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Effect of $\text{Ni}(\text{NO}_3)_2$ Precursor Concentration on Electrodeposited NiMn Electrocatalysts for HMF Oxidation to DFF

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Abstract: This study aims to develop a Ni–Mn bimetallic electrocatalyst for the selective oxidation of 5-hydroxymethylfurfural (HMF) to 2,5-diformylfuran (DFF) via electrodeposition. The NiMn electrocatalyst was synthesized by varying precursor concentrations to tune its structural and electrochemical properties. The results show that an optimal concentration produces a highly crystalline NiMn structure with improved electrochemical performance. The optimized catalyst achieves 98.8% selectivity and 8.5% yield for DFF at 1.75 V vs. RHE over 6 hours, while simultaneously enabling hydrogen evolution at the cathode, demonstrating efficient biomass upgrading with value-added hydrogen production.

Keywords: 2,5-diformylfuran; 5-hydroxymethylfurfural; electrocatalyst; electrodeposition; oxidation

1. Introduction

Electrochemistry is one of the manufacturing processes that has several benefits, such as the capability to be utilized on an industrial scale, the capacity to easily manage the output that is required, and the capability to operate on green energy as a source of electrical power^{1–4}. There is now a significant amount of research being carried out on a wide range of electrode materials, including both precious and non-precious metals⁵. It is believed that the various types of materials employed can influence the activities occurring at the electrode. These processes include the acceleration of their kinetics^{6,7}, the control of the course of reactions in complicated reactions^{8,9}, and the improvement of Faraday efficiency in electrochemical systems^{10,11}. The shortage of precious metals is a distinct problem that has to be addressed, although they are still widely utilized as electrodes in industry due to the better qualities they provide^{12,13}. Because of this, it is necessary to conduct research into other ways to substitute valuable metals that are restricted. It is well known that materials used for electrodes made of transition metals exhibit powerful catalytic activity in various electrochemical processes, such as the splitting of water or the transformation of biomass^{14–18}. As an additional point of

interest, transition metals are well-known for their vast availability, low cost, and environmental safety^{19,20}.

According to several studies that have been conducted on transition metal-based materials that include catalytic capabilities, the combination of nickel and manganese is incredibly intriguing since it possesses much higher performance than it does when it is in its solitary condition^{21,22}. In addition, previous studies have demonstrated that the utilization of NiMn as a heterogeneous catalyst is the most effective method for the production of DFF (2,5-furandicarboxaldehyde)²¹, a chemical platform that is crucial in a number of different industrial sectors, including medicines, fungicides, furan-urea-based resins, and the ligand industry^{23–26}. However, the commercial use of homogeneous systems still needs further separation processes^{27–30}, while heterogeneous catalysts are limited due to the existence of high-pressure and high-temperature gases that are required for their development^{31–33}.

Nickel- and manganese-based materials have attracted considerable attention as electrocatalysts due to their abundance^{34,35}, low cost^{36,37}, and relatively low toxicity³⁸ compared with noble metals. Both metals are widely available transition metals and exhibit good electrochemical activity as well as chemical stability in

alkaline or mildly alkaline electrolytes^{39–41}). When synthesizing NiMn catalysts as electrodes, particularly for the creation of electrochemical DFF, it is necessary to pay particular attention to the aforementioned requirements. For the purpose of this inquiry, NiMn electrocatalysts were made by the process of electrodeposition because of the ease and accuracy with which it governs structure, thickness, and other features^{42–44}). As an additional point of interest, the findings of this research indicate that the concentration of the precursor during the electrodeposition process has a substantial influence on the crystal structure of the NiMn. Additionally, this study investigates the effect of precursor concentration on the formation of the NiMn crystal structure and its influence on the electrochemical oxidation of HMF to DFF. The process simultaneously produces hydrogen gas at the cathode, highlighting the coupled value-added production of DFF and clean hydrogen energy.

2. Materials and Methods

2.1. Materials

Chemicals utilized in this study include the following: 5-hydroxymethyl-2-furaldehyde (HMF) (99%, Sigma-Aldrich); 2,5-furandicarboxaldehyde (DFF) (97%, Sigma-Aldrich); $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (99%, Sigma-Aldrich); $\text{MnSO}_4 \cdot \text{H}_2\text{O}$ (99%, Sigma-Aldrich); $\text{Na}_2\text{B}_4\text{O}_7$ (pH 9.6) (98%, Merck); Nickel Plate; Aquadest. The electrochemical system employs a cell without a separator and a three-electrode arrangement, with Ag/AgCl 3 M KCl and platinum mesh operating as the reference and counter electrodes, respectively. This study employed a potentiostat (PalmSens) for driving the electrochemical process. The material was characterized using X-ray powder diffraction (D8 Advance (Bruker)), and the product was examined using high-performance liquid chromatography (Nexera LC-40) with a UV detector and the C18 column at 55°C, with an eluent containing 5 mM sulfuric acid.

2.2. Methods

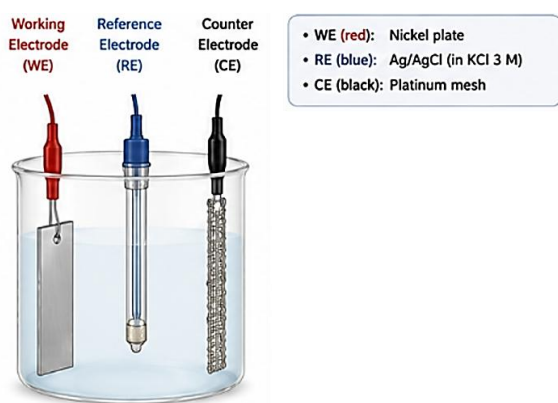


Fig. 1: Schematic diagram of the NiMn electrodeposition process using a three-electrode system

The nickel plate went through cleaning with a sonicator in 3 M HCl, acetone, and ethanol for 15 minutes each prior to usage⁴⁵). The nickel plate was subsequently dried and restricted to an area of 1 cm² on each side. This study employed an electrodeposition method to produce a NiMn electrocatalyst on a nickel plate substrate as in Figure 1.

A constant voltage of -1.0 V vs. Ag/AgCl⁴⁶) was applied to the electrochemical cell for a duration of 60 seconds, utilizing the nickel plate as the working electrode. This investigation employed a precursor solution comprising $\text{Ni}(\text{NO}_3)_2$ at varying concentrations (0.03 M, 0.10 M, 0.15 M, and 0.20 M) and MnSO_4 in a 1:2⁴⁷) ratio relative to $\text{Ni}(\text{NO}_3)_2$. The resulting NiMn deposits were designated as NiMn-0.03, NiMn-0.10, NiMn-0.15, and NiMn-0.20 according to the $\text{Ni}(\text{NO}_3)_2$ concentration used, respectively. The NiMn deposit on the nickel plate's surface was subsequently washed with distilled water and dried at room temperature prior to use. The i-v curve presented in this study was obtained through cyclic voltammetry (CV) within a potential range of 0.0 to 1.0 V relative to Ag/AgCl. The measurements employed a potential step of 0.01 V and a scan rate of 0.01 V/s across three scans using a 0.1 M $\text{Na}_2\text{B}_4\text{O}_7$ electrolyte. The NiMn electrocatalyst exhibiting the lowest overpotential will subsequently be utilized to generate DFF from the HMF oxidation reaction over a duration of 6 hours, employing the chronoamperometry method. The parameters utilized included room temperature, a 1 mM concentration of HMF, and stirring with a magnetic stirrer. For product analysis, 200 μL of electrolyte was collected before and after the reaction, diluted with distilled water to a final volume of 2 mL, and subsequently injected into the HPLC^{48,49}). The HPLC curve will be analyzed in relation to the previously obtained standard curves for HMF and DFF. The HPLC results will be employed to calculate the HMF conversion, DFF yield, and DFF selectivity according to equations (1), (2), and (3), respectively⁵⁰).

$$C_{\text{HMF}} = (n_{0,\text{HMF}} - n_{x,\text{HMF}}) / n_{0,\text{HMF}} \times 100\% \quad (1)$$

$$Y_{\text{DFF}} = n_{x,\text{DFF}} / n_{0,\text{HMF}} \times 100\% \quad (2)$$

$$S_{\text{DFF}} = n_{x,\text{DFF}} / (n_{0,\text{HMF}} - n_{x,\text{HMF}}) \times 100\% \quad (3)$$

3. Results and Discussion

3.1. Effect of precursor concentration on electrochemical characteristics

Changing the concentration of the precursor has a significant influence on the electrochemical performance of the NiMn electrocatalyst, particularly on the overpotential required for the water oxidation reaction. This effect is evident from the i-v curves (Figure 2), which were recorded from the anodic scan using the cyclic voltammetry (CV) technique. Among the tested precursor

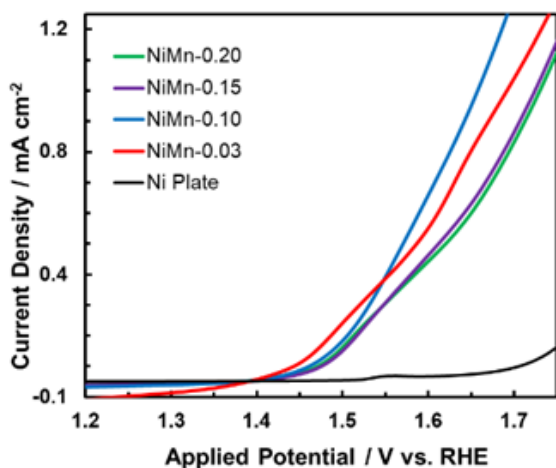


Fig. 2: LSV curves of NiMn electrodes prepared using different precursor concentrations for electrodeposition.

concentrations, the electrocatalyst synthesized from 0.10 M $\text{Ni}(\text{NO}_3)_2$ and 0.05 M MnSO_4 exhibited the most superior catalytic activity, indicating an optimal balance between metal ion availability and deposition kinetics. At this concentration, the electrode displays the highest current density, signifying an enhanced charge transfer rate during oxidation reaction⁵¹.

In contrast, at lower precursor concentrations (e.g., NiMn-0.03), the electrode exhibits a higher overpotential to reach 1.0 mA/cm^2 , suggesting that insufficient ion concentration during electrodeposition results in a thinner or less compact catalyst layer with fewer active centers^{52,53}. Conversely, when the precursor concentration becomes too high (NiMn-0.15 or NiMn-0.20), although the current density initially increases, excessive deposition may lead to denser films that hinder electrolyte penetration and reduce the number of accessible active sites, ultimately limiting performance⁵⁴. Notably, the overpotential of NiMn-0.10 decreases by approximately 400 mV compared to NiMn-0.03 at a current density of 1.0 mA/cm^2 . This substantial reduction confirms that optimizing precursor concentration is a key factor in achieving efficient oxidation reaction catalysis.

3.2. Effect of precursor concentration on material characteristics

Furthermore, the XRD result shown in Figure 3 reveals how varying the precursor concentration affects the crystal structure of the NiMn electrocatalyst. The diffraction peaks at 44.69° , 52.02° , and 76.61° correspond to the (111), (200), and (220) planes of nickel^{55,56}, while those at 44.69° and 76.61° are also related to the (400) and (622) planes of NiMn oxide⁵⁷. The higher intensity of these peaks in NiMn-0.10 indicates better crystallinity and improved lattice ordering⁵⁸ compared to NiMn-0.03.

A new peak appearing at 16.91° is observed only in NiMn-0.10, which may correspond to either the (111) plane of

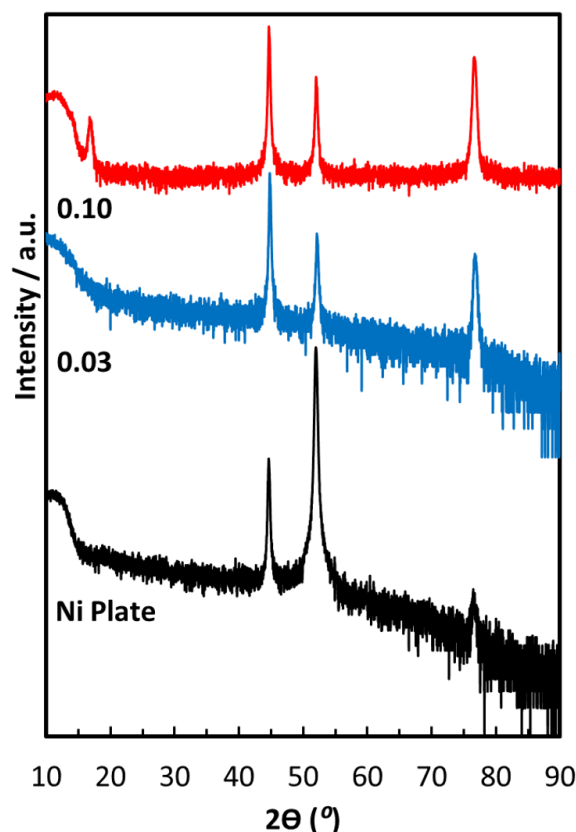


Fig. 3: XRD pattern of NiMn-0.10, NiMn-0.03, and its substrate (nickel plate)

NiMn_2O_4 ⁵⁹ or metal hydroxide (001)^{60–62}. The overlapping nature of these possible assignments suggests that this feature likely originates from structural modification induced by Mn incorporation, rather than providing definitive evidence for a specific crystalline phase. This observation suggests that an optimal precursor concentration promotes improved structural organization and possible short-range ordering within the Ni–Mn system. In contrast, the broader peaks of NiMn-0.03 indicate a more amorphous nature with smaller crystallite domains. At excessively high precursor concentrations, however, rapid nucleation and deposition can hinder atom rearrangement into an ordered lattice, resulting in disordered growth and lattice defects⁶³. Thus, while a moderate concentration (NiMn-0.10) enhances crystallinity, overly concentrated solutions may instead yield a less ordered, amorphous structure. Overall, NiMn-0.10 presents the most balanced crystalline structure, supporting better catalytic performance.

The FTIR spectra shown in Figure 4 display the characteristic vibration bands of NiMn electrocatalysts synthesized with different precursor concentrations. The broad absorption band around 3400 cm^{-1} corresponds to the O–H stretching vibration^{64,65}, indicating the presence of surface hydroxyl groups and adsorbed water molecules. The band at approximately 1630 cm^{-1} is attributed to the bending vibration of –OH, confirming the existence of interlayer or surface-bound water^{66,67}.

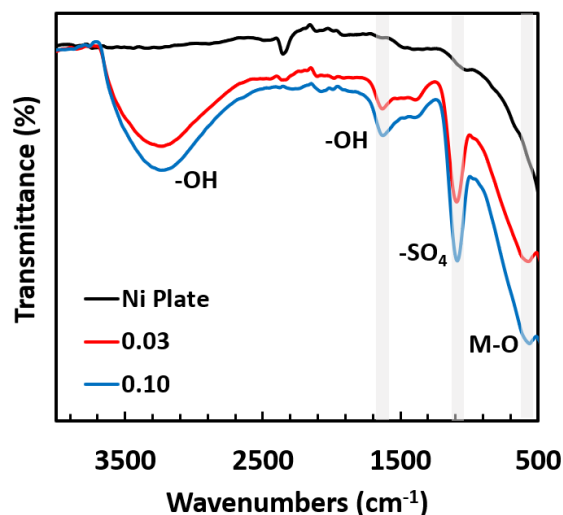


Fig. 4: FTIR of NiMn-0.10, NiMn-0.03, and its substrate (nickel plate)

A distinct absorption around 1100–1200 cm^{-1} is assigned to the SO_4^{2-} stretching mode^{68,69}, derived from the intercalated anion⁷⁰. In the lower wavenumber region (around 550–600 cm^{-1}), the peaks correspond to metal–oxygen (M–O) vibrations⁷¹, characteristic of Ni–O and Mn–O bonds within the oxide or hydroxide lattice. The NiMn-0.10 sample exhibits a stronger M–O band compared to NiMn-0.03, implying a higher degree of metal–oxygen network formation or stronger metal–oxygen bonding at NiMn-0.10. This could be related to variations in nucleation behavior and electrodeposition process, resulting in a better-defined oxide structure⁷².

3.3. Application for HMF oxidation reaction

The electrochemical oxidation of HMF can proceed through two possible reaction pathways. In the first pathway, HMF is sequentially oxidized to 2,5-Diformylfuran (DFF), followed by the formation of 5-Formyl-2-furancarboxylic acid (FFCA), and finally 2,5-Furandicarboxylic acid (FDCA)^{73–75}. In the second pathway, HMF is first converted to 5-Hydroxymethyl-2-furancarboxylic acid (HMFA), which is subsequently oxidized to FFCA and FDCA^{76–78}. The two possible HMF oxidation pathways are illustrated in Figure 5.

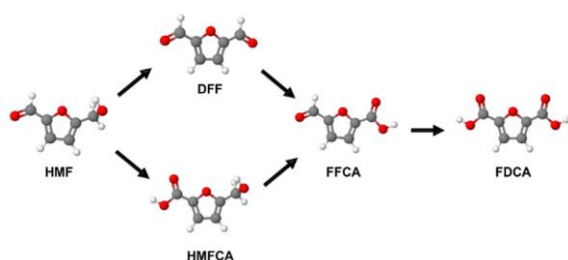


Fig. 5: Schematic illustration of the two possible pathways for the oxidation of HMF

Selective formation of DFF requires the reaction to proceed through the first pathway. This route is generally favored under acidic to near-neutral conditions⁷⁹, whereas increasingly alkaline environments tend to promote the second pathway, leading to the formation of HMFA as the primary intermediate⁸⁰.

In this study, Sodium tetraborate ($\text{Na}_2\text{B}_4\text{O}_7$) was employed as the electrolyte to direct the oxidation of HMF through the DFF pathway rather than the HMFA pathway. The electrolyte provides a mildly alkaline environment, which remains sufficiently stable for the operation of transition-metal-based electrocatalysts^{81,82} while still allowing the reaction to proceed via the DFF intermediate. Consequently, the use of $\text{Na}_2\text{B}_4\text{O}_7$ helps maintain suitable reaction conditions for achieving selective oxidation toward DFF.

The effectiveness of NiMn-0.10 as an electrocatalyst was further assessed by its employment in the electrochemical reaction of HMF oxidation to create DFF. DFF is a compound that has a high economic value and is employed as an intermediary in the production of various chemical products and materials. Following a period of 6 hours during which the reaction process was carried out, the electrolyte solution that contained the products of the reaction was subsequently examined. Figure 6 exhibits the product analysis of HMF electro-oxidation every 2 hours for 6 hours. Furthermore, the DFF's yield has increased over time due to more charges passing into the system. However, the selectivity of the DFF also increases over time, indicating that the HMF transformation to become DFF is more effective.

The study has provided an overview of the major impact that the change in precursor concentration during electrodeposition has on the crystal structure of the electrocatalyst that is produced as a result of the process. The crystallinity of the material can be greatly increased, and new crystal planes can form, if the ideal precursor

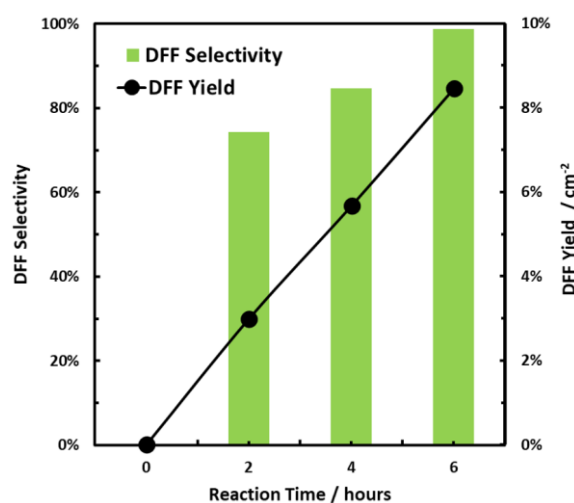


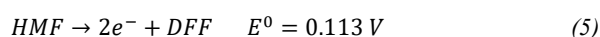
Fig. 6: Product analysis of 1 mM HMF 50 mL oxidation using NiMn-0.10 in $\text{Na}_2\text{B}_4\text{O}_7$ at 1.75 V vs. RHE

Table 1: DFF production comparison from HMF oxidation reaction

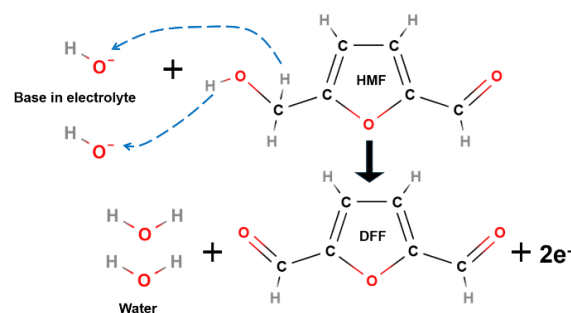
System	Condition	DFF Selectivity	Reference
metal bromide with acetic acid	75 °C, 7000 kPa air	61.00 %	27)
V ₂ O ₅ /TiO ₂	90 °C, 1600 kPa air	85.00%	28)
immobilized vanadyl pyridine	130 °C	82.00 %	29)
Fe ₃ BiMo ₁₂ O ₄₂	140 °C, 100 kPa O ₂	99.00 %	31)
β-MnO ₂	140 °C, 100 kPa O ₂	99.00 %	32)
AC-SO ₃ H@Ag	140 °C	77.90 %	33)
Pt/Fe ₃ O ₄ /rGO	140 °C, 500 kPa O ₂	32.80 %	83)
Fe ₃ O ₄	Room temperature	94.40 %	
Fe ₃ O ₄	80 °C	99.3 %	84)
Fe-Co	100 °C, 1 MPa O ₂	99.0 %	85)
Pt	Room temperature	26.51 %	86)
Pt	50 °C, 150 kPa O ₂	73.00 %	87)
Ru/γAl ₂ O ₃	50 wt% in DMF, 35 °C, 0.5 MPa O ₂	70-80%	88)
SGH-TiO ₂	UV light (λ=515 nm)	87.0 %	89)
Mn/UiO-66(Zr/Hf)-NH ₂	300 W xenon lamp, 10 mL / min O ₂	84.8 %	90)
CdS@MXene	Light Emitting Diode (LED) lamp (λ > 420 nm)	91.63 %	91)
Nb ₂ O ₅ /g-C ₃ N ₄	Sunlight Irradiation	70.00 %	92)
MnOx	60 °C	68.21 %	93)
Ru-NiO	Room temperature, 1.283 V	90.00 %	94)
Co ₈ Ce ₂ Ox	Room temperature, 1.50 V	92.00 %	95)
NiMn	Room temperature, 1.75 V	98.8 %	This work

concentration is used in the production of NiMn-0.10. A contribution from the (111) plane, which is exclusively present in the NiMn-0.10 sample, is thought to be responsible for the improvement in electrocatalyst performance, according to estimates. Compared to other studies using electrocatalysts, the utilization of NiMn-0.10 for the oxidation of HMF shows highly favorable results. A comparison of DFF production from HMF oxidation using different electrocatalytic systems is presented in Table 1. With a DFF selectivity of 98.8% and a product yield of 8.5%, the NiMn-0.10 electrocatalyst demonstrates superior performance in HMF conversion, highlighting its efficiency among electrocatalytic approaches.

Although this study demonstrates the high selectivity and efficiency of the NiMn-0.10 electrocatalyst for the oxidation of HMF to DFF, the scope of this work primarily focuses on the anodic reaction and the resulting DFF selectivity and yield. Therefore, hydrogen production at the cathode was not quantitatively evaluated in this study. Thermodynamically, the anodic oxidation of HMF must be balanced by a cathodic reduction reaction⁹⁶). Under aqueous conditions, this reaction is the Hydrogen Evolution Reaction (HER) in equation (4)⁹⁷):



In this work, an anodic potential of 1.75 V vs. RHE was applied to the working electrode to drive HMF oxidation (equation (5)⁹⁸), while the counter electrode functions as the cathode where HER is expected to occur, producing

**Fig. 7:** Mechanism for the transformation of HMF to DFF under alkaline conditions

Hydrogen as the complementary reaction. In addition, many previous studies have also confirmed the generation of H₂ gas at the cathode during electrochemical HMF oxidation systems^{10,98–101}). Furthermore, the mechanism of the transformation of HMF into DFF under alkaline conditions is illustrated in Figure 7.

3.4. Synergistic Effect of Ni and Mn

The Mott–Schottky plots in the Figure 8 show the relationship between 1/C² and potential for nickel hydroxide and manganese hydroxide. Both materials exhibit a positive slope, indicating that they are n-type semiconductors, where electrons are the majority charge carriers. The main difference is observed in the intercept position along the potential axis, which is related to the flat band potential. Manganese hydroxide shows a more negative flat band potential compared to nickel hydroxide, indicating that its electronic energy level is higher (more reductive). However, after overcoming the activation

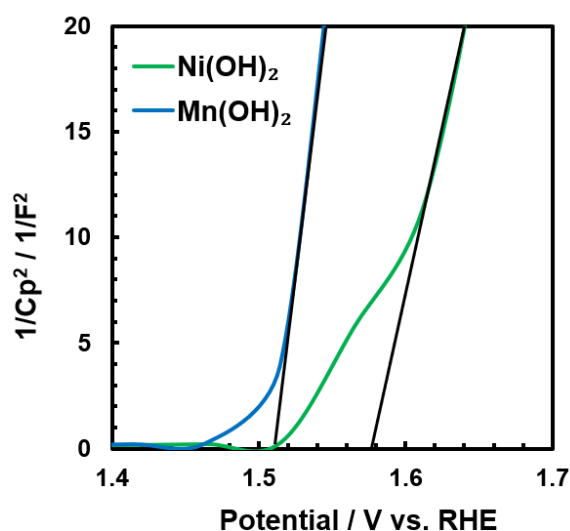


Fig. 8: Mott–Schottky plot of nickel hydroxide and manganese hydroxide

potential, nickel hydroxide exhibits higher electrocatalytic activity.

In addition, differences in the slope of the plots indicate differences in charge carrier density between the two materials. A smaller slope corresponds to a higher carrier density, which contributes to improved conductivity and electron transfer kinetics. In this case, nickel hydroxide tends to exhibit better charge transfer capability compared to manganese hydroxide. Overall, the Mott–Schottky analysis suggests that although manganese hydroxide is more easily activated electronically (more negative flat band potential), nickel hydroxide possesses characteristics more favorable for electrocatalytic processes, particularly in terms of charge transfer and active species formation. This indicates a potential synergistic effect in the Ni–Mn bimetallic system, where Mn facilitates the initial activation, while Ni acts as the primary active site for the oxidation reaction.

4. Conclusion

This study identified the optimal $\text{Ni}(\text{NO}_3)_2$ concentration for the preparation of NiMn electrocatalysts and examined its influence on the crystal structures. The enhancement in electrocatalyst performance, indicated by a reduction in overpotential, is attributed to improved crystallinity and structural modification. These important findings offer comprehensive details about the development of transition metal-based electrocatalysts through precursor concentration optimization. The use of the NiMn electrocatalyst in DFF production demonstrated 98.8% selectivity for the resulting product, achieving a 8.5% yield of 8.57% conversion of HMF. This achievement demonstrates that NiMn-0.10 can selectively and efficiently produce DFF, despite a low HMF conversion rate. This electrochemical approach to DFF synthesis is

simpler and more environmentally friendly than previous methods documented in the literature, which necessitate extreme conditions such as high temperatures or pressurized oxygen gas. It can be conducted at room temperature and atmospheric pressure without the need for external oxidants. This work demonstrates the significant potential of NiMn-0.10 for applications in the conversion of biomass into high-value compounds through a more sustainable process. Furthermore, the integration of HMF oxidation with the hydrogen evolution reaction at the cathode presents a promising strategy for improving the overall efficiency and sustainability of electrochemical biomass valorization.

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Nomenclature

Symbol	Description (Unit)
C_{HMF}	HMF conversion (%)
$n_{0,\text{HMF}}$	HMF initial amount (mole)
$n_{x,\text{HMF}}$	HMF final amount (mole)
Y_{DFF}	DFF yield (%)
$n_{x,\text{DFF}}$	DFF final amount (mole)

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