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Comparative Study of the Catalytic Activities of Ni, NiCo, and PtNiCo in Ethanol Electrooxidation: Contributions of Individual Metals

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Abstract

This study compares the catalytic activity of Ni, NiCo, and PtNiCo in the ethanol electrooxidation reaction to elucidate the role of each metal. The catalysts were synthesized via electrodeposition at room temperature and characterized using energy-dispersive X-ray spectroscopy and X-ray diffractometry techniques. Catalytic activity was evaluated through various electrochemical tests, including electrochemical impedance spectroscopy, linear sweep voltammetry, and chronoamperometry. Ethanol electrooxidation tests conducted via cyclic voltammetry over 1000 cycles revealed a progressive increase in catalytic activity among the three catalysts, with PtNiCo exhibiting the highest performance. The synergistic effect of the three metals resulted in a low charge transfer resistance (3.46 Ω), a small Tafel slope (156 mV/decade), a high peak current density (32.06 mA/cm²), a low I_f/I_b ratio, and an enhanced reactant diffusion coefficient. These findings provide valuable insights for designing efficient and high-performance tri-metallic catalysts.

Keywords: PtNiCo, ethanol electrooxidation, electrocatalyst

1. Introduction

Direct ethanol fuel cells (DEFCs) are a promising renewable energy source due to their potential to generate clean energy, flexibility, and relatively high-power output by utilizing the ethanol electrooxidation reaction to produce electricity [1]. Ethanol, as a fuel for DEFCs, offers several advantages, including mass producibility, environmental friendliness, non-toxicity, and non-explosiveness [2]. Despite these benefits, the development of DEFCs faces several challenges, primarily their low fuel cell efficiency [3]. This limitation arises from the high activation energy required for ethanol electrooxidation, necessitating the use of an efficient catalyst to accelerate the reaction rate within the fuel cell [4].

Platinum (Pt) is a superior catalyst for ethanol oxidation reactions due to its high catalytic activity [5]. However, its scarcity leads to high production costs [6]. Moreover, Pt is susceptible to CO poisoning, which can degrade its catalytic performance [6]. A promising approach to reducing Pt usage while enhancing its catalytic activity is alloying it with transition metals such as Ni, Co, Zn, Cu, and Ag. Studies on bimetallic and trimetallic Pt-based catalysts have demonstrated significant improvements in catalytic activity.

Platinum-based trimetallic catalysts, such as PtNiCo, have been applied in methanol oxidation [7], acetaldehyde oxidation [8], and hydrogen and oxygen reduction [9], demonstrating superior performance compared to monometallic and bimetallic catalysts. The electronic structure modification of Pt through alloying with nickel (Ni) and cobalt (Co) is thought to weaken the binding of catalytic activity-inhibiting species on Pt [9]. However, previous studies have not thoroughly examined the specific role of each metal in the reaction.

In this study, Ni, NiCo, and PtNiCo catalysts were synthesized via electrodeposition at room temperature. Their catalytic activities were compared to identify the role

of each metal. The results demonstrated a progressive increase in catalytic activity from Ni to NiCo to PtNiCo, influenced by the metal composition in the samples. This research provides an understanding the specific role of each metal in this trimetallic catalyst, which can serve as a guideline for designing an optimal catalyst composition with high efficiency and performance.

2. Materials and Methods

2.1 Materials

The materials used were nickel sulfate hexahydrate ($NiSO_4.6H_2O$), cobalt sulfate heptahydrate ($CoSO_4.6H_2O$), chloroplatinic acid hexahydrate ($K_2PtCl_6.6H_2O$), ethanol (C_2H_5OH), sulfuric acid (H_2SO_4), sodium hydroxide (NaOH), potassium chloride (KCl), and distilled water (H_2O).

2.2 Methods

2.2.1 Synthesis of Ni, NiCo, and PtNiCo

A copper wire substrate with a diameter of 2 mm was surface-smoothed using 5000-grit sandpaper. The smoothed substrate was subsequently sonicated for 10 minutes. The substrate was rinsed with distilled water and dried at room temperature. Subsequently, precursor solutions of Ni, NiCo, and PtNiCo were prepared in an acidic medium (0.04 M H_2SO_4) with the following concentration compositions.

Table 1. Composition of the electrolyte solutions used for catalyst deposition.

Sample	Precursors		
	$K_2PtCl_6.6H_2O$	$NiSO_4.6H_2O$	$CoSO_4.7H_2O$
Ni	-	0.05 M	-
NiCo	-	0.01 M	0.04 M
PtNiCo	0.001 M	0.04 M	0.009 M

Electrodeposition was carried out using a three-electrode cell configuration. The working electrode used was a Cu wire substrate, the counter electrode was a Pt wire, and the reference electrode was Ag/AgCl (3 M KCl). Deposition was performed at a potential of -1.6 V for 10 minutes at room temperature. Upon completion of the deposition process, the samples were rinsed with ethanol. The diagram of synthesis processes is shown in Fig. 1.

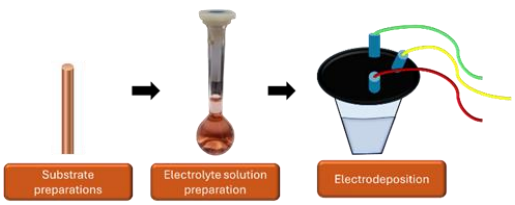

Figure 1. The diagram of the synthesis of Ni, NiCo, and PtNiCo via electrodeposition.

2.2.2 Characterizations

The elemental composition of the samples was analyzed using an energy-dispersive X-ray spectrometer (Bruker, Quantax). Meanwhile, phase analysis was performed using X-ray diffraction (XRD, PANalytical Aeris Cu K_α with PIXcel1D-Medipix3 detector).

2.2.3 Electrochemical impedance spectroscopy test

Electrochemical impedance spectroscopy (EIS) technique was performed in a 0.5 M KCl solution. EIS measurement was conducted in the frequency range of 0.1 Hz to 100 kHz

2.2.4 Linear sweep voltammetry test

Linear sweep voltammetry (LSV) test was performed in a 1 M ethanol solution in alkaline medium. The potential range studied was from -0.6 V to 0.6 V vs Ag/AgCl, and the measurements were conducted using an electrochemical working station.

2.2.5 Electrooxidation ethanol performance test

The electrooxidation of ethanol performance was evaluated in a 1 M ethanol solution in an alkaline medium. This evaluation employed cyclic voltammetry (CV) with a potential range of -0.75 V to 0.75 V at a scan rate of 250 mV/s for 1000 cycles.

3. Results and Discussion

The EDX measurement results of the samples demonstrated the successful deposition of the constituent elements of the Ni, NiCo, and PtNiCo catalysts onto the Cu wire substrate surface, as evidenced by the EDX spectrum in Fig. 2. The percentage of elements within the samples is presented in Table 2. The elemental compositions contained in samples were influenced by the precursor ion concentrations and the reduction potential standard of each metal. Pt composition was significantly higher due to Pt's relatively lower reduction potential compared to the other metals, despite the low precursor ion concentration of Pt [10], Table 1.

Table 2. The elemental composition of the synthesized Ni, CoNi, and PtNiCo coatings on Cu wire substrate.

Sample	Composition (at.%)		
	Pt	Ni	Co
Ni	-	100	-
NiCo	-	23.50	76.50
PtNiCo	66.63	29.40	3.97

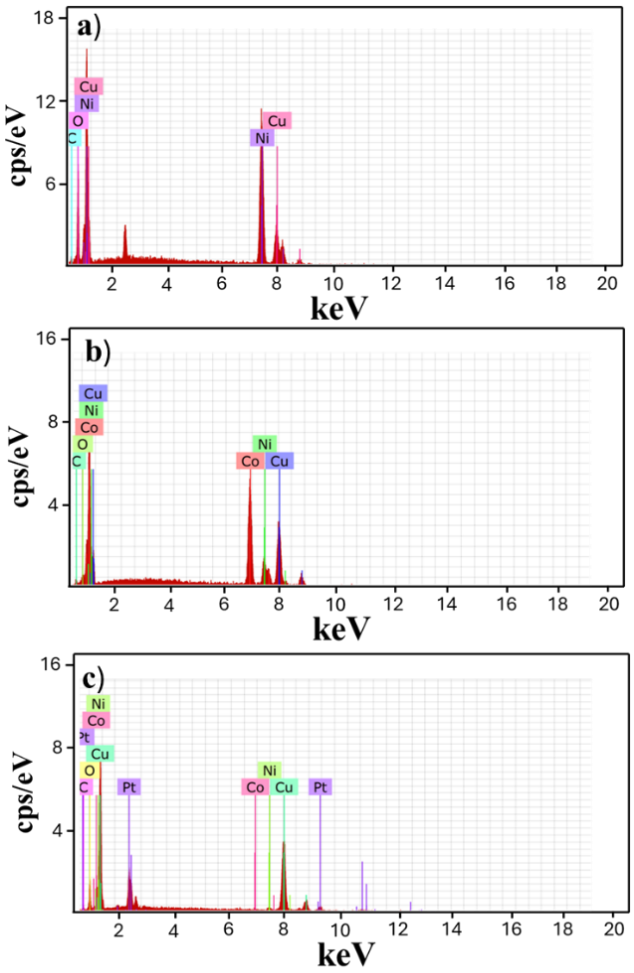

Figure 2. EDX spectra of the samples of (a) Ni, (b) CoNi, and (c)PtNiCo.

The formation of the PtNiCo trimetallic was confirmed through X-ray diffraction (XRD) analysis, as presented in Fig. 3. Low-intensity peak at 2θ 40.85° suggests the presence of the PtNiCo thin-film catalyst, which aligns with the reference COD No 1525469. Meanwhile, the diffraction peaks at 2θ 43.61°, 50.72°, 74.36°, 90.14°, and 95.35° indicate the presence of the copper wire substrate, which corresponds to the reference COD No. 7101269. The diffraction pattern reveals that the intensity of the Cu peaks is significantly higher than the PtNiCo catalyst peaks. This phenomenon can be attributed to the thin film wire of the sample restricting the penetration of X-rays, thereby resulting in lower catalyst peak intensity. Additionally, the high crystallinity of the copper substrate also influences the diffraction pattern. The copper substrate exhibits high crystallinity and an ordered structure, leading to a higher diffraction intensity compared to the catalyst [11].

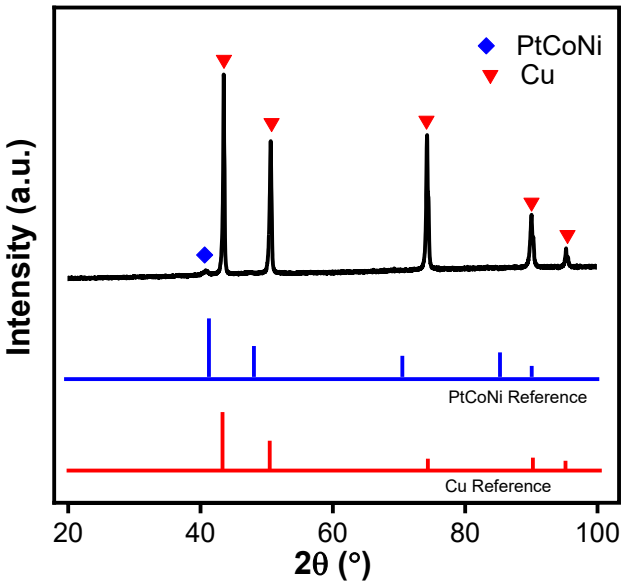

Figure 3. Diffraction pattern of PtNiCo electrodeposits formed on a copper wire substrate.

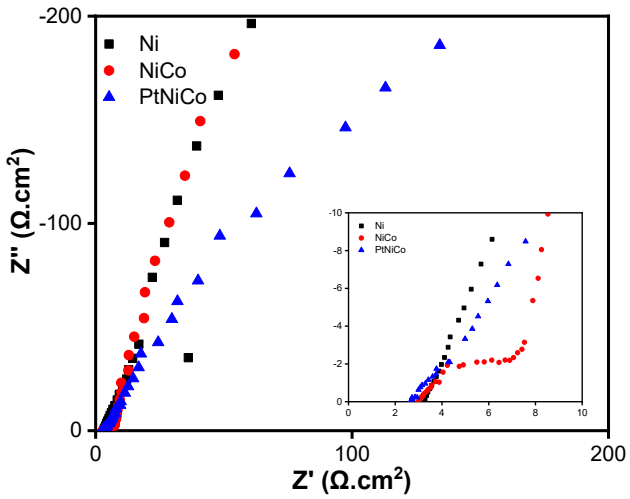

Figure 4. Nyquist plots illustrate the electrochemical impedance behavior of Ni, NiCo, and Pt-NiCo at low frequency and high frequency (inset).

Nyquist plot profiles of the catalysts are presented in Fig. 4. Based on the fitting results, the charge transfer resistance (R_{ct}) values are summarized in Table 3. A low R_{ct} value indicates efficient charge transfer within the sample. The PtNiCo catalyst exhibited a relatively lower R_{ct} value compared to the other catalysts, attributed to the presence of Pt. Pt possesses superior electronic properties due to the d-band center of Pt is located at an energy level very close to the Fermi level [12]. The NiCo catalyst also demonstrated a lower R_{ct} value than Ni, resulting from the synergistic effect between cobalt and nickel. These two metals have complementary electronic properties, leading

to a lower R_{ct} value compared to the Ni monometallic catalyst [13].

Table 3. R_{ct} values obtained from the EIS measurements.

Sample	R_{ct} (Ω)
Ni	75.80
CoNi	5.20
PtNiCo	3.46

Tafel slope values describe the reaction kinetics of the catalysts. The Tafel slope is defined as the amount of voltage (mV) required to increase the current by one decade, expressed in mV/decade [14]. A lower Tafel slope value indicates a more active catalyst, which requires a smaller voltage to achieve a significant current response. The addition of Pt to the catalyst directly enhances the current density and reduces the overpotential. Furthermore, NiCo exhibited higher kinetic activity compared to Ni, attributed to the improvement of the electronic structure [13]. These results are consistent with the impedance analysis.

Figures 6a-d illustrate the CV test results for evaluating the catalyst performance in the ethanol electrooxidation reaction. The CV test results over 1000 cycles demonstrated that the Ni catalyst exhibited the lowest catalytic performance compared to the other catalysts, Fig. 6a. Nickel is known to possess catalytic activity towards organic compounds in an alkaline environment; however, its activity remains suboptimal for ethanol electro-oxidation [15]. Although an increase in current was observed at a potential of 0.36 V vs. Ag/AgCl, this current increase was not attributed to the ethanol electro-oxidation reaction but rather to the oxidation of nickel species to NiOOH, which was subsequently reduced during the reverse scan [16]. This is evidenced by the presence of a reduction peak at a voltage of 0.23 V vs. Ag/AgCl. The Ni catalyst produced a maximum current density of 2.06 mA/cm², which continuously decreased as the CV cycles progressed. This decrease is attributed to CO_{ads} poisoning on the catalyst surface [17]. Furthermore, excessive formation of Ni-OOH species during the test can induce surface deformation of the catalyst, reducing the active sites of the nickel monometallic catalyst [18].

The NiCo alloy exhibited enhanced catalytic activity compared to the Ni catalyst. As shown in Fig. 6b, the peak oxidation current density for the NiCo catalyst was 11.05 mA/cm². The role of Co serves as a co-catalyst to improve resistance to CO_{ads} poisoning, as cobalt can oxidize CO_{ads} that cover the active sites of the catalyst [19]. As shown in the voltammogram profile, the oxidation peak in cycles 1-50 experienced a decrease due to the CO_{ads} poisoning phenomenon. However, after the 100th cycle, the catalytic activity increased because of the presence of cobalt atoms, which oxidized the CO_{ads} adsorbed on the catalyst surface. The CO_{ads} oxidation reaction by Co is as follows [20].

$$CO_{ads} + Co - OH_{ads} \rightarrow Co - CO - OH \rightarrow Co - COOH \quad (1)$$

In addition to mitigating CO poisoning, the Co metal in the NiCo alloy also functions as an adsorber of OH- ions during the ethanol oxidation reaction process. This facilitates the reaction between Co-OH_{ads} species and Ni-(CH_3CHO_{ads}) species to form carboxylic acids [21]. The mechanism involved is as follows.

$$Ni + CH_3CH_2OH \leftrightarrow Ni - (CH_3CH_2OH)_{ads} \quad (2)$$

$$Ni - (CH_3CH_2OH)_{ads} + 3OH^- \rightarrow Ni - (CH_3CO)_{ads} + 3H_2O + 3e^- \quad (3)$$

$$Ni - (CH_3CO)_{ads} + Co - OH_{ads} \rightarrow Ni - CH_3COOH + Co \quad (4)$$

$$Ni - CH_3COOH + OH^- \rightarrow NI + CH_3COO^- + H_2O \quad (5)$$

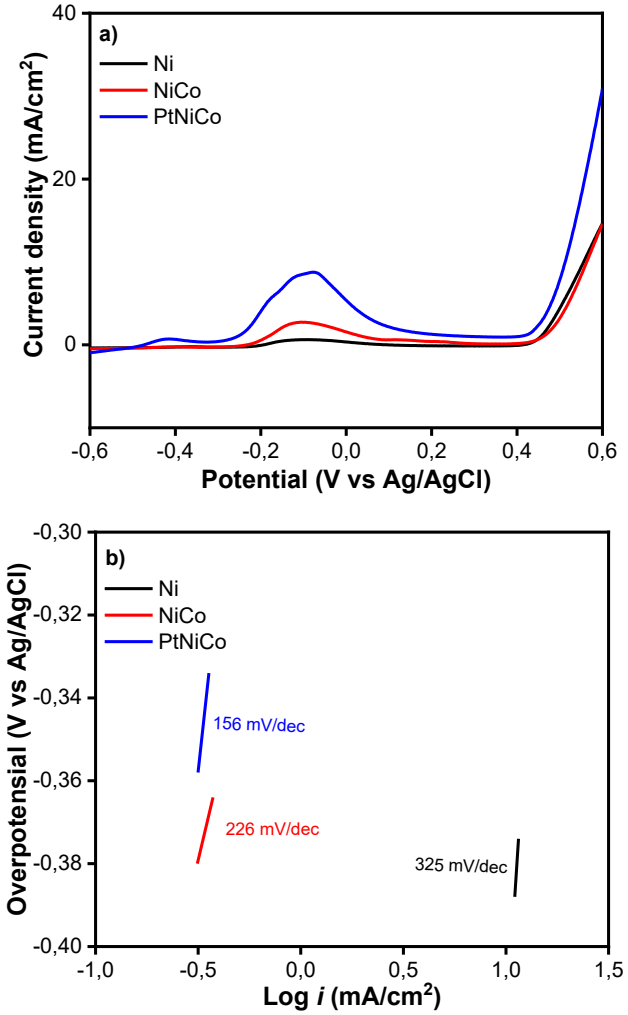

Figure 5. LSV curve (a) and the Tafel slope values (b) derived from the LSV potentiodynamic curves of Ni, CoNi, and PtNiCo.

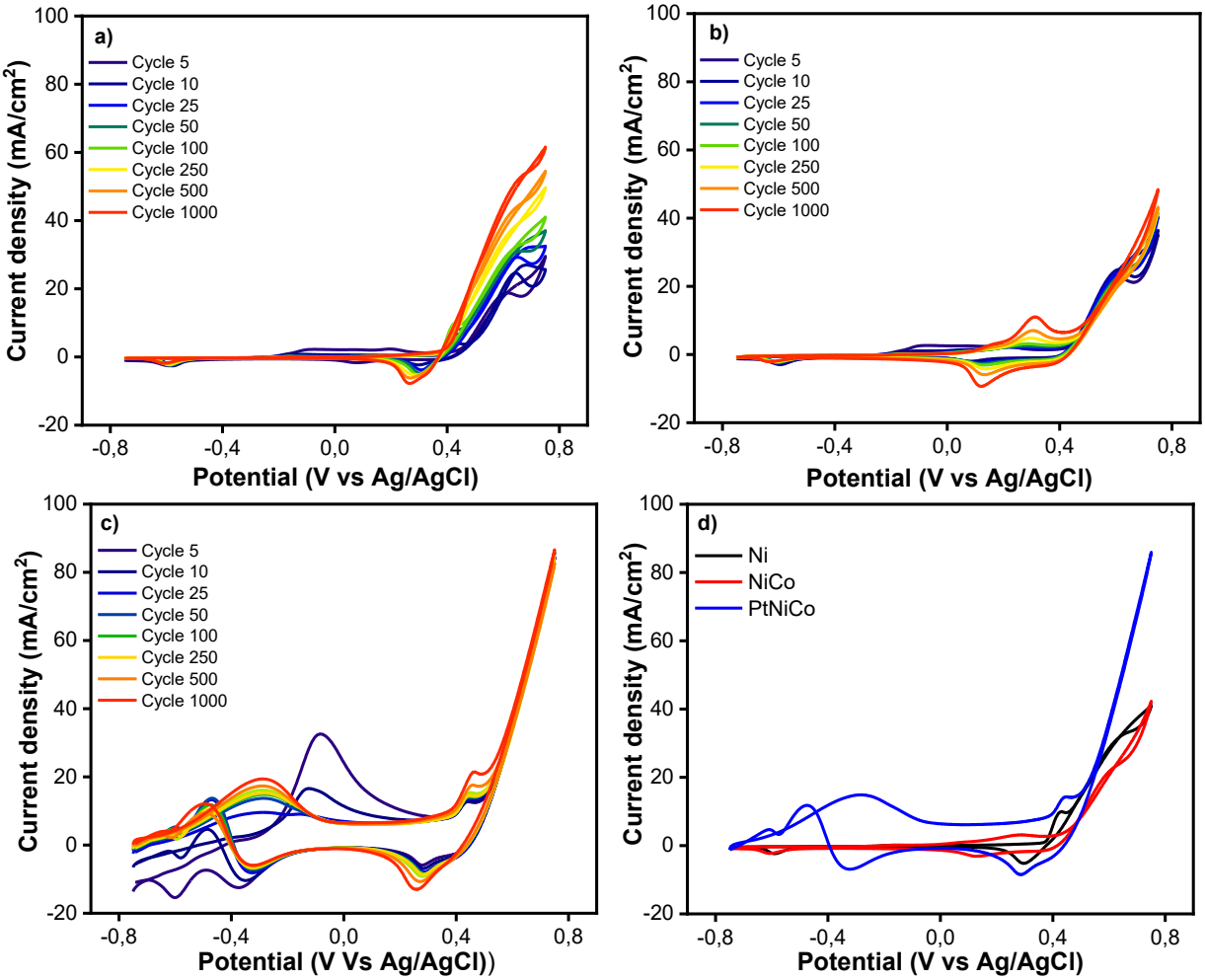

Figure 6. Cyclic voltammetry for the ethanol electrooxidation until 5-1000 cycle of (a) Ni, (b) CoNi, (c) PtCoNi and (d) comparison CV cycle 100 of Ni, NiCo, and PtNiCo samples was performed at a scan rate of 250 mV/s.

The catalytic performance of NiCo remained relatively lower compared to the PtNiCo catalyst. The presence of Pt atoms in the catalyst enhanced the active sites for C-C bond cleavage. This is supported by the high oxidation peak current density, reaching 32.60 mA/cm², as shown in Fig. 6c. Furthermore, the lowest I_f/I_b ratio of the PtNiCo catalyst indicated that PtNiCo exhibited superior tolerance to CO_{ads} poisoning [22]. This observation is substantiated by platinum's ability to generate high catalytic activity, coupled with the synergistic effect of NiCo. Specifically, Ni contributed to an increase in reaction active sites, while Co facilitated the oxidation of CO_{ads} on the catalyst surface.

The diffusion coefficient value is utilized to describe the rate of a substance within a medium and serves as a benchmark for catalyst performance. The diffusion coefficient was calculated using the following Randles-Sevcik equation [24].

$$I_p = 2.69 \times 10^5 n^{3/2} A D^{1/2} v^{1/2} C \quad (6)$$

Where I_p maximum current (A), n number of electrons involved in the reaction, A is the surface area of the catalyst (cm^2), D is the diffusion coefficient (cm^2s^{-1}), v is the scan rate (V/s), dan the concentration of the electrolyte (mol/cm^3).

Table 3. I_f/I_b ratio of the Ni, NiCo, and PtNiCo catalysts.

Sampel	I_f/I_b
Ni	1.30
NiCo	0.71
PtNiCo	0.47

Based on the calculated diffusion coefficient values for each sample (Table 4), it was observed that the PtNiCo catalyst exhibited the best reaction kinetics, characterized by a high ion diffusion value. This can be attributed to platinum's high conductivity. High conductivity implies rapid electron transfer, enabling more efficient reaction

progression, enhancing the chemical reaction rate in ethanol electro-oxidation, and directly increasing the sample's diffusion coefficient [25].

Table 4. Diffusion coefficient of Ni, CoNi, PtNiCo catalysts.

Sample	Diffusion coefficient
Ni	5.96×10^{-15} cm^2/s
CoNi	2.43×10^{-13} cm^2/s
PtNiCo	2.11×10^{-9} cm^2/s

4. Conclusion

In this study, Ni, NiCo, and PtNiCo were successfully synthesized and compared for their catalytic activities. The enhanced catalytic activities were observed in NiCo and PtNiCo, attributed to the synergistic effects between the metals. The research findings demonstrate that the addition of Pt and Co to the Ni catalyst significantly improves the catalytic activity and ethanol electro-oxidation performance. Co plays a role in enhancing catalytic activity by oxidizing CO_{ads} and adsorbing OH^- ions, while Pt functions as a booster for the active sites involved in C–C bond breaking. Understanding the individual roles of each metal within the PtNiCo trilayer catalyst provides valuable insights for the development of more efficient catalysts for DEFCs applications.

Author contributions

Devi Indrawati Syafei: Writing-original draft and Investigation. Yasir Arafat Hutapea: Writing-review and editing. Liana Christiani: Writing-review and editing.

Conflicts of interest

There are no conflicts to declare.

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