(1)*1000,p02_1(1)*1000,p03_1(1)*1000,p171_1(1)*1000,p172_1(1)*1000, ...
p173_1(1)*1000,p331_1(1)*1000,p332_1(1)*1000,p333_1(1)*1000,p51_1(1)*1000, ...
p52_1(1)*1000,p53_1(1)*1000,p671_1(1)*1000,p672_1(1)*1000,p673_1(1)*1000 ...
,p831_1(1)*1000,p832_1(1)*1000,p833_1(1)*1000,p31_1(1)*1000,p32_1(1)*1000 ...
,p33_1(1)*1000)

%-----
%TAFEL SLOPE DETERMINATION
%20th CYCLE
%Co9S8

```

```

%Replicate 1
x01_20=700; %starting value in tafel region
y01_20=770; %ending value in tafel region
[x01_020,E01t_20,C01t_20,p01_20,a01_20]=tafel_slope(x01_20,y01_20,E01_20,C01_20); %p0_1 gives
tafel slope (p0_1(1)) and intercept (p0_1(2))
%Replicate 2
x02_20=610; %starting value in tafel region
y02_20=850; %ending value in tafel region
[x02_020,E02t_20,C02t_20,p02_20,a02_20]=tafel_slope(x02_20,y02_20,E02_20,C02_20); %p0_1 gives
tafel slope (p0_1(1)) and intercept (p0_1(2))
%Replicate 3
x03_20=570; %starting value in tafel region
y03_20=800; %ending value in tafel region
[x03_020,E03t_20,C03t_20,p03_20,a03_20]=tafel_slope(x03_20,y03_20,E03_20,C03_20);
%-----
%-----

%Ni.17Co.83S
%Replicate 1
x171_20=550; %starting value in tafel region
y171_20=650; %ending value in tafel region 680
[x171_020,E171t_20,C171t_20,p171_20,a171_20]=tafel_slope(x171_20,y171_20,E171_20,C171_20); %p0_1
gives tafel slope (p0_1(1)) and intercept (p0_1(2))
%Replicate 2
x172_20=570; %starting value in tafel region
y172_20=750; %ending value in tafel region
[x172_020,E172t_20,C172t_20,p172_20,a172_20]=tafel_slope(x172_20,y172_20,E172_20,C172_20); %p0_1
gives tafel slope (p0_1(1)) and intercept (p0_1(2))
%Replicate 3
x173_20=560; %starting value in tafel region
y173_20=670; %ending value in tafel region 710
[x173_020,E173t_20,C173t_20,p173_20,a173_20]=tafel_slope(x173_20,y173_20,E173_20,C173_20) ;
%-----
%-----

%Ni.33Co.67S
%Replicate 1
x331_20=550; %starting value in tafel region
y331_20=650; %ending value in tafel region
[x331_020,E331t_20,C331t_20,p331_20,a331_20]=tafel_slope(x331_20,y331_20,E331_20,C331_20); %p0_1
gives tafel slope (p0_1(1)) and intercept (p0_1(2))
%Replicate 2
x332_20=580; %starting value in tafel region
y332_20=730; %ending value in tafel region
[x332_020,E332t_20,C332t_20,p332_20,a332_20]=tafel_slope(x332_20,y332_20,E332_20,C332_20) ;
%Replicate 3
x333_20=580; %starting value in tafel region
y333_20=750; %ending value in tafel region
[x333_020,E333t_20,C333t_20,p333_20,a333_20]=tafel_slope(x333_20,y333_20,E333_20,C333_20) ;
%-----
%-----

%Ni.5Co.5S
%Replicate 1
x51_20=550; %starting value in tafel region
y51_20=820; %ending value in tafel region
[x51_020,E51t_20,C51t_20,p51_20,a51_20]=tafel_slope(x51_20,y51_20,E51_20,C51_20) ;%p0_1 gives

```

```

tafel slope (p0_1(1)) and intercept (p0_1(2))
%Replicate 2
x52_20=550; %starting value in tafel region
y52_20=830; %ending value in tafel region
[x52_020,E52t_20,c52t_20,p52_20,a52_20]=tafel_slope(x52_20,y52_20,E52_20,c52_20);
%Replicate 3
x53_20=550; %starting value in tafel region
y53_20=770; %ending value in tafel region
[x53_020,E53t_20,c53t_20,p53_20,a53_20]=tafel_slope(x53_20,y53_20,E53_20,c53_20);
%-----
%-----

%Ni.67Co.33S
%Replicate 1
x671_20=580; %starting value in tafel region
y671_20=800; %ending value in tafel region
[x671_020,E671t_20,C671t_20,p671_20,a671_20]=tafel_slope(x671_20,y671_20,E671_20,c671_20) ;%p0_1
gives tafel slope (p0_1(1)) and intercept (p0_1(2))
%Replicate 2
x672_20=580; %starting value in tafel region
y672_20=730; %ending value in tafel region
[x672_020,E672t_20,C672t_20,p672_20,a672_20]=tafel_slope(x672_20,y672_20,E672_20,c672_20);
%Replicate 3
x673_20=580; %starting value in tafel region
y673_20=800; %ending value in tafel region
[x673_020,E673t_20,C673t_20,p673_20,a673_20]=tafel_slope(x673_20,y673_20,E673_20,c673_20);
%-----
%-----

%Ni.83Co.17S
%Replicate 1
x831_20=580; %starting value in tafel region
y831_20=720; %ending value in tafel region
[x831_020,E831t_20,C831t_20,p831_20,a831_20]=tafel_slope(x831_20,y831_20,E831_20,c831_20) ;%p0_1
gives tafel slope (p0_1(1)) and intercept (p0_1(2))
%Replicate 2
x832_20=580; %starting value in tafel region
y832_20=750; %ending value in tafel region
[x832_020,E832t_20,C832t_20,p832_20,a832_20]=tafel_slope(x832_20,y832_20,E832_20,c832_20);
%Replicate 3
x833_20=580; %starting value in tafel region
y833_20=730; %ending value in tafel region
[x833_020,E833t_20,C833t_20,p833_20,a833_20]=tafel_slope(x833_20,y833_20,E833_20,c833_20);
%-----
%-----

%Ni3S2
%Replicate 1
x31_20=600; %starting value in tafel region
y31_20=680; %ending value in tafel region
[x31_020,E31t_20,c31t_20,p31_20,a31_20]=tafel_slope(x31_20,y31_20,E31_20,c31_20) ;%p0_1 gives
tafel slope (p0_1(1)) and intercept (p0_1(2))
%Replicate 2
x32_20=630; %starting value in tafel region
y32_20=670; %ending value in tafel region
[x32_020,E32t_20,c32t_20,p32_20,a32_20]=tafel_slope(x32_20,y32_20,E32_20,c32_20);
%Replicate 3

```

```

x33_20=600; %starting value in tafel region
y33_20=740; %ending value in tafel region
[x33_20,E33t_20,c33t_20,p33_20,a33_20]=tafel_slope(x33_20,y33_20,E33_20,c33_20);
%-----
%displaying the tafel slopes for all nanofibers
fprintf(['The 20th cycle tafel slopes (mv/dec) for compositions: Co_9S_8 ' ...
'rep 1 %0.1f , Co_9S_8 rep 2 %0.1f, Co_9S_8 rep 3 %0.1f \n,Ni.17 rep 1' ...
' %0.1f , Ni.17 rep 2 %0.1f,Ni.17 rep 3 %0.1f Ni.33 rep 1 %0.1f mv/dec,' ...
' Ni.33 rep 2 %0.1f, Ni.33 rep 3 %0.1f \n, Ni.5 rep1 %0.1f, Ni.5 rep 2 ' ...
'%0.1f, Ni.5 rep 3 %0.1f, Ni.67 rep 1 %0.1f, Ni.67 rep 2 %0.1f ,Ni.67 ' ...
'rep 3 %0.1f\n, Ni.83 rep 1 %0.1f, Ni.83 rep 2 %0.1f, Ni.83 rep 3 ' ...
'%0.1f\n, Ni3 rep 1 %0.1f, Ni3 rep 2 %0.1f, Ni3 rep 3 %0.1f\n'], ...
p01_20(1)*1000,p02_20(1)*1000,p03_20(1)*1000,p171_20(1)*1000,p172_20(1) ...
*1000,p173_20(1)*1000,p331_20(1)*1000,p332_20(1)*1000,p333_20(1)*1000, ...
p51_20(1)*1000,p52_20(1)*1000,p53_20(1)*1000,p671_20(1)*1000,p672_20(1) ...
*1000,p673_20(1)*1000,p831_20(1)*1000,p832_20(1)*1000,p833_20(1)*1000, ...
p31_20(1)*1000,p32_20(1)*1000,p33_20(1)*1000)

%-----
%Plots to see if tafel region is covered prior to tafel plot determination
%so as to adjust the starting and ending indeces
figure(8)
tiledlayout(2,2)
%Co9S8
%top plot
ax03=nexttile;
plot(E01t_1,C01t_1,'-',Linewidth=1.5)
hold on
plot(E02t_1,C02t_1,'-',Linewidth=1.5)
plot(E03t_1,C03t_1,'-',Linewidth=1.5)
plot(E01t_20,C01t_20,'-',Linewidth=1.5)
plot(E02t_20,C02t_20,'-',Linewidth=1.5)
plot(E03t_20,C03t_20,'-',Linewidth=1.5)
ylabel('j [mA/cm^2]','FontSize', 12)
xlabel('E-iR (V vs RHE)','FontSize', 12)
title('Co_9S_8')
legend('rep1:1','rep2:1','rep3:1','rep1:20','rep2:20','rep3:20',Location='northwest')
xlim([0.8 2.0])
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

%Ni.17Co.83S
%right plot
ax173=nexttile;

```

```

plot(E171t_1,C171t_1,'-',Linewidth=1.5)
hold on
plot(E172t_1,C172t_1,'-',Linewidth=1.5)
plot(E173t_1,C173t_1,'-',Linewidth=1.5)
plot(E171t_20,C171t_20,'-',Linewidth=1.5)
plot(E172t_20,C172t_20,'-',Linewidth=1.5)
plot(E173t_20,C173t_20,'-',Linewidth=1.5)
ylabel('j [mA/cm^2]','FontSize', 12)
xlabel('E-iR (V vs RHE)','FontSize', 12)
title('Ni_0._1_7Co_0._8_3S')
legend('rep1:1','rep2:1','rep3:1','rep1:20','rep2:20','rep3:20',Location='northwest')
xlim([0.8 2.0])
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

%Ni.33Co.67S
%bottom left plot
ax33=nexttile;
plot(E331t_1,C331t_1,'-',Linewidth=1.5)
hold on
plot(E332t_1,C332t_1,'-',Linewidth=1.5)
plot(E333t_1,C333t_1,'-',Linewidth=1.5)
plot(E331t_20,C331t_20,'-',Linewidth=1.5)
plot(E332t_20,C332t_20,'-',Linewidth=1.5)
plot(E333t_20,C333t_20,'-',Linewidth=1.5)
ylabel('j [mA/cm^2]','FontSize', 12)
xlabel('E-iR (V vs RHE)','FontSize', 12)
title('Ni_0._3_3Co_0._6_7S')
legend('rep1:1','rep2:1','rep3:1','rep1:20','rep2:20','rep3:20',Location='northwest')
xlim([0.8 2.0])
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

%bottom right plot
ax53=nexttile;
plot(E51t_1,C51t_1,'-',Linewidth=1.5)
hold on
plot(E52t_1,C52t_1,'-',Linewidth=1.5)
plot(E53t_1,C53t_1,'-',Linewidth=1.5)
plot(E51t_20,C51t_20,'-',Linewidth=1.5)

```

```

plot(E52t_20,C52t_20,'-',Linewidth=1.5)
plot(E53t_20,C53t_20,'-',Linewidth=1.5)
ylabel('j [mA/cm^2]','FontSize', 12)
xlabel('E-iR (V vs RHE)','FontSize', 12)
legend('rep1:1','rep2:1','rep3:1','rep1:20','rep2:20','rep3:20',Location='northwest')
title('Ni_0._5Co_0._5S')
xlim([0.8 2.0])
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

figure(9)
tiledlayout(2,2)
%top right plot
ax673=nexttile;
plot(E671t_1,C671t_1,'-',Linewidth=1.5)
hold on
plot(E672t_1,C672t_1,'-',Linewidth=1.5)
plot(E673t_1,C673t_1,'-',Linewidth=1.5)
plot(E671t_20,C671t_20,'-',Linewidth=1.5)
plot(E672t_20,C672t_20,'-',Linewidth=1.5)
plot(E673t_20,C673t_20,'-',Linewidth=1.5)
ylabel('j [mA/cm^2]','FontSize', 12)
xlabel('E-iR (V vs RHE)','FontSize', 12)
legend('rep1:1','rep2:1','rep3:1','rep1:20','rep2:20','rep3:20',Location='northwest')
title('Ni_0._6_7Co_0._3_3S')
xlim([0.8 2.0])
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

%top left plot
ax833=nexttile;
plot(E831t_1,C831t_1,'-',Linewidth=1.5)
hold on
plot(E832t_1,C832t_1,'-',Linewidth=1.5)
plot(E833t_1,C833t_1,'-',Linewidth=1.5)
plot(E831t_20,C831t_20,'-',Linewidth=1.5)
plot(E832t_20,C832t_20,'-',Linewidth=1.5)
plot(E833t_20,C833t_20,'-',Linewidth=1.5)
ylabel('j [mA/cm^2]','FontSize', 12)
xlabel('E-iR (V vs RHE)','FontSize', 12)

```

```

legend('rep1:1','rep2:1','rep3:1','rep1:20','rep2:20','rep3:20',Location='northwest')
title('Ni_0_8_3Co_0_1_7S')
xlim([0.8 2.0])
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

%bottom left plot
ax33=nexttile;
plot(E31t_1,C31t_1,'-',Linewidth=1.5)
hold on
plot(E32t_1,C32t_1,'-',Linewidth=1.5)
plot(E33t_1,C33t_1,'-',Linewidth=1.5)
plot(E31t_20,C31t_20,'-',Linewidth=1.5)
plot(E32t_20,C32t_20,'-',Linewidth=1.5)
plot(E33t_20,C33t_20,'-',Linewidth=1.5)
ylabel('j [mA/cm^2]','FontSize', 12)
xlabel('E-iR (V vs RHE)','FontSize', 12)
legend('rep1:1','rep2:1','rep3:1','rep1:20','rep2:20','rep3:20',Location='northwest')
title('Ni_3S_2')
xlim([0.8 2.0])
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';
%-----

%Linearized tafel plots
%1st cycle
figure(10)
tiledlayout(2,2)
%top left plot
ax05=nexttile;
plot(log(abs(C01t_1)),a01_1,'Linewidth',1.5)
hold on
plot(log(abs(C02t_1)),a02_1,'Linewidth',1.5)
plot(log(abs(C03t_1)),a03_1,'Linewidth',1.5)
plot(log(abs(C01t_20)),a01_20,'Linewidth',1.5)
plot(log(abs(C02t_20)),a02_20,'Linewidth',1.5)
plot(log(abs(C03t_20)),a03_20,'Linewidth',1.5)
title('Co_9S_8')
legend('1:1','2:1','3:1','1:20','2:20','3:20',Location='northwest')
ylabel('Overpotential (V vs RHE)','FontSize', 12)

```

```

xlabel('log(j[ $\text{mA}/\text{cm}^2$ )]','FontSize', 12)
ylim([0.3 0.7])
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

%top right plot
ax175=nexttile;
plot(log(abs(C171t_1)),a171_1,'Linewidth',1.5)
hold on
plot(log(abs(C172t_1)),a172_1,'Linewidth',1.5)
plot(log(abs(C173t_1)),a173_1,'Linewidth',1.5)
plot(log(abs(C171t_20)),a171_20,'Linewidth',1.5)
plot(log(abs(C172t_20)),a172_20,'Linewidth',1.5)
plot(log(abs(C173t_20)),a173_20,'Linewidth',1.5)
title('Ni_0._1_7Co_0._8_3S')
legend('1:1','2:1','3:1','1:20','2:20','3:20',Location='northwest')
ylabel('Overpotential (V vs RHE)','FontSize', 12)
xlabel('log(j[ $\text{mA}/\text{cm}^2$ )]','FontSize', 12)
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

%bottom left plot
ax335=nexttile;
plot(log(abs(C331t_1)),a331_1,'Linewidth',1.5)
hold on
plot(log(abs(C332t_1)),a332_1,'Linewidth',1.5)
plot(log(abs(C333t_1)),a333_1,'Linewidth',1.5)
plot(log(abs(C331t_20)),a331_20,'Linewidth',1.5)
plot(log(abs(C332t_20)),a332_20,'Linewidth',1.5)
plot(log(abs(C333t_20)),a333_20,'Linewidth',1.5)
title('Ni_0._3_3Co_0._6_7S')
legend('1:1','2:1','3:1','1:20','2:20','3:20',Location='northwest')
ylabel('Overpotential (V vs RHE)')
xlabel('log(j[ $\text{mA}/\text{cm}^2$ )]','FontSize', 12)
title('Ni_0._3_3Co_0._6_7S','FontSize', 12)
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';

```

```

ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

%bottom right plot
ax55=nexttile;
plot(log(abs(C51t_1)),a51_1,'Linewidth',1.5)
hold on
plot(log(abs(C52t_1)),a52_1,'Linewidth',1.5)
plot(log(abs(C53t_1)),a53_1,'Linewidth',1.5)
plot(log(abs(C51t_20)),a51_20,'Linewidth',1.5)
plot(log(abs(C52t_20)),a52_20,'Linewidth',1.5)
plot(log(abs(C53t_20)),a53_20,'Linewidth',1.5)
legend('1:1','2:1','3:1','1:20','2:20','3:20',Location='northwest')
ylabel('Overpotential (V vs RHE)','FontSize', 12)
xlabel('log(j[mA/cm^2])','FontSize', 12)
title('Ni_0._5Co_0._5S')
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

figure(11)
tiledlayout(2,2)
%top left plot
ax675=nexttile;
plot(log(abs(C671t_1)),a671_1,'Linewidth',1.5)
hold on
plot(log(abs(C672t_1)),a672_1,'Linewidth',1.5)
plot(log(abs(C673t_1)),a673_1,'Linewidth',1.5)
plot(log(abs(C671t_20)),a671_20,'Linewidth',1.5)
plot(log(abs(C672t_20)),a672_20,'Linewidth',1.5)
plot(log(abs(C673t_20)),a673_20,'Linewidth',1.5)
legend('1:1','2:1','3:1','1:20','2:20','3:20',Location='northwest')
ylabel('Overpotential (V vs RHE)','FontSize', 12)
xlabel('log(j[mA/cm^2])','FontSize', 12)
title('Ni_0._6_7Co_0._3_3S')
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

%top right plot

```

```

ax835=nexttile;
plot(log(abs(C831t_1)),a831_1,'Linewidth',1.5)
hold on
plot(log(abs(C832t_1)),a832_1,'Linewidth',1.5)
plot(log(abs(C833t_1)),a833_1,'Linewidth',1.5)
plot(log(abs(C831t_20)),a831_20,'Linewidth',1.5)
plot(log(abs(C832t_20)),a832_20,'Linewidth',1.5)
plot(log(abs(C833t_20)),a833_20,'Linewidth',1.5)
legend('1:1','2:1','1:20','2:20',Location='northwest')
ylabel('Overpotential (V vs RHE)','FontSize', 12)
xlabel('log(j[ $\text{mA}/\text{cm}^2$ ])','FontSize', 12)
title('Ni_0_.8_3Co_0_.1_7S')
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

%top right plot
ax35=nexttile;
plot(log(abs(C31t_1)),a31_1,'Linewidth',1.5)
hold on
plot(log(abs(C32t_1)),a32_1,'Linewidth',1.5)
plot(log(abs(C33t_1)),a33_1,'Linewidth',1.5)
plot(log(abs(C31t_20)),a31_20,'Linewidth',1.5)
plot(log(abs(C32t_20)),a32_20,'Linewidth',1.5)
plot(log(abs(C33t_20)),a33_20,'Linewidth',1.5)
legend('1:1','2:1','3:1','1:20','2:20','3:20',Location='northwest')
ylabel('Overpotential (V vs RHE)','FontSize', 12)
xlabel('log(j[ $\text{mA}/\text{cm}^2$ ])','FontSize', 12)
title('Ni_3S_2')
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 1.5;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

%-----
%-----
%ONSITE POTENTIAL CALCULATION (@ 10  $\text{mA}/\text{cm}^2$ )
%Co9S8
%1st cycle
y01_onsite_1=((p01_1(1)*log(10))+ p01_1(2))*1000;
y02_onsite_1=((p02_1(1)*log(10))+ p02_1(2))*1000;
y03_onsite_1=((p03_1(1)*log(10))+ p03_1(2))*1000;

```

```

y0_onsite_1=[y01_onsite_1,y02_onsite_1,y03_onsite_1];

%Ni.17Co.83S
%1st cycle
y171_onsite_1=((p171_1(1)*log(10))+ p171_1(2))*1000;
y172_onsite_1=((p172_1(1)*log(10))+ p172_1(2))*1000;
y173_onsite_1=((p173_1(1)*log(10))+ p173_1(2))*1000;
y17_onsite_1=[y171_onsite_1,y172_onsite_1,y173_onsite_1];

%Ni.33Co.67S
%1st cycle
y331_onsite_1=((p331_1(1)*log(10))+ p331_1(2))*1000;
y332_onsite_1=((p332_1(1)*log(10))+ p332_1(2))*1000;
y333_onsite_1=((p333_1(1)*log(10))+ p333_1(2))*1000;
y330_onsite_1=[y331_onsite_1,y332_onsite_1,y333_onsite_1];
%Ni.5Co.5S
y51_onsite_1=((p51_1(1)*log(10))+ p51_1(2))*1000;
y52_onsite_1=((p52_1(1)*log(10))+ p52_1(2))*1000;
y53_onsite_1=((p53_1(1)*log(10))+ p53_1(2))*1000;
y5_onsite_1=[y51_onsite_1,y52_onsite_1,y53_onsite_1];

%Ni.67Co.33S
y671_onsite_1=((p671_1(1)*log(10))+ p671_1(2))*1000;
y672_onsite_1=((p672_1(1)*log(10))+ p672_1(2))*1000;
y673_onsite_1=((p673_1(1)*log(10))+ p673_1(2))*1000;
y67_onsite_1=[y671_onsite_1,y672_onsite_1,y673_onsite_1];

%Ni.83Co.17S
y831_onsite_1=((p831_1(1)*log(10))+ p831_1(2))*1000;
y832_onsite_1=((p832_1(1)*log(10))+ p832_1(2))*1000;
y833_onsite_1=((p833_1(1)*log(10))+ p833_1(2))*1000;
y83_onsite_1=[y831_onsite_1,y832_onsite_1,y833_onsite_1];

%Ni3S2
y31_onsite_1=((p31_1(1)*log(10))+ p31_1(2))*1000;
y32_onsite_1=((p32_1(1)*log(10))+ p32_1(2))*1000;
y33_onsite_1=((p33_1(1)*log(10))+ p33_1(2))*1000;
y3_onsite_1=[y31_onsite_1,y32_onsite_1,y33_onsite_1];

%20th cycle
%Co9S8
%1st cycle
y01_onsite_20=((p01_20(1)*log(10))+ p01_20(2))*1000;
y02_onsite_20=((p02_20(1)*log(10))+ p02_20(2))*1000;
y03_onsite_20=((p03_20(1)*log(10))+ p03_20(2))*1000;
y0_onsite_20=[y01_onsite_20,y02_onsite_1,y03_onsite_1];

%Ni.17Co.83S
y171_onsite_20=((p171_20(1)*log(10))+ p171_20(2))*1000;
y172_onsite_20=((p172_20(1)*log(10))+ p172_20(2))*1000;
y173_onsite_20=((p173_20(1)*log(10))+ p173_20(2))*1000;
y17_onsite_20=[y171_onsite_20,y172_onsite_20,y173_onsite_20];

%Ni.33Co.33S

```

```

y331_onsite_20=((p331_20(1)*log(10))+ p331_20(2))*1000;
y332_onsite_20=((p332_20(1)*log(10))+ p332_20(2))*1000;
y333_onsite_20=((p333_20(1)*log(10))+ p333_20(2))*1000;
y330_onsite_20=[y331_onsite_20,y332_onsite_20,y333_onsite_20];

```

%Ni.5Co.5S

```

y51_onsite_20=((p51_20(1)*log(10))+ p51_20(2))*1000;
y52_onsite_20=((p52_20(1)*log(10))+ p52_20(2))*1000;
y53_onsite_20=((p53_20(1)*log(10))+ p53_20(2))*1000;
y5_onsite_20=[y51_onsite_20,y52_onsite_20,y53_onsite_20];

```

%Ni.67Co.33S

```

y671_onsite_20=((p671_20(1)*log(10))+ p671_20(2))*1000;
y672_onsite_20=((p672_20(1)*log(10))+ p672_20(2))*1000;
y673_onsite_20=((p673_20(1)*log(10))+ p673_20(2))*1000;
y67_onsite_20=[y671_onsite_20,y672_onsite_20,y673_onsite_20];

```

%Ni.83Co.17S

```

y831_onsite_20=((p831_20(1)*log(10))+ p831_20(2))*1000;
y832_onsite_20=((p832_20(1)*log(10))+ p832_20(2))*1000;
y833_onsite_20=((p833_20(1)*log(10))+ p833_20(2))*1000;
y83_onsite_20=[y831_onsite_20,y832_onsite_20,y833_onsite_20];

```

%Ni3S2

```

y31_onsite_20=((p31_20(1)*log(10))+ p31_20(2))*1000;
y32_onsite_20=((p32_20(1)*log(10))+ p32_20(2))*1000;
y33_onsite_20=((p33_20(1)*log(10))+ p33_20(2))*1000;
y3_onsite_20=[y31_onsite_20,y32_onsite_20,y33_onsite_20];

```

%STATISTICAL ANALYSIS

%Average calculations

%Co9S8

```

y0_onsite_1avg=mean(y0_onsite_1);
y0_onsite_20avg=mean(y0_onsite_20);

```

%Ni.17Co.83S

```

y17_onsite_1avg=mean(y17_onsite_1);
y17_onsite_20avg=mean(y17_onsite_20);

```

%Ni.33Co.67S

```

y330_onsite_1avg=mean(y330_onsite_1);
y330_onsite_20avg=mean(y330_onsite_20);

```

%Ni.5Co.5S

```

y5_onsite_1avg=mean(y5_onsite_1);
y5_onsite_20avg=mean(y5_onsite_20);

```

%Ni.67Co.33S

```

y67_onsite_1avg=mean(y67_onsite_1);
y67_onsite_20avg=mean(y67_onsite_20);

```

%Ni.83Co.17S

```

y83_onsite_1avg=mean(y83_onsite_1);
y83_onsite_20avg=mean(y83_onsite_20);

```

```

%Ni3S2
y3_onsite_1avg=mean(y3_onsite_1);
y3_onsite_20avg=mean(y3_onsite_20);

%Standard deviation calculations
%Co9S8
y0_onsite_1std=std(y0_onsite_1);
y0_onsite_20std=std(y0_onsite_20);

%Ni.17Co.83S
y17_onsite_1std=std(y17_onsite_1);
y17_onsite_20std=std(y17_onsite_20);

%Ni.33Co.67S
y33_onsite_1std=std(y33_onsite_1);
y33_onsite_20std=std(y33_onsite_20);

%Ni.5Co.5S
y5_onsite_1std=std(y5_onsite_1);
y5_onsite_20std=std(y5_onsite_20);

%Ni.67Co.33S
y67_onsite_1std=std(y67_onsite_1);
y67_onsite_20std=std(y67_onsite_20);

%Ni.83Co.17S
y83_onsite_1std=std(y83_onsite_1);
y83_onsite_20std=std(y83_onsite_20);

%Ni3S2
y3_onsite_1std=std(y3_onsite_1);
y3_onsite_20std=std(y3_onsite_20);

%error
err_onsite_1=[y3_onsite_1std,y83_onsite_1std,y67_onsite_1std,y5_onsite_1std,y33_onsite_1std,y17_onsite_1std,y0_onsite_1std]; %standard deviation for cycle 1 with Ni composition in descending order
err_onsite_20=[y3_onsite_20std,y83_onsite_20std,y67_onsite_20std,y5_onsite_20std,y33_onsite_20std,y17_onsite_20std,y0_onsite_20std]; %standard deviation for cycle 20

%generation of average plots with error bars
%tafel slopes
x=[0,0.17,0.33,0.5,0.67,0.83,1]; %Co fraction

%onsite potential error bar plot
y1=[y3_onsite_1avg,y83_onsite_1avg,y67_onsite_1avg,y5_onsite_1avg,y33_onsite_1avg,y17_onsite_1avg,y0_onsite_1avg]; %average onsite potential for 1st cycle for all catalysts with Ni composition in descending order
y20=[y3_onsite_20avg,y83_onsite_20avg,y67_onsite_20avg,y5_onsite_20avg,y33_onsite_20avg,y17_onsite_20avg,y0_onsite_20avg]; %average onsite potential for 20th cycle for all catalysts

%Tafel slope statistical analysis
%Co9S8

```

```

y0_tafel_1=[p01_1(1),p02_1(1),p03_1(1)].*1000; %1st cycle
y0_tafel_avg1=mean(y0_tafel_1); %average tafel slope
y0_tafel_20=[p01_20(1),p02_20(1),p03_20(1)].*1000; %1st cycle
y0_tafel_avg20=mean(y0_tafel_20); %average tafel slope

%Ni.17Co.83S
y17_tafel_1=[p171_1(1),p172_1(1),p173_1(1)].*1000; %1st cycle
y17_tafel_avg1=mean(y17_tafel_1); %average tafel slope
y17_tafel_20=[p171_20(1),p172_20(1),p173_20(1)].*1000; %1st cycle
y17_tafel_avg20=mean(y17_tafel_20); %average tafel slope

%Ni.33Co.67S
y33_tafel_1=[p331_1(1),p332_1(1),p333_1(1)].*1000; %1st cycle
y33_tafel_avg1=mean(y33_tafel_1); %average tafel slope
y33_tafel_20=[p331_20(1),p332_20(1),p333_20(1)].*1000; %1st cycle
y33_tafel_avg20=mean(y33_tafel_20); %average tafel slope

%Ni.5Co.5S
y5_tafel_1=[p51_1(1),p52_1(1),p53_1(1)].*1000; %1st cycle
y5_tafel_avg1=mean(y5_tafel_1); %average tafel slope
y5_tafel_20=[p51_20(1),p52_20(1),p53_20(1)].*1000; %1st cycle
y5_tafel_avg20=mean(y5_tafel_20); %average tafel slope

%Ni.67Co.33S
y67_tafel_1=[p671_1(1),p672_1(1),p673_1(1)].*1000; %1st cycle
y67_tafel_avg1=mean(y67_tafel_1); %average tafel slope
y67_tafel_20=[p671_20(1),p672_20(1),p673_20(1)].*1000; %1st cycle
y67_tafel_avg20=mean(y67_tafel_20); %average tafel slope

%Ni.83Co.17S
y83_tafel_1=[p831_1(1),p832_1(1),p833_1(1)].*1000; %1st cycle
y83_tafel_avg1=mean(y83_tafel_1); %average tafel slope
y83_tafel_20=[p831_20(1),p832_20(1),p833_20(1)].*1000; %20th cycle
y83_tafel_avg20=mean(y83_tafel_20); %average tafel slope

%Ni3S2
y3_tafel_1=[p31_1(1),p32_1(1),p33_1(1)].*1000; %1st cycle
y3_tafel_avg1=mean(y3_tafel_1); %average tafel slope
y3_tafel_20=[p31_20(1),p32_20(1),p33_20(1)].*1000; %1st cycle
y3_tafel_avg20=mean(y3_tafel_20); %average tafel slope

%standard deviation
%Co9S8
y0_tafel_1std=std(y0_tafel_1);
y0_tafel_20std=std(y0_tafel_20);

%Ni.17Co.83S
y17_tafel_1std=std(y17_tafel_1);
y17_tafel_20std=std(y17_tafel_20);

%Ni.33Co.67S
y33_tafel_1std=std(y33_tafel_1);
y33_tafel_20std=std(y33_tafel_20);

```

```

%Ni.5Co.5S
y5_tafel_1std=std(y5_tafel_1);
y5_tafel_20std=std(y5_tafel_20);

%Ni.67Co.67S
y67_tafel_1std=std(y67_tafel_1);
y67_tafel_20std=std(y67_tafel_20);

%Ni.83Co.17S
y83_tafel_1std=std(y83_tafel_1);
y83_tafel_20std=std(y83_tafel_20);

%Ni3S2
y3_tafel_1std=std(y3_tafel_1);
y3_tafel_20std=std(y3_tafel_20);

%error
err_tafel_1=[y3_tafel_1std,y83_tafel_1std,y67_tafel_1std,y5_tafel_1std,y33_tafel_1std,y17_tafel_1std,y0_tafel_1std]; %standard deviations for cycles 1
err_tafel_20=[y3_tafel_20std,y83_tafel_20std,y67_tafel_20std,y5_tafel_20std,y33_tafel_20std,y17_tafel_20std,y0_tafel_20std]; %standard deviations for cycles 20

%tafel slope error bar plot
yt1=[y3_tafel_avg1,y83_tafel_avg1,y67_tafel_avg1,y5_tafel_avg1,y33_tafel_avg1,y17_tafel_avg1,y0_tafel_avg1]; %average tafel slope for 1st cycle for all nanofibers
yt20=[y3_tafel_avg20,y83_tafel_avg20,y67_tafel_avg20,y5_tafel_avg20,y33_tafel_avg20,y17_tafel_avg20,y0_tafel_avg20]; %average onsite potential for 20th cycle for all nanofibers

%onsite potential error bar plot
figure(12)
p1=plot(x,y1,'k^',Linewidth=0.8);
hold on
p20=plot(x,y20,'kx',Linewidth=0.8);
errorbar(x,y1,err_onsite_1,'k^',Linewidth=0.8)
errorbar(x,y20,err_onsite_20,'kx',Linewidth=0.8)
legend([p1 p20],{'Cycle 1','Cycle 20'})
lgd = legend;
lgd.FontSize = 12;
xticks([0 0.17 0.33 0.5 0.67 0.83 1])
xlim([0 1])
ylim([250 850])
xlabel('Co fraction in Co_xNi_1-_xS_y','FontSize', 12)
ylabel('Overpotential at 10 mAcm^-^2 [mV]','FontSize', 12)
xlim([-0.1 1.1])
%Removing major ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 2;
ax.FontSize = 14;
ax.TickLength = [0 0];
ax.XMinorTick = 'off';
ax.YMinorTick = 'off';
%removing legend box
leg = ax.Legend;

```

```

leg.Box = 'off';

%Tafel slope error bar plot
figure(13)
pt1=plot(x, yt1, 'k^', Linewidth=0.8);
hold on
pt20=plot(x, yt20, 'kx', Linewidth=0.8);
errorbar(x, yt1, err_tafel_1, 'k^', Linewidth=0.8)
errorbar(x, yt20, err_tafel_20, 'kx', Linewidth=0.8)
legend([pt1 pt20], {'cycle 1', 'cycle 20'})
lgd = legend;
lgd.FontSize = 12;
xticks([0 0.17 0.33 0.5 0.67 0.83 1])
xlim([-0.1 1.1])
ylim([5 170])
xlabel('Co fraction in Co_xNi_1-_xs_y', 'FontSize', 14)
ylabel('Tafel slope [mv dec^-1]', 'FontSize', 14)
%Removing major ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.Linewidth = 2;
ax.FontSize = 14;
ax.TickLength = [0 0];
ax.XMinorTick = 'off';
ax.YMinorTick = 'off';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

%COMPOSITE LSV AND CV ANALYSIS WITH AVERAGED 3 REPLICATES
%Co9S8
[J0_1] = OER_rep_avg(I01_1, I02_1, I03_1);
[JL0_1] = OER_rep_avg(C01_1, C02_1, C03_1);

%Ni.17Co.83S
[J17_1] = OER_rep_avg(I171_1, I172_1, I173_1);
[JL17_1] = OER_rep_avg(C171_1, C172_1, C173_1);

%Ni.33Co.67S
[J33_1] = OER_rep_avg(I331_1, I332_1, I333_1);
[JL33_1] = OER_rep_avg(C331_1, C332_1, C333_1);

%Ni.5Co.5S
[J5_1] = OER_rep_avg(I51_1, I52_1, I53_1);
[JL5_1] = OER_rep_avg(C51_1, C52_1, C53_1);

%Ni.67Co.33S
[J67_1] = OER_rep_avg(I671_1, I672_1, I673_1);
[JL67_1] = OER_rep_avg(C671_1, C672_1, C673_1);

%Ni.83Co.17S
[J83_1] = OER_rep_avg(I831_1, I832_1, I833_1);
[JL83_1] = OER_rep_avg(C831_1, C832_1, C833_1);

```

```

%Ni3S2
[J3_1] = OER_rep_avg(I31_1,I32_1,I33_1);
[JL3_1] = OER_rep_avg(C31_1,C32_1,C33_1);

%Composite CV and plots
%LSV plots
figure(14)
plot(E01_1,JL0_1,LineWidth=1.5)
hold on
plot(E171_1,JL17_1,LineWidth=1.5)
plot(E331_1,JL33_1,LineWidth=1.5)
plot(E51_1,JL5_1,LineWidth=1.5)
plot(E671_1,JL67_1,LineWidth=1.5)
plot(E831_1,JL83_1,LineWidth=1.5)
plot(E31_1,JL3_1,LineWidth=1.5)
xlabel('Potential-iR (V vs RHE)','FontSize', 14)
ylabel('Current density (mA/cm^2)','FontSize', 14)
legend('Co_9S_8', 'Ni_0_.1_7Co_0_.8_3S', 'Ni_0_.3_3Co_0_.6_7S', ...
       'Ni_0_.5Co_0_.5S', 'Ni_0_.6_7Co_0_.3_3S', 'Ni_0_.8_3Co_0_.1_7S', ...
       'Ni_3S_2',Location='northwest')
lgd = legend;
lgd.FontSize = 12;
xlim([0.8 2.1])
%adding minor ticks and thickening plot's border
ax = gca;
ax.Box = 'on';
ax.LineWidth = 1.5;
ax.FontSize = 14;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

%CV plots
figure(15)
plot(V01_1,J0_1,LineWidth=1.5)
hold on
plot(V171_1,J17_1,LineWidth=1.5)
plot(V331_1,J33_1,LineWidth=1.5)
plot(V51_1,J5_1,LineWidth=1.5)
plot(V671_1,J67_1,LineWidth=1.5)
plot(V831_1,J83_1,LineWidth=1.5)
plot(V31_1,J3_1,LineWidth=1.5)
xlabel('Potential-iR (V vs RHE)','FontSize', 14)
ylabel('Current density (mA/cm^2)','FontSize', 14)
legend('Co_9S_8', 'Ni_0_.1_7Co_0_.8_3S', 'Ni_0_.3_3Co_0_.6_7S', ...
       'Ni_0_.5Co_0_.5S', 'Ni_0_.6_7Co_0_.3_3S', 'Ni_0_.8_3Co_0_.1_7S', ...
       'Ni_3S_2',Location='northwest')
lgd = legend;
lgd.FontSize = 12;
xlim([0.8 2.1])
%adding minor ticks and thickening plot's border
ax = gca;

```

```

ax.Box = 'on';
ax.Linewidth = 1.5;
ax.FontSize = 14;
ax.XMinorTick = 'on';
ax.YMinorTick = 'on';
%removing legend box
leg = ax.Legend;
leg.Box = 'off';

```

The 1st cycle tafel slopes (mV/dec) for compositions:Co_9S_8 rep 1 79.8,Co_9S_8 rep 2 96.9, Co_9S_8 rep 3 87.8

Ni.17 rep 1 85.3, Ni.17 rep 2 63.8,Ni.17 rep 3 85.2,
 Ni.33 rep 1 129.3, Ni.33 rep 2 108.6, Ni.33 rep 3 97.6
 , Ni.5 rep1 106.3, Ni.5 rep 2 127.2, Ni.5 rep 3 95.5,
 Ni.67 rep 1 101.2, Ni.67 rep 2 88.3, Ni.67 rep 3 92.9
 , Ni.83 rep 1 102.8 ,Ni.83 rep 2 84.2, Ni.83 rep 3 130.7
 ,Ni3 rep 1 98.9, Ni3 rep 2 91.0, Ni3 rep 2 89.8

The 20th cycle tafel slopes (mV/dec) for compositions: Co_9S_8 rep 1 81.9 , Co_9S_8 rep 2 108.9, Co_9S_8 rep 3 102.8

,Ni.17 rep 1 40.2 , Ni.17 rep 2 30.8,Ni.17 rep 3 54.5 Ni.33 rep 1 55.6 mV/dec, Ni.33 rep 2 59.9, Ni.33 rep 3 72.8
 , Ni.5 rep1 72.7, Ni.5 rep 2 78.5, Ni.5 rep 3 75.4, Ni.67 rep 1 81.4, Ni.67 rep 2 54.8 ,Ni.67 rep 3 60.8
 , Ni.83 rep 1 79.1, Ni.83 rep 2 68.3, Ni.83 rep 3 86.9
 , Ni3 rep 1 83.4, Ni3 rep 2 54.9, Ni3 rep 3 74.2

