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Hardware Article

# Low cost 3D printable flow reactors for electrochemistry

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ABSTRACT

Transition to carbon neutrality requires the development of more sustainable pathways to synthesize the next generation of chemical building blocks. Electrochemistry is a promising pathway to achieve this goal, as it allows for the use of renewable energy to drive chemical transformations. While the electroreduction of carbon dioxide ($CO_2$) and hydrogen evolution are attracting significant research interest, fundamental challenges exist in moving the research focus toward performing these reactions on scales relevant to industrial applications. To bridge this gap, we aim to facilitate researchers' access to flow reactors, which allow the characterization of electrochemical transformations under conditions closer to those deployed in the industry. Here, we provide a 3D-printable flow cell design (manufacturing cost < $5), which consists of several plates, offering a customizable alternative to commercially available flow reactors (cost > $6,000). The proposed design and detailed build instructions allow the performance of a wide variety of chemical reactions in flow, including gas and liquid phase electroreduction, electro (less)plating, and photoelectrochemical reactions, providing researchers with more flexibility and control over their experiments. By offering an accessible, low-cost reactor alternative, we reduce the barriers to performing research on sustainable electrochemistry, supporting the global efforts necessary to realize the paradigm shift in chemical manufacturing.

## Specifications table

| Hardware name | *3D printable flow reactor* |
|---|---|
| Subject area | • Engineering and materials science<br>• Chemistry and biochemistry |
| Hardware type | • Other [please specify] - *Electrochemistry* |
| Closest commercial analog | *https://dioxidematerials.com/product/complete-5-cm2-fa-electrolyzer/* |
| Open-source license | *Creative Commons Attribution 4.0 International* |
| Cost of hardware | *Flow reactor only <$5; reactor with the supporting equipment - $1,207.12 - $3,867.97* |
| Source file repository | *https://doi.org/10.5281/zenodo.8435816* |

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**Table 1**
***Google Scholar*** results for each keyword/phrase relating to $CO_2$ electrolysis and hydrogen production. Results are collected from publications released between January 1, 2022, and January 1, 2023. Results were collected by searching for keywords in $CO_2$ electrolysis and hydrogen evolution. The final "Total Number of Results" row summarizes the total number of search results recorded, but the overlap between said search results is not accounted.

| Keyword or Searched Phrase | Number of Results from January 1, 2022, to January 1, 2023 |
|---|---|
| Flow Cell | 187,000 |
| Hydrogen Evolution | 140,000 |
| Electrochemical Reduction | 125,000 |
| Gas Diffusion Electrode | 56,200 |
| $CO_2$ Electrolysis | 28,100 |
| H-Cell | 19,500 |
| $CO_2$ Electroreduction | 22,300 |
| Hydrogen Cell | 14,700 |
| $CO_2$ Electrolyzer | 10,400 |
| Total Number of Results | 603,200 |

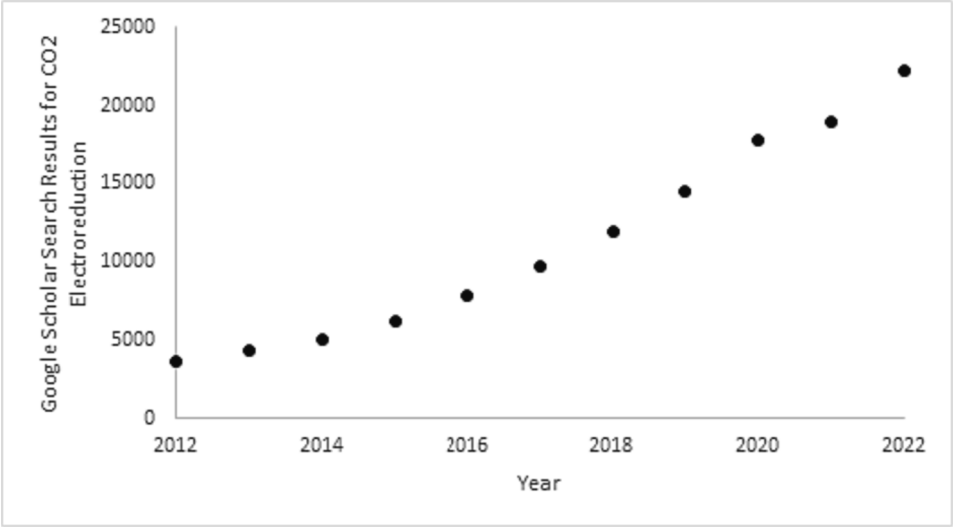


**Fig. 1.** Google Scholar results for the keywords "$CO_2$ Electroreduction". Each data point indicates the number of articles published within its respective year, indicating a growing interest in $CO_2$ electroreduction over the past decade.

## Hardware in context

The ramifications of climate change continue to intensify, leading laboratories worldwide to investigate methodologies to reduce carbon emissions, among which electrochemistry is attracting significant attention. $CO_2$ electrolysis and hydrogen evolution are the commonly studied electrochemical reactions for effective carbon mitigation; the first technique converts the greenhouse gas $CO_2$ into valuable products via an electrocatalytic transformation, while hydrogen evolution provides a fuel source to bypass carbon usage altogether [1,2]. The demand for $CO_2$ electrolysis and hydrogen evolution-based research is illustrated by publication statistics extracted from ***Google Scholar*** (Table 1). Looking only at articles published between January 1, 2022, and January 1, 2023, our search yielded 597,000 results, indicating significant research potential in $CO_2$ electrolysis and hydrogen evolution. Fig. 1 illustrates a drastic increase in Google Scholar results for the "$CO_2$ Electroreduction" keywords – up to 25,000 results in 2022.

Despite growing interest, multiple challenges must be addressed to enable the broader use of electrochemical methods in real-life applications. Factors such as the experimental cost reduce the number of entities able to conduct research in these fields and, in turn, limit the progress in electrochemistry. A solution to reducing the experimental cost is to create comparatively inexpensive equipment, thus, we turn to 3D printing as an alternative method to produce electrochemical reactors [3–6]. 3D printing offers a fast, low-cost alternative to purchasing instrument pieces through manufacturers, and models can be customized to suit a laboratory's needs better. Such examples of 3D model customizations in electrochemistry are most clearly demonstrated through the use of 3D printed electrodes [7,8] and biosensors [9,10] in the field of bioelectronics. Regarding environmentally focused electrochemistry, as described above, 3D printing membrane reactors for electrochemical conversion has been reported to have numerous benefits in addition to lower costs, including improved test capacities and better chemical identification; however, no 3D print design files were attached to these reports [11–13].

**Table 2**
Potential experiments that can be run using the 3D printed flow cell. Please note that not all of the suggested experiments in Table 2 have -been conducted in this paper, but the proposed flow reactor design allows the establishment of leak-free interfaces needed for these applications. It is recommened to substitute Plate_A with Plate_F and Plate_B with Plate_E if using a membrane in the reactor to minimize potential friction damage. Plate_B can also be substituted with Plate_D if a reference electrode is not required.

| *Application* | *Phases* | *Recommended plates* | *Additional equipment* |
|---|---|---|---|
| *Electrochemical reactions* | *Gas/liquid/liquid* | *A + B + A* | *Gas flowmeters, peristaltic pump* |
| | *Gas/liquid* | *A + A / A + B + C* | |
| | *Liquid/liquid* | *A + B + C* | |
| *Photoelectrochemical reactions* | *Liquid* | *A + B with a transparent window attached inside of the gasket* | *Peristaltic pump, UV–vis light source* |
| *Hydrogen production* | *Gas* | *A + B + A* | *Gas flowmeters, peristaltic pump* |
| *Electroplating* | *Liquid* | *A + C* | *Peristaltic pump* |
| *Electro-less plating* | *Liquid* | *A + C* | *Peristaltic pump* |
| *Light-sensitive reactions* | *Gas* | *A + B + A* | *Peristaltic pump* |
| | *Gas/liquid* | *A + A / A + B + C* | |
| | *Liquid* | *A + B + C* | |

In addition to significant cost reduction, the proposed reactor design offers the advantage of performing reactions in flow. Flow reactors allow high precision, reproducibility, and selectivity in electrochemical experiments [14]. A flow reactor is a reactor in which chemicals of interest are carried as a continuous stream: reactants enter the flow cell through tubing at a constant flow rate, and the products are continuously removed from the system in a similar fashion [15]. In contrast to frequently deployed H-cells, where the electrochemical materials (cathodes and anodes) are immersed in liquid solutions, flow reactors provide an opportunity to expose the electrochemical materials to a well-controlled interphase with an intensified mass transfer, ultimately allowing electrochemical reactions to be conducted in a more controlled manner. This enables, e.g., a more selective synthesis of specific hydrocarbons [16–18]. Besides $CO_2$ electrolysis and hydrogen evolution, flow reactors can also be deployed for nitrogen reduction and many chemical reactions relevant to fine and pharmaceutical engineering applications [19].

Though the need to adopt flow reactors more frequently was highlighted in the literature [20], these reactors are still not used as widely as they might be, primarily due to the limited accessibility of the reactors themselves and the additional challenges in operating the experimental set-up where the potential gas and liquid leaks need to be carefully controlled. Therefore, we provide a detailed overview of operating and troubleshooting procedures in addition to the flow reactor design. With a customizable and more accessible version of the flow reactor, more research can be dedicated to understanding sustainability-focused procedures such as $CO_2$ electrolysis and hydrogen production, thus hastening the transition towards carbon neutrality [21–23].

A basic commercially available flow reactor consisting of two titanium and stainless-steel plates costs $6,050; however, it does not include the flowmeter, pump, or connections necessary to run most experiments [24]. In this paper, we propose a 3D printed flow reactor that offers the ability to perform diverse gas and liquid phase reactions, and offer more accessible alternatives to the components needed to successfully run multiple electrochemical experiments as shown in Table 2 of Hardware Description. While the application of the 3D printed flow reactor will be inherently limited to the chemical compatibility of the used filament and the quality of the print itself, we envision a broad range of chemistries compatible with the commercially available filaments, as outlined in the section “Filament choice.”.

## Hardware description

**Design overview.** 3D printable files are provided for several basic plates to form a flow cell: a plate with a serpentine-type flow field and inlet/oulet holes (Plate_A), a plate with a hexagonal hole in the center and its inlet/outlet connections shifted to the sides (Plate_B), and a flat supportive plate (Plate_C) (Fig. 2.a). These plates can be subsequently arranged in different ways, depending on the phases involved in each experiment (gas/liquid) and what interphases need to be created, summarized in Table 2. As an exemplary reactor arrangement, Fig. 2.b depicts the reactor assembly for $CO_2$ electrolysis experiments, where the reactive environment consists of three phases (gas/first-liquid/second-liquid), separated by a cathode, anion exchange membrane, and anode. Importantly, the first-liquid phase must simultaneously be exposed to both upper and lower channels. Therefore, we use a combination of a plate with serpentine flow (for gas supply), a plate with openings on both sides (for the first liquid phase), and another plate with serpentine flow for the second liquid supply. Plates with serpentine flow fields incorporate an O-ring opening to further improve the sealing.

An essential part of the proposed designare tube connectors. Instead of threaded connectors, which require a high resolution of 3D print (and thus would be associated with a higher cost), we incorporate a simple cylindrical connector that silicone tubing (1/8 in.) can be directly pushed on top of (see pictures under Building Instructions).

**Gasket choice and their dual role.** To ensure a proper seal between the plates, we deploy two elements: rubber gaskets of carefully adjusted thickness and external clamps. The gaskets (Fig. 2.a) play a dual role: they both allow for an accurate seal between the plates and hold an opening for the electrocatalytic material, which shall be exposed to the gas or liquid flow circulating on top/above. The thickness of the gasket must be equal to the thickness of the electrocatalytic material being held (to avoid any irregularities in the assembled reactor). It is vital to design your rubber gasket to properly fit all non-3D printed materials and accommodate the number of desired electrodes, otherwise a leak can occur and the experiment’s validity will be compromised.

**Two and three electrode experiments.** To accommodate diverse electrochemical measurements, our reactors can be used both

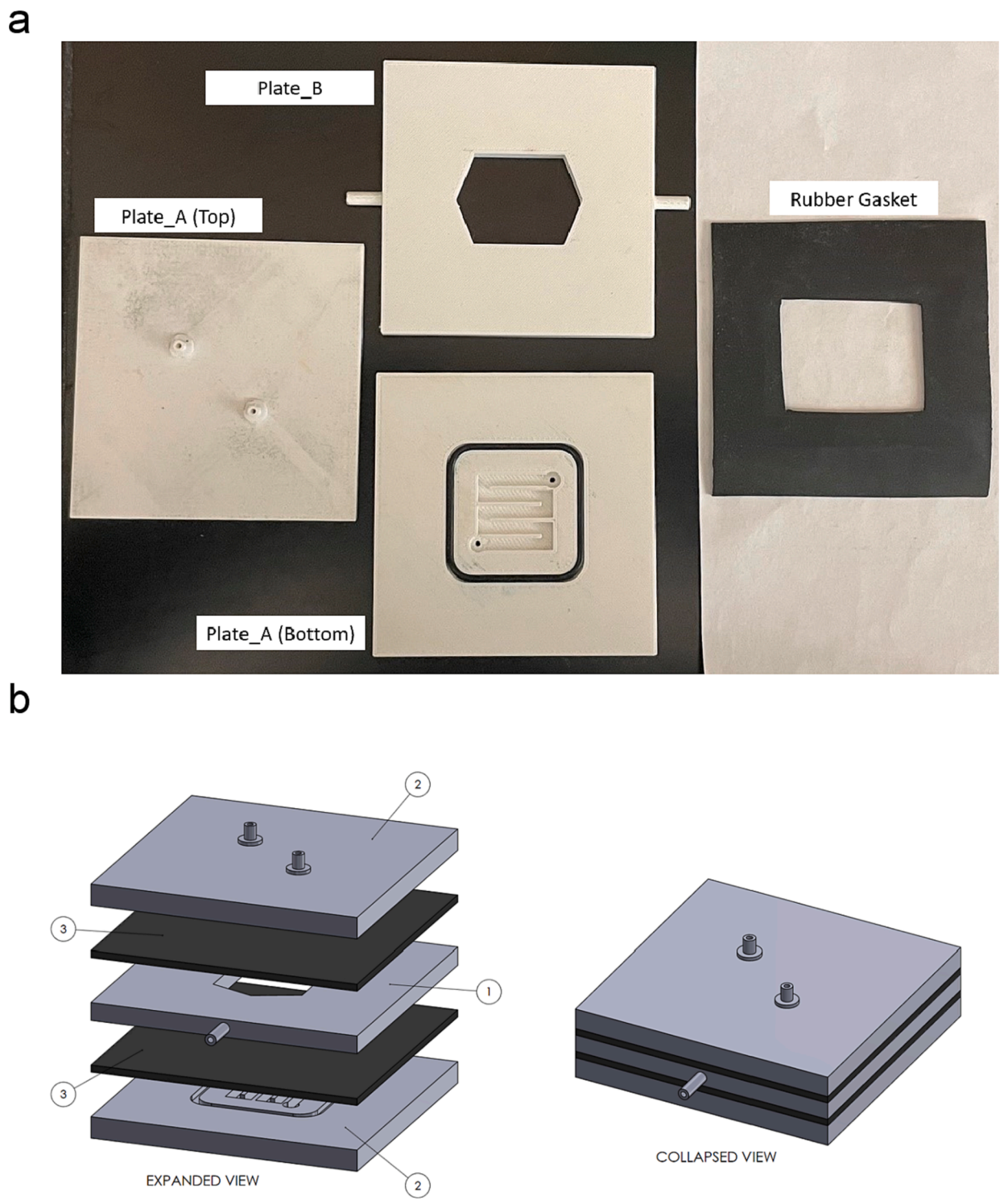


**Fig. 2.** a. Overview of the proposed flow field designs and example of a rubber gasket; b. The basic structure of the 3D printed flow cell. The numbered components are as follows: 1. Plate_B 2. Plate_A 3. Rubber gasket.

for two and three-electrode arrangement set-ups [17]. A solar cell or DC power can directly power a two-electrode system. In contrast, three-electrode systems are typically powered by a potentiostat that can be used for in-depth studies of the electrochemical properties of cathode and anode materials. In the case of three electrode experiments, it is necessary to connect an additional reference electrode to the system. Thus we provide a small opening in Plate_B to analyze electrochemical properties close to the reactive interphase. For

**Table 3**
Commonly used filaments, their cost in US dollars per oz, and their chemical compatibility with commonly used chemicals are presented below. A rating of "A" indicates the polymer resists the chemical very well with little impact, while a "D" indicates poor performance, up to and including the complete destruction of the filament. For more information regarding the rating system, visit PRUSA Polymers' "Chemical Resistance of 3D Materials" [28].

| | PLA | PVB | PETG | ASA | ABS | PC | PA | PP |
|---|---|---|---|---|---|---|---|---|
| Price ($/oz) | 0.57 | 0.96 | 0.62 | 0.74 | 0.62 | 0.85 | 0.96 | 1.02 |
| Water ($H_2O$) | A | A | A | A | B | A | A | A |
| IPA 75% (Isopropyl Alcohol, $C_3H_8O$) | C | D | A | B | B | B | A | A |
| IPA 99% (Isopropyl Alcohol, $C_3H_8O$) | C | D | B | B | B | B | A | A |
| Acetic acid 8% (vinegar, $C_2H_4O_2$) | B | C | A | A | B | A | C | A |
| Sodium chloride 10% (salt, NaCl) | B | B | A | B | A | A | A | A |
| Citric acid ($C_6H_8O_7$) | B | B | A | A | – | A | B | A |
| Hydrochloric acid 37% (HCl) | C | – | A | C | C | A | D | A |
| Hydrogen Peroxide 30% ($H_2O_2$) | B | – | A | A | A | A | D | A |
| Phosphoric acid 85% ($H_3PO_4$) | C | – | C | – | A | A | D | A |
| Nitric acid 69% ($HNO_3$) | D | – | D | – | A | D | D | A |
| Sulphuric acid 96% ($H_2SO_4$) | D | – | D | – | – | D | D | A |
| Fridex (Ethylene glycol, $C_2H_6O_2$) | B | B | A | A | – | A | – | – |
| Savo 1:10 (NaClO) | B | B | A | A | – | B | A | A |
| Ethanol ($C_2H_6O$) | C | D | B | B | A | B | A | A |
| Acetone ($C_3H_6O$) | D | D | C | D | D | C | A | A |

more information on choosing the optimal reference electrode, we recommend the article "Judicious selection, validation, and use of reference electrodes for in situ and operando electrocatalysis studies" by Alnoush et al. [25].

**External sealing**. Last, we propose a strategy to ensure proper sealing by external clamps instead of the typical threaded connectors, again in response to the need to minimize complex, high-resolution elements and allow for successful printing of the reactor design even by low-cost 3D printers.

For applications that include a fragile anion exchange membrane, we recommend using screws instead of clamps as these will impart less strain on the membrane than the shifting of plates that occurs when using clamps. In such cases, Plate_F should be used in place of Plate_A and Plate_E in place of Plate_B. Plates E and F have holes along the plate perimeter that are designed to be put together via screws (in this case, users will need to purchase the screws detailed in the Bill of Materials instead of the clamps).

**Current collectors**. In addition to the elements discussed above, some high current density applications might require the inclusion of metal foil elements (similar in shape to rubber gaskets) as current collectors. The procedure of placing these elements is described in detail in the supplementary videos attached to a recent report on the operation of gas diffusion-electrode-based reactors for $CO_2$ electroreduction [26].

**Ohmic losses.** Minimizing Ohmic losses is critical to ensuring the energy efficiency of electrochemical reactions. Therefore, the proposed plates are as thin as possible, thus significantly reducing the distance between the cathode and the anode.

**Filament choice**. The filament is critical to the safe operation of the reactor and should be determined based on the goals of each experiment, most importantly, their chemical compatibility and melting point. For most reactions, we recommend PETG; it has a large range of commonly used chemicals that are safe to use with the printing material, a printing temperature between 210 and 230 °C, is hydrophobic, and does not deform easily [27]. However, PETG is unsuitable for all applications and each laboratory must determine the best filament for their application needs. Standard filaments and their chemical compatibilities are provided by PRUSA Polymers and depicted in Table 3 [28]. The impact resistance of each filament is beyond the scope of this paper, but interested readers can read the following papers for further information [29,30].

**Table 4**
Recommended printing parameters for each plate (Plate_A–F).

| *Printing Parameter* | *Value/Unit* |
|---|---|
| Material | 1.75 mm PETG filament |
| Printing Temperature | 245 °C |
| Layer Height | 0.1 mm |
| Shell Width | 0.8 mm |
| Infill Percentage | 15 % |
| Infill Speed | 70 mm/s |

Utilizing the recommended PETG filament and all necessary equipment to test for a proper seal provided in the Bill of Materials (including pumps, mass flow controllers, mass flow meters, catholyte, and anolyte bottles), the entire experimental set-up (with additional screw components) costs approximately $3867.97. In addition to customization and reduced cost, these 3D printable models can bypass the shipping process, and the flow cell will be ready to use in a comparably shorter amount of time. Printing the three primary plates provided in this paper using PETG filament takes approximately 9 hours, as opposed to the weeks it can take to receive commercially available equipment.

**Scope of the hardware.** Diverse electrochemical experiments listed in Table 2 require using potentiostats and analytical chemistry tools, such as gas chromatography, high-performance liquid chromatography, or mass spectrometry. Given the variety of the potential uses of the flow reactors, we do not aim to cover the connection and use of this equipment. Instead, we focus on the capability of the flow reactors to provide specific gas and liquid flows to subsequently allow for performing more complex experiments.

## Design files

| Design file name | File type | Open source license | Location of the file |
|---|---|---|---|
| *Plate_A.stl* | *STL* | *Creative Commons Attribution 4.0 International* | *https://doi.org/10.5281/zenodo.8435816* |
| *Plate_B.stl* | *STL* | *Creative Commons Attribution 4.0 International* | *https://doi.org/10.5281/zenodo.8435816* |
| *Plate_C.stl* | *STL* | *Creative Commons Attribution 4.0 International* | *https://doi.org/10.5281/zenodo.8435816* |
| *Plate_D.stl* | *STL* | *Creative Commons Attribution 4.0 International* | *https://doi.org/10.5281/zenodo.8435816* |
| *Plate_E.stl* | *STL* | *Creative Commons Attribution 4.0 International* | *https://doi.org/10.5281/zenodo.8435816* |
| *Plate_F.stl* | *STL* | *Creative Commons Attribution 4.0 International* | *https://doi.org/10.5281/zenodo.8435816* |

*Plate_A.stl*

- This file contains the 3D-printable model of a square plate with an open serpentine flow channel. The serpentine channel has a hole on either end to enable fluid flow with a diameter of 3.0 mm.

*Plate_B.stl*

- This file contains the 3D-printable model of a square plate with a hollow, hexagonal center. The hexagon has holes on opposing sides to enable fluid introduction and flow. This plate contains a small opening near the fluid inlet for a wire-based reference electrode with a diameter of 0.5 mm.

*Plate_C.stl*

- This file contains a flat, square plate with no components, which serves as a support for the experiments where only one or two flow fields are needed.

Plate_D.stl

- This file contains the 3D-printable model of a square plate with a hollow, hexagonal center. The hexagon has holes on opposing sides to enable fluid introduction and flow. This plate does not contain the reference electrode component from Plate_B.

Plate_E.stl

- This file contains the 3D-printable model of a square plate with a hollow, hexagonal center. The hexagon has holes on opposing sides to enable fluid introduction and flow. This plate does not contain the reference electrode component from Plate_B. This plate has eight holes along its perimeter for screw-based clamping.

Plate_F.stl

- This file contains the 3D-printable model of a square plate with an open serpentine flow channel. The serpentine channel has a hole on either end to enable fluid flow with a diameter of 3.0 mm. This plate has eight holes along its perimeter for screw-based clamping.

All plates detailed above are modeled to be 75 mm x 75 mm x 6 mm, with the inlet tubes of the hexagonal plates (Plate_B, Plate_D, and Plate_E) increasing the length to 95 mm.

**Bill of materials summary**

| Designator | Component | Number | Cost per unit -currency | Total cost - currency | Source of materials | Material type |
|---|---|---|---|---|---|---|
| Plate_A.stl | *Plate A, estimated cost to print with PETG* | *2* | *$1.64 /plate* | *$3.28* | https://tinyurl.com/33w59j8f | Polymer |
| Plate_B.stl | *Plate B, estimated cost to print with PETG* | *1* | *$1.48 /plate* | *$1.48* | https://tinyurl.com/33w59j8f | Polymer |
| Plate_C.stl | *Plate C, estimated cost to print with PETG* | *1* | *$1.51 /plate* | *$1.51* | https://tinyurl.com/33w59j8f | Polymer |
| Silicone Tubing, Pump | *Tubing + Pump* | 1 | $56.30 /pump | $56.30 | https://tinyurl.com/2yu92c69 | Polymer |
| Bottle 1, Bottle 2 | *Bottle + Cap + Tubing* | 2 | $166.50 /bottle | $330.00 | https://www.sigmaaldrich.com/US/en/product/sigma/cls11665 | Polymer |
| Gasket (1, 2, 3, 4) | *Rubber Sheeting for Gaskets* | 1 | $11.96 /sheet | $11.96 | https://tinyurl.com/2jwby8bw | Polymer |
| O-Ring | *O-Ring, 38mmx42mmx2mm* | 2 | $0.84/ring | $1.68 | https://www.amazon.com/gp/product/B07GJG1CB7/ref=ppx_yo_dt_b_asin_title_o02_s00?ie=UTF8&psc=1 | Polymer |
| Clamp | *Clamp* | 4 | $5.50 /clamp | $21.99 | https://tinyurl.com/5n89ta3b | Polymer |
| Screw | *M3 x 40 mm screw with nut* | 8 | $0.24 /screw and bolt | $1.87 | https://tinyurl.com/5n73fyrw | Metal |
| Washers | *¼" stainless steel flat washer* | 8 | $0.06 /washer | $0.49 | https://tinyurl.com/3uvmwx5k | Metal |
| Copper Tape | *Copper Tape* | 1 | $10.99 /roll | $10.99 | https://tinyurl.com/3pbf9rww | Metal |
| PVC Tubing | *PVC Tubing, 1/16" ID, 1/8" OD* | 1 | $17.34 /tubing | $17.34 | https://www.amazon.com/gp/product/B07K7RRW93/ref=ppx_yo_dt_b_asin_title_o05_s00?ie=UTF8&psc=1 | Polymer |
| Flowmeter | *Alicat Flowmeter* | 1 | $720.00 /flow meter | $720.00 | https://www.instrumart.com/products/48580/alicat-scientific-p-series-pressure-gauge | Non-specific |
| Mass Flow Controller | *Alicat Mass Flow Controller* | 1 | $1,778.9/Mass Flow Controller | $1,778.96 | https://www.fishersci.com/shop/products/mass-flow-controller-0-200sccm/NC2112095#?keyword=alicat%20mass%20flow%20controller | Non-specific |
| Rotameter | *Rotameter* | 1 | $30.59 /rotameter | $30.59 | https://tinyurl.com/4urnfxuk | Non-specific |

The cost of each plate was estimated using the free software ideaMaker.4.3.2. To calculate estimated costs, open the desired plate file in ideaMaker and complete the following steps:

1. Click on Printer in the upper left-hand corner.
2. Under Filament Settings, insert the desired filament (in this case PETG) and adjust the cost of filament per kg (~$0.059/kg for a 2.2 lbs coil of PETG on Amazon.com), and click Save.
   a. It is also possible to adjust potential printing parameters in this window for more accurate printing time estimations.
3. Under Slice, select Start Slice, double-check your template, and click Slice to receive a cost and time estimate for printing.

## Build instructions

### *Printing*

SAFETY HAZARDS – Check your selected filament's compatibility before running any experiments to avoid unwanted reactions and spillage.

The parameters recommended for printing plates A–F with PETG filament are provided in Table 4 below. The estimated time needed to print Plates A, B, and C with PETG is approximately 9 h and consumes ~ 50 g of filament.

If printing directly onto glass to create a photoreactor, the parameters listed in Table 4 may need to be adjusted to account for less filament adhesion to the printing tray.

### *Flow Cell Assembly*

This procedure should be performed for the specific reactor arrangement chosen for the electrochemical experiment. We provide an assembly procedure for the reactor arrangement depicted in Fig. 2.b for illustrative purposes.

1. Print two copies of ***Plate_A,*** and one of ***Plate_B***
2. Outline ***Plate_A*** with a marker on the rubber sheet for gaskets. Cut along the marker outline to create a gasket. Repeat this step two more times for a total of 3 gaskets. *Note*: if you want to use this flow cell for gas-based experiments, print two copies of

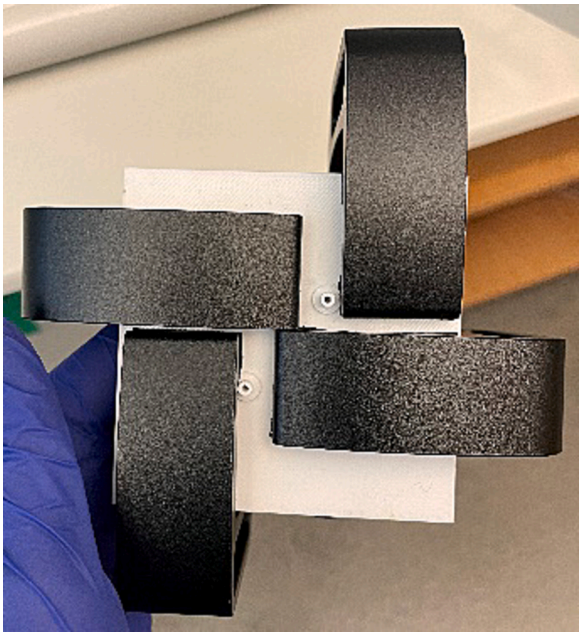

**Fig. 3.** Orientation of clamps for assembling the recommended flow cell for gas phase reactions.

***Plate_C*** and cut out two more gaskets **(Gasket 4, Gasket 5).** These two gaskets will not need to be cut in the middle, instead acting as a seal for leakage tests.

a. Before creating your gaskets, remember that the thickness of your gasket's material should equal the thickness of your catalyst.

3. Align one gasket with ***Plate_B*** and use a precision knife to cut the hexagonal center out of your gasket. To ensure the plate does not shift during this process, firmly clasp the gasket to the plate using binder clips. *Note*: It is better to have a small boarder inside the hexagonal center than to over-cut around the edges.
4. Measure the dimensions of the inner square of ***Plate_A*** and cut an identical square into the same location on two gaskets (***Gasket 1, Gasket 3***).
5. Attach your anode to ***Gasket 1*** using ***Copper Tape.*** Using another piece of copper tape, attach this piece of tape to the inner square of copper tape on ***Gasket 1***, near the middle of the square's edge is preferable, and create a long copper tape tail outside of the gasket**.** Fold the copper tape tail onto itself so the adhesive portions stick together. This serves as a point of contact for aligator clips in electrochemical reactions.
6. Repeat step 5 using ***Gasket 3***.
7. Attach a porous membrane over the hexagonal hole in ***Gasket 2*** with copper tape.
8. Attach an **O-Ring** to the hollow square outline on both copies of ***Plate_A*** (two o-rings total).
9. Layer the printed plates and gaskets from bottom to top in the following order to create the flow cell. The final results should be similar to those depicted in Fig. 2b in Hardware Description.
   a. ***Plate_A***
   b. ***Gasket 1***
   c. ***Gasket 2***
   d. ***Plate_B***
   e. ***Gasket 3***
   f. ***Plate_A***
10. Use the four **Clamps** (avoiding the tubing fittings on the outside of the top and bottom of the flow cell) to seal the system; aim to apply equal, firm pressure without damaging the plates. It is best to orient the clamps near the middle of the plate as shown in Fig. 3 below for a gas tight seal along the o-rings, but placing the clamps closer to the perimeter is viable for strictly liquid components (Fig. 4, Fig. 7) and may make tube and alligator clip attachment easier. *Note*: If using screw-based compression, insert and tighten the screws now and do not use any clamps.

## Operation instructions

Before conducting any experiments, perform the necessary leak tests provided below.

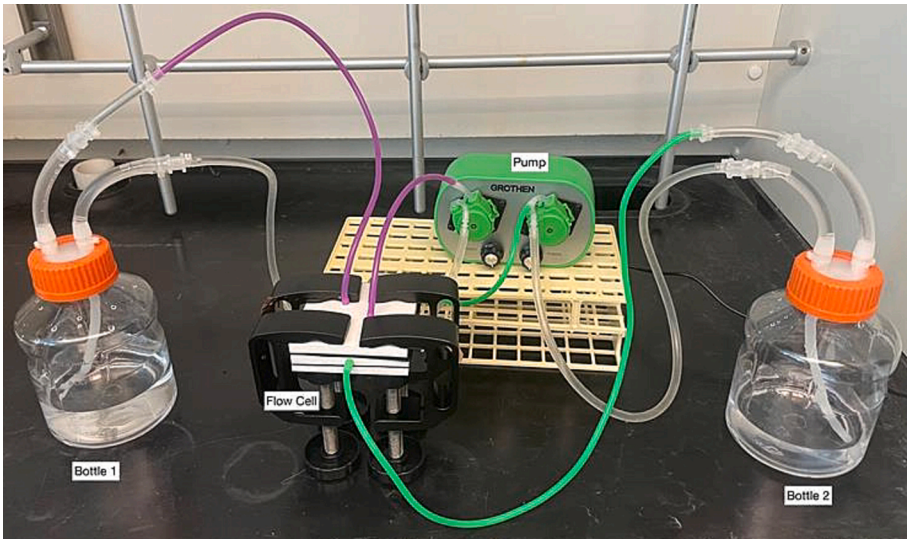


**Fig. 4.** Setup to check for liquid leaks in the flow cell. The clamps are situated differently than shown in Fig. 3. to more clearly show inlet/outlet streams.

*Procedure for Checking for Liquid Leaks*

SAFETY HAZARDS – This procedure uses water near electrical instruments. Be cautious of spills, keep your benchtop orderly, and follow general laboratory safety procedures. Solutions of high concentration and pH have the potential to erode and weaken filament – we again encourage users to select the proper filament for their experiment and instill safety measures in case of broken plates.

1. Fill ***Bottle 1*** and ***Bottle 2*** with ~ 250 ml of water each (Fig. 4).
2. Create a loop on the flow cell by connecting the two tube fittings on one ***Plate_A*** with a small ***Silicone Tube***.
3. Assemble the flow cell as described in steps 9 and 10 of **Flow Cell Assembly**. Put the ***Plate_A*** with a small tube on the bottom of the flow cell.
4. Attach one ***Silicone Tube*** to either side of ***Plate_B*** and connect that same tube directly to the ***Pump***. Attach a different ***Silicone Tube*** to the remaining pump fixture and connect that new tube's remaining end to ***Bottle 1***. Ensure the connected tube inside ***Bottle 1*** is submerged in water. This serves as the source of water for ***Plate_B.***
5. Attach a different ***Silicone Tube*** to the remaining side of ***Plate_B,*** then connect the same tube to the remaining ***Bottle 1*** tube that is not submerged in water. This acts as an outlet for the water pumped into ***Plate_B.***
6. Turn on the ***Pump*** and set it to the lowest possible flow rate (stop when the ***Pump*** "clicks" on). When water from ***Bottle 1*** has passed through ***Plate_B*** it will reenter ***Bottle 1*** and drip into the supply of water at the bottom of the bottle.
7. Allow the ***Pump*** to run for ~ 5 min. If any water leaks from the system during this time, note where the leakage occurs, turn off the ***Pump***, and consult "Troubleshooting Leaks" below**.** Regardless, after the 5 min is over, turn off the pump.
   a. Troubleshooting Leaks:
      i. The most common fix for leakage is to reassemble the flow cell and ensure the gaskets are properly aligned to form a tight seal on the plates.
      ii. Check if your plates have any signs of damage or larger gaps between layers at the locations of leakage. If this is the case, reprint the necessary plate(s).
      iii. Tighten the clamps on the plate or relocate them as needed.
      iv. Double check you have an O-Ring attached to each ***Plate_A.***
   b. Repeat steps 2–7 if there is a leak. If no leakage occurs, proceed to step 8.
8. Remove the short tube on the top ***Plate_A***. Connect that top plate to the ***Pump*** and ***Bottle 2*** in the same way described in steps 4–5.
9. When the ***Plate_B*** is connected to ***Bottle*** **1** and the top ***Plate_A*** is connected to ***Bottle*** **2**, repeat steps 6–7. An image of this configuration is shown in Fig. 4.
10. Repeat steps 8–9 but this time using the bottom ***Plate_A*** in place of the top one so all three plates are checked for leaks**.**
11. When all three plates are leak free the flow cell is ready for use in liquid applications.

*Procedure for Checking for Gaseous Leaks*

SAFETY HAZARDS – Perform all gas-based experiments in a fume hood and test with a non-flammable gas (e.g., air).

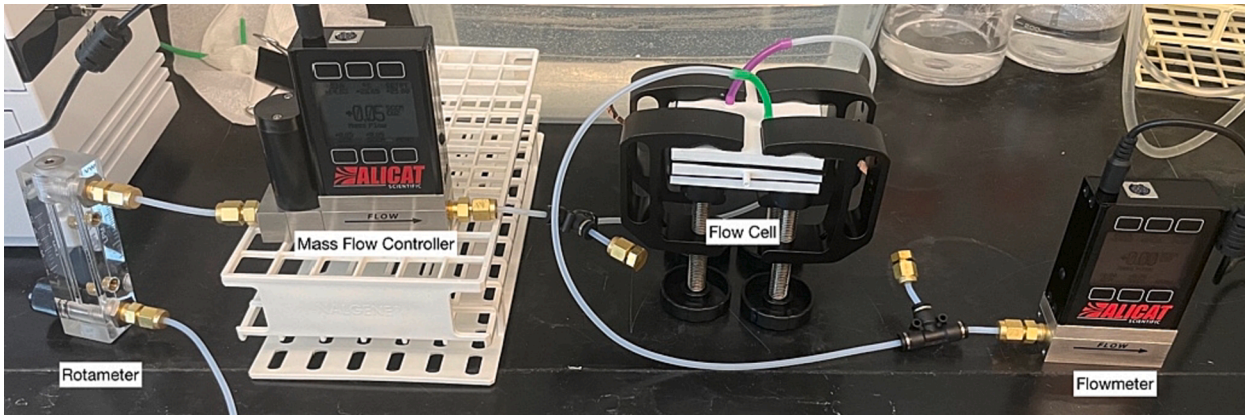


**Fig. 5.** Setup to check for gaseous leaks in the flow cell. The clamps are situated differently than shown in Fig. 3. to more clearly show inlet/outlet streams.

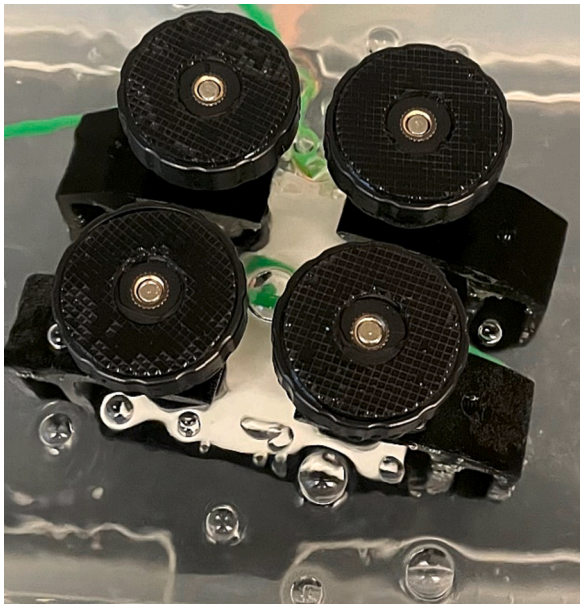

**Fig. 6.** Bubbles emerging from a submerged flow cell due to gas leakage.

1. Calibrate the ***Mass Flow Controller*** and ***Flowmeter*** (Fig. 5) by choosing the gas selected for the procedure from the calibration catalogue. If your device does not incorporate a calibration catalogue, use a bubble flow meter for calibration.
2. Connect the reactor plates as necessary for your experiment. For illustration purposes, this procedure uses the following reactor setup:
   a. ***Plate_A***
   b. ***Gasket 4***
   c. ***Plate_C***
3. Attach one small (~0.5in) piece of silicone tubing to the top of each tube-fitting on ***Plate_A*** (2 pieces total).
4. Connect a PVC tube to each small piece of silicone tubing on ***Plate_A*** (2 pieces total).
5. Connect one end of the PVC tube to the ***Mass Flow Controller*** and connect the other PVC tube to the ***Flowmeter.***.
6. Connect a known gas to the ***Rotameter*** and reduce output to 0.1GPM. Using a PVC tube, connect the outlet of the ***Rotameter*** to the inlet of the ***Mass Flow Control***. This setup is shown in Fig. 5.
7. Adjust the setpoint of the ***Mass Flow Controller*** to 25sccm.
8. Check the reading of the ***Flowmeter***. If it is equivalent to the setpoint of the ***Mass Flow Controller*** (25sccm), ***Plate_A*** is leak free and ready for use in gaseous applications. If the reading rate is not equivalent, refer to "Troubleshooting Leaks" below:

Troubleshooting leaks:

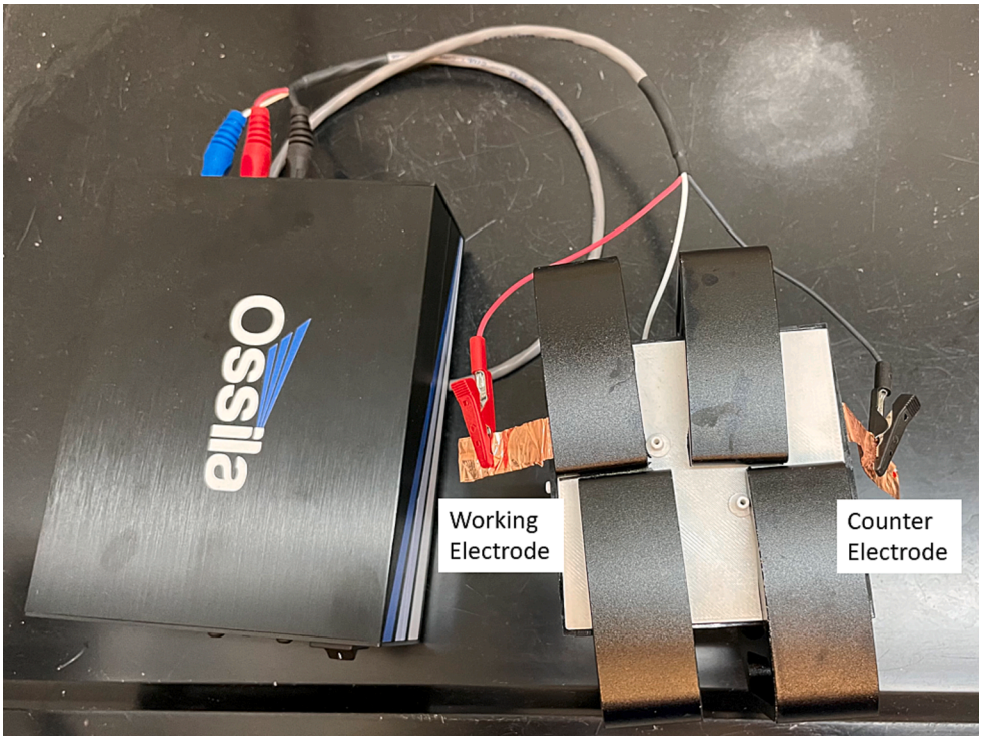


**Fig. 7.** Connection of the reactor to the potentiostat. The clamps are situated differently than shown in Fig. 3. to more clearly show alligator clip connections.

a. To check the potential location of your gas leak, fully submerge the flow cell in water and continue to pump air into the system. Bubbles will indicate a source of lost gas (see Fig. 6).
b. Check each connection in your system for gas leaks using Snoop or an equivalent. If bubbles appear on any connection point after the application of Snoop, there is a gas leak at that location.
c. Ensure your gaskets are properly aligned, and the clamps fully tightened. Consider changing the orientation of your clamps.
d. Check your plates for any signs of damage or space between each layer of filament – gas could be escaping through the plate itself.

9. Disassemble your flow cell.
10. Repeat steps 2–9 with a different ***Plate_A***.
11. Layer the printed plates and gaskets from bottom to top in the following order to create the flow cell:

a. ***Plate_A***
b. ***Gasket 1***
c. ***Plate_B***
d. ***Gasket 4***
e. ***Plate_C***

12. Form a closed loop by attaching a ***Silicone Tube*** to each end of ***Plate_B***.
13. Repeat steps 3–8. If your ***Flowmeter*** and ***Mass Flow Controller*** have the same readings, then both of your ***Plate_A***s and your ***Plate_B*** are gas tight and ready to construct the flow cell depicted in **Flow Cell Assembly**.
14. Check for gas leaks by closing off two plates with ***Silicone Tubes*** and running gas through the ***Mass Flow Controller,*** flow cell, ***Flowmeter*** set up. If the ***Mass Flow Controller*** and ***Flowmeter*** are equivalent, the flow cell is gas tight and ready for use in gaseous experiments.

*Procedure for Electrochemical Experiments*

SAFETY HAZARDS – Perform all gas-based experiments in a fume hood and test with a non-flammable gas (e.g., air). Apply secondary containers to hold potential liquid leakage.

For all electrochemical experiments, the procedure of connecting the reactor elements and providing the liquid flow follows the steps described under the leak test sections, except for the fact that the electrical current is supplied through a DC power source or a potentiostat. Therefore, the cathode and the anode need to be connected by copper tape to the electrodes as depicted in Fig. 7. Subsequently, any electrochemical measurement can be performed.

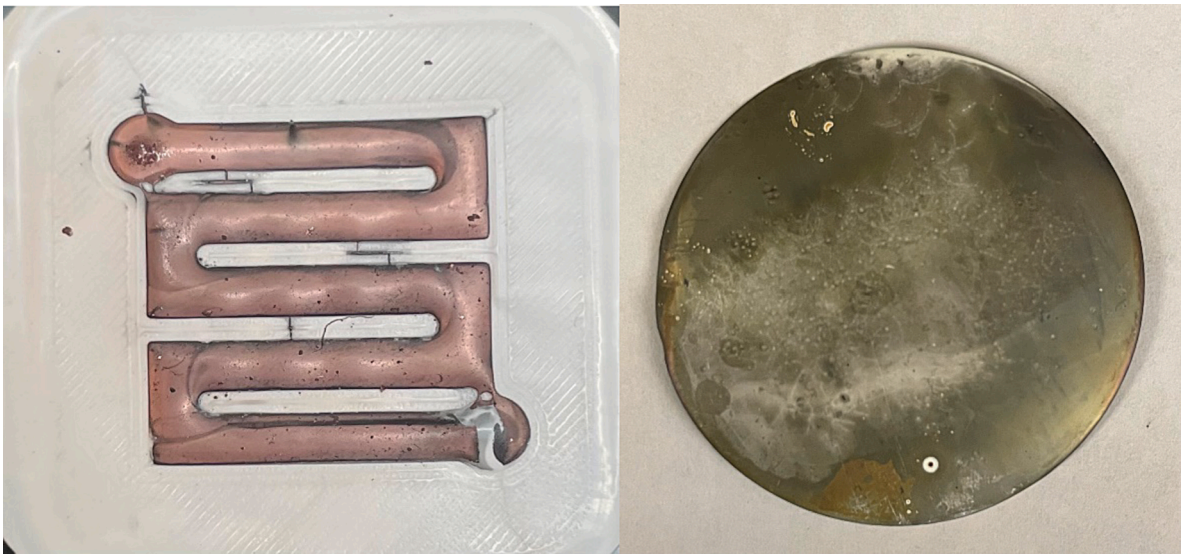

**Fig. 8.** Electroless deposition of copper plating solution using the serpentine pattern on the 3D printed flow cell is shown on the left. On the right is electroless deposition of copper plating solution onto a PES membrane using typical submersion techniques.

## Validation and characterization

Provided that the correct filament and gaskets are used, all applications listed in Table 2 can be explored.

*Electroless Deposition of Copper using 3D Printed Flow Cell*

Here we demonstrate the ability to force a specific liquid flow pattern by circulating $SnCl_2$, $PdCl_2$, and a copper plating solution through the reactor consisting of Plate_A and Plate_C. Without the flow reactor, it is naturally not possible to achieve the same controlled contact between the liquid phase and adjacent material. The "Procedure for Checking for Liquid Leaks" above was used as the basis for this experiment, exchanging water from the test procedure with $SnCl_2$, $PdCl_2$, and a copper plating solution [31].

Before beginning an experiment to deposit copper onto a Polyethersulfone (PES) membrane, plates A and C were printed – the goal was to provide only one channel, enabling one flow of an electroless plating solution at a time. To demonstrate the ability to provide a serpentine flow pattern, Plate_A was selected, while Plate_C was chosen to apply a consistent flat pressure to the membrane. With the goal to deposit material on one side of the membrane, a single flow chamber (Plate_A) was needed. Both plates were printed using PETG.

An adhesive, square gasket of similar size to the plates was applied to Plate_C to enhance the seal. Due to the PES's negligible thickness, no other gasket was needed for this system and the membrane was placed in the center of the gasket-side of Plate_C. The final order of the flow cell from bottom to top is as follows:

1. Plate_C
2. Adhesive Gasket
3. PES Membrane
4. Plate_A

Pressure was then applied to the middle of the flow cell via four clamps in the orientation shown in Fig. 3. to ensure the patterned section of Plate_A was firmly pressed to the membrane. The Procedure for Testing for Liquid Leaks was enacted, and the PES membrane was observed to be wet, indicating fluids were passing through the desired channels. The flow cell was disassembled, a new membrane was attached, and then the flow cell was reassembled to begin electroless deposition of copper.

Utilizing the procedure reported by Cao, Wu, Yang, et. al, the following electroless deposition of copper experiment was conducted [32]:

Solution $SnCl_2$ comprised of 50 mmol/L $SnCl_2$ and 30 ml/L HCl, solution $PdCl_2$ of 0.75 g/L $PdCl_2$ and 3 ml/L HCl, and the copper plating solution were prepared. The $SnCl_2$ solution was pumped through the system at approximately 15 ml/s for 10 min. DI water was then pumped through for 1 min as a rinse at the same flow rate. This simple procedure was then repeated with the $PdCl_2$ solution, then the copper plating solution to deposit a serpentine pattern onto a PES membrane seen in Fig. 8. For copper(II) electro-less deposition, the relevant half-cell reaction is [33]:

$$Cu^{2+} + 2e^{-} \rightarrow Cu^{0} \quad E = +0.34V$$

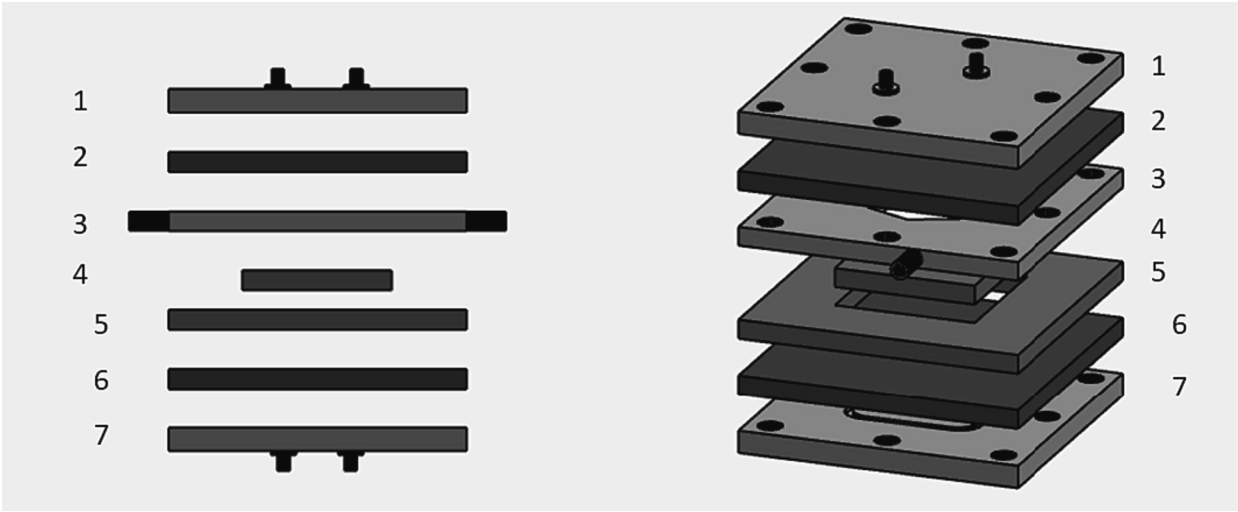


**Fig. 9.** Two diagrams of the 3D printed electrolyzer layout for electrochemical characterization. The numbered components are as follows: 1. Plate_F 2. Rubber gasket + cathode 3. Plate_E 4. Membrane 5. Rubber gasket with a center square removed 6. Rubber gasket + anode 7. Plate_E.

For comparison, a similar procedure was enacted using a submersion technique, where a new PES membrane was submerged in each of the above solutions for equivalent amounts of time. It is clear that using the flow cell for electroless deposition of copper onto the PES membrane results in a controlled surface area being coated.

*Electrochemical Characterization*

To demonstrate the applicability of the 3D printed reactor for electrochemical applications, a combined $CO_2$ capture and hydrogen evolution reaction was performed using a) a 3D printed flow cell and b) the titanium/stainless steel flow cell listed as a commercial analog.

Three inlet and outlet flows are needed for this experiment: two for a KOH electrolyte and one for pure gaseous $CO_2$. The commercially available electrolyzer consists of a catholyte plate (containing an inlet/outlet for one flow of KOH and one of $CO_2$), an anolyte plate (containing an inlet/outlet for KOH), and a flow plate provided by the same company for KOH flow from the catholyte plate.

The following order of the commercial flow cell from top to bottom is as follows:

1. Cathode plate (inlets for both gas and liquid flow)
2. Rubber gasket + cathode held in place with copper tape; a "tag" was made from the copper tape and attached to the copper tape inside the flow cell to stick outside the flow cell for alligator clip attachment; the copper tape holding the cathode in place was face down.
3. Plastic middle flow plate for KOH flow from the catholyte plate
4. Membrane
5. Rubber gasket with a square cut out of its center
6. Rubber gasket + anode held in place with copper tape; a "tag" was made from the copper tape and attached to the copper tape inside the flow cell to stick outside the flow cell for alligator clip attachment; the copper tape holding the anode in place was face down.
7. Anode plate (inlet for only liquid flow)

To mimic the commercial analog, two copies of plate F and one copy of plate E were printed; plates F and E were chosen over plates A and B to reduce friction on the membrane from shifting plates, as discussed previously. The following order of the 3D printed flow cell from top to bottom is as follows:

1. Plate_F
2. Rubber gasket + cathode held in place with copper tape; a "tag" was made from the copper tape and attached to the copper tape inside the flow cell to stick outside the flow cell for alligator clip attachment; the copper tape holding the cathode in place was face down.
3. Plate_E
4. Membrane
5. Rubber gasket with a square cut out of its center

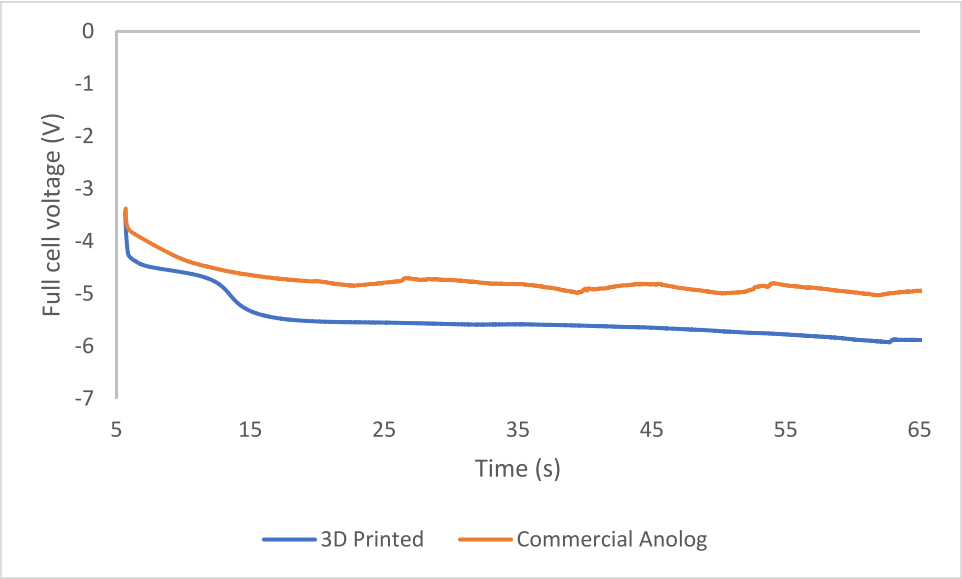


**Fig. 10.** Full cell voltage for the 3D printed electrolyzer (blue, bottom line) and its commercial analog (orange, top line). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

6. Rubber gasket + anode held in place with copper tape; a "tag" was made from the copper tape and attached to the copper tape inside the flow cell to stick outside the flow cell for alligator clip attachment; the copper tape holding the anode in place was face down.
7. Plate_F

A diagram of the 3D-printed electrolyzer's layout is depicted in Fig. 9 below.

The following procedure was conducted using both electrolyzers:

Preparation of the membrane begins a minimum of 12 h in advance, with the submersion of the membrane in 1 M KOH solution. The membrane FAA-3–50 from Fuel Cell was used and a 1 M KOH solution was prepared, using solid potassium hydroxide from Fisher Chemical and DI $H_2O$.

The electrolyzer was assembled as described above, ensuring a tight seal on the system with screw clamps to reduce friction on the membrane. MGL370 carbon paper from Fuel Cell was cut into a 1 in x 1 inch square as the cathode and nickel foam with a thickness of 1 mm from Fuel Cell was similarly cut for the anode.

A check for liquid and gaseous leaks was then conducted following the procedures in **Operation Instructions**. It is important to note that 0.1 M KOH was used for the liquid leak test to ensure the membrane was kept at an optimum pH and that this KOH was not removed from the system during the gaseous leak test to avoid drying out the membrane.

After ensuring the flow cell was leak-free, 0.1 M of KOH solution was pumped through two ports (into the middle and bottom plates) in the electrolyzer using a syringe pump with silicone tubes at 16.875 uL/s (approximately 1 ml/min). No gas was introduced to the system until the outlet tubing of each KOH port began to drip KOH solution into a waste beaker.

Pure, gaseous $CO_2$ was then run through an Allicat Mass Flow Controller to set the inlet flow rate of the gas to 5 SCCM $CO_2$. Alligator clips were attached to each flow cell's aforementioned copper tape tags, and an Autolab Potentiostat and NOVA 2.1.6 software were used for the electrochemical analysis in galvanostat mode. After a 5 second delay, a current of −0.1 A was applied for 60 s. The results of each experiment are shown below in Fig. 10.

Full cell voltage for the 3D printed cell and the commercial analog differ only by ~ 0.81 V. The full cell voltage for the 3D printed cell is more stable than the commercially available analog. Additionally, the gaseous outlet flow for both electrolyzers increased past the inlet flow of 5 SCCM $CO_2$, with the 3D printed electrolyzer's flow rate reading ~ 6.09 SCCM $CO_2$ and its commercial analog reading ~ 6.30 SCCM $CO_2$. The increased outlet gaseous flow rate indicates that hydrogen was produced.

**Capabilities and Limitations:**

- Filament and quality of the 3D print largely determine the hardware's capabilities, particularly regarding gas-based experiments that must be air-tight. Alternatively, CNC machining could be used if sufficient print quality cannot be obtained. Resin-based printing could benefit users interested in intricate detailing or smaller components, and flexible printing filaments could be used to print specialized gaskets.
- The sealing of the reactor is critical to safe and accurate electrochemical measurements. Be sure to perform leak tests before starting any experiment.

- The choice of equipment supporting the reactor, such as gas flow meters, pumps, etc., are critical, and necessary flowrates and the accuracy of the measurement needs to be reviewed based on the goal of the electrochemical experiment.
- Ohmic loss, flow rate maximums, and electrochemical performance will vary significantly between each experimental setup. We strongly encourage users to share their printing procedures and customized plate designs to ensure the reproducibility of experiments.

## CRediT authorship contribution statement

**Erin Heeschen:** Writing – original draft, Validation, Formal analysis, Investigation, Data curation. **Elena DeLucia:** Methodology. **Yilmaz Arin Manav:** Methodology, Design, Fabrication. **Daisy Roberts:** Visualization. **Benyamin Davaji:** Supervision, Resources. **Magda Barecka:** Supervision, Resources, Writing – review & editing, Project administration.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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