How to Make Cathodes with a Diffusion Layer for Single-Chamber Microbial Fuel Cells

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Introduction

The use of a cathode with a diffusion layer in a single-chamber microbial fuel cell (MFC) can significantly improve the performance of the MFC by increasing its coulombic efficiency and maximum power density while simultaneously decreasing its loss of water. The coulombic efficiency is increased by the increased power output and by the increased operation time of the MFC. The operation time is likely increased due a reduction in the amount of oxygen flow to the anode chamber where bacteria preferentially use oxygen directly as an electron acceptor instead of using oxygen indirectly via the anode of the MFC. Oxygen flow, however, is still necessary for the catalyst-side, which is opposite the air-exposed side, of the cathode. Thus, an optimum thickness for the diffusion layer exists and is the subject of a paper by Cheng et al (2006) The instructions below describe how to make 12 cm^2 circular cathodes with a polytetrafluoroethylene (PTFE) diffusion layer of optimum thickness. This process takes several hours due to a number of steps that require wait time.

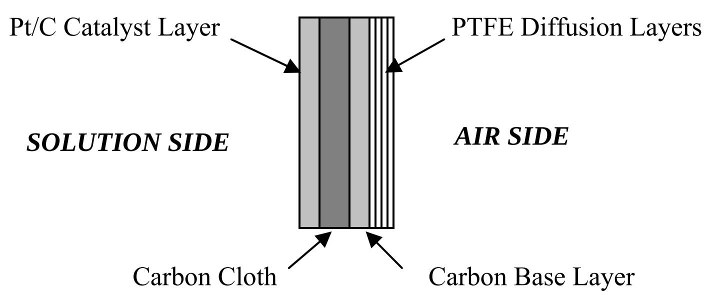

***Figure 1*:** Exaggerated side-view of a cathode with a four-coating PTFE diffusion layer

Materials

- Carbon Cloth with 30% by Weight PTFE Wet-Proofing (E-TEK B-1/B/30WP)
- Carbon Black Powder (Cabot Vulcan XC-72)
- 40% by Weight PTFE Solution (diluted from 60% solution)
- 60% by Weight PTFE Solution (Aldrich PTFE 60 wt. % dispersion in water)
- 10% by Weight Platinum on Carbon Powder (ETEK C1-10 10% Pt on Vulcan XC-72)
- 5% by Weight Nafion® Solution (Aldrich Nafion® perfluorinated ion-exchange resin)
- Pure Iso-propanol (99%) (Alfa Aesar)
- De-ionized (DI) Water
- Solid glass beads, 3mm in diameter (Propper Manufacturing Co. Inc.)

Equipment

- Metric Ruler

- Sharp Utility Knife
- Electronic Balance (Resolution in milligrams)
- Flat Spatula
- 1000 µL Micropipettor with Several Pipette Tips
- Piece of Cardboard (Approximately 1 ft. × 1 ft.)
- Small Paintbrush (1/4 in. Wide Tip)
- Furnace (Capable of Heating at 370 ºC)
- High-Temperature Fiberglass Gloves
- High-Temperature Ceramic Plate
- 3.8 cm Diameter Punch
- Small Mallet or Hammer
- Wooden Cutting Board
- Plastic sample vial with cap. 8mL,
- Touch mini Vortexer.

Applying the Carbon Base Layer

NOTE: Inhomogeneities and variations in carbon black and PTFE content can lead to variations in performance from cathode to cathode. Thus, it is recommended that care be taken to ensure that as homogeneous a coating as possible is applied with as little as possible losses of the carbon black base coat mixture.

1) Cut a piece of carbon cloth, using the ruler and utility knife, to make 2 cathodes (4 cm × 8 cm).

2) Measure 1.56 mg of carbon black for every 1 cm^2 of cathode surface area using the electronic balance.

EXAMPLE: A two-cathode piece of carbon cloth, having an area of 32 cm^2, would require 1.56 $mg/cm^2 \times 32\ cm^2 = 50$ mg of carbon black.

3) Measure, using the micropipettor, 12 µL of 40% PTFE solution for every 1 mg of carbon black used in the step above.

EXAMPLE: A two-cathode piece of carbon cloth, using 50 mg of carbon black, would require 50 mg × 12 µL/mg = 600 µL of 40% PTFE solution.

4) Put carbon black into a plastic sample vial, add 6-8 glass beads and the PTFE solution, cap the vial, vortex for 20 seconds.

5) Coat all carbon suspension onto the one side of carbon cloth using small paintbrush, gently paint to prevent getting the carbon suspension into another side of carbon cloth.

Figure 2**:** Glass beads(left), paintbrush(center), and plastic vial(right).

6) Allow the coating to air-dry on the piece of cardboard for at least 2 hours. The drying process can be expedited using a hair dryer.

CAUTION: Before using the furnace, note that hot surfaces and radiating heat when operating the furnace can cause serious injury to exposed body parts. Be sure to exercise care and to wear high-temperature gloves when handling this equipment.

7) Place the piece of carbon cloth on a high-temperature ceramic plate in a pre-heated furnace at 370 ºC for about 20-30 minutes.

Figure 3**:** Three two-cathode pieces of carbon cloth, ready to be placed in the furnace.

Figure 4**:** The furnace used to heat the pieces of carbon cloth.

8) Remove the ceramic plate and carbon cloth allowing them to cool to room temperature on high-temperature tiles before handling. Alternatively, these items may be cooled, albeit more

slowly, by turning off the furnace and leaving the furnace door open slightly.

NOTE: Curling of the carbon cloth after it has cooled, as seen in Figure 4, is normal and becomes more pronounced as PTFE diffusion layers are added.

Figure 5**:** two-cathode piece of carbon cloth that curled after cooling from 370 ºC to room temperature.

Applying the Diffusion Layer

1) Shake the 60% PTFE solution well since this solution is actually a suspension of PTFE particles that needs to be well mixed in order to ensure evenly distributed coatings.

2) Apply one coat of 60% PTFE solution in a uniform fashion to the previously coated side of the carbon cloth using the small paintbrush.

3) Smooth out any bubbles and remove any chunks of PTFE using the paintbrush.

4) Allow the PTFE coating to air-dry for at least 5-10 minutes. The coating should turn white when dry.

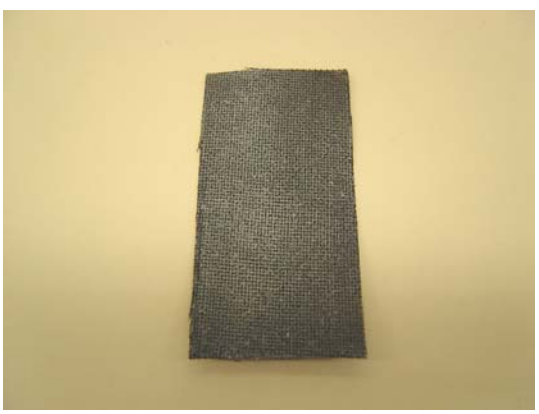

Figure 6**:** two-cathode pieces of carbon cloth with white PTFE layer after air-drying.

Figure 7**:** two-cathode pieces of carbon cloth with black PTFE layer after heat treating in furnace.

5) Place the piece of carbon cloth on a high-temperature ceramic plate in a pre-heated furnace at 370 ºC for about 10-15 minutes.

6) Remove the ceramic plate and carbon cloth allowing them to cool to room temperature on high-temperature tiles before handling. The PTFE coating should turn glossy black.

7) Repeat steps 1 through 5 above three more times to add and heat a total of 4 PTFE coatings. This number of coatings provides the optimum thickness for the diffusion layer.

8) Punch out the circular cathodes using the small hammer to hit the metal punch through the carbon cloth on top of a wooden cutting board. The wooden board preserves the sharp edge of punch since impact of the punch with a hard surface dulls this edge, or cut out cathode to right size using a scissors.

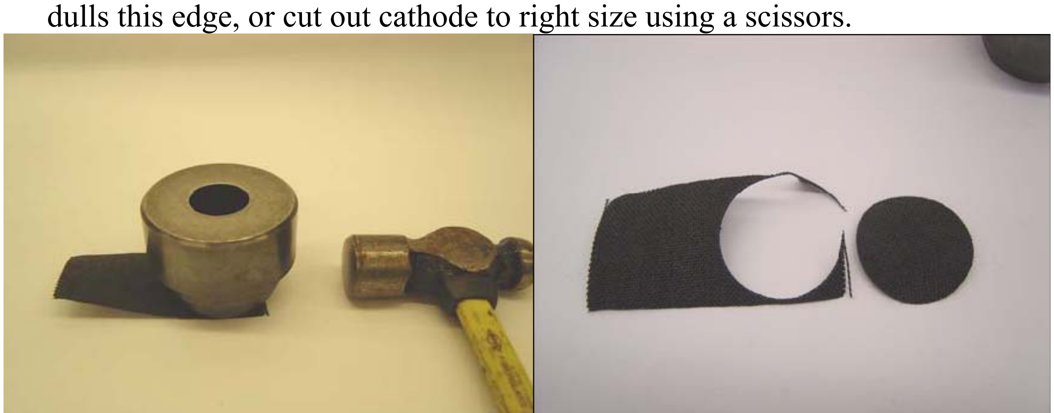

Figure 8**:** A circular 12 cm^2 cathode that has been cut out of a two-cathode piece of carbon cloth.

Applying the Catalyst Layer

1) Measure the amount of 10% Pt/C corresponding to 0.5 mg of Pt for every 1 cm^2 of cathode surface area using the electronic balance.

   EXAMPLE: A single cathode piece of carbon cloth, having an area of 12 cm^2, would require 0.5 $mg/cm^2 \times 12\ cm^2 = 6$ mg of Pt catalyst. For 10% Pt/C powder, 6/0.10 = 60 mg of 10% Pt/C would be required.

2) Put the required Pt/C into the plastic sample vial, Add about 0.83 µL of DI water for every 1 mg of Pt/C in a dropwise fashion into the vial, add 6-8 glass beads, cap vial, and then vortex for couple of seconds. This "water treats" the Platinum catalyst.

   EXAMPLE: A single piece of carbon cloth, using 60 mg of Pt/C powder, would require 60 mg × 0.83 µL/mg = 50 µL of DI water.
   WARNING: Insufficient amounts of water or insufficient mixing may cause the Pt catalyst to react with the alcohol present in the Nafion® solution resulting in an excessive heat release and possibly even fire.

3) Add 6.67 µL of Nafion® solution and 3.33 µL of pure iso-propanol for every 1 mg of Pt/C into the vial using the micropipettor, vortex for 20 seconds.

   EXAMPLE: A single piece of carbon cloth, using 60 mg of Pt/C powder, would require 60 mg × 6.67 µL/mg = 400 µL of 5% Nafion solution, and 60 mg × 3.33 µL/mg = 200 µL of pure iso-propanol.

4) Coat the catalyst paste-like mixture on the side opposite the diffusion layer coatings maintaining as even and homogenous a consistency as possible using paintbrush. Use the piece of cardboard to prevent getting the catalyst coating on surfaces such as tabletops.

5) Allow the coating to air-dry for at least 24 hours. The drying process can be expedited using a hair dryer set to LOW heat.

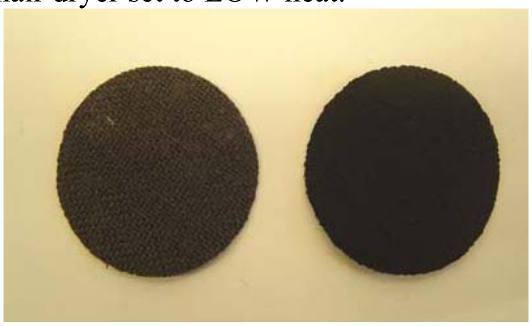

***Figure 9*:** A circular cathode before catalyst coating (left) and after coating (right).

   CAUTION: Using a hair dryer at HIGH heat settings can damage the Pt catalyst and the Nafion® binder.

Troubleshooting

The carbon base layer came through the carbon cloth to leave blots on carbon/PTFE on the other side.

This happens occasionally, especially for those making cathodes for the first time. This is a problem because the presence of electrically insulating PTFE on the electroactive side of the cathode can hinder the conductivity of the cathode. To remedy this problem, the blots of carbon/PTFE that have bled through the carbon cloth can be scraped away using the spatula so as to expose carbon cloth beneath.

The piece of carbon cloth frayed around the edges during the coating process.

Usually this is not an issue since the edges of the piece of carbon cloth are cut away when punching out the circular cathodes. If the fraying is severe, plan the punching out of cathodes around this frayed region accordingly, accepting the fact that the number of cathodes obtained from the piece of carbon cloth may be less than intended.

References

Cheng, S.; Liu, H.; Logan, B. E. (2006). Increased performance of single-chamber microbial fuel cells using an improved cathode structure. *Electrochemistry Communications 8*, 489-494.

Cheng, S., H. Liu and B.E. Logan. (2006). Power densities using different cathode catalysts (Pt and CoTMPP) and polymer binders (Nafion and PTFE) in single chamber microbial fuel cells. *Environ. Sci. Technol.* **40(1):364-369.**