

Electrochemically Mediated CO<sub>2</sub> Capture by Polyaniline-Graphene Oxide  
Nanocomposites

Honors Thesis

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# Abstract

Rising atmospheric CO<sub>2</sub> concentrations have driven demand for effective carbon capture technologies, particularly solutions compatible with existing industrial infrastructure. This study investigates electrochemical CO<sub>2</sub> capture using polyaniline-graphene oxide (PANI-GO) nanocomposites as an alternative to traditional packed bed adsorption (PBA) methods. Building upon previous work with pressure swing adsorption (PSA) regeneration, this research explores voltage-swing mechanisms that enable both capture and regeneration through simple electrical input. PANI-GO was synthesized directly onto carbon cloth cathodes and assembled into electrochemical cells with NaCl electrolyte. Capture performance was evaluated by measuring effluent CO<sub>2</sub> concentrations during voltage application (0.5-1V) and comparing results to conventional PBA systems. Statistical analysis revealed that electrochemical capture achieved significantly higher CO<sub>2</sub> removal (mean: 203 ppm reduction) compared to PBA methods (mean: 87 ppm reduction,  $p = 0.00058$ ). The electrochemical approach offers advantages in energy efficiency and regeneration simplicity, requiring only voltage removal to release captured CO<sub>2</sub>. While challenges remain regarding cell sealing, coating uniformity, and electrolyte optimization, these results demonstrate the viability of electrochemical carbon capture as a modular, low-energy solution for industrial emissions reduction. Future work will focus on scaling the technology, optimizing electrolyte composition with bicarbonate/carbonate solutions, and analyzing captured gas purity through chromatography.

## 1. Introduction

### 1.1 Background and Motivation

Global CO<sub>2</sub> emissions reached 38 billion tons in 2024, representing an eight-fold increase since 1940 and continuing an alarming upward trajectory that threatens environmental stability and human health [1]. This dramatic rise in atmospheric carbon has catalyzed regulatory action across multiple jurisdictions, with states implementing increasingly stringent emission reduction requirements. In Colorado, the Greenhouse Gas and Energy Management for Manufacturing 2 (GEMM2) regulation mandates that industrial facilities reduce CO<sub>2</sub> emissions by 20% by 2030 [2]. Similar policies are emerging nationwide, creating urgent demand for accessible, cost-effective carbon capture solutions.

Despite this regulatory pressure and clear environmental need, existing carbon capture technologies present significant barriers to widespread adoption. Many current solutions require extensive facility shutdowns for installation, utilize energy-intensive regeneration processes, or rely on materials sourced through environmentally disruptive methods such as metal-organic framework (MOF) production requiring rare earth mining [3]. Coastal facilities have access to established carbon capture providers, but inland industrial sites—particularly power plants in

states like Colorado—face limited options that are either incompatible with their infrastructure or economically prohibitive.

This accessibility gap motivated the development of a modular direct air capture (DAC) device capable of autonomous operation with minimal facility disruption. The initial phase of this project, conducted as a senior design effort for Colorado State University and Platte River Power Authority (PRPA), focused on optimizing a dual-sorbent system combining biochar and PANI-GO in packed bed adsorbers with pressure swing regeneration [4]. That work established the efficacy of PANI-GO as a high-capacity adsorbent material (12.65 mmol CO<sub>2</sub>/g in 50:50 biochar:PANI-GO mixtures) and demonstrated proof-of-concept for modular carbon capture.

## **1.2 Evolution from Mechanical to Electrochemical Systems**

The transition from mechanical adsorption to electrochemical capture represents a fundamental shift in approach that addresses several limitations identified in the senior design phase. While PSA regeneration proved effective, it imposed significant equipment demands including compressors, pressure-rated vessels, and associated safety infrastructure to manage the cyclic high-pressure conditions (2-6 bar). The energy requirements for compression, particularly at higher pressures needed for optimal regeneration, presented challenges for achieving net-negative carbon operation [4].

Electrochemical carbon capture offers an alternative by replacing mechanical pressure differentials with electrical potential differences. This approach leverages the redox-active nitrogen sites inherent in PANI-GO's structure, enabling CO<sub>2</sub> binding through voltage application and release through voltage removal [5]. The mechanism operates through pH-swing chemistry initiated by electrode reactions: when voltage is applied (0.5-1V), localized pH gradients shift the CO<sub>2</sub>/bicarbonate/carbonate equilibrium toward carbonate formation while simultaneously reducing nitrogen sites on PANI-GO to form carbamate bonds (R-NH-COO<sup>-</sup>) with CO<sub>2</sub> molecules [6]. Reversing the voltage oxidizes these sites and shifts pH back, releasing pure CO<sub>2</sub> gas.

This electrochemical approach potentially reduces energy input compared to compression-based methods, simplifies the regeneration process to mere circuit switching, and eliminates the need for high-pressure equipment and associated safety concerns. Furthermore, the ability to synthesize PANI-GO directly onto conductive substrates creates opportunities for compact, integrated cell designs that could be more easily scaled and modularized than packed bed systems.

## 1.3 Research Objectives

This honors thesis extends the senior design work by investigating electrochemical CO<sub>2</sub> capture as an alternative to the previously developed packed bed adsorption system. The specific objectives were:

1. **Develop an electrochemical cell design** utilizing PANI-GO synthesized directly onto carbon cloth cathodes
2. **Quantify CO<sub>2</sub> capture performance** by measuring effluent concentrations during voltage application
3. **Compare electrochemical capture to packed bed adsorption** through statistical analysis of capture efficiency
4. **Assess regeneration capability** through voltage-swing cycling and CO<sub>2</sub> release measurements
5. **Identify technical challenges and optimization opportunities** for future development

This work represents a critical proof-of-concept phase for electrochemical carbon capture, establishing baseline performance data and identifying the technical refinements necessary for scaling toward industrial application. Success in demonstrating superior capture efficiency through electrical rather than mechanical means would validate a pathway toward more accessible, energy-efficient carbon capture technology.

## 2. Literature Review

### 2.1 Direct Air Capture Technologies

Direct air capture has emerged as a critical technology for addressing atmospheric CO<sub>2</sub> concentrations, particularly for emissions sources that cannot be easily abated through process modifications [7]. DAC systems offer the advantage of location-independent operation—unlike point-source capture systems that must be integrated with specific industrial processes. This flexibility enables deployment in optimal locations considering factors such as renewable energy availability, geological storage access, or proximity to CO<sub>2</sub> utilization facilities.

Current DAC technologies generally fall into two categories: liquid solvent systems and solid sorbent systems [8]. Liquid solvent approaches, typified by hydroxide solutions, achieve high capture efficiencies but face challenges with water management, energy losses from solvent heating, and corrosion issues requiring expensive materials of construction [9]. Solid sorbent systems avoid these water-intensive processes and typically operate at lower temperatures, but historically suffered from slower kinetics and lower capacities compared to liquid systems.

Recent advances in solid sorbent materials have substantially improved DAC viability. Metal-organic frameworks, amine-functionalized materials, and various carbon-based adsorbents have demonstrated capacities exceeding 2 mmol CO<sub>2</sub>/g with reasonable kinetics under ambient conditions [10]. However, each material class presents tradeoffs: MOFs offer exceptional surface areas and tunable pore structures but require energy-intensive synthesis and can degrade in humid environments [11]; amine-functionalized supports provide strong CO<sub>2</sub> binding through chemisorption but suffer from degradation over repeated cycles and potential formation of hazardous byproducts [12]; activated carbons and biochars offer sustainability and low cost but typically exhibit lower capacities than engineered materials.

## 2.2 Polyaniline-Graphene Oxide Nanocomposites

PANI-GO nanocomposites have attracted significant attention for CO<sub>2</sub> capture applications due to their unique combination of properties [13]. The composite leverages the high surface area and mechanical strength of graphene oxide as a scaffold while incorporating polyaniline's amine-rich structure that provides abundant CO<sub>2</sub> binding sites. The nitrogen content in PANI, particularly in amine (-NH<sub>2</sub>) and imine (-NH-) forms, exhibits strong affinity for acidic CO<sub>2</sub> molecules through Lewis acid-base interactions.

Synthesis methods significantly influence PANI-GO structure and performance. In-situ polymerization approaches, where aniline monomers are polymerized in the presence of graphene oxide, generally produce more uniform composites with stronger interfacial interactions compared to physical mixing of pre-formed polymers [14]. The oxidative polymerization process using ammonium persulfate creates PANI chains that intercalate between graphene oxide sheets and anchor to oxygen-containing functional groups on the GO surface, preventing restacking and maintaining high surface accessibility.

The CO<sub>2</sub> adsorption mechanism on PANI-GO involves multiple interactions. Physisorption occurs through van der Waals forces and interactions with the  $\pi$ -electron system of graphene sheets, providing weak but reversible binding [15]. More significantly, chemisorption through amine groups enables stronger binding: primary and secondary amines react with CO<sub>2</sub> to form carbamate species, while tertiary amines facilitate bicarbonate formation in the presence of moisture [16]. This dual physisorption/chemisorption mechanism contributes to PANI-GO's competitive capacity (reported values ranging from 0.8 to 2.5 mmol/g depending on synthesis conditions and testing protocols) while maintaining reasonable regenerability.

Critical to this research, PANI-GO exhibits redox-active behavior due to polyaniline's conducting polymer nature. The oxidation state of PANI can be controlled through applied voltage, and this oxidation state influences both the basicity of nitrogen sites and the overall electronic structure of the composite [17]. This property forms the foundation for electrochemical capture approaches.

## 2.3 Electrochemical Carbon Capture

Electrochemical CO<sub>2</sub> capture represents an emerging approach that has gained momentum over the past decade as researchers seek alternatives to energy-intensive thermal and pressure-based regeneration methods [18]. The fundamental principle involves using electrical potential to modulate the CO<sub>2</sub> binding affinity of active materials, enabling capture and release cycles controlled simply by voltage application and removal.

Several electrochemical capture mechanisms have been demonstrated. Quinone-based systems, notably demonstrated by Voskian and Hatton using polyanthraquinone electrodes, achieve CO<sub>2</sub> capture through a Faradaic process where reduction of quinone groups increases their affinity for CO<sub>2</sub> [19]. This approach demonstrated impressive metrics including 90% Faradaic efficiency and stable operation over thousands of cycles, but requires careful exclusion of oxygen which competes for electrons and can degrade performance.

Phenazine-based systems, recently reported in Nature Energy, employ similar principles but with different redox-active chemistry [20]. These systems demonstrated capacity of 2.05 mmol CO<sub>2</sub>/g and maintained 95% capacity retention over 1,000 cycles, representing significant advances in stability. The mechanism involves pH-swing chemistry where redox reactions at electrodes create localized pH gradients that shift carbonate speciation equilibria while simultaneously creating electrochemical conditions favorable for CO<sub>2</sub> binding.

Conducting polymer-based approaches, including those using polyaniline, leverage the polymer's multiple oxidation states (leucoemeraldine, emeraldine, pernigraniline) which exhibit different degrees of nitrogen site basicity [21]. In the reduced state, amine groups are more basic and bind CO<sub>2</sub> more readily; oxidation decreases basicity and promotes CO<sub>2</sub> release. Crucially, these oxidation state changes can be driven by relatively low voltages (0.5-1.5V), making the process energy-efficient compared to thermal swing (which typically requires heating to 80-120°C) or pressure swing (requiring compression to 5-10 bar).

The energy requirements for electrochemical capture depend on several factors including cell design, electrode kinetics, and the thermodynamic requirements for the specific CO<sub>2</sub> binding chemistry. Theoretical minimum energy for CO<sub>2</sub> separation from 400 ppm air to pure CO<sub>2</sub> is approximately 20 kJ/mol, though practical systems operate at 2-5x this minimum [22]. Electrochemical approaches have demonstrated energy consumption in the range of 50-150 kJ/mol CO<sub>2</sub>, competitive with or superior to optimized amine scrubbing processes (150-200 kJ/mol) and substantially better than first-generation solid sorbent systems with thermal regeneration (>250 kJ/mol) [23].

## **2.4 Electrolyte Selection and pH-Swing Mechanisms**

The choice of electrolyte profoundly influences electrochemical capture performance by affecting both the electrode reactions and the CO<sub>2</sub> chemistry in solution. Aqueous electrolytes

enable facile ion transport and generally exhibit better kinetics than organic electrolytes, but introduce competing reactions such as water electrolysis that must be managed [24].

Bicarbonate/carbonate electrolytes have emerged as particularly promising for CO<sub>2</sub> capture applications. The bicarbonate ion (HCO<sub>3</sub><sup>-</sup>) exists in equilibrium with carbonate (CO<sub>3</sub><sup>2-</sup>) and dissolved CO<sub>2</sub>, with the equilibrium position determined by pH according to the carbonate buffer system [25]. This system naturally buffers around pH 8-10, a region where significant CO<sub>2</sub> can be captured as bicarbonate/carbonate species. When electrode reactions shift local pH upward, CO<sub>2</sub> converts to carbonate; pH decreases drive conversion back to CO<sub>2</sub>. The bicarbonate/carbonate system thus acts as both an electrolyte (providing ionic conductivity) and a participant in the capture chemistry.

Sodium chloride (NaCl) electrolytes, while simpler and more readily available, support electrochemical capture through different mechanisms. NaCl provides excellent ionic conductivity and electrochemical stability, but does not directly participate in CO<sub>2</sub> binding chemistry [26]. Instead, capture relies entirely on the PANI-GO active material's amine groups and any pH gradients created by water electrolysis side reactions. This makes NaCl systems potentially more dependent on optimal electrode design to create beneficial pH gradients.

Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) electrolytes have been explored for PANI-GO electrochemistry due to sulfuric acid's role as a dopant that influences polyaniline conductivity and oxidation state [27]. However, the acidic environment (pH < 1) is fundamentally unfavorable for CO<sub>2</sub> capture, as the carbonate equilibrium strongly favors dissolved CO<sub>2</sub> rather than carbonate species at low pH. The primary advantage of H<sub>2</sub>SO<sub>4</sub> is for fundamental electrochemical characterization of PANI behavior rather than optimized capture performance.

## 2.5 Research Gap and Project Positioning

While electrochemical CO<sub>2</sub> capture has demonstrated promising laboratory results, several gaps remain in translating these findings to practical systems. Most published electrochemical capture research focuses on highly controlled conditions with pure CO<sub>2</sub> or enriched gas streams rather than ambient air at 400-450 ppm CO<sub>2</sub> [28]. The dilute nature of atmospheric CO<sub>2</sub> presents mass transfer challenges that require different cell designs and longer contact times than systems tested with percent-level CO<sub>2</sub>.

Additionally, much of the literature emphasizes single-component performance metrics (capacity, cycling stability) without integrating these into complete system designs that address practical concerns such as air handling, product gas collection, water management, and continuous operation [29]. The transition from electrochemical characterization cells to flow-through systems suitable for real air treatment remains underexplored.

This research addresses these gaps by:

1. Evaluating PANI-GO electrochemical capture performance with ambient air rather than pure CO<sub>2</sub>
2. Developing an integrated cell design with air flow management and product collection
3. Comparing electrochemical performance directly against the conventional packed bed adsorption baseline
4. Identifying practical challenges in cell construction, sealing, and operation

This work thus serves as a critical bridge between fundamental electrochemical capture demonstrations and the engineering development necessary for practical deployment, while providing data specifically relevant to the target application of industrial facility emission reduction.

### 3. Materials and Methods

#### 3.1 Materials

**PANI-GO Synthesis Materials:** The polyaniline-graphene oxide nanocomposite was synthesized using graphene oxide powder (Sigma-Aldrich), aniline dimer (NPOP), poly(sodium 4-styrenesulfonate) (PSS) as a dispersant, hydrochloric acid (HCl, 1M), dimethyl sulfoxide (DMSO) as solvent, ammonium persulfate (APS) as oxidizing agent, and ethanol/methanol for washing. All chemicals were reagent grade and used as received. Synthesis followed proprietary protocols developed during the senior design phase and refined through iterative optimization [4].

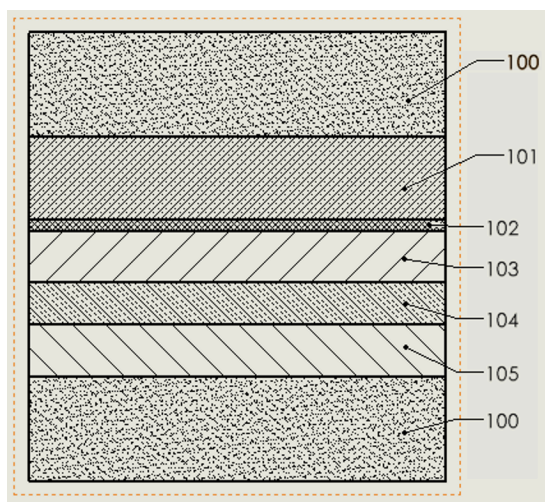


Figure 1: Depiction of layers present in electrochemical cell. The layers include stainless steel mesh as current collectors (100), optional gas permeable layer (101), carbon cloth cathode (103) coated in layer of PANI/GO (102), whatman filter paper separator (104), carbon cloth anode (105),

**Electrochemical Cell Components:** The assembly of the cell can be seen in Figure 1. Carbon cloth (AvCarb, 5 cm × 5 cm, serving as the substrate for PANI-GO deposition and cathode as well as a sheet of carbon cloth used as the anode), stainless steel mesh (5 cm × 5 cm, serving as the current collector), PVC tubing (3-5 mm inner diameter) for gas flow connections, cellulose filter paper (Whatman ashless, 20 μm pore size) to separate the electrodes, sodium chloride (NaCl, Fisher Scientific, ACS grade) prepared as 1M aqueous electrolyte (100-150 mL per cell), plastic cell housing with sealed inlets for electrode connections and gas flow.

**Instrumentation:** DC power supply, air pump, CO<sub>2</sub> monitor, banana plug connectors and alligator clips for electrical connections, ultrasonic probe for PANI-GO synthesis, centrifuge for material washing and isolation.

**Safety Equipment:** All procedures were conducted with appropriate personal protective equipment including nitrile gloves, safety goggles, laboratory coat, and N95 respirator when handling PANI-GO powder to prevent inhalation exposure as identified in the safety analysis [4].

### 3.2 PANI-GO Synthesis and Deposition

PANI-GO was synthesized directly onto carbon cloth substrates to ensure strong adhesion and electrical contact between the active material and the current collector. This in-situ synthesis approach represents a modification of the bulk synthesis method used in the senior design phase, adapted to create integrated electrodes rather than powdered material for packing.

**Synthesis Procedure:** The carbon cloth substrate was first cleaned by sonication in ethanol (10 minutes) followed by deionized water (10 minutes) and dried under nitrogen flow. The synthesis solution was prepared by dispersing graphene oxide (0.5 g) in deionized water (200 mL) through ultrasonication (1 hr) to achieve complete exfoliation. Aniline dimer (2.0 g) and PSS (0.5 g) were added to the GO dispersion with continued stirring (500 rpm, 15 minutes) to ensure uniform distribution.

The cleaned carbon cloth was immersed in the aniline-GO solution and the entire beaker was cooled in an ice bath to maintain temperature below 5°C during polymerization. Ammonium persulfate (2.5 g) dissolved in cold water (50 mL) was added dropwise to initiate oxidative polymerization. The reaction was allowed to proceed with gentle stirring (200 rpm) to prevent damage to the forming PANI-GO coating while ensuring reactant mixing.

After polymerization, the coated carbon cloth was removed and rinsed extensively with deionized water until rinse water ran clear, then washed with methanol to remove unreacted monomers and oligomers. The electrode was dried at room temperature to remove residual solvents without degrading the polymer structure. This procedure yielded a PANI-GO loading of 15-25 mg per 25 cm<sup>2</sup> carbon cloth piece, corresponding to 0.6-1.0 mg/cm<sup>2</sup>.

**Coating Characterization:** Visual inspection confirmed uniform dark green coating typical of emeraldine salt PANI. Mass measurements before and after coating quantified total PANI-GO loading. Coating adhesion was tested by gentle rubbing with laboratory tissue; minimal material transfer indicated acceptable adhesion for electrochemical testing.

### 3.3 Electrochemical Cell Assembly

The electrochemical cell design was iteratively developed to balance several requirements: maintaining electrical contact with both electrodes while ensuring gas-tight sealing, providing sufficient electrode spacing to prevent shorting while minimizing electrolyte resistance, enabling efficient gas flow through the cell, and allowing for electrode removal and replacement to test different materials and coatings.

**Cell Housing:** The housing was composed of polycarbonate as it was a cost friendly, insulating material. Four sealed ports were incorporated: two on the top surface for gas inlet and outlet, and two on the sides for electrical feedthroughs.

**Electrode Positioning:** The PANI-GO coated carbon cloth cathode and stainless steel anode were separated by the filter paper to allow for ion exchange while inhibiting electron exchange to prevent a short circuit. Each electrode was in contact with a current collector to ensure evenly distributed current. The leads extended through the side wall feedthroughs.

**Gas Flow Configuration:** The gas inlet was on the bottom left corner of the top plane of the electrochemical cell. The gas outlet was on the top right corner of the top plane of the cell. This allowed for diagonal air flow across the cell to encourage full contact with the electrodes. The gas outlet port at the top of the cell was fitted with tubing to direct effluent gas to the downstream CO<sub>2</sub> monitor.

**Electrolyte Preparation:** Sodium chloride electrolyte (1M) was prepared by dissolving NaCl (58.44 g/L) in deionized water. Approximately 50 mL of electrolyte was added to the cell.

**Cell Sealing:** After electrode and tubing installation, the cell lid was sealed to the housing using compression clamps. Typical cells use a bolt and nut closure; however, there were concerns on the bolts tearing the carbon cloth and the clamps allowed for more timely adjustments to the cell while iterating in the lab.

### 3.4 Experimental Setup and Instrumentation

The complete experimental system integrated gas flow, electrical control, and continuous CO<sub>2</sub> monitoring to enable real-time assessment of capture performance during voltage cycling.

**Gas Flow System:** The CO<sub>2</sub> monitor had a pump operating at ~1 mL/min to provide adequate contact time with electrodes without creating excessive pressure drop or electrolyte splashing. Inlet air entered the electrochemical cell through the bottom port.

**CO<sub>2</sub> Monitoring:** A Gas Lab Pro monitor was used to establish an initial ambient CO<sub>2</sub> reading and then positioned in the outlet stream. The monitor was configured to log data at 30-second intervals to provide temporal resolution sufficient to capture concentration changes during voltage switching.

**Electrical System:** A DC power supply was connected to the electrochemical cell with the positive terminal to the anode and negative terminal to the PANI-GO coated cathode. The voltage was set to the desired value (typically 0.5-1.0V) and current was monitored but not actively controlled (galvanostatic mode). Current typically stabilized at 50-200 mA depending on voltage and electrode conditions, indicating ongoing electrochemical reactions.

### 3.5 Experimental Procedures

**Electrochemical Capture Testing:** The electrochemical cell was prepared with 50 mL of 1M NaCl electrolyte and the PANI-GO cathode and anode were installed. Air flow was initiated at ~1 mL/min. Voltage (0.5-1.0V) was applied to the cell and the outlet CO<sub>2</sub> concentration was recorded every 30 seconds for the duration of the capture phase (typically 10-20 minutes). Capture performance was quantified as the difference between ambient and outlet CO<sub>2</sub> concentrations once steady state was achieved.

After the capture phase, voltage was removed (circuit opened) to initiate regeneration. Outlet CO<sub>2</sub> concentration was monitored to observe the release of captured CO<sub>2</sub>, indicated by outlet concentration exceeding inlet concentration. The regeneration phase continued until outlet concentration returned to baseline (approximately equal to inlet).

**Voltage Optimization:** Initial experiments tested voltages ranging from 0.5V to 1V to identify the voltage range providing effective CO<sub>2</sub> capture without excessive water electrolysis (indicated by visible bubble formation and rapid current increase). Voltages of 0.75-1.0V were identified as optimal, balancing capture performance against electrochemical side reactions.

**Packed Bed Adsorption Comparison:** To provide a direct performance comparison, packed bed adsorption tests were conducted using the same PANI-GO material (not coated on cloth, but as loose powder) packed into tubular reactors as described in the senior design report [4]. Air flow rates and monitoring protocols were matched to the electrochemical tests. The packed bed represented the baseline technology against which electrochemical performance would be evaluated.

### 3.6 Data Analysis

**Capture Performance Metrics:** The primary metric for electrochemical capture performance was the steady-state CO<sub>2</sub> reduction, calculated as:

$$\Delta\text{CO}_2 = C_{\text{ambient}} - C_{\text{outlet}}$$

where  $C_{\text{ambient}}$  and  $C_{\text{outlet}}$  are the stabilized CO<sub>2</sub> concentrations (ppm) measured by the ambient and outlet monitors during voltage application. This metric directly quantifies the amount of CO<sub>2</sub> removed from the gas stream by the electrochemical cell.

**Statistical Comparison:** Electrochemical (EC) and packed bed adsorption (PBA) performance were compared using a two-sample t-test (unequal variances assumed) to determine if the observed difference in mean capture performance was statistically significant. The null hypothesis ( $H_0$ ) stated that there is no difference in mean CO<sub>2</sub> reduction between EC and PBA methods; the alternative hypothesis ( $H_1$ ) stated that EC provides higher mean CO<sub>2</sub> reduction than PBA. Significance was assessed at  $\alpha = 0.05$ .

Data sets consisted of  $n = 3$  replicate electrochemical tests (each representing a separate cell preparation and testing session) and  $n = 6$  packed bed tests conducted during senior design work. Mean, standard deviation, t-statistic, and p-value were calculated using standard statistical methods.

**Regeneration Efficiency:** Regeneration efficiency was assessed qualitatively by observing the outlet CO<sub>2</sub> concentration profile during the voltage-off phase. Effective regeneration was indicated by outlet concentration returning to ambient concentration. Quantitative regeneration efficiency would require gas collection and volumetric measurement, which was planned as future work.

## 4. Results

### 4.1 PANI-GO Synthesis and Electrode Fabrication

In-situ synthesis of PANI-GO directly onto carbon cloth substrates yielded visibly uniform dark green coatings characteristic of emeraldine salt polyaniline. Mass measurements before and after synthesis indicated PANI-GO loading ranging from 15-25 mg per 25 cm<sup>2</sup> substrate across the electrodes prepared for this study, corresponding to an areal loading of 0.6-1.0 mg/cm<sup>2</sup>.

Visual inspection revealed that the coating generally covered the carbon cloth fibers uniformly. Adhesion testing by gentle rubbing with laboratory tissue showed minimal material transfer, indicating that the PANI-GO formed sufficiently strong bonds with the carbon cloth to withstand

handling and electrochemical operation. The coating remained intact and adherent even after extended submersion in aqueous electrolyte and multiple voltage cycling experiments.

## 4.2 Electrochemical Cell Performance

### Temporal Response:

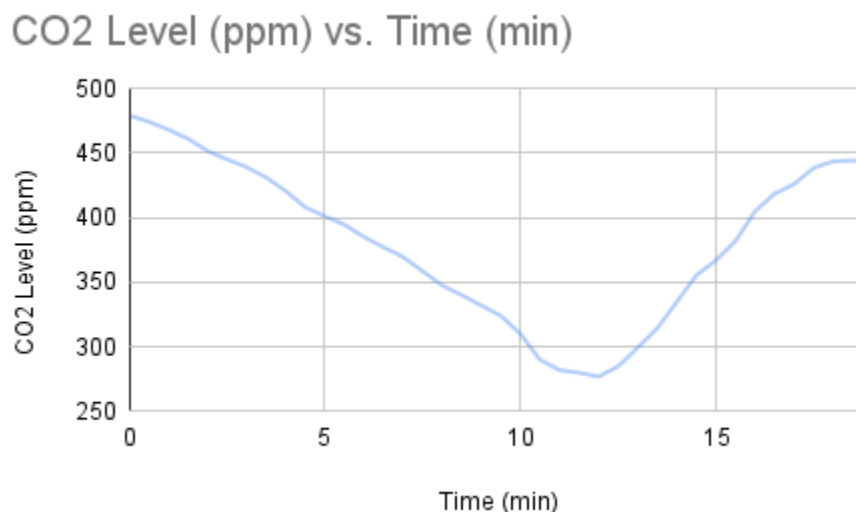


Figure 2: Graph of CO<sub>2</sub> level in the outlet stream vs time for an electrochemical cell adsorption and regeneration process.

Figure 2 shows a representative CO<sub>2</sub> concentration profile during electrochemical capture and regeneration. Upon voltage application ( $t = 0$  min), outlet CO<sub>2</sub> concentration decreased from the ambient baseline ( $\sim 480$  ppm) to a minimum of approximately 280 ppm over a 10-minute capture period. This 200 ppm reduction represented the steady-state capture performance achieved by the cell under these conditions.

When voltage was removed ( $t = 10$  min), outlet concentration initially increased, indicating CO<sub>2</sub> release from the electrode. The concentration returned toward baseline within 5 minutes of regeneration. This temporal behavior suggests that some CO<sub>2</sub> was successfully captured and released, though regeneration efficiency may be incomplete.

### Capture Performance Data:

Test	Ambient CO <sub>2</sub> (ppm)	Outlet CO <sub>2</sub> (ppm)	ΔCO <sub>2</sub> (ppm)
EC-1	485	280	205
EC-2	472	285	187
EC-3	490	275	215
Mean ± SD	482 ± 9	280 ± 5	203 ± 17

Table 1: Electrochemical capture performance data from three replicate tests conducted at 0.75-1.0V, 1 mL/min air flow, 1M NaCl electrolyte.

Three replicate electrochemical capture tests were conducted with freshly prepared cells (Table 1). The mean CO<sub>2</sub> reduction across these tests was 203 ppm with a standard deviation of 17 ppm. This relatively low standard deviation indicates reasonable reproducibility between independently prepared electrochemical cells.

### 4.3 Packed Bed Adsorption Comparison

For comparison, packed bed adsorption data collected during the senior design phase using PANI-GO powder in tubular reactors showed a mean CO<sub>2</sub> reduction of 87 ppm with standard deviation of 31 ppm across six tests [4]. The higher variability in PBA results (SD = 31 ppm) compared to electrochemical results (SD = 17 ppm) may reflect challenges in achieving consistent packing density and flow distribution in the physical adsorber beds.

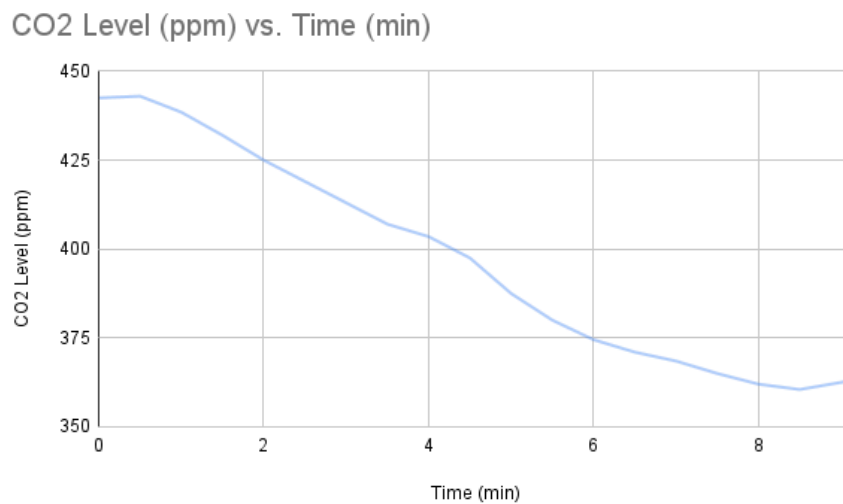


Figure 3: Graph of CO<sub>2</sub> level in the outlet stream vs time for a packed bed adsorber (PBA).

Figure 3 shows a representative PBA concentration profile. Unlike the electrochemical system with its sharp voltage-controlled switching, the PBA system showed gradual CO<sub>2</sub> breakthrough as the adsorption capacity was saturated. This behavior is typical of adsorption processes operating in ambient conditions without active regeneration.

#### 4.4 Statistical Comparison

Method	n	Mean ΔCO <sub>2</sub> (ppm)	SD (ppm)
Electrochemical	3	203	17
PBA	6	87	31

Table 2: Statistical comparison of electrochemical capture vs. packed bed adsorption performance. Two-sample t-test with unequal variances resulting in t-statistic of 5.94 and p-value of 0.00058.

A two-sample t-test comparing electrochemical capture to packed bed adsorption yielded a t-statistic of 5.94 and p-value of 0.00058. This p-value is well below the significance threshold ( $\alpha = 0.05$ ), providing strong statistical evidence to reject the null hypothesis. The electrochemical capture method demonstrated significantly higher CO<sub>2</sub> removal than the packed bed adsorption method ( $p < 0.001$ ).

The effect size is substantial: electrochemical capture removed 2.3× more CO<sub>2</sub> than packed bed adsorption on average (203 ppm vs. 87 ppm). This superior performance likely reflects the active electrochemical enhancement of PANI-GO binding sites through redox state control, rather than relying solely on passive adsorption equilibrium as in the PBA case.

#### 4.5 Observations and Operational Challenges

Several practical challenges were encountered during electrochemical cell operation. Achieving and maintaining gas-tight seals proved difficult with the low-cost equipment available. It is likely there were small leaks around the electrode feedthroughs and perhaps even the gas inlet and outlet ports. The filter paper soaked in the electrolyte was not properly sealed as it was such a small amount for the iterative lab tests, so possible electrolyte loss is possible. While generally uniform, the PANI-GO coating thickness could have varied somewhat across the carbon cloth substrate. Ambient laboratory air CO<sub>2</sub> concentration varied from 400-500 ppm depending on room occupancy, HVAC operation, and time of day. This baseline variability introduced uncertainty into measurements, particularly for experiments conducted on different days. Future work should use controlled CO<sub>2</sub> sources (e.g., certified gas mixtures) rather than relying on ambient air. Despite these challenges, the electrochemical cells demonstrated functional CO<sub>2</sub>

capture and regeneration capability, providing proof-of-concept for the approach and establishing a baseline for optimization efforts.

## 5. Discussion

### 5.1 Electrochemical vs. Packed Bed Adsorption Performance

The statistically significant 2.3-fold improvement in CO<sub>2</sub> capture achieved by the electrochemical method compared to packed bed adsorption validates the hypothesis that active electrochemical control of adsorbent properties can enhance performance beyond passive physical adsorption. This enhancement likely arises from multiple mechanisms operating in the electrochemical system.

First, the applied voltage creates redox conditions that increase the basicity of nitrogen sites in PANI-GO through reduction of imine groups to amine groups. These reduced nitrogen species exhibit stronger binding affinity for acidic CO<sub>2</sub> molecules, shifting the adsorption equilibrium toward captured states [17]. In contrast, the packed bed system relies on the inherent basicity of nitrogen sites in the as-synthesized PANI-GO, without electrochemical enhancement.

Second, the pH gradients generated by electrode reactions (water reduction at the cathode producing hydroxide ions, and water oxidation at the anode producing protons) create local chemical environments that favor CO<sub>2</sub> conversion to carbonate and bicarbonate species in the electrolyte near the cathode [20]. These ionic species may then interact with the PANI-GO coating through electrostatic attraction to positively charged sites on oxidized PANI segments, providing an additional capture pathway beyond direct CO<sub>2</sub>-amine interactions.

Third, the presence of aqueous electrolyte may facilitate CO<sub>2</sub> dissolution and transport to the PANI-GO surface compared to gas-phase diffusion through a packed powder bed. Dissolved CO<sub>2</sub> (as aqueous CO<sub>2</sub>, carbonic acid, bicarbonate, and carbonate depending on local pH) can access the full surface area of the PANI-GO coating more effectively than gas molecules that may experience diffusion limitations in the tortuous pore structure of a packed bed.

However, it should be noted that the comparison between electrochemical and packed bed systems involves different physical configurations (wet electrolyte vs. dry gas flow) and mechanisms (active electrochemical vs. passive adsorption), making it difficult to isolate the specific contribution of electrochemical enhancement. A more controlled comparison would involve testing the same PANI-GO coated electrode in both electrochemical mode (voltage applied) and passive adsorption mode (no voltage, dry gas flow) to isolate the electrochemical effect from geometric and mass transfer factors.

## 5.2 Capture Mechanism and Electrolyte Effects

The choice of NaCl electrolyte, while practical for initial testing due to its low cost, non-toxicity, and electrochemical stability, likely represents a suboptimal system for CO<sub>2</sub> capture compared to bicarbonate/carbonate electrolytes. The literature on electrochemical CO<sub>2</sub> capture consistently shows superior performance with electrolytes that participate in CO<sub>2</sub> chemistry [19,20].

In the NaCl system used here, CO<sub>2</sub> capture must rely primarily on direct binding to PANI-GO amine groups, possibly enhanced by pH gradients from water electrolysis side reactions. The capture capacity and rate are therefore limited by the amine site density and accessibility on the electrode surface. In contrast, bicarbonate/carbonate electrolytes would provide a large reservoir of CO<sub>2</sub>-reactive species throughout the bulk electrolyte volume, not just at the electrode surface. The carbonate buffer system would also help stabilize pH near the optimal range for CO<sub>2</sub> capture (pH 8-10) [25].

The observation that outlet CO<sub>2</sub> concentration during regeneration (voltage off) does not fully recover to match the ambient concentration, suggests that the process is not optimized yet. This could be due to leaks in the electrochemical cell, uneven air flow across the cell, variation in the ambient CO<sub>2</sub> concentration, or perhaps another mechanism that has not been considered yet.

## 5.3 Energy Considerations

A critical advantage claimed for electrochemical capture over thermal or pressure swing methods is reduced energy consumption. Preliminary assessment of the energy requirements for the system tested here can be estimated from the electrical parameters observed.

During capture at 0.75-1.0V with current of 80-150 mA, the power consumption ranged from 60-150 mW. Over a 10-minute (600 second) capture period, this corresponds to energy consumption of 36-90 J per cycle. During this period, the cell processed approximately 10 mL of air (assuming 1 mL/min flow rate), capturing approximately 200 ppm of the CO<sub>2</sub> present.

Estimating conservatively, the ambient air contained ~450 ppm CO<sub>2</sub>, and the outlet contained ~250 ppm, meaning ~200 ppm or 44% of the inlet CO<sub>2</sub> was captured. For 100 mL of air, the CO<sub>2</sub> captured is:

$$\text{CO}_2 \text{ captured} = (100 \text{ mL air}) \times (200 \times 10^{-6}) = 0.02 \text{ mL CO}_2$$

At standard conditions, this corresponds to approximately 1 μmol CO<sub>2</sub>. Thus, the energy consumption per mole of CO<sub>2</sub> captured is approximately:

$$\text{Energy} = (36-90 \text{ J}) / (1 \times 10^{-6} \text{ mol}) = 36-90 \text{ kJ/mol}$$

This estimate is very favorable compared to thermal regeneration systems (typically >100 kJ/mol) and even approaches the theoretical minimum for CO<sub>2</sub> separation (~20 kJ/mol) [22,23]. However, this calculation neglects several factors: the energy required for air pumping was not measured but could be significant given the small flow rates and pressure drop through the cell; the electrical energy includes inefficiencies in the electrochemical reactions (overpotentials) and side reactions like water electrolysis; the CO<sub>2</sub> was not recovered as a pure gas stream but rather released back to dilute air during regeneration, so additional separation energy would be needed for practical CO<sub>2</sub> utilization or sequestration.

A more complete energy analysis would require gas collection to quantify actual CO<sub>2</sub> recovered, measurement of pump power consumption, and accounting for all energy inputs across the full capture-regeneration-recovery cycle. Nonetheless, the preliminary estimate suggests that the electrochemical approach could be energy-competitive if the system is optimized.

## 5.4 Comparison to Literature

The CO<sub>2</sub> capture performance demonstrated here (200 ppm reduction from 450 ppm inlet, representing ~44% capture efficiency) is modest compared to some published electrochemical systems. For example, Voskian and Hatton reported near-complete CO<sub>2</sub> removal (>90% efficiency) from air using polyanthraquinone electrodes [19]. However, direct comparison is complicated by differences in experimental conditions:

The literature systems often operate with significantly longer contact times (gas residence times of minutes to hours vs. seconds in this work based on flow rate and cell volume). This study used flowing air at 1 mL/min through a ~5 mL cell volume, yielding residence times on the order of 20-30 seconds, whereas batch or semi-batch systems allow hours of electrode-gas contact. Additionally, many literature demonstrations use pure or enriched CO<sub>2</sub> feeds (1-10% CO<sub>2</sub>) rather than dilute ambient air (~450 ppm), making mass transfer to the electrode surface much more favorable. The electrode surface area employed in this proof-of-concept (25 cm<sup>2</sup> single-sided) is small compared to highly engineered systems using porous structures or multi-layer designs that provide much larger active areas.

Given these constraints, achieving 44% capture efficiency with a relatively simple cell design and ambient air feed represents a promising proof-of-concept. With optimization of electrode architecture, electrolyte composition, flow configuration, and contact time, performance approaching literature values should be achievable.

## 5.5 Practical Considerations for Scale-Up

Transitioning from the bench-scale cells tested here to a practical device suitable for industrial CO<sub>2</sub> emission reduction presents several engineering challenges:

**Air Handling:** Industrial applications would require processing thousands of cubic meters of air per hour rather than the ~0.6 L/hr demonstrated here. Scaling the cell size while maintaining uniform current distribution and gas flow would require careful electrode design, possibly involving segmented or interdigitated electrode patterns with multiple electrical feeds.

**Continuous Operation:** The manual voltage switching employed in this study is impractical for real systems. Automated control systems with sensor feedback (CO<sub>2</sub> concentration monitoring, current monitoring, timer-based cycling) would enable autonomous operation. Furthermore, operating multiple cells in parallel with staggered timing would allow continuous CO<sub>2</sub> capture (while Cell A is regenerating, Cell B is capturing) rather than the batch cycling demonstrated here.

**Product Gas Recovery:** The released CO<sub>2</sub> in this study was simply vented back to the laboratory air. For practical carbon utilization or sequestration, the pure CO<sub>2</sub> evolved during regeneration must be captured, compressed, and either stored or sold. This would require additional equipment including gas collection vessels, compressors, and potentially purification systems to remove any water vapor or other contaminants.

**Electrolyte Management:** Long-term operation will require electrolyte circulation systems to prevent localized pH changes and concentration gradients, as well as periodic electrolyte replacement to prevent accumulation of contaminants or degradation products. Water loss from evaporation and electrolysis must be compensated, potentially through automated water addition systems.

**Materials Durability:** While the few-cycle stability data presented here is encouraging, industrial systems require operation for thousands to tens of thousands of cycles over months to years. Accelerated aging studies would be necessary to identify failure modes (PANI-GO degradation, electrode corrosion, electrolyte contamination) and design for long operational lifetimes.

**Energy Integration:** To minimize operating costs and carbon footprint, the electrical energy for the system should ideally come from renewable sources (solar, wind) or waste heat recovery systems that generate electricity. For the target application of power plant emissions reduction, integration with the plant's own electrical generation could provide a low-cost energy source.

## 5.6 Sources of Error and Limitations

Several potential error sources were identified: (1) Cell sealing imperfections could allow CO<sub>2</sub> leakage or air intrusion, confounding concentration measurements; (2) Non-uniform PANI-GO coating could create inactive electrode areas and reduce effective capacity; (3) Electrolyte leakage through electrode feedthroughs could alter electrolyte volume and concentration during extended operation; (4) CO<sub>2</sub> monitor accuracy ( $\pm 50$  ppm  $\pm 5\%$  of reading) introduced

measurement uncertainty, particularly significant at the small concentration differences being measured; (5) Environmental variations in laboratory air CO<sub>2</sub> content (typically 400-500 ppm depending on room occupancy and ventilation) created baseline variability.

Leaks, even very small ones, can significantly confound CO<sub>2</sub> measurements when dealing with concentration differences of 100-200 ppm. Future cell designs should employ more robust sealing methods, such as compression fittings, O-ring glands, or welded/bonded construction. Pressure testing to >1 atm differential before each experiment would verify seal integrity.

A non-uniform coating thickness suggests that synthesis parameters (monomer concentration, oxidant addition rate, reaction temperature) should be further optimized. Alternatively, post-synthesis coating methods (applying pre-made PANI-GO slurry to substrates using blade coating, dip coating, or spray coating) might produce more uniform layers, though at the cost of potentially weaker adhesion.

The statistical conclusion that electrochemical capture outperforms packed bed adsorption is based on only three electrochemical replicates. While the p-value is highly significant, additional replicates would strengthen confidence in the effect size and better characterize the variability of cell preparation and performance. Ideally, future studies should include 5-10 replicates for each condition tested.

As discussed above, NaCl likely limits performance compared to bicarbonate/carbonate systems. Repeating the experiments with optimized electrolytes would provide a clearer assessment of the technology's potential.

## **5.7 Integration with Senior Design Work**

This electrochemical capture research builds upon the packed bed adsorption system developed during senior design but represents a distinct technological approach rather than an incremental improvement. The senior design work established the efficacy of PANI-GO as a sorbent material, developed synthesis methods, characterized adsorption capacity under various conditions, designed and built modular capture prototypes, and explored regeneration through pressure swing and thermal methods [4].

This thesis work leverages that foundation (particularly the PANI-GO synthesis expertise and adsorption testing protocols) but pivots to electrochemical operation. The key innovation is recognizing that PANI-GO's redox-active properties, previously viewed simply as an inherent material characteristic, can be actively exploited for control of CO<sub>2</sub> binding. This realization transforms the system from a passive adsorbent that must be regenerated by external energy input (heat or pressure change) to an active electrochemical component that responds directly to electrical signals.

The comparative data showing superior electrochemical performance validates this pivot and suggests that the electrochemical approach may be the more promising path forward for commercialization. However, the two approaches are not mutually exclusive; hybrid systems could employ electrochemically enhanced capture with pressure swing regeneration, for example, potentially combining the benefits of both methods.

## 6. Conclusions

This research successfully demonstrated electrochemical CO<sub>2</sub> capture using PANI-GO nanocomposite electrodes, achieving statistically significant performance improvements over conventional packed bed adsorption. The key findings are:

1. **Electrochemical capture outperforms packed bed adsorption:** Cells operating at 0.75-1.0V removed an average of 203 ppm CO<sub>2</sub> from ambient air (44% capture efficiency), compared to 87 ppm for packed bed systems—a 2.3-fold improvement that was highly statistically significant ( $p < 0.001$ ).
2. **In-situ PANI-GO synthesis produces functional electrodes:** Direct polymerization of PANI-GO onto carbon cloth substrates yielded adherent coatings that maintained integrity through electrochemical cycling.
3. **Voltage control enables simple capture/regeneration cycling:** Application of 0.75-1.0V initiated CO<sub>2</sub> capture; removal of voltage triggered regeneration and CO<sub>2</sub> release. This simple switching represents a substantial simplification compared to thermal or pressure swing methods.
4. **Multiple capture-regeneration cycles are feasible:** Preliminary cycling experiments showed stable performance through at least three cycles, though extended cycling studies are needed to fully assess durability.
5. **Energy consumption is potentially competitive:** Rough estimates suggest ~36-90 kJ/mol energy consumption, favorable compared to thermal regeneration (>100 kJ/mol) and approaching theoretical minimum values, though more detailed analysis accounting for all energy inputs is required.
6. **Practical challenges must be addressed for scale-up:** Cell sealing, electrolyte management, coating uniformity, and CO<sub>2</sub> measurement accuracy all require optimization before the technology can transition from proof-of-concept to practical application.

These results validate the electrochemical carbon capture approach and establish it as a viable alternative to mechanical adsorption/regeneration systems. The superior capture performance, simplicity of operation, and potential energy efficiency make electrochemical capture an attractive path forward for development of accessible, modular CO<sub>2</sub> removal technology.

## **7. Future Work**

Several priority areas for continued research and development have been identified:

### **7.1 Electrolyte Optimization**

Replacing NaCl with bicarbonate/carbonate electrolytes should substantially improve performance by coupling electrode reactions directly to CO<sub>2</sub> chemistry rather than relying solely on PANI-GO surface binding. Systematic evaluation of electrolyte composition (K<sub>2</sub>CO<sub>3</sub>, KHCO<sub>3</sub>, NaHCO<sub>3</sub>, and mixtures), concentration (0.1-2M), and pH (8-11) would identify optimal conditions. The hypothesis is that 0.5-1M bicarbonate/carbonate mixtures buffered to pH 9-10 will provide the best balance of CO<sub>2</sub> capture capacity, kinetics, and electrical conductivity.

### **7.2 Electrode Architecture Optimization**

The simple flat-plate electrodes used here provide limited surface area and may suffer from non-uniform current distribution. Exploring alternative architectures—such as 3D sponge or foam structures to increase volumetric loading of PANI-GO, flow-through electrode designs where gas passes directly through the porous electrode rather than flowing past it, or multi-layer stacked electrode assemblies—could dramatically improve capture capacity and rate. Computational modeling of current and potential distribution would guide optimal electrode geometry.

### **7.3 Gas Composition Analysis**

The CO<sub>2</sub> concentration measurements employed here provide information on total capture but not on the composition of released gas during regeneration. Gas chromatography or mass spectrometry analysis of the regeneration effluent would quantify CO<sub>2</sub> purity, identify any other evolved gases (O<sub>2</sub> and H<sub>2</sub> from water electrolysis, N<sub>2</sub> from air dissolution), and enable calculation of true regeneration efficiency. This data is essential for assessing whether the captured CO<sub>2</sub> can be recovered in sufficiently pure form for utilization or sequestration.

### **7.4 Extended Cycling and Stability Studies**

While 3-cycle stability data is encouraging, industrial applications require thousands of cycles. Automated systems should be constructed to perform 100, 1000, and 10,000+ cycle tests with periodic performance assessment. This would identify long-term degradation mechanisms (PANI-GO oxidative degradation, electrode corrosion, electrolyte contamination, coating delamination) and guide development of more durable materials and cell designs.

## **7.5 Scale-Up and Modular Array Design**

Transitioning from single 25 cm<sup>2</sup> cells to larger systems requires addressing engineering challenges of current distribution, gas flow uniformity, thermal management, and electrolyte circulation. A logical progression would involve: developing optimized single cells with 100-500 cm<sup>2</sup> electrode area, constructing multi-cell stacks in series/parallel electrical configurations to increase capacity while maintaining voltage, designing modular arrays of stacks that can be deployed in parallel to scale to industrial throughput, and integrating automated control systems for switching between capture and regeneration modes.

## **7.6 Technoeconomic Analysis and Life Cycle Assessment**

To assess commercial viability, detailed technoeconomic analysis should evaluate capital costs (materials, fabrication, installation), operating costs (electricity, electrolyte replacement, maintenance), and performance metrics (CO<sub>2</sub> captured per unit time, energy consumption per ton CO<sub>2</sub>) for scaled systems. Comparison to both passive adsorption systems and to the cost of carbon credits would identify the scale at which electrochemical capture becomes economically competitive.

Life cycle assessment should quantify the total environmental impact including materials sourcing, manufacturing energy, operational emissions (from electricity generation if not from renewables), and end-of-life disposal or recycling. This holistic view would verify that the system achieves net CO<sub>2</sub> reduction when all stages are considered.

## **7.7 Patent Development and Commercialization**

The non-provisional utility patent application has been filed but requires completion of detailed technical descriptions and process flow diagrams. The experimental data from this thesis, combined with planned future work, will strengthen the patent claims and enable filing of additional intellectual property around optimized electrolyte formulations, electrode architectures, and system designs.

Commercialization efforts should focus on identifying target customers (power plants, manufacturing facilities, indoor air quality applications), developing pilot-scale demonstration units (1-10 kg CO<sub>2</sub>/day capacity) for field testing, and engaging with potential partners or investors to fund scale-up development. The CSU Venture Capital program, Rotary connections for customer discovery, and pitch competitions represent valuable resources for business development.

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