A Comparison of Photocatalysis and Electrocoagulation for Azo Dye Treatment and the Use of H_2 PEM Fuel Cells to Increase Electrocoagulation Efficiency

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Research Plan

Environmental Engineering

A. Rationale

Textile industries produce a significant amount of the world's wastewater, with Bangladesh alone contributing more than 200 million cubic meters of textile wastewater per year (Hossain et al., 2018). This textile wastewater can contain strong color, suspended particles, and non-biodegradable materials, which are often toxic or carcinogenic (Nandi, 2013). Two potential methods for breaking down these dyes are photocatalysis and electrocoagulation, but while the latter is an efficient way to purify this wastewater, it uses large amounts of electricity. About 80% of the world's energy comes from fossil fuels, which cause air pollution as well as global warming (Çokay and Gürler, 2016). Global energy usage is also increasing, which makes it necessary to make a cleaner source of electricity. One way to do all of these things may be to collect the hydrogen from electrocoagulation, which can be used to generate electricity and increase the efficiency of electrocoagulation.

Hydrogen gas is becoming a promising future source of energy (Santos et al, 2017). Hydrogen is most commonly produced using electrolysis, a method of splitting H_2O into H_2 and ½ O_2 using electricity (Chakik et al, 2016). While hydrogen can be generated in other ways, water is by far the most abundant and a very efficient of hydrogen. One of the most common and efficient methods for electrolysis and using hydrogen as fuel is using a proton exchange membrane fuel cell (PEMFC). To generate hydrogen, an electric current is applied and H_2O splits into H^+ and ½ O_2. To generate electricity, H_2 is fed on one side of the proton exchange membrane while O_2 (air) is fed on the other. When a power draw is applied, H_2 splits into H^+ ions. The H^+ ions cross the membrane while the electrons are left behind. H^+ reacts with O_2 to form H_2O while the electrons are used to create the electric current (Derbeli et al, 2017). The amount of current produced by the fuel cell is proportional to the flow rate of hydrogen and oxygen. For a PEM fuel cell to generate electricity, the reactants (hydrogen and oxygen) must be at a pressure of at least 0.1 to 2 psi relative to the air pressure. The air or hydrogen can be humidified to increase the efficiency of the fuel cell (Spiegel, 2017). Because a PEMFC uses a membrane, it can withstand a wide range of temperatures and pressures (Santos et al, 2017).

One method of electrolysis that also functions to purify water is electrocoagulation. Electrocoagulation is the process by which an electric current is run through two electrodes (anode and cathode) submersed in water. The electrodes are separated so electricity runs through the water, allowing for contaminants in the water to become polarized and for the following reactions to happen: At the cathode, hydrogen gas and OH^- are produced through electrolysis. (Nandi et al, 2013) The more chemically reactive the metal is (the less stable its electron configuration is), the more H^+/OH^- it will generate (Chakik et al, 2016). At the anode, metal ions are released. These ions combine with the OH^- generated at the cathode to form metal hydroxides. The hydroxides attach via charge differential to any polarized particle in the water to form flocs (complexes of contaminants and metal hydroxides) that are large enough to settle or be filtered out of the water. The most common metals used in electrocoagulation are aluminum and iron (Nandi et al, 2013).

Another water purification process is photocatalysis - the use of light to activate a catalyst which participates in the acceleration of a chemical reaction. In the process of photocatalysis, light hits the catalyst (normally TiO_2 or ZnO), and an electron is excited. The light must be of a specific wavelength depending on the photocatalyst in order to have enough energy to initiate the process, and the boundary for the energy needed from a light source for this to happen is known as the band gap width (Patsoura et al., 2006). In other words, a photon which possesses energy greater than or equal to the band gap width of the catalyst is needed for photocatalysis to occur (Saggoioro et al., 2011). If a photon with greater energy than the band gap width is absorbed, an electron (e^-_{cb}) "jumps" from the valence band to the conduction band of the catalyst, leaving behind a hole (h^+_{vb}) in the valence band (Saggoioro et al., 2011). The band gap can vary drastically depending on the photocatalyst, but a large band gap is generally preferred, as this leads to the photocatalyst having a high enough energy to break down pollutants (Chen et al., 2017). The band gap for titanium dioxide is very large at 3.2 eV (Zinc oxide is also very large at 3.37 eV), which makes them widely used models (Romao et al., 2016). However, not all forms of titanium dioxide behave the same, as the band gap for the anatase form is 3.2 eV, while the band gap for the rutile form is only 3.0 eV (Tian et al., 2015).

The electrons and holes are critical for the overall process due to the way they react with water and oxygen. In this step, both hydroxyl radicals (OH^-) and superoxide ions (O_2^{2-}) are produced (Badawy et al., 2015). These compounds are not very stable, but they are very good at degrading organic pollutants, such as textile dyes. In this process, the ions first oxidize the organic pollutants, which are often dyes. Secondly, a mineralization process occurs, in which the dye is broken down into carbon dioxide and harmless, inorganic ions (Badawy et al., 2015).

In 2019, Çokay and Gürler studied the effects of voltage (1-5V) on the amount of hydrogen produced and the purity of the hydrogen collected in the electrocoagulation of nickel, copper, and chrome plating wastewater. The total organic carbon was measured after each trial to test the effectiveness of electrocoagulation. Increased voltage was shown to increase H_2 production and H_2 purity in all cases. Increasing voltages also dramatically increased TOC removal.

Chakik et al in 2017 tested the effects of the cathode material on hydrogen gas formation in electrolysis. Electrodes were made mainly out of zinc combined with 5-15% (by mass) of the electrode being either iron, copper, cobalt, or chromium. It was found that, while hydrogen production decreased with increasing percentages of copper, hydrogen production increased with increased percentages of iron.

Chen et al. in 2017 studied the effects of initial pH, dye concentration, catalyst concentration, and mole ratio of oxalic acid to zinc acetate during preparation of a zinc oxide photocatalyst on dye degradation. It was found that acidic pH's, low concentrations of dye, high doses of zinc oxide, and a 4:1 mole ratio led to the highest degradation rates.

B. Research Question, Hypothesis, and Expected Outcomes

Research Question:

What effect will chemical factors, such as pH, dye concentration, and catalyst concentration have on the photocatalytic degradation of methyl orange? How do voltage, pH, and dye concentration affect the efficiency of electrocoagulation? What is the maximum ratio of hydrogen power generated to input power for electrocoagulation that can be reached (percent energy recapture)?

Alternate Hypothesis: An acidic pH, higher catalyst concentration, and lower dye concentration will lead to the most efficient photocatalytic system. Electrocoagulation efficiency will increase with an increase in voltage, a decrease in initial dye concentration, and a high pH. The percent energy recapture of electrocoagulation with hydrogen will be around 11%.

Null Hypothesis: pH, dye concentration, and titanium dioxide concentration will have no effect on the photocatalytic degradation system. Voltage, dye concentration, and pH will have no effect on the efficiency of electrocoagulation and hydrogen production. Percent energy recapture from electrocoagulation using hydrogen gas will be negligible.

Engineering Goals: Lids will be 3D printed to hold the electrodes for electrocoagulation and capture hydrogen.

Expected Outcomes:

For photocatalysis, upon decreasing pH, increasing concentration of titanium dioxide, and decreasing dye concentration, a greater percentage of dye breakdown will be observed (Chen et al., 2017, Saggoioro et al., 2017). During electrocoagulation, an increase in voltage, a decrease in dye concentration, and a higher (more basic) pH will increase the efficiency of the process due to increased power input, more metal hydroxides for every molecule of contaminant, and more hydroxide ions present (Nindi et al, 2013). The maximum increase will be 11%, as hydrogen produced from electrocoagulation can be captured and used in the fuel cells. This hypothesis was obtained through a calculation of specifications of the hydrogen fuel cell and previous research (Chakik et al., 2017).

C

- **Procedures**

a. Experimental Design

Experimentation will be structured such that many variables present in the electrocoagulation, photocatalysis, and hydrogen production processes can be optimized. These variables include voltage, pH, and dye concentration for electrocoagulation, and TiO_2 concentration, pH, and dye concentration during photocatalysis.

b. Preliminary

1. System Construction

The Electrocoagulation system will use a pair of iron electrodes in a 150 mL beaker (5.7 cm x 8.6 cm) with a 3D printed lid that is different depending on the phase. For dye breakdown trials, lids will be printed out of PLA plastic and will hold two electrodes. The electrodes will be secured to the lid using rubber bands. For trials that will be collecting hydrogen gas, a different lid will be 3D printed to hold the electrodes in a position ideal for hydrogen capture. There will be a hole in the middle for one silicone tube to be attached and an adaptor to attach two silicone tubes to the lid to transport the hydrogen gas to the hydrogen fuel cell.

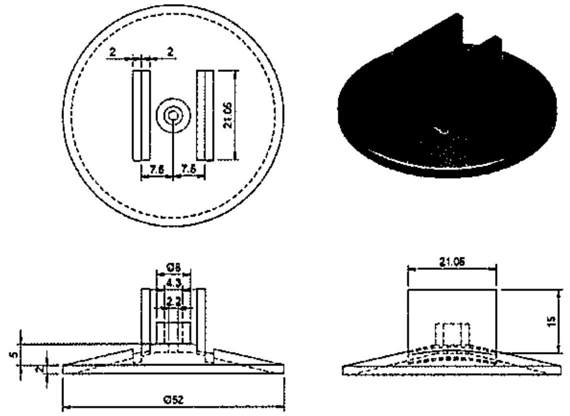

Figure 1: Schematic of Electrocoagulation Lid for Dye Breakdown (Diagrams Created by Research Entrants)

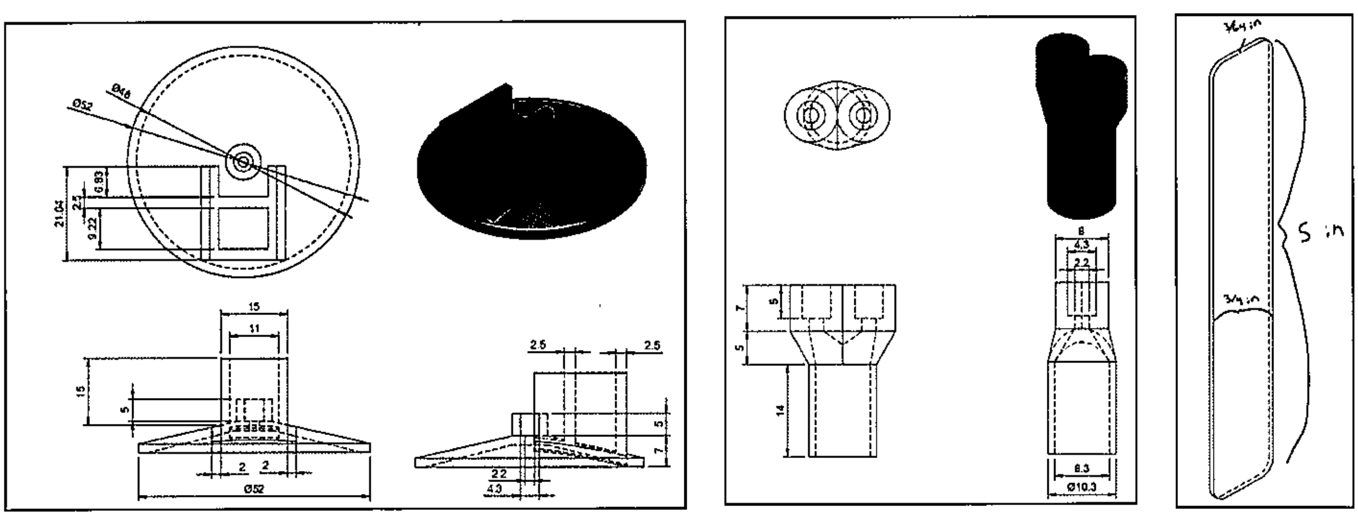

Figure 2: Schematic of Electrocoagulation Lid for Hydrogen Capture and adapter for two fuel cells (mm) and electrode (in.) (Diagrams Created by Research Entrants)

The hydrogen fuel cell will take in hydrogen produced during electrolysis and oxygen from the air. When the hydrogen and oxygen react, water and electricity will be produced.

2. Supplying Power

A DC power supply (Carolina Supply) will be used to power electrocoagulation. A 3D-printed box will direct the produced hydrogen to the PEM fuel cell (Horizon) to generate electricity during hydrogen trials.

3. Making and Measuring Methyl Orange Solutions

Methyl orange solutions will be made by adding 0.05% methyl orange by mass aqueous solution (Carolina Supply) to distilled water. methyl orange (1.2 L, 0.05% methyl orange by mass) will be transferred via a small graduated cylinder into a 150 mL beaker. Each trial will consist of either a 50 or 100 mL of solution, with each solution having a concentration of 50, 100, or 150 mg/L of methyl orange.

4. Measuring Dye Concentration

The optimal wavelength to measure the methyl orange concentration will be determined in a spectrophotometer (Vernier Go-Direct UV Vis spectrophotometer) by measuring the absorbance spectrum of methyl orange. The wavelength at the peak of the absorbance spectrum will be the wavelength in which all the other absorbance values are measured. A Beer's Law plot will be made, using a spectrophotometer, correlating the absorbance at a specific wavelength of light to concentration. Concentrations of methyl orange between 0 and 200 mg/L will be measured and plotted. The Beer's Law Plot will be used for all trials to determine dye concentration and the efficiency of dye removal by using the linear regression (line of best fit) between concentration and absorbance.

c. Testing- Photocatalysis

First, the amount of distilled water needed (depending on the dye concentration) to make 50 mL of solution will be added to a 100 mL beaker, which will be where photocatalysis will take place (Saggoioro et al., 2011). Then, if needed, pH will be altered through the application of 0.1 molar HCl or NaOH, which will be purchased from Carolina Biological (see pH testing below for more information). Titanium dioxide (0.1, 0.5, or 1 g/L) and methyl orange (50, 100, or 150 mg/L) will be added to each trial of photocatalysis. The titanium dioxide will be purchased as a mixture of rutile and anatase from Sigma Aldrich, and methyl orange from Carolina Biological. The solution will be kept in a cabinet under the UV radiation lamp (UVB will be used) to ensure that the light from the UV lamp is the only light they will receive and to keep our eyes safe. An 8.5 cm distance will be kept between the UV light source and the solution (Chen et al., 2017), resulting in an average light intensity of 1.8 W/m^2. Samples for each trial will be taken from solution after 24 hours, and the solution will be centrifuged at 12,000 rpm for 5 minutes to create a pellet of TiO_2 that can be filtered out and will not interfere with absorbance readings. The absorbance at the most effective wavelength will be measured using a Vernier GoDirect UV-Vis Spectrophotometer (Saggoioro et al., 2011). The absorbance of each trial in a spectrophotometer will be compared to its initial absorbance to assess what percentage of the orignal dye had been degraded (% left = $(A_{final} / A_{initial})*100$).

To alter the pH of the solution, 0.1 molar HCl and NaOH will be added with the required volumes to the 50 mL solution of distilled water prior to the addition of the titanium dioxide or the dye. The pH's that will be tested for this study are 3, 5, 7, 9, and 11 (Chen et al., 2017). To make sure that the pH is accurate, the Vernier pH probe will be used before starting the period of trials. When changing other independent variables in the study, the pH will be altered to its optimal value. The concentration of the titanium dioxide photocatalyst will then be altered, with concentrations of 0.1, 0.5, and 1 g/L (Saggoioro et al., 2011) in solutions with volumes of 50 mL. When other variables are being altered and TiO_2 concentrations are being kept constant, 0.1 g/L will be the concentration used. Lastly, the concentrations of methyl orange will be tested at 50 mg/L, 100 mg/L, and 150 mg/L (Saggoioro et al., 2011). Dye concentration will be a manipulated variable in order to see if high degradation rates can be maintained when there is more methyl orange to degrade. When the concentration of dye is kept constant, 50 mg/L, or 5 mg per 50 mL of solution, will be the concentration used.

d. Testing- Electrocoagulation

1. Dye Breakdown

The effects of voltage, pH, and dye concentration will be tested for, with each variable being utilized for the total length of the trial. Voltage will be tested first, then pH, then dye concentration. The effects of voltage at 2.5 and 5 volts will be tested, with the voltage being set using the power supply. The concentration of methyl orange will be 100 mg/L and the pH will be 7. Next, trials testing pH at 3, 5, 7, 9, and 11 will be run with 100 mg/L methyl orange and the optimal voltage for electrocoagulation. Lastly, the effects of dye concentration at 50, 100, and 150 mg/L will be measured using the optimal voltage and pH for dye breakdown.

Each trial will be run for 20 minutes and then filtered using Whatman filter paper. After this, the amount of methyl orange remaining will be calculated by measuring the solution's absorbance at the optimal wavelength in a spectrophotometer. Percent removal will be calculated by $(A_{final}/A_{initial})*100$. The absorbance of the solution will be recorded and compared against the Beer's Law plot to obtain the concentration of methyl orange.

2. Percent of Recaptured Energy

This phase will be done to test how much hydrogen (gas) power can be generated while electrocoagulation is taking place to find the percent of energy recaptured for the process. Each trial will be run using 100 mL of distilled water with 3.5g of NaCl. The salt concentration was chosen because it is the average concentration of salts in seawater: 35g/L (NOAA, 2019). Electrocoagulation will take place with the second lid design with the adapter (Figure 2). The lid, using silicone tubes, will direct hydrogen generated from electrocoagulation to two fuel cells (Horizon PEM Mini Fuel Cell). The fuel cells will be connected in series to produce twice the output voltage of one fuel cell. The output of this will be connected to a Horizon Energy Monitor to measure the amount of voltage, wattage, and amperage being produced. A motor will be used to draw current, which is needed for current to be drawn. After the voltage from the hydrogen fuel cell increases to its apparent maximum, the motor will be connected to the fuel cell monitor, and the output power will be measured in Watts. This will then be divided by the input power (W) from the power supply to find the percent of of recaptured energy (percent recaptured energy = $100*W_{output} / W_{input}$).

- **Risk and Safety**

1. *Human participants research: N/A*
2. *Vertebrate animal research: N/A*
3. *Potentially hazardous biological agents research: N/A*

4. Hazardous chemicals, activities & devices:

Methyl Orange, Indicator, Sodium Salt, 0.05% Aqueous, Laboratory Grade, 1.2 L

- Concentration - 0.05%
- Volume- 1.2 L
- Risk assessment- not a harmful substance according to OSHA standards
- Supervision in the lab will always be maintained by a trained adult professional
- For safety precautions, nitrile gloves, goggles, and a lab apron will be worn
- Disposal- will be disposed of in accordance with local regulations- a permitted waste disposer will be contacted to ensure compliance
- www.carolina.com/teacher-resources/Document/msds-methyl-orange-005-percent/tr-msds-methyl-orange-005-percent.tr.

Titanium Dioxide, mixture of rutile and anatase (TiO_2)

- Concentration- pure powder will be purchased, solutions of 0.1 g/L, 0.5 g/L, and 1 g/L in solvent of 50, 100, or 150 mg/L of methyl orange dye
- Volume- roughly 3 g of total powder needed
- Risk assessment- not a harmful substance or mixture (MSDS, Titanium IV Oxide)
- Supervision will always be present from research supervisor

- Nitrile gloves, goggles, and a lab apron will be worn at all times
- Surplus powder will be offered to a licensed disposal service, while contaminated packaging can be disposed of as unused product
- https://www.sigmaaldrich.com/catalog/product/aldrich/700339?lang=en&region=US

Hydrochloric Acid

- Concentration- 0.1 Molar, volume- roughly 20 mL needed for this study
- Risk assessment- skin and eye irritation, potential toxicity if inhaled
- Always will be adult supervision when working in the lab
- Goggles, nitrile gloves, and a lab apron will be used for safety precaution
- Permitted waste disposal will be contacted for waste disposal
- https://www.carolina.com/pdf/msds/hcl01mghs.pdf

Sodium Hydroxide

- Concentration- 0.1 Molar, volume- roughly 20 mL needed for this study
- Risk assessment- can cause severe skin burns and eye damage, may be harmful to aquatic life
- Goggles, nitrile gloves, and a lab apron will be used for safety precaution
- Permitted waste disposer must be contacted for waste disposal
- https://www.carolina.com/pdf/msds/naoh01mghs.pdf

Hydrogen Gas- projected volume of 1.4 mL produced in each 20 minute trial

- Flammable and can form explosive mixtures when combined with air, incompatible with oxidizers
- Lab supervision will be in place whenever working with H_2 gas
- Hydrogen will be handled at atmospheric pressure and cannot be stored anywhere where sparks can form
- All hydrogen will be converted directly into H_2O in the PEM fuel cell.
- https://www.carolina.com/pdf/msds/hcl01mghs.pdf

Iron Electrodes

- Not a harmful substance
- Supervision will be present in the lab while working with these electrodes
- Vinyl gloves will be work to prevent touching any iron hydroxides or dye that washes onto the electrodes
- Can be disposed of normally by just throwing it out in the garbage

DC Power Supply

- Can be extremely harmful if both the positive and negative leads are touched at the same time while this is on
- Like all other chemicals/devices, lab supervision will be present while we are using this
- Will be stored on a countertop
- Extreme caution will be used to make sure to never touch both caps of the leads at the same time

Electrocoagulation

- Current running through water could be harmful if touched
- A lid will be used to hold electrodes and prevent electrocution from touching the water
- Adult supervision will always be present when running these trials
- The electrocoagulation apparatus will have a lid to prevent any possible exposure to an electric current during electrocoagulation

UV Light

- UV-B light will be used
- Can cause eye irritation/damage if stared directly into
- Light source will be kept only in a closed cabinet when turned on, so it is never exposed to our eyes; light will be turned off before opening cabinet again
- Adult supervision will always be present in the lab

- **Data Analysis**

A Beer's Law Plot will be made, using a spectrophotometer, to correlate absorbance at a specific wavelength to the dye concentration. The wavelength used will be found by measuring the dye's absorption spectrum and choosing the wavelength with the highest absorbance. After every trial, the absorbance of the filtrate will be measured, and the percent dye remaining will be calculated with the following equation: % left = $100^* (A_{final} / A_{initial})$. For energy recapture trials, $100^*(W_{output} / W_{input})$, will be done to determine the percent of energy recaptured for electrocoagulation. Averaged data will be graphed using Microsoft Excel. Statistical analysis will be determined using IBM SPSS v. 26 with a one-way ANOVA followed by a post-hoc Scheffe with $p<0.05$ to measure significance in each test group. The efficiencies of photocatalysis and electrocoagulation will be measured and compared by comparing the wattage (J/s) over time for both.

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Project Summary

NO ADDENDUMS EXIST