H₂O Systems
Initial Prototype

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Prototype Design Parameters

Water flow
- Flow Rate
- Residence Time

Electricity
- Breakdown of water
- Anodizing Ti
- Circuit Diagram

Biology
- Bacterial Culture Prep
- Water Sample Prep
- Bacterial Quantification

Prototype
Electrical Behavior

• So far, we’ve shown that our proposed electric field of $1-5 \times 10^5$ V/m is lower than the dielectric strengths of water and air ($100-300 \times 10^5$ and $30 \times 10^5$ V/m respectively)

• Here we describe our efforts in preventing electrolysis of water by insulating the electrodes. Also, we provide a comprehensive electrical description of our device through a circuit model which guides our materials selection for the device.
**Electrolysis of Water**

- Not to confuse with dielectric breakdown potential of water, which pertains to the threshold at which “electron avalanches” occur.
  

- Electrolysis of water involves electron transfer between water molecules and electrically charged metal electrodes.

- Hydrogen and oxygen gas (the bubbles) are produced in the cathode (-) and anode (+) respectively. OH\(^-\) and H\(^+\) are left in the solution.

- **GOAL:** Prevent electrolysis of water

\[
\text{At the Cathode (-):} \quad 2\text{H}_2\text{O} + 2e^- \rightarrow \text{H}_2 + 2\text{OH}^- \\
\text{At the Anode (+):} \quad 2\text{OH}^- \rightarrow \text{H}_2\text{O} + 1/2\text{O}_2 + 2e^- \\
\text{Total Reaction:} \quad \text{H}_2\text{O} \rightarrow \text{H}_2 + 1/2\text{O}_2
\]
The device can be electrically represented by a circuit diagram in which one capacitor is in series with two other in parallel to one another.
This treatment assumes the ideal non-conducting behavior (no electrolysis) and is useful to estimate the electric field and potential in each component, especially in the water.

κ is dielectric constant and ε is permittivity, which are materials properties.

The values for the dielectric constants are:

- TiO$_2$; κ$_1$=14-170 or Polyester κ$_1$=κ$_3$ = 3.2
- H$_2$O; κ$_2$ = 80

Ref. www.matweb.com
Solving the Circuit

The equivalent capacitance for this circuit is:

$$C_{eq} = \frac{C_1(C_2 + C_3)}{C_1 + C_2 + C_3}$$

Q = CV

Capacitance can be expressed as:

$$C_i = \frac{\varepsilon_i A}{d}$$

$$\kappa_i = \frac{\varepsilon_i}{\varepsilon_0}$$, where $$\varepsilon_0 = 8.854 \times 10^{-12} \text{ C}^2/\text{Nm}^2$$ is the permittivity of free space, $$\varepsilon_i$$ is permittivity of material
A is area and d distance
Solving the Circuit

Under these assumptions, the voltage across each component is:

\[ V_1 = \frac{V_{\text{app}}(C_2 + C_3)}{C_1 + C_2 + C_3} \]

\[ V_2 = V_3 = \frac{V_{\text{app}} C_1}{C_1 + C_2 + C_3} \]

The electric field in each component can be obtained through:

\[ V_f - V_i = -\int \vec{E} \cdot d\vec{s} \]

But each capacitor models one material. The voltage drop can be treated as linear

\[ E_i = \frac{V_i}{d_i} \]
Solving the Circuit

Therefore, the electric field across each component can be expressed as:

\[ E_1 = \frac{V_{app} (C_2+C_3)}{(C_1+C_2+C_3)d_1} \]

\[ E_2 = E_3 = \frac{V_{app} C_1}{(C_1+C_2+C_3)d_{2,3}} \]

This enables us to predict the effects of the insulating (electrolysis protection) on the actual electric field on water. As depicted earlier, capacitor 2 (or 3) stands for water. If the capacitance of capacitor 1 is low (as in polymers) the field through water will decrease. More quantitatively…
1 - Surface Area of insulating layer (TiO$_2$ or polyester shimstock): $5 \times 10^{-3} \text{ m}^2$, $d \approx 1 \times 10^{-9} \text{ m}$ for TiO$_2$ or $d = 12.5 \times 10^{-6} \text{ m}$ for shimstock.

2 - Surface area of water layer: $3 \times 10^{-3} \text{ m}^2$, $d = 127 \times 10^{-6} \text{ m}$.

3 - Surface area of shimstock window: $2 \times 10^{-3} \text{ m}^2$, $d = 127 \times 10^{-6} \text{ m}$.

Therefore, the capacitances are:

1.1 - $C_{1,\text{TiO}_2} = 6.2 \times 10^{-6}$ to $7.5 \times 10^{-5} \text{ F}$

and $C_{1,\text{ST}} = 1.13 \times 10^{-8} \text{ F}$

2.1 - $C_{2,\text{H}_2\text{O}} = 2.8 \times 10^{-8} \text{ F}$

3.1 - $C_{3,\text{ST}} = 1.12 \times 10^{-9} \text{ F}$ (Shimstock window)
Expected E-Fields

• Therefore, the expected E-Field across the water (capacitor 2), for the different insulating options under 25V are:
  – Using Shimstock: \( E_2 = 359.23 \text{ V/m} \)
  – Using TiO\(_2\): \( E_2 = 1.9\times10^5 \) to \( 23.8\times10^5 \text{ V/m} \) which falls in our targeted range

• There is a clear advantage in using anodized Ti under these considerations, as it allows for lower voltages in order to obtain the lysing electric field. The changes in electrical field in each capacitor is caused by the distribution on potential between each capacitor, that in series must add to the total voltage. However, this distribution depends on the dielectric constants, which can guide our materials selection.
Insulating Attempts

• As shown above, due to dielectric constraints, anodized Ti makes a better suited insulator to minimize the electrolysis of water.

• Although the ohmeter measured TiO₂ and polyester shimstock resistances cannot be resolved (too high), their calculated resistances differ substantially. Through $R = \rho l/A$, where $\rho$ is resistivity, $l$ length and $A$ cross-sectional area, we obtain:
  - TiO₂ $\rho=10^{11}$ to $10^{16}$ Ωm thus, $R_{\text{TiO}_2} = 2\text{M}\Omega$ to $2\times10^5 \text{M}\Omega$
  - Polyester coating $\rho=10^{13}$ Ωm thus $R = 524.3 \text{ M}\Omega$

• Therefore, anodized Ti provides both high resistance and good dielectric properties. However, we observed poor prevention of electrolysis with anodized Ti when compared to polyester shimstock covered electrodes.
Insulating the Electrodes: Anodized Ti

- Titanium anodization occurs by placing a piece of titanium as the anode in an electrolytic cell. TiO$_2$ is formed on the surface of the sample, which provides corrosion resistance and electrical insulation.

- Anodization took place with 30V for 1h. The current raised from ~0.32A to 0.80A. The solution employed was 0.1M NaOH, with pH~13, as specified by SAE AMS 2488.

Color change observed in early stages of anodization.
Insulating the Electrodes: Anodized Ti

- The resistances of the anodized layers and the titanium were measured on the surface by placing the voltmeter probes on the surface, separated by 1cm

<table>
<thead>
<tr>
<th>Part</th>
<th>Resistance [Ω]</th>
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<tbody>
<tr>
<td>Bare Ti</td>
<td>0.22</td>
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<tr>
<td>Blue layer</td>
<td>50</td>
</tr>
<tr>
<td>Grey region</td>
<td>Not resolved</td>
</tr>
<tr>
<td>Resistivity of TiO$_2$</td>
<td>$10^{13}$-$10^{18}$ Ωcm</td>
</tr>
</tbody>
</table>

Ref. www.matweb.com
Insulating the Electrodes: Anodized Ti

The plot depicts the change in current as voltage was applied across the Ti electrodes, one of them anodized (the cathode in this case). Below we see the set up used.

![Diagram of the setup with an anodized electrode and water flow](image-url)
Insulating the Electrodes: Anodized Ti

• Currently, we observe bubbles when running water through the anodized device, which means electron transfer occurs between water molecules and the electrode plates.
• This can be due to the small films formed, because of voltage limitations
Insulating the Electrodes: Anodized Ti

• Ideally, the sharp increase in current should be observed at voltages higher than our target operating potential of 25V. This way electrolysis of water would be avoided.

• Power supply capable of higher voltages is necessary, in order to assess the feasibility of anodized titanium as means to prevent electrolysis of water.

• Possibility of TiO$_2$ interacting with ions in water?
Insulating the Electrodes: Polyester Shimstock

- We also tested the performance of the shimstock coating the steel prototype electrodes under increasing voltages.
- The shimstock proved to be an efficient barrier for electrolysis at the voltages analysed:
- No bubbles were observed throughout the experiment
Insulating the Electrodes: Summary

• As shown above, anodized Ti has great potential as an insulating layer. However, new attempts to create better films must be performed. The use of polyester shimstock has been proven to be effective, however, higher voltage supplies will be necessary in order to create the target electric field through water.
Biology: Bacterial Culture

Want bacteria to be in exponential growth phase during experiment

For every experimental run:
1. Inoculate overnight culture day before
2. Dilute o/n culture in fresh media morning of run
3. Prepare water sample from culture and run experiment in afternoon

www.qiagen.com
Biology: Water Sample

Target concentration ~ 272 cfu/ml
- Average E. coli concentration in Charles River
- Concentration controlled by dilutions performed the morning of the experiment

**Stainless Steel system:**
- 40 ml total volume (20 ml/syringe)
- prepare 45 ml sample = 9 ml culture + 36 ml water

**Ti system:**
- 30 ml total volume
- prepare 35 ml sample = 7 ml culture + 28 ml water
Biology: Bacterial Quantification

• To be performed before and after each experimental run
• 2 methods of bacterial concentration quantification:
  – LB Amp plates
  – Spectrophotometer
• Decrease in bacterial concentration = indication of cell lysis

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**LB Amp plates**
1. Plate 100µL water sample
   - Perform dilution beforehand if necessary
2. Allow 1 day to grow
3. Count colonies

**Spectrophotometer**
1. Transfer 1ml sample to cuvette
   - Perform dilution beforehand if necessary
2. Measure OD$_{660}$
3. Calculate concentration by Beer’s Law: $A = εcl$
To control flow rate, will apply a constant load » constant pressure
Water Flow: Controlling flow rate

Constant Load

Constant Pressure

Constant Flow Rate

Couette Flow

\[ P = \frac{mg}{Area} = \frac{3\mu LQ}{2Wd^3} \]

Flow Rate vs Applied Mass

\[ y = 3.4586x - 2.9095 \]
\[ R^2 = 0.9593 \]
Water Flow: Setting flow rate

Flow Rate [ml/s] = 3.46(Applied Mass [kg]) – 2.91
Target Flow Rate = 1 liter/hour = 0.278 ml/s

Residence Time = Volume_{water flow region} / Flow Rate
Target Residence Time >> 17 ms

Taking the target flow rate and required residence time into consideration, we conclude that:

a \textbf{\sim 1.5 kg} weight will yield our desired results at a flow rate of \textbf{\sim 2.3 ml/s} and a residence time of \textbf{\sim 134 ms}
Initial Prototype

Testing procedure:
1) Setup system design and attach onto a stand.
2) Prepare water sample for testing.
3) Determine bacterial concentration of pre-treatment water sample.
4) Load water sample (20mL/syringe in 2-syringe SS system; 30mL in 1-syringe Ti system) into syringe(s) and remove air bubbles.
5) Clamp syringes onto stand to stabilize.
6) Set voltage at 25 V.
7) Apply load onto syringe(s) and allow entire volume of sample to run through system.
8) Determine bacterial concentration of post-treatment water sample.
# Gantt Chart

<table>
<thead>
<tr>
<th>Task</th>
<th>Start</th>
<th>End</th>
<th>Start</th>
<th>End</th>
<th>Start</th>
<th>End</th>
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<tbody>
<tr>
<td>Modification</td>
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<td>4/27-5/10</td>
<td>5/11-5/18</td>
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<td>Final Presentation Preparation</td>
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Questions?

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Titanium Electrode Setup