



Copper foam modified electrodes for CO₂ electroreduction: A study on deposition potential effect and flow cell performance

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ABSTRACT

Background: The development of effective electrochemical conversion technologies is imperative due to the rising global CO₂ emissions. A promising platform for CO₂ reduction to formate is copper electrode, which can stabilize the carbon dioxide radical that is essential for CO₂ conversion. **Methods:** In this work, Cu foam was electrodeposited in situ on a copper plate with sodium citrate acting as a capping agent (CuF@Cu), with variation of potential deposition were 3V and 5V. **Findings:** The foam structure of Cu in Cu electrode was confirmed with SEM and XRD measurements for both potential deposition variations. Furthermore, CO₂ electroreduction was carried out in a flow cell under ideal conditions, which included aeration for 20 minutes, a flow rate of 75 mL min⁻¹, and an applied potential of -0.33 V vs. Ag/AgCl. For formic acid conversion, the Faradaic efficiency rose from 14.18% (Cu bare) to 26.73% (CuF@Cu 3V) which an 88.7% improvement over bare copper. **Conclusion:** The enhanced performance is attributed to the increased surface area and three-dimensional foam structure, which augments active sites for CO₂ activation. This work demonstrates that simple electrodeposition of copper foam is an effective strategy for improving electrochemical CO₂ reduction efficiency. **Novelty/Originality of this article:** These findings demonstrate that CuF@Cu makes using this straightforward electrodeposition technique a viable option for CO₂ to formate conversion.

KEYWORDS: carbon dioxide; Cu electrode; Cu foam; flow cell.

1. Introduction

Carbon dioxide (CO₂) emissions remain one of the principal drivers of global climate change. According to recent estimates, global CO₂ emissions from fossil fuel consumption reached 37.41 Gt in 2024, reflecting a continued increase in atmospheric carbon levels and underscoring the need for effective mitigation strategies (Commission, 2025; Riyanto & Sylvia, 2025). Among the various approaches proposed to address this challenge, the electrochemical reduction of CO₂ has attracted considerable attention because it enables the conversion of an abundant greenhouse gas into value-added chemicals using electricity from renewable sources (Jiwanti et al., 2020; Khalil et al., 2019; Riyanto & Sanjaya, 2024;

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Zhang et al., 2022). Formate and formic acid are particularly attractive products owing to their broad applications in chemical synthesis, hydrogen storage, and fuel-cell technologies.

Despite these advantages, CO₂ electroreduction is intrinsically challenging because the first electron-transfer step to form the CO₂ radical anion is thermodynamically unfavorable and requires a substantial overpotential (Kang et al., 2020; Khan et al., 2025). Catalyst selection is therefore critical for lowering the activation barrier and directing the reaction toward desirable products. Among the metallic electrocatalysts investigated to date, copper is unique in its ability to stabilize key adsorbed intermediates and to produce a wide range of reduced carbon products (Diksy et al., 2020; Jiang et al., 2023; Saprudin et al., 2023). The catalytic behavior of copper is highly sensitive to surface structure and morphology, and significant improvements in activity and selectivity have been reported for nanowires, nanocubes, and nanosheets (Maniam et al., 2023; Nitopi et al., 2019; Zhu et al., 2019).

Porous copper foam has emerged as an attractive electrode architecture because its three-dimensional interconnected network can provide a high density of accessible active sites while facilitating electrolyte penetration and reactant transport. These structural features may also create local reaction environments that suppress the competing hydrogen evolution reaction and improve selectivity toward formate. Nevertheless, compared with other copper nanostructures, copper foam remains underexplored, particularly in flow-cell systems that offer enhanced mass transport and are more relevant to practical CO₂ conversion processes (Kottakkat et al., 2019).

In the present work, copper foam was synthesized using sodium citrate as a capping agent to control particle growth and pore formation, followed by the electrodeposition onto a copper plate to produce a three-dimensional Cu foam/Cu electrode (CuF@Cu). The structure and composition of the resulting electrodes were characterized by scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy and X-ray diffraction, while their electrochemical behavior was evaluated by cyclic voltammetry. The electrocatalytic performance of CuF@Cu towards CO₂ reduction was subsequently investigated in a flow-cell configuration using formic acid as the target product. This research aims to clarify the influence of copper foam morphology on catalytic activity and selectivity and to assess a simple and reproducible strategy for improving the formate production under mild operating conditions.

2. Methods

2.1 Materials

Copper plates (99.8% purity, 1 mm thickness; Findustri, Indonesia) were used as the substrate for electrode preparation. Anhydrous CuSO₄, KHCO₃, KOH, NaOH, HClO₄, formic acid, methanol, and isopropanol were purchased from Merck. Sodium citrate dihydrate (Na₃C₆H₅O₇·2H₂O) was obtained from Wako, hydrazine monohydrate (N₂H₄·H₂O) from Sigma-Aldrich, and gelatin from Nitta Inc. All chemicals were used as received without further purification. All aqueous solutions were prepared using deionized water. High-purity nitrogen (N₂) and carbon dioxide (CO₂) were used for electrolyte purging and electrochemical reduction experiments.

The morphology and elemental composition of the electrodes were examined by scanning electron microscopy equipped with energy-dispersive X-ray spectroscopy (SEM-EDS, Hitachi SU-3500). Crystal structures were identified by X-ray diffraction (XRD, PANalytical X'Pert Pro 2318). Electrochemical measurements were performed using a PGSTAT204 potentiostat/galvanostat (Metrohm Autolab). Liquid and gaseous products were analyzed using high-performance liquid chromatography (HPLC, PG LC 210) and gas chromatography (GC, Shimadzu), respectively.

2.2 Cu/Cu foam electrode preparation

Cu foam particles were synthesized with a chemical reduction method. First, 0.957 g of

anhydrous CuSO_4 and 1.764 g of sodium citrate dihydrate were dissolved in 30 mL of deionized water. Gelatin (75 mg) was then added as a pore-directing agent, followed by 1.8 g of NaOH to create an alkaline medium. The mixture was stirred at room temperature for 30 min until a homogeneous solution was obtained. Next, 9 mL of 10 M hydrazine monohydrate was added dropwise over approximately 10 min while the solution was continuously stirred. Hydrazine acted as the reducing agent, converting Cu^{2+} ions into metallic Cu particles. After the addition was complete, the suspension was stirred for a further 2 h at room temperature to ensure complete reduction.

The resulting Cu particles were collected by vacuum filtration using a PTFE membrane filter (0.22 μm , Advantec) and washed several times with deionized water. The washed particles were then redispersed in a fresh solution containing 30 mL of deionized water, 1.8 g of NaOH, 75 mg of gelatin, and 9 mL of 10 M hydrazine monohydrate. This second dispersion step was carried out to obtain a stable suspension for electrodeposition and to maintain the Cu particles in a reduced state prior to deposition. Circular copper plates with a diameter of 3.0 cm were used as substrates. The total geometric area of each plate was 7.07 cm^2 , and the entire surface was exposed to the deposition solution and subsequently used as the active cathode area. Before deposition, the copper plates were cleaned sequentially with methanol, isopropanol, and deionized water. Cu foam was deposited onto the copper substrates in a two-electrode configuration using the copper plate as the cathode and a platinum spiral as the counter electrode. Deposition was carried out at constant cell voltages of 3 and 5 V for 5 min at room temperature. After deposition, the electrodes (denoted as CuF@Cu) were rinsed thoroughly with deionized water and dried at room temperature before characterization by Scanning Electron Microscopy–Energy-Dispersive X-ray Spectroscopy (SEM–EDS) and X-ray Diffraction (XRD).

2.3 Electrocharacterization of Cu/Cu foam electrode

Electrochemical characterization was performed in a conventional three-electrode cell using bare Cu and CuF@Cu as the working electrodes, Ag/AgCl as the reference electrode, and a platinum spiral as the counter electrode. The geometric area of the working electrode exposed to the electrolyte was 7.07 cm^2 . The electrolyte was 0.1 M KHCO_3 . Prior to each measurement, the electrolyte was purged with N_2 at 200 mL min^{-1} for 15 min to remove dissolved oxygen, followed by CO_2 purging with the same flow rate for 20 min to achieve saturation. Cyclic voltammetry was recorded in the potential range from 0 to -1.2 V versus Ag/AgCl at a scan rate of 50 mV s^{-1} .

2.4 Carbon dioxide conversion with flow cell system

CO_2 electroreduction was carried out in a custom-built three-electrode flow cell using bare Cu and CuF@Cu as cathodes, a platinum spiral as the anode, and Ag/AgCl as the reference electrode. The exposed geometric area of the cathode was 7.07 cm^2 . The catholyte and anolyte were 0.1 M KHCO_3 and 0.1 M KOH, respectively. The flow cell consisted of two acrylic compartments directly connected without an ion-exchange membrane. PTFE spacers with a thickness of 1 mm were placed between the electrodes to maintain a fixed interelectrode distance. Each compartment contained 50 mL of electrolyte.

Before electrolysis, the catholyte was purged with N_2 at 200 mL min^{-1} for 15 min to remove dissolved oxygen and then with CO_2 at the same flow rate for 20 min to saturate the solution. Preliminary experiments were first conducted using bare Cu to establish a baseline and identify suitable operating conditions. The selected conditions were subsequently applied to CuF@Cu to enable a direct comparison between the two electrodes. The applied potential was varied from -0.33 to -0.93 V versus Ag/AgCl, and the electrolyte flow rate was varied from 50 to 100 mL min^{-1} .

Based on the optimization results, CO_2 electroreduction was performed at -0.33 V versus Ag/AgCl with an electrolyte flow rate of 75 mL min^{-1} for 60 min. Liquid products were analyzed by HPLC, and gaseous products were analyzed by GC. Quantification was

performed using the external standard method. Standard solutions of formic acid were prepared over a concentration range of 0.1–50 mM, and calibration curves showed linear responses with correlation coefficients (R^2) greater than 0.999. Hydrogen was quantified by GC using certified gas standards, with calibration curves also showing R^2 values greater than 0.999. Each analysis was performed at least in duplicate, and the relative deviation between measurements was less than 5%.

3. Results and Discussion

3.1 Cu foam/Cu electrode fabrication and characterization

Copper foam was prepared through a two-step process involving chemical reduction and electrodeposition. In the first step, Cu^{2+} ions from CuSO_4 were complexed with sodium citrate to control the reduction rate and particle growth. Hydrazine monohydrate was then added as a reducing agent to convert Cu^{2+} into metallic Cu particles. Gelatin was introduced as a pore-directing agent to stabilize hydrogen bubbles and promote the formation of a uniform porous structure during deposition. In the second step, the resulting Cu suspension was electrodeposited onto a copper plate under an applied voltage, where hydrogen bubbles served as dynamic templates to generate a three-dimensional foam structure.

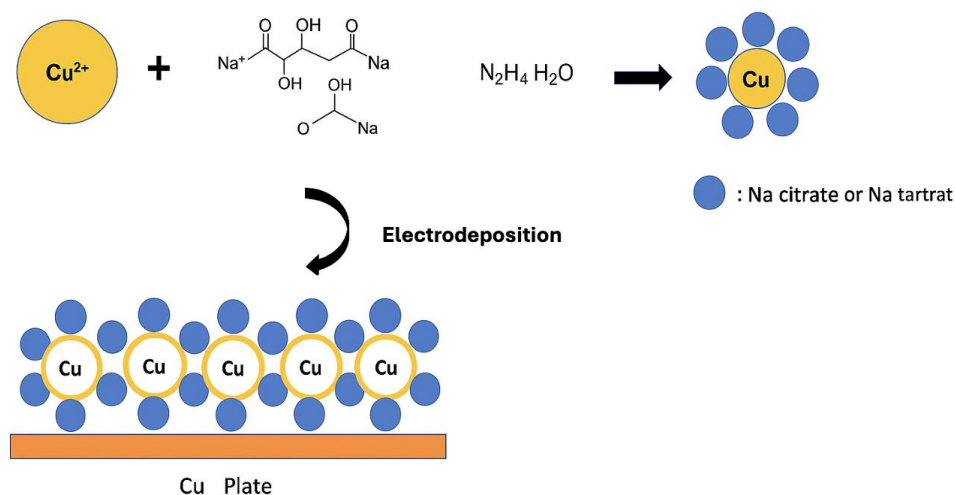


Fig. 1. Schematic diagram of Cu foam synthesis on the surface of Cu plate

As illustrated in Figure 1, sodium citrate coordinates with Cu^{2+} ions to form a stable copper–citrate complex, which moderates nucleation and leads to more homogeneous Cu particles. After chemical reduction by hydrazine, the Cu particles are dispersed in the deposition bath and deposited onto the Cu plate. Simultaneously, hydrogen bubbles generated at the cathode create temporary templates that define the porous architecture through the dynamic hydrogen bubble template (DHBT) mechanism.

SEM images show that the CuF@Cu electrode has been successfully deposited on the Cu plate, as shown in Figure 2. The pores formed in the Cu foam are quite small, while those in the CuF@Cu citrate 5 V are very small. Citrate has three carboxyl groups ($-\text{COOH}$) and one hydroxyl group ($-\text{OH}$). This structure causes citrate to form a stable complex with Cu^{2+} ions. The Cu citrate complex has a high stability constant ($\log \beta_{10} = 5.7\text{--}6.1$) (Duangsa et al., 2021). Citrate can form chelate structures with Cu^{2+} through multidentate coordination, involving both carboxylate and hydroxyl groups simultaneously, resulting in better charge distribution and high stability. Citrate also has extensive cross-linking capabilities, resulting in a great heat release during the decomposition process and facilitating the formation of homogeneous structures and small pores (Cao et al., 2015; Jiwanti et al., 2019).

Anhydrous CuSO_4 is used as a precursor for the formation of Cu foam on the surface of Cu plates, while $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ (Na citrate) functions as a capping agent to maintain the

stability of Cu foam formation on the surface of Cu plate electrodes. These capping agents play an important role in controlling the morphology and size of the copper particles formed through complexation and electrostatic stabilization mechanisms. Gelatin compounds are added to control the pore size of the Cu foam formed, the more gelatin added, the smaller the pore size formed. This mechanism occurs because gelatin reduces the surface energy of the dispersion solution and suppresses the coalescence of hydrogen bubbles formed during the electrodeposition process, resulting in a smaller and more uniform pore structure (Besharati & Paydar, 2023; Shin & Liu, 2004).

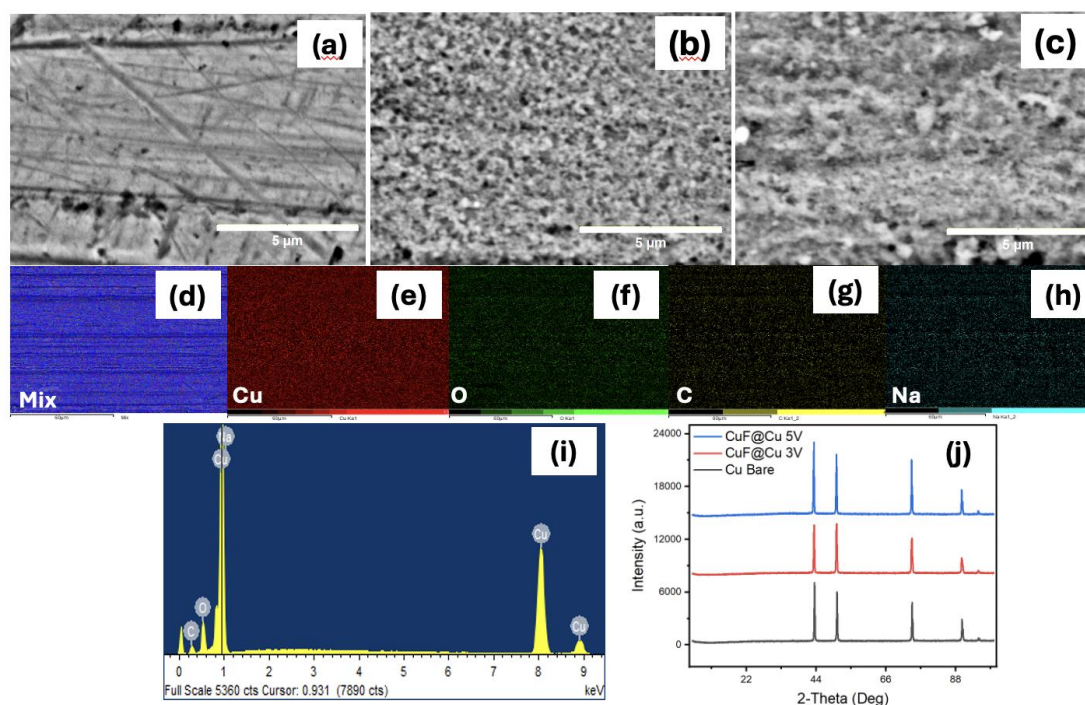


Fig. 2. SEM image of (a) Cu Bare, (b) CuF@Cu 3 V, and (c) CuF@Cu 5 V, (d-h) EDS mapping of CuF@Cu 3V with mix atom, Cu (Copper), O (Oxygen), C (Carbon), and Na (Sodium), (i) EDS point of CuF@Cu 3V and (j) XRD measurement of all materials

A 0.015 M NaOH solution is used as a pH stabilizer because metals are less stable in solutions that are too acidic. In an alkaline environment, Cu^{2+} ions can form soluble hydroxide complexes and prevent premature precipitation. $\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$ acts as a reducing agent that converts Cu(II) species from CuSO_4 to Cu(0). The application of potential serves to create hydrogen bubbles when the electrodeposition method is underway; the presence of these bubbles promotes the formation of a Cu foam structure deposited on the Cu plate through the dynamic hydrogen bubble template (DHBT) mechanism. The mechanism is illustrated in Figure 1 (Wang & Ma, 2023).

Table 1. EDS point analysis of synthesized materials

Electrode	Element				Atomic (%)			
	Weight (%)							
	C K	O K	Na K	Cu K	C K	O K	Na K	Cu K
Cu Bare	-	5.82	-	94.18	-	19.72	-	80.28
CuF@Cu 3V	10.09	9.99	0.77	79.15	30.62	22.76	1.21	45.41
CuF@Cu 5V	9.74	11.12	0.41	78.73	29.35	25.17	0.64	44.84

The XRD diffractograms of bare Cu and Cu foam show 2θ values for all electrodes that are identical to JCPDS Card 003-1005 or JCPDS Card 04-0836 for Cu, as illustrated in Figure 2j. The characteristic diffraction peaks of Cu are found at $2\theta \approx 43.6^\circ$ (111), 50.8° (200), and 74.4° (220), confirming the face-centered cubic (FCC) structure of Cu. These results indicate that the Cu foam that has been successfully deposited on the unmodified Cu plate is Cu(0)

species, without the presence of significant Cu_2O or CuO oxide phases (Duangsa et al., 2021; Xun et al., 2019; Zhang et al., 2006).

3.2 Electrochemical Study of Cu/Cu foam electrode

Electrochemical characterization of Cu/Cu foam material was performed using cyclic voltammetry. The measurement results are shown in the following figure. In Figure 3, a Cu peak is observed at a potential of approximately -0.43 to -0.63 V, indicating the reduction of Cu^+ to Cu^0 (Hussain et al., 2025; S.Vidhya, 2017). Cu foam with Na citrate at a deposition potential of 3 V shows the highest S/B background current of 1.334 A. This is because its pores are quite small, resulting in a larger surface area compared to other electrodes, which indirectly has more active sites for electrochemical reactions (Li et al., 2022; Wang et al., 2019).

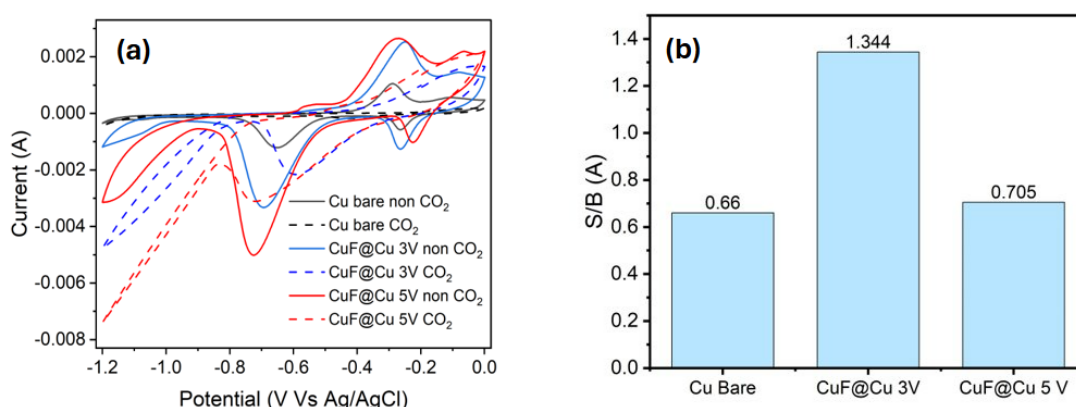


Fig. 3. Electrochemical study of CuF@Cu using Na-Citrate (CuF@Cu-NaC) electrode in the presence of CO_2 gas

3.3 The electroreduction of carbon dioxide

The electrochemical reduction of carbon dioxide was optimized by varying the CO_2 purging time, electrolyte flow rate, and applied potential, as shown in Figure 4. The amount of dissolved CO_2 available at the cathode is strongly influenced by both the gas saturation time and the electrolyte circulation rate. Among the conditions examined, 20 min of CO_2 purging and an electrolyte flow rate of 75 mL min^{-1} produced the highest cathodic current (Figure 4a,b), indicating an appropriate balance between CO_2 availability and mass transport to the electrode surface. The effect of applied potential was subsequently evaluated using bare Cu, and the highest Faradaic efficiency for formic acid was obtained at $-0.33 \text{ V vs. Ag/AgCl}$ (Figure 4c). At this relatively mild potential, the competition between CO_2 reduction and the hydrogen evolution reaction (HER) appears to be minimized, and these conditions were therefore used for subsequent experiments with the CuF@Cu 3 V electrode (Girichandran et al., 2024; Syauqi et al., 2023).

The products formed during CO_2 electroreduction were analyzed by using high-performance liquid chromatography and gas chromatography. Formic acid was identified as the principal liquid product, while hydrogen was detected as the main gaseous by-product for both bare Cu and CuF@Cu 3 V electrodes (Figure 5a). Under the optimized conditions, the CuF@Cu 3 V electrode exhibited higher Faradaic efficiency and carbon conversion efficiency toward formic acid than bare Cu, increasing the Faradaic efficiency from 23.28% to 26.73% (Figure 5b). Notably, the formic acid production rate increased significantly from 142.05 mmol/h for bare Cu to 435.55 mmol/h for CuF@Cu 3V. Although the improvement in Faradaic efficiency is moderate, the most notable result is the substantial suppression of hydrogen evolution, with a hydrogen Faradaic efficiency of only 0.07%. Reproducibility testing gave a relative standard deviation of 5.16%, indicating that

the electrode preparation and electrochemical performance were reasonably consistent (Figure 5c).

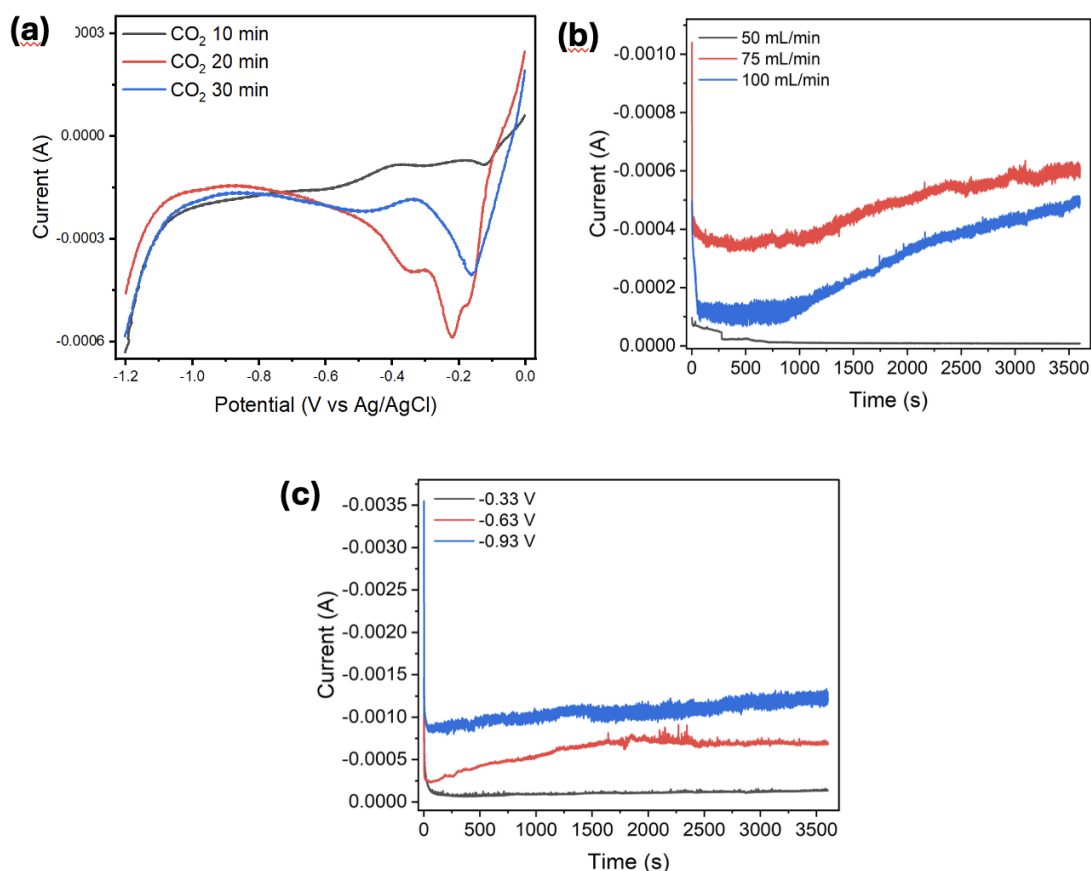


Fig. 4. Optimization of CuF@Cu for CO₂ reduction Flow-Cell: (a) aeration time, CO₂ purging time; (b) flow rate; and (c) applied potential

The improved selectivity toward formic acid can be understood in terms of the reaction pathway on the Cu surface. On copper-based catalysts, formate formation is generally associated with the stabilization of the *OCHO intermediate, whereas multicarbon (C₂⁺) products are formed through adsorption of *CO intermediates followed by C–C coupling. Under the relatively low applied potential used in this study, the adsorption and dimerization of *CO species are expected to be less favorable, while the *OCHO pathway remains kinetically accessible. The predominance of formic acid and the absence of detectable C₂⁺ products therefore suggest that the CuF@Cu surface provides reaction sites that preferentially stabilize oxygen-bound intermediates rather than strongly adsorbed *CO species.

The enhanced performance of CuF@Cu 3 V is closely related to its three-dimensional porous architecture. The interconnected pore network increases the electrochemically accessible surface area and provides a high number of exposed catalytic sites for CO₂ adsorption and activation. In addition, the porous structure facilitates electrolyte penetration and shortens diffusion distances, thereby improving the transport of dissolved CO₂ to the active surface. The confined microenvironment within the pores may also increase the local pH near the electrode surface, which suppresses the competing HER and favors the stabilization of intermediates involved in formate production. Smaller and more uniform pores produced in the presence of sodium citrate are likely to further enhance the reactant residence time and intermediate confinement, contributing to the improved selectivity observed for CuF@Cu 3 V (Elgazzar et al., 2025; Fernández-Caso et al., 2023).

A comparison with previously reported Cu-based electrodes is presented in Table 2. Direct comparison of Faradaic efficiencies among different studies should be interpreted

with caution because electrocatalytic performance is strongly affected by electrolyte composition, pH, current density, reactor configuration, and electrolysis duration. The data in Table 2 are therefore intended to provide general context rather than a strict ranking of catalytic performance. Within this context, the CuF@Cu electrode developed in this work achieved a formic acid Faradaic efficiency of 26.73% in a flow-cell configuration at a relatively low applied potential, together with near-complete suppression of hydrogen evolution. These results indicate that the porous Cu foam structure improves the selectivity of copper toward formate formation and demonstrate a clear relationship between electrode architecture and electrocatalytic behavior.

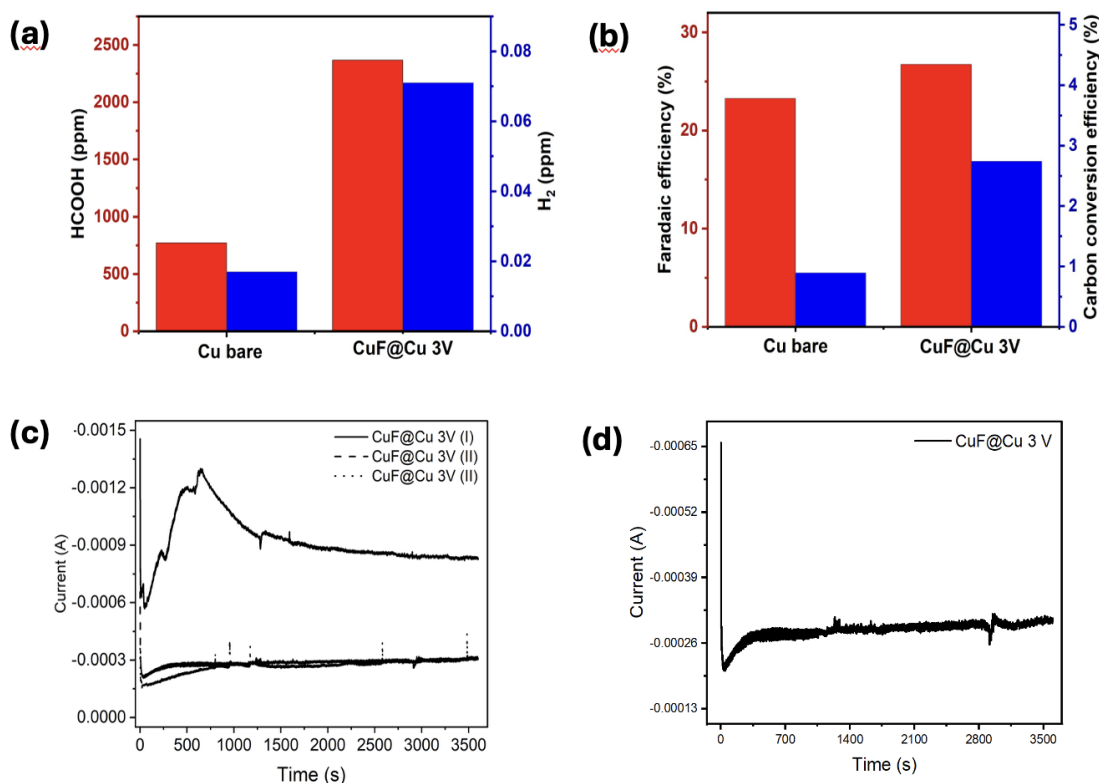


Fig. 5 (a) Application of developed electrode for CO₂ reduction; (b) efficiency; and (c) reproducibility (electrode performance)

The specific structure determines the distinctive performance of Cu materials in CO₂ electroreduction; therefore, a comparative analysis was conducted to provide further insight into the CO₂ electroreduction reactions. The evaluation is based on the Faradaic efficiency (FE) produced in the formation process of formic acid (HCOOH) and hydrogen (H). The test was carried out using a variety of electrode and electrolyte modification types in two system configurations, namely the batch system (H-type cell) and the Flow cell system.

Based on the data in Table 2, it can be concluded that the surface modification of the Cu electrode has a significant effect on the selectivity of the CO₂ electroreduction reaction, especially in increasing the formation of formic acid (HCOOH) and reducing the side reaction of hydrogen gas formation (H₂). In batch systems (H-type cells), Cu electrodes without modification or with a porous structure (Cu foam@Cu) exhibit relatively low Faradaic efficiency for HCOOH and dominant H₂ formation, indicating the strength of the hydrogen evolution reaction (HER) on the surface of pure Cu. In contrast, modification of Cu with Sn and F (Sn@Cu and CuF@Cu) overall can increase selectivity against HCOOH while suppressing H₂ formation, indicating the important role of electronic effects and active site isolation in stabilizing formate-forming intermediate reactions.

In flow cell systems, increased CO₂ mass transport and changes in the reaction environment result in higher Faradaic efficiency of HCOOH than batch systems, especially

in Cu foil and Cu@Bi electrodes, with FE HCOOH values above 70%. This shows that the configuration of the reactor plays an equally important role as the modification of the electrode material in determining electrocatalytic performance. Overall, the combination of Cu surface modification and the use of flow systems have been shown to be effective in increasing the electroreduction selectivity of CO₂ towards HCOOH by suppressing hydrogen evolution reactions.

Table 2. Comparison of performance of the developed materials with other reports

Electrode	System	Red time (min.)	Electrolyte	%Faradaic efficiency	
				HCOOH	H ₂
Cu@Cu ₂ O (Li & Kanan., 2012)	Batch	60	0.5 M KHCO ₃	33.0	29.4
Sn@Cu (Zhang et al., 2018)	Batch	60	0.5 M NaHCO ₃	61.1	37.2
Cu foil (Li et al., 2020)	Flow	40	0.5 M KHCO ₃	27.1	64.1
Cu@Bi (Zhang et al., 2020)	Flow	30	1.0 M KOH	71.3	9.6
CuF@Cu (This work)	Flow	60	0.1 M KHCO ₃ / 0.1 M KOH	26.7	0.07

Abbreviation: Red time = Reduction time

4. Conclusions

Cu foam was successfully deposited on the Cu bare using a capping agent in the form of Na citrate with a voltage of 3 V and 5 V. The optimum condition of CO₂ electroreduction in a flow cell system was at a potential of 0.33 V and a flow rate of 75 mL/min, which produced a Faradaic efficiency of formic acid of 23.28% and 26.73% for Cu bare and CuF@Cu 3 V, respectively. The CuF@Cu 3 V electrode has higher Faradaic efficiency due to the larger surface area and three-dimensional structure of Cu foam, which improves active sites for electrochemical reaction. Other variations are needed, such as pH, capping agent concentration, and deposition time in the synthesis of Cu foam to obtain a more optimal Faradaic efficiency of CO₂ conversion.

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Author Contribution

H.G.R., and F.T.: Data curation, formal analysis, and writing the original draft. L.P., O.M., and A.R.S.: Analysis, validation, writing –review and editing. F.T., and O.M.: Data curation and formal analysis. A.R.S., and O.M.: validation, funding acquisition, and supervision.

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Ethical Review Board Statement

Not available.

Informed Consent Statement

Not available.

Data Availability Statement

Not available.

Conflicts of Interest

The authors declare no conflict of interest.

Declaration of Generative AI Use

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