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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A. Introduction

a. Need, Knowledge Base, Literature Review

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 $\frac{1}{2} O_2$ 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 $\frac{1}{2} O_2$. 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, which causes contaminants in the water to become polarized as well as reactions that occur at the electrodes themselves. 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_2/OH^- it will generate (Chakik et al, 2016). At the anode, metal ions are released. These ions combine with the OH⁻ generated from the cathode to form metal hydroxides. The hydroxides attach via charge differential to any polarized particle in the water, clumping contaminants together into particles (complexes of contaminants and metal hydroxides) large enough to settle or be filtered. 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 in turn participates in the acceleration of a chemical reaction. In the process of photocatalysis, light, which must be of a specific wavelength depending on the photocatalyst in order to have enough energy to initiate the process, hits the catalyst (normally TiO_2 or ZnO), and an electron is excited. There is a boundary for the energy needed from a light source for this to happen 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 varies 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 in it of themselves, but they are very good at degrading organic pollutants, such as textile dyes. In this process, the ions first oxidize these 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. Purpose

The purpose of this study was to compare photocatalysis and electrocoagulation in the remediation of methyl orange and to determine how the following variables affected those two processes:

- pH, TiO2 concentration, and dye concentration were tested for photocatalysis
- voltage, pH, and dye concentration were tested for electrocoagulation

- Maximum percent energy recapture was tested for the electrocoagulation process, using hydrogen fuel cells ($100*W_{out}/W_{in}$)

c. Hypotheses and Engineering Goals

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 maximum energy recapture of electrocoagulation 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.
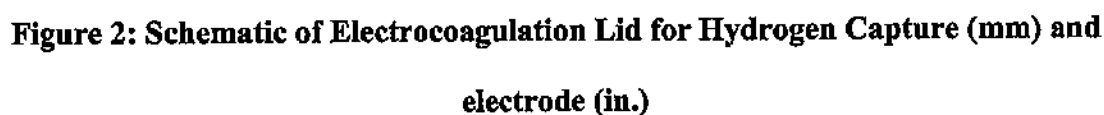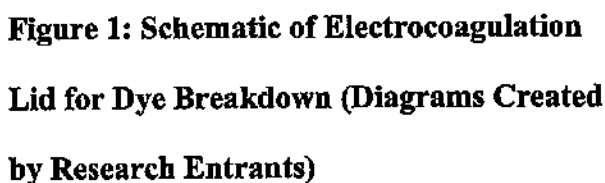
B. Methodology

a. Experimental Design

Experimentation was structured such that many variables present in the electrocoagulation, photocatalysis, and hydrogen production processes were 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 used 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 were printed out of PLA plastic and held two electrodes. The electrodes were secured to the lid using rubber bands. For trials that were collecting hydrogen gas, a different lid was 3D printed to hold the electrodes in a position ideal for hydrogen capture. There was 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 (mm) and electrode (in.)

The hydrogen fuel cell was meant to take in hydrogen produced during electrolysis and oxygen from the air. When the hydrogen and oxygen react, water and electricity were produced.

2. Supplying Power

A DC power supply (Carolina Supply) was used to supply 0, 2.5, or 5V at 40 A/m^2 to the electrodes using alligator clips. The 3D-printed lid directed the produced hydrogen to the PEM fuel cell (Horizon), which used the hydrogen to generate electricity.

3. Making and Measuring Methyl Orange Solutions

Methyl orange solutions were 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) was transferred via a small graduated cylinder into a 150 mL beaker. Each trial consisted 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 was determined in a spectrophotometer (Vernier Go-Direct UV Vis spectrophotometer) by measuring the absorbance spectrum of methyl orange. This specific wavelength was the wavelength in which all the other absorbance values are measured. A Beer's Law plot was 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 were measured and plotted. The Beer's Law Plot was

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

1. Photocatalysis- General Process/Procedure

First, the amount of distilled water needed (depending on the dye concentration) to make 50 mL of solution were added to a 100 mL beaker, which were where photocatalysis took place (Saggoioro et al., 2011). Then, if needed, pH was altered through the application of 0.1 molar HCl or NaOH, which were purchased from Carolina Biological (see pH testing below for more information). The concentrations of titanium dioxide (ranging from 0.01 to 1 gram per liter of solution) and methyl orange dye (ranging from 50 to 150 mg per liter of solution) needed for each trial was measured and then put into the 50 mL solution to initiate the process. The Titanium Dioxide was purchased as a mixture of rutile and anatase from Sigma Aldrich, and methyl orange from Carolina Biological. The solution was kept in a cabinet under the UV radiation lamp (UVB) to ensure that the light from the UV lamp was the only light they received. An 8.5 cm distance was 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 were taken from solutions after 24 hours, and the solutions were centrifuged at 12,000 rpm for 10 minutes to create a pellet of TiO$_2$ that could be filtered out. The absorbance at the most effective wavelength was measured using a Vernier GoDirect UV-Vis Spectrophotometer (Saggoioro et al., 2011). Absorbance in a spectrophotometer was compared to the initial absorbance of that concentration of dye 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 were 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 were tested for this study were 3, 5, 7, 9, and 11 (Chen et al., 2017). To make sure that the pH is accurate, the Vernier pH probe was used before starting the period of trials. When changing other independent variables in the study, the pH was altered to its optimal value. The concentration of the titanium dioxide photocatalyst was then altered, with concentrations of 0.1 g/L, 0.5 g/L, 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, or 5 mg per 50 mL of solution, was the concentration used. The concentrations methyl orange were 50 mg/L, 100 mg/L, and 150 mg/L (Saggoioro et al., 2011). Dye concentration was a manipulated variable in order to see if high degradation rates can be maintained when there is more to degrade and because it has been frequently tested in journal articles (Chen et al., 2017, Saggoioro et al., 2011). When the concentration of dye was kept constant, 50 mg/L, or 5 mg per 50 mL of solution, was the concentration used.

d. Testing- Electrocoagulation

1. Dye Breakdown

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

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

2. Percent of Recaptured Energy

This phase was done to test how much hydrogen (gas) power would be generated while electrocoagulation was taking place to find the percent of energy recaptured for the process. Each trial was 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). The electrocoagulation took place with the second lid design and the adapter (Figure 2). The lid, using silicone tubes, directed hydrogen generated from electrocoagulation to two fuel cells (Horizon PEM Mini Fuel Cell). The fuel cells were connected in series to produce twice the output voltage of one fuel cell. The output of this was connected to a Horizon Energy Monitor to measure the amount of voltage, wattage, and amperage produced. A motor was used to draw current, which was needed for current to be drawn. After the voltage from the hydrogen fuel cell increased to its apparent maximum, the motor was connected to the fuel cell monitor, and the output power was measured in Watts. This was then divided by the input power (W) from the power supply to find the percent of of recaptured energy (percent recaptured energy = $(100*W_{output} / W_{input})$.

C. Results
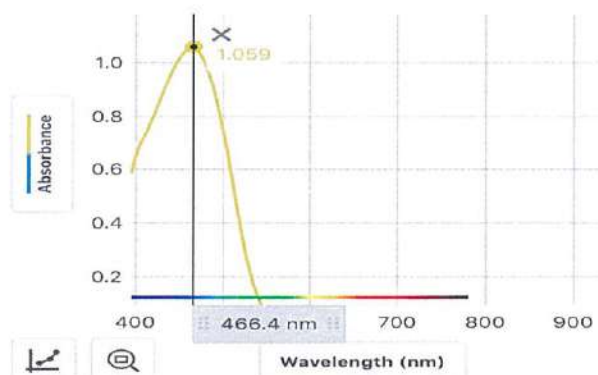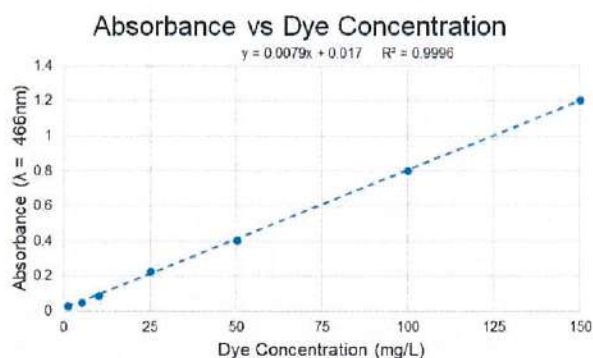
a. Absorbance Spectrum

Figure 3- The Absorbance Spectrum of Methyl Orange

Methyl orange reached a maximum absorbance at 466 nm, so this became the wavelength the absorbance was measured at for all other trials.

b. Beer's Law Plot

Figure 4- Absorbance of methyl orange as a function of its concentration (mg/L)

(Beer's Law Plot)

The correlation between the concentration of methyl orange and absorbance was very strong, with an r^2 value of 0.999. The line of best fit was $A = 7.96*10^{-3}C + .0134$, where C= concentration of methyl orange (mg/L), and A= absorbance.
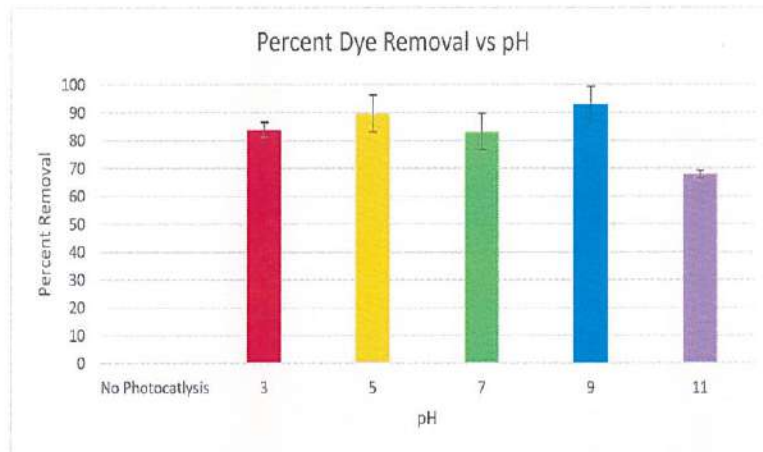
c. Photocatalysis Trials

Figure 5- The Effect of pH on Percentage of Dye Broken Down (N=3)

From this figure, it can be concluded that all pH values were successful at breaking down dye, as all groups were significant to no photocatalysis, just to slightly different extents. pH 9 had the highest proportion of dye broken down (approx. 94%), although not significantly. pH 11 performed significantly ($p < .05$) worse than pH 3, pH 5, and pH 9, as less dye was broken down.
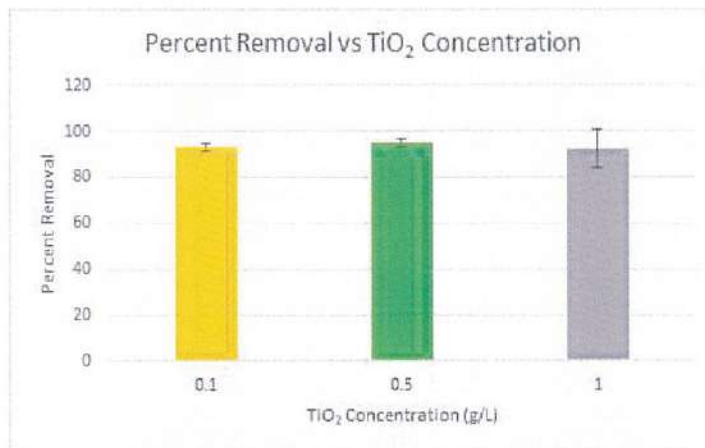

Figure 6-The Effect of TiO$_2$ Concentration on Percentage of Dye Broken Down (N=3)

This figure shows that all groups were successful in breaking down dye, as they all broke down over 90% of it. However, the groups were not significant to one another when run through SPSS, as the p-values were not less than 0.05. This means that they all broke down dye at roughly the same rate, and that TiO2 concentration had little effect.
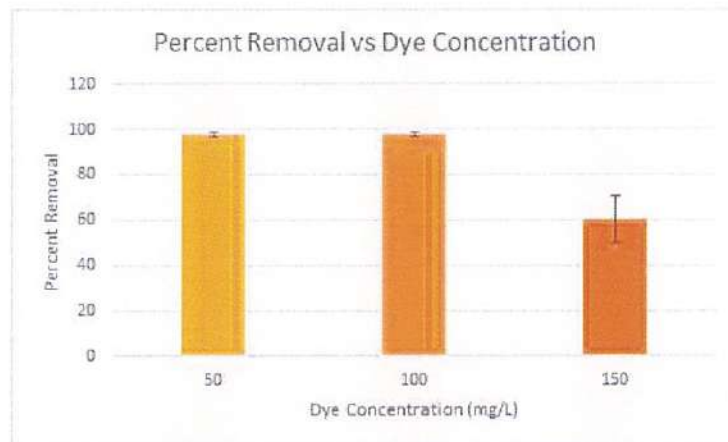
Figure 7- The Effect of Initial Dye Concentration on Percent Removal of Dye Breakdown (N=3)

From this, it is evident that while all the dye was broken down in the 50 mg/L and 100 mg/L trials, much less (significantly) was broken down when the concentration of dye was 150 mg/L. This suggests that there is some sort of "falling off point", where the concentration of dye becomes too much for the limited amount of TiO_2 to break down. This point exists somewhere between 100 and 150 mg/L of dye.

d. **Electrocoagulation for Dye Breakdown Trials**
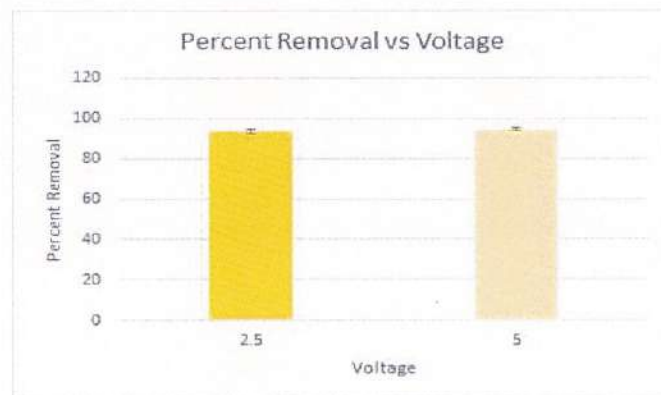

Figure 8- The Effect of Input Voltage on the Percent Removal of Methyl Orange (N=3)

From figure 8, it is demonstrated that while the percent of dye removed was slightly greater for the 5 V group than the 2.5 V group, the data is insignificant. Both groups ended up removing nearly 100% of the dye when run for 20 minutes at their specific voltage.
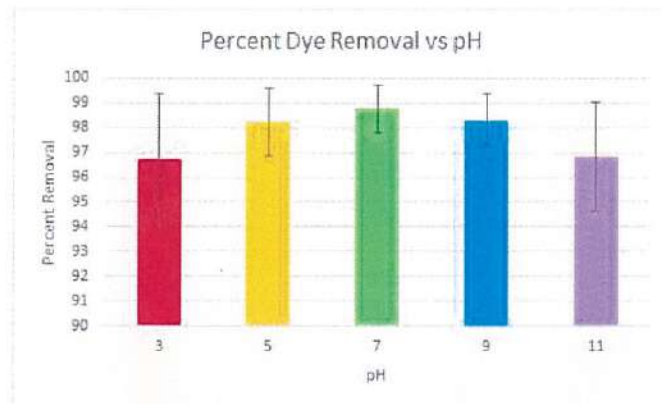

Figure 9- The Effect of pH on the Percent Removal of Dye (N=3)

Figure 9 is slightly different in that the y-axis, while still percent removal, is now on a scale from 90-100. The data between groups was not significant, and every group removed over 95% of the dye from the 50 mg/L methyl orange solution. However, the percent removal was the highest, slightly, at ph=7. This was determined to be the optimal pH, so for the rest of the electrocoagulation phase, when other variables were being altered, a pH of 7 was used.
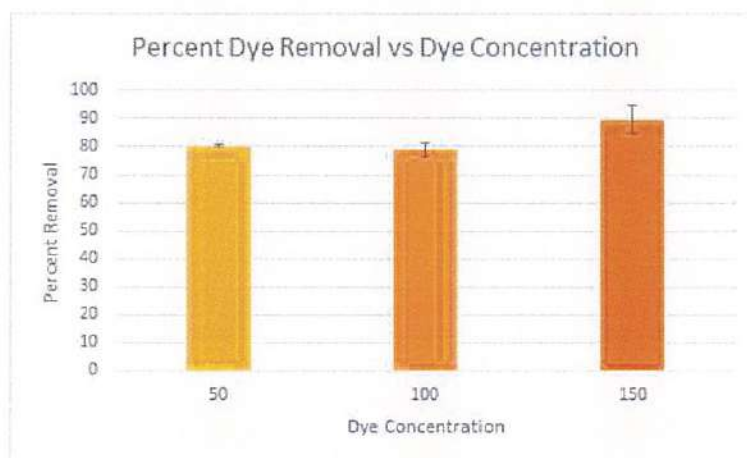

Figure 10- The Effect of Initial Dye Concentration on Percent Removal of Dye In Electrocoagulation (N=3)

Figure 10 demonstrates that there was actually an increase in percent removal of dye with higher concentrations, as 150 mg/L was significantly higher than either of the other two groups. While in photocatalysis, there was a point at which percent dye breakdown decreased significantly, for electrocoagulation, it was the opposite.

e. **Percent of Energy Recaptured Trials**

Percent Energy Recapture vs Input Energy

$y = \{x < 1.4496: x*12.0391, x \geq 1.4496: 25.2970/x\}$ $R^2 = 0.8608$
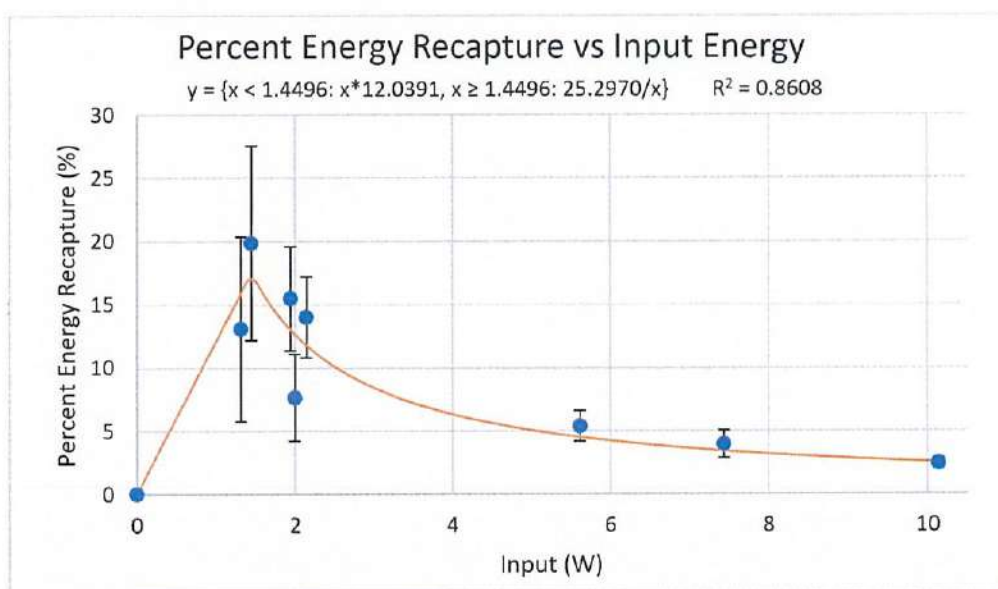
Figure 11- Percent of Energy Recaptured in Electrocoagulation (N=3)

From figure 11, it is shown that the electrocoagulation process could recapture up to 20% of its energy, reaching a maximum around 1.5 W. The efficiency shoots up as the voltage is raised from 0 to this maximum, but then exhibits an inverse relationship when the wattage was increased to over this maximum value.

D. Discussion

The purpose of this study is to compare the effects of and test the operational parameters of photocatalysis and electrocoagulation. The effects of pH, TiO_2 concentration, and dye concentration was measured for photocatalysis, and the effects of voltage, pH, and methyl orange concentration was measured for their impact on the efficiency of electrocoagulation and hydrogen production.

The photocatalysis worked, and was able to break down the vast majority of the dye no matter what the pH, but the most effective pH values were 9 and 5 while pH 11 was the least

effective, significantly. The results of Chen et al. in 2017 support this somewhat, as in both studies, the most basic pH value served as the worst medium, but the results were mostly contradictory, as in their study, acidic pH values worked the best, while in ours, pH 9 was the best group. The difference may be as a result of the photocatalyst used. In Chen et al., ZnO was used as the primary catalyst, while our study used titanium dioxide. Depending on the type of photocatalyst used, different solution conditions may be optimal, and this provides good evidence for that.

There seemed to be no significant change in efficiency as TiO_2 concentration increased. This may be because relatively little amounts of TiO_2 are needed to purify water. These results are supported by Saggioro et al., where the difference in dye removal between 0.1g/L and 1g/L is very small. Therefore, in a real-life application of photocatalysis, it is optimal to use lower concentrations of TiO_2, as the purification works just as well, and less powder must be removed from the solution to yield potable water.

Dye breakdown was high for the 50 and 100 mg/L of dye, but significantly lower for the group with a dye concentration of 150 mg/L. This makes sense, because the breakdown of dye is a reaction between the TiO_2 and dye, and at some point, the concentration of dye becomes too large for the small amount of TiO_2 to handle. This point occurred somewhere between 100 and 150 mg/L.

Electrocoagulation also worked, as it was able to coagulate the vast majority of the dye. While both the 2.5 V and 5 V groups demonstrated this, the two groups were not significant to one another. This was because the electrodes produced enough iron hydroxides at 2.5 V for the process to work efficiently. In both groups, roughly 94% of dye was removed, indicating that once a certain voltage is achieved, increasing it more does not have much of an effect.

For pH testing, all groups resulted in the removal of nearly all dye, but they were not significant to one another. This is supported by Nandi et al., in 2013, who also found that in the pH range of 3-11, electrocoagulation does not significantly differ in the percentage of dye it can remove from solution. While H^+ and OH^- ions do play a role in the process of electrocoagulation, it is at pHs above 3, enough OH^- radicals are produced to neutralize the solution and produce

metal hydroxides, while pHs below that significantly hinder the ability of iron hydroxides to form.

Lastly, the group with a 150 mg/L initial dye concentration removed significantly more dye than the 50 mg/L or 100 mg/L group. This was because, unlike photocatalysis, electrocoagulation was able to produce enough metal hydroxides to remove all of the dye. Therefore, since all the final absorbances were roughly the same but the initial absorbances were higher for larger concentrations, the resulting removal rate was higher.

Electrocoagulation was more energy efficient than photocatalysis: even though it required 11.18x more energy (J/s), at any given moment, it ran for 72x shorter than photocatalysis, making it more efficient. However, photocatalysis could potentially be run with only the light from the sun, while electrocoagulation requires a power supply.

Electrocoagulation also was able to recapture a significant portion of its energy because our design allowed for two hydrogen fuel cells to be run simultaneously. A large amount of hydrogen was captured, and the fuel cells produced a significant amount of power, resulting in a relatively high value for ($100*W_{output}$ / W_{input}). This method was novel, and the percent of energy recaptured was nearly twice the ore-study predictions. Potentially, the efficiency of electrocoagulation could be increased by 20%, which would have profound implications on the successfulness of the process.

One limitation of this study was that photocatalysis was not tested with direct sunlight. UV light was used, and while the sun does release some UV light, it would be important to know whether solely light from the sun can accomplish this process. Also, the process was small scale; not enough hydrogen was generated to run the electrocoagulation off of power from only the PEM fuel cell. Finally, the hydrogen fuel cell had a small maximum output, so it was difficult to know whether further increases in amperage would have resulted in more power being produced.

E. Conclusion

The optimal conditions for photocatalysis were pH values between 3 and 10, a TiO_2 concentration between 0.1 and 1 g/L, and methyl orange dye concentration between 50 and 100

mg/L. For electrocoagulation, the conditions were higher voltages, pH's between 3 and 11, and dye concentrations less than 150 mg/L or less. The engineering goals were successful- enough hydrogen was produced and captured to use in fuel cells. Lastly, electrocoagulation was more energy efficient than photocatalysis, but it requires a power source, while photocatalysis can potentially be run off of only sunlight.

F. Future Studies

In the future, electrocoagulation can be run at a larger scale, generating enough watts to increase the efficiency of itself significantly. Also, to better compare electrocoagulation to photocatalysis, electrocoagulation could be run using solar panels. This would mimic the light from the sun needed for photocatalysis. Finally, photocatalysis could be tested out in the sunlight instead of in lab settings, to gain a better understanding of its real world implications.

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