



# **Utilizing UV/TiO<sub>2</sub> Advanced Oxidation to Degrade Organic Compounds in Runoff Water**

Major Qualifying Project completed  
in partial fulfillment of the requirements for the  
Degree of Bachelor of Science

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## Abstract

Organic compounds have been in storm water runoff, contaminating lakes, streams and groundwater for decades now, with no feasible, cost effective and efficient removal techniques thus far. Non-point source pollution from things such as automobiles can be the origin of many organic substances like oil and gasoline. These contaminants are then washed into surrounding grass and soil by rainfall, which negatively impacts nearby waters. The objective of this project was to evaluate the removal of these harmful organic contaminants from storm water through the use of titania ( $\text{TiO}_2$ ) in combination with UV light. Past studies have shown that titania when subjected to UV light, will produce oxidants that can degrade organic contaminants in water. In this research, we evaluated the feasibility of using titania/UV light to reduce organic compound concentrations in a small scale detention pond.

## Acknowledgements

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## 1.0 Introduction

Currently there is a demand for a safe and effective way to eliminate harmful organic compounds in storm water runoff. The human race lives in an age where organic compounds such as gasoline, oils and dyes are commonly found in water at dangerous concentrations. Areas where water runs off typically include parking lots, roads and other modernized areas like factories and commercial buildings. These organics are typically deposited into detention ponds, where the run-off water collects. If this polluted water goes untreated it can affect not only humans but also other species. Altogether, the effect of organic pollutants in water supplies is a global issue that requires immediate attention.

One effective way to induce the breakdown of organics in solution is the use of photocatalysis. Research suggests that titania ( $\text{TiO}_2$ ) can lower the concentration of organic compounds in solution when subjected to UV light. When photons from UV light come into contact with the titania, it demonstrates photocatalytic behavior for the oxidation of the organic compound. At this point it is apparent that there are multiple applications for this novel technology. It has been used to coat buildings in order to degrade air-borne contaminants and clean the surrounding air. However, we have identified a potential novel application for this process. Since titania is light-activated, it is feasible that storage collection reservoirs could be coated with this compound to destroy the harmful hydrocarbons that are found in runoff water.

For this research we hypothesized that titania when used in combination with UV light will degrade organic contaminants in runoff water. First, this involved finding a surface to adhere the titania. The ideal surface is something that can be easily put on the bottom of a reservoir but also adheres well to the titania on a long term basis. Some sample surface materials

are concrete, glass and ceramics. The next step was finding a sample organic compound that can be used in this testing to evaluate the feasibility of the titania system. The compound must be an organic but not a volatile chemical in order to avoid loss of the contaminant due to volatilization. Possible compounds for these experiments are sugar (glucose), ethanol and methylene blue.

A small scale collection reservoir with a titania coating was fabricated to test our hypothesis. Tests were then conducted with known concentrations in the reservoir. After an allotted amount of time analysis with a spectrophotometer was performed to measure the treated organic concentration in the water in the reservoir. According to the hypothesis, the final concentration was expected to be significantly lowered due to the presence of the titania and UV light. This work evaluated the feasibility of coating run-off collection basins with titania in order to reduce the concentration of harmful organics in water.

## 2.0 Background

Titania subjected to UV light can produce oxidants that can degrade organic contaminants in water. The ability of Ti/UV to mineralize organics is the focus of the project and the goal was to create a detention pond with titania attached to the surface that can be used to treat storm run-off water. Contaminated run-off water can be stored and treated with the Ti/UV process to remove organic contaminants in the water.

The real world problem that needs to be addressed involves contamination of ground and surface waters resulting from storm runoff from pavements. Storm water that originates in parking lots and roads may contain various organic compounds that can be released into the environment. Vehicles, such as cars and trucks, contribute to storm water pollution by leaking oil and gasoline onto roads, driveways, and parking lots. Spills of petroleum products in refineries, fuel deposits, and gasoline stations, as well as containers in hazardous waste storage areas, may also contribute to the release of pollutants. The biggest area of impact of contamination is, however, surface water. Surface water is water in surface water bodies such as oceans, rivers, streams, and lakes. When hazardous substances come into contact with surface waters, they may cause pollution due to contact with the water. Sediments, including sand and soils, on the bottom of these surface water sources, tend to become contaminated as well. There are many ways that surface water can come in contact with hazardous substances. For example, these substances can be discharged directly from an outfall pipe from industrial sources or from older sewer systems that may overflow during precipitation events.

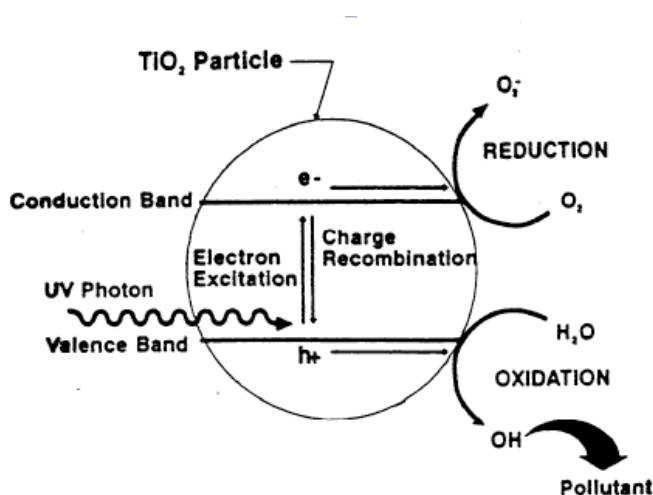
This water quality change because of surface water contamination affects all levels of an ecosystem negatively. It directly impacts the health of the lower food chain organisms. This, in

turn, impacts the availability of the food supply throughout the rest of the food chain. Surface water contamination also affects the health of wetlands, which depletes its ability to support healthy ecosystems, control flooding, and filter pollutants from storm water runoff. When directly drinking or bathing in contaminated water, the health of animals and humans may be impacted. When it comes to aquatic organisms, specifically fish, surface water contamination is very dangerous when it becomes ingested.

In order to minimize the impact and movement of these pollutants, existing methods have been applied but have not been completely successful. A few examples would be overland flow channels, grassy swales, retention basins, and wetlands. However, many of these methods have drawbacks and downfalls. The success of retention basins at removing pollutants, for example, is more effective with increasing size. Since larger basins cost more, there comes a point where the price of the basin does not translate to a significant increase in treatment efficiency of the basin. Furthermore, these previous methods all share common problems such as the amount of maintenance required, larger required system space, and high cost with construction. Titania coating, on the other hand, may successfully decontaminate storm water run-off, preserving the water quality of ground and surface waters without some of these additional setbacks.

Titania is a photocatalytic compound in the form of titanium dioxide. Titanium dioxide has been studied extensively due to the unique properties and large variety of applications, such as gas sensors, self-cleaning materials, and as pigments with enhanced photodegradation activity with visible light. Titanium dioxide has been used previously as photocatalysts for solar energy conversion because it is a low cost, non-toxic substance with excellent photoactivity. When  $\text{TiO}_2$  is irradiated by sunlight in the ultraviolet range, electrons pass across the band gap into the

conduction band, leaving holes in the valence band. These electron holes have strong oxidizing power and can react with hydroxide ions or oxidize water adsorbed at the surface to form hydroxyl radicals. The conduction band electron can reduce adsorbed oxygen to form superoxide ion radicals which can further break down to form hydroxyl radicals through various pathways. In the absence of oxygen the conduction band electron may react directly with the contaminant via reductive processes. The redox reactions occurring from the hole-electron pairs at the surface of the semiconductor can then degrade organic compounds into oxidized or reduced products. This process is shown below in Figure 1.



**Figure 1: Primary reactions occurring at the surface of an illuminated  $\text{TiO}_2$  particle. (Mehos and Turchi, 1993 p. 8))**

The most abundant form of titania occurs in nature as rutile. This is the predominant type and is used in industrial applications. Anatase is another form of titania and according to research, shows the highest photoactivity. Brookite is the third and final form of titania, but is not very common and scarcely used. A specific procedure must be followed in order to produce titania. It is purified by converting titanium tetrachloride in the chloride process where crude ore is reduced with carbon and oxidized with chlorine (Bakardjieva, 2005 p. 192).

The goal of this project was to develop a titania-coated retention pond to remove organic contaminants from storm-water. This was done by creating a bench-scale retention pond coated with a thin layer of titanium dioxide, introducing contaminated water to the pond, and then measuring the amount of hazardous material left in the water after the titanium dioxide has had time to react with the contaminants.

The first task was to adhere titanium dioxide to a small-scale basin simulating a retention pond. Past research has verified that titanium dioxide can be adhered to concrete and concrete could feasibly be the foundation of a retention pond. The next task involved deciding which method would best adhere the titanium dioxide to the concrete. The most effective method was found to create a paste with the titanium dioxide, cement water, and sand, and then cover the concrete surface with this paste. For this small scale experiment, petri dishes were used as small scale retention ponds and the  $\text{TiO}_2$  was attached to the petri dish surface with glue.

Previous research and experiments have been conducted with titania to observe its ability to remove pollutants from air and water. The Catalytic Pavement Borders Research Report (1996) was a study conducted in cooperation with the U.S. Department of Transportation, Federal Highway Administration and was written by Alexander Jones and Richard Watts. Titanium dioxide was used as an amendment incorporated into pavement surfaces for controlling hydrocarbon runoff. Six different materials were evaluated for fixing the titanium dioxide onto pavements. Two of these six materials, paint base and concrete sealant, proved to be the most effective and had the highest photocatalytic ability. With these materials, contaminants such as hexadecane, anthracene, and 2,4,6-trichlorophenol were effectively removed. As a result, this report illustrated the potential for photocatalytic pavements for alleviating nonpoint highway runoff (Watts, 1996 Rep. no. 391.1)

Another report, titled The Effect of Application Methods on the Effectiveness of Titanium Dioxide as a Photocatalyst Compound to Concrete Pavement (2010) evaluated three methods of titanium dioxide application to concrete pavement. The report was written by Marwa Hassan, Heather Dylla, Louay Mohammad, and Tyson Rupnow for the Louisiana Transportation Research Center and LSU. The first method involved applying a thin coating to the concrete surface at a titanium dioxide content of 3% and 5%. This surface mixture consisted of ultrafine titanium dioxide, cement, filler and water. The second method applied a water-based TiO<sub>2</sub> surface treatment, PURETI, to the hardened concrete surface. PURETI forms an invisible ultra-thin coating that exposes the nano-sized titanium dioxide particles to the atmosphere. Finally, the third method required sprinkling nano-sized titanium dioxide particles to the fresh concrete surface at a TiO<sub>2</sub> content of 3% and 5% prior to hardening. After the experiments had been carried out, multiple conclusions were drawn. The coating with 5% TiO<sub>2</sub> and PURETI product were the most efficient in removing nitrogen oxide from air. The use of a thin coating would be more susceptible to abrasion than using the sprinkling method or PURETI product, according to a rotary abrasion test. Also, the samples with PURETI product had a more uniform distribution and higher concentration of TiO<sub>2</sub>. Overall, the method involving PURETI produced the most positive results and greater NO removal efficiency (Hassan, 2010 Tech. No. 10-0746)

A report entitled “The Evaluation of Titanium Dioxide as a Photocatalytic in Asphalt Binder” (2011) explored the use of titanium dioxide coating as a photocatalysis on surfaces such as flexible pavements rather than concrete pavement. It was written by Marwa Hassan, Louay Mohammad, Samuel Cooper, and Heather Dylla for the Louisiana Transportation Research Board and LSU. The main purpose of this experiment was to evaluate Ti coating on asphalt pavement because approximately 94% of the road network in US is surfaced with hot-mix

asphalt (flexible pavement), not concrete pavement. The impacts of integrating the additives on the rheological properties of the binder was investigated. Also, a commercial crystallized anatase-based titanium dioxide powder was blended with a conventional asphalt binder at three modification rates. At the end of this experiment, it was found that the use of TiO<sub>2</sub> as a modifier to asphalt binder was effective in removing part of the NO<sub>x</sub> pollutants in air. Rheological tests indicated the addition of TiO<sub>2</sub> didn't affect the physical properties of conventional binder.

(Hassan, 2010 Tech. no. 11-1111)

A report titled “Photocatalytic Activity of Nano-TiO<sub>2</sub> on Glass in Building Envelope” also provided valuable background information in regards to this project. It was written by K.D.G Fernando, A.A.P de Alwis, V. Karunaratne, and W.A.P.J. Premaratne for NANCO Ltd in Sri Lanka. In this investigation nano-TiO<sub>2</sub> and pigmentary TiO<sub>2</sub> were synthesized using titanyl sulfate precursor. Synthesized nano-TiO<sub>2</sub> was characterized by Raman spectroscopy, X-ray diffraction, scanning electron microscopy (SEM), and scanning transmission electron microscopy (STEM) methods. To access the photocatalytic activity of the nano-TiO<sub>2</sub>, the degradation of bromothymol blue in aqueous solution was observed under diffused light. This was conducted in a Petri dish two different ways. The first method involved taking one piece of 50 mm by 20 mm glass slide with thin films of TiO<sub>2</sub> on both sides. This was then dipped in a Petri dish containing the indication and then exposed to diffused light for 48 hours. The other method was the same experiment but instead, was carried out by adding 0.5 g of nano-TiO<sub>2</sub> powder in to a Petri dish rather than the glass slide. Many things were concluded at the end of the report. First, Nano-TiO<sub>2</sub> synthesized is in pure anatase form where it shows a higher level of photocatalytic activity. Also, the Nano-TiO<sub>2</sub> coated on glass showed a higher photocatalytic activity and self-cleaning effect that would be used effectively in building envelopes. This



method can be applied on a wide range of applications, involving the deposition of photocatalytic TiO<sub>2</sub> films on low thermally resistant materials, such as plastics (Fernando, 2010).

The background research summarized helped form a basic understanding of previous research conducted on the TiO<sub>2</sub>/UV process and many results were taken into consideration for the development of our methodology. Gathering background on the chemical itself also provided insight as to the specific behaviors of TiO<sub>2</sub> which is important for the completion of this project.

## 3.0 Methodology

### 3.1 Standard Curve

A standard curve was needed to relate the light absorption of methylene blue (MB) to the concentration of the solution. In order to accomplish this, 5 different samples were produced.

These were at the concentrations of:

- 0.001% (17.57 mg/L)
- 0.002% (35.14 mg/L)
- 0.003% (52.71 mg/L)
- 0.004% (70.28 mg/L)
- 0.005% (87.85 mg/L)

For each different concentration, a separate sample was made. The following masses of MB were measured out and added to 5 separate Erlenmeyer flasks:

- 0.001757 g
- 0.003514 g
- 0.005271 g
- 0.007028 g
- 0.008785 g

Then 100 mL of pure water from the lab was added to each Erlenmeyer flask. Then each sample was thoroughly mixed to ensure homogeneity. Using separate pipettes, a singular tapered cuvette was filled with each solution that was made.

After each cuvette was filled, the light absorbance at the wavelength of 395 nm was measured for each cuvette. A cuvette of pure water was used to zero the instrument used to measure the light absorbance to ensure reproducible results.

To make the standard curve, the 5 concentrations were plotted on the x-axis with the measured absorbance along the y-axis. A line of best fit was then added to the plotted points to form the standard curve that was used for the remainder of testing.

## 3.2 Small Scale Testing

Small scale testing began by following these steps in order to make a MB solution of known concentration:

1. Measure approximately 0.005 g of MB
2. Add measured MB to Erlenmeyer flask
3. Add 100 mL of pure water to flask
4. Mix until color is uniform throughout solution
5. Test absorbance at wavelength of 395 nm for solution in a spectrophotometer
6. Record absorbance as initial condition

### 3.2.1 First Trial

The first step was to prepare the 6 Petri dishes used for testing. These samples were for the following contact times:

- 0.5 hour
- 1 hour
- 8 hour
- 24 hour
- Control (24 hour)
- Control w/ Glue (24 hour)

The 0.5, 1, 8 and 24 hour samples were prepared in the following way:

1. Open a sterile Petri Dish
2. Cover the base of the dish with Superglue

3. Use a Q-tip or cotton swab to spread the Superglue evenly around the base
4. Apply Sigma-Aldrich 99.9% Titanium(IV) Oxide (Anatase) to wet Superglue by shaking enough onto the base to cover the entire surface (about 0.2 g)
5. Wait 5 minutes for Superglue to harden
6. Once hardened, use purified water to wash off any extra titania from the dish

The control w/ glue sample follows the prior steps 1-3, as to avoid any contact with the titania.

The Control sample only uses a plain sterile Petri dish, to avoid any interference from the Superglue and titania. Then all samples were subjected to the following procedure:

1. Pipette 5 mL of the MB solution into each Petri dish
2. Cover each sample with the top half of the Petri dish
3. Place each Petri dish directly under UVP an Analytik Jena Company 245 nm UV lamp for specified amount of time
4. When the contact time has been reached, use a syringe to fill a tapered cuvette with sample
5. Measure and record absorbance (in the spectrophotometer) for that sample immediately
6. Dispose of the Petri dish and cuvette for that sample

### 3.2.2 Second Trial

The next set of experiments was meant to test the effect of covering the samples. So, the first step was to prepare 5 Petri dishes used for testing. These samples were:

- Control with cover

- Control
- 5 mL sample with cover
- 5 mL sample
- 3 mL sample

With the exception of the control samples, each Petri dish underwent the same preparation, which was as follows:

1. Open a sterile Petri Dish
2. Cover the base of the dish with Superglue
3. Use a Q-tip or cotton swab to spread the Superglue evenly around the base
4. Apply titania to wet Superglue by shaking enough onto the base to cover the entire surface (about 0.2 g)
5. Wait 5 minutes for Superglue to harden
6. Once hardened, use pure water to wash off any extra titania from the dish

Then, all samples (including controls) were subjected to the following procedure:

1. Pipette 5 mL (3 mL for the “3 mL sample”) of the MB solution into each Petri dish
2. Cover the “Control with cover” and “5 mL sample with cover,” with the top half of the Petri dish
3. Place all samples under UVP an Analytik Jena Company 245 nm UV lamp
4. Use syringe to fill tapered cuvette with sample at the following times:
  - a. 0 hours
  - b. 1 hour
  - c. 2 hours

- d. 4 hours
  - e. 6 hours
  - f. 8 hours
5. Measure the light absorbance in the spectrophotometer at a wavelength of 395 nm
  6. Empty the liquid from the cuvette back into the Petri dish for that sample
  7. Place back under UVP an Analytik Jena Company 245 nm UV lamp and repeat at each testing time mentioned in Step 4

### 3.2.3 Third Trial

The next set of experiments was meant to test the effect of using a syringe filter when extracting the samples. Also for this testing, all of the samples remained uncovered for consistency. So, the first step was to prepare 4 Petri dishes used for testing. These samples were:

- Control
- Sample 1
- Sample 2
- Sample 3

With the exception of the control sample, each Petri dish underwent the same preparation, which was as follows:

1. Open a sterile Petri dish
2. Cover the base of the dish with Superglue
3. Use a Q-tip or cotton swab to spread the Superglue evenly around the base
4. Apply titania to wet Superglue by shaking enough onto the base to cover the entire surface (about 0.2 g)

5. Wait 5 minutes for Superglue to harden
6. Once hardened, use pure water to wash off any extra titania from the dish

Then, all samples (including controls) were subjected to the following procedure:

1. Pipette 5 mL of the MB solution into each Petri dish
2. Place all samples under UVP an Analytik Jena Company 245 nm UV lamp
3. Use a syringe to extract the sample from the Petri dish
4. Then attach the Acrodisc® Syringe Filter with HT Tuffryn® Membrane (.2 µm pore size) to the syringe tip and fill tapered cuvette with sample at the following times:
  - a. 0 hours
  - b. 2 hours
  - c. 3 hours
  - d. 4 hours
5. Measure the light absorbance in the spectrophotometer at a wavelength of 395 nm
6. Empty the liquid from the cuvette back into the Petri Dish for that sample
7. Place back under UVP an Analytik Jena Company 245 nm UV lamp and repeat at each testing time mentioned in Step 4

### **3.2.4 Fourth Trial**

The fourth trial mostly followed the exact same procedure as the third trial, with some slight alterations. For this round of testing, pure water was added to the sample to keep it at a constant volume of 5 mL for the duration of testing. There were 5 samples being tested for this trial, and they were:



- Control
- Sample 1
- Sample 2
- Sample 3
- Sample 4

Again, with the exception of the control sample, each Petri dish underwent the same preparation, which was as follows:

1. Open a sterile Petri dish
2. Cover the base of the dish with Superglue
3. Use a Q-tip or cotton swab to spread the Superglue evenly around the base
4. Apply titania to wet Superglue by shaking enough onto the base to cover the entire surface (about 0.2 g)
5. Wait 5 minutes for Superglue to harden
6. Once hardened, use pure water to wash off any extra titania from the dish

Then, all samples (including controls) were subjected to the following procedure:

1. Pipette 5 mL of the MB solution into each Petri dish
2. Place all samples under UVP an Analytik Jena Company 245 nm UV lamp
3. Use a syringe to extract the sample from the Petri dish
4. Then attach the Acrodisc® Syringe Filter with HT Tuffryn® Membrane (.2  $\mu\text{m}$  pore size) to the syringe tip and fill tapered cuvette with sample at the following times:
  - a. 0 hours
  - b. 1 hours

- c. 2 hours
  - d. 4 hours
  - e. 8 hours
5. Measure the light absorbance in the spectrophotometer at a wavelength of 395 nm
  6. Empty the liquid from the cuvette back into the Petri dish for that sample
  7. FOR SAMPLES 1 & 2 – At the 4 hour mark, add a necessary amount of pure water to bring the sample back up to a volume of 5 mL
  8. FOR SAMPLES 3 & 4 – At each hour mark, add a necessary amount of pure water to bring the sample back up to a volume of 5 mL
  9. Control sample needs no treatment
  10. Place back under UVP an Analytik Jena Company 245 nm UV lamp and repeat at each testing time mentioned in Step 4

### 3.2.5 Fifth Trial

The fifth trial was used to test the effectiveness of using a syringe filter on the titania/UV experiment. So, the first step was to prepare 4 Petri dishes used for testing. These samples were:

- Control
- Filtered Sample
- Unfiltered Sample

Again, with the exception of the control sample, both Petri dishes underwent the same preparation, which was as follows:

1. Open a sterile Petri dish
2. Cover the base of the dish with Superglue

3. Use a Q-tip or cotton swab to spread the Superglue evenly around the base
4. Apply titania to wet Superglue by shaking enough onto the base to cover the entire surface (about 0.2 g)
5. Wait 5 minutes for Superglue to harden
6. Once hardened, use pure water to wash off any extra titania from the dish

Then, all samples (including controls) were subjected to the following procedure:

1. Pipette 5 mL of the MB solution into each Petri dish
2. Place all samples under UVP an Analytik Jena Company 245 nm UV lamp
3. Use a syringe to extract the sample from the Petri Dish
4. For the filtered sample, attach the Acrodisc® Syringe Filter with HT Tuffryn® Membrane (.2 µm pore size) to the syringe tip and fill tapered cuvette with sample
5. For the unfiltered sample, just used the unfiltered syringe to fill the tapered cuvette
6. Extract liquid and take measurements for absorption at the following times:
  - a. 0 hours
  - b. 1 hours
  - c. 2 hours
  - d. 3 hours
  - e. 4 hours
7. Measure the light absorbance in the spectrophotometer at a wavelength of 395 nm
8. Empty the liquid from the cuvette back into the Petri dish for that sample
9. Place back under UVP an Analytik Jena Company 245 nm UV lamp and repeat at each testing time mentioned in Step 4

All samples were subjected to the same experimental set-up that can be seen in Figure 2 below. The UV light was positioned on top of two overturned beakers so there was enough room for the petri dishes to fit underneath.

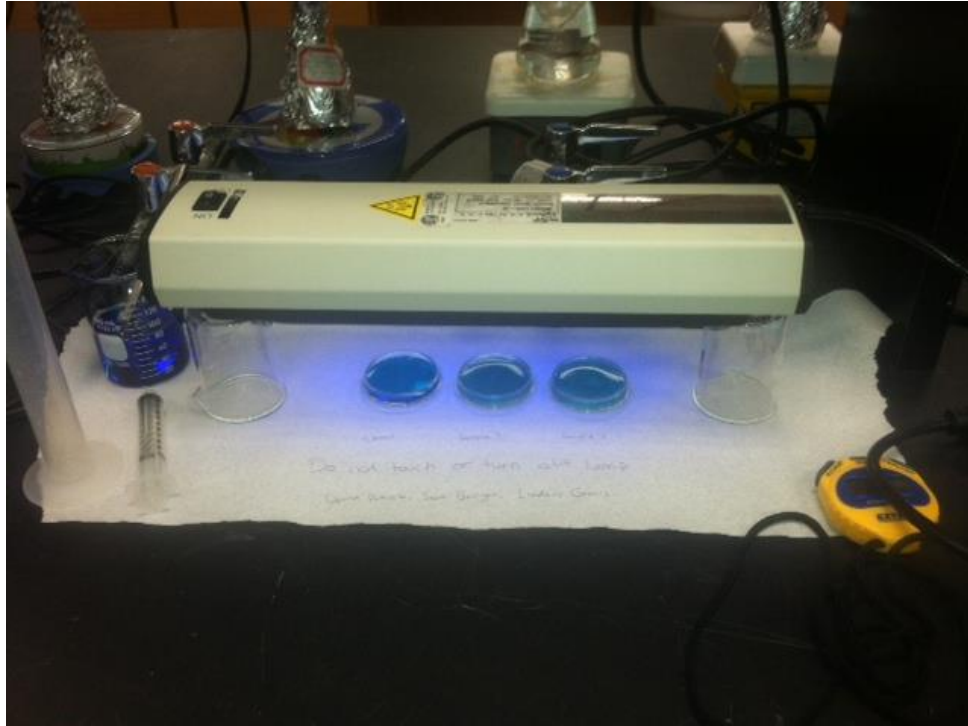


Figure 2 : Experimental apparatus

## **4.0 Results and Discussion**

### **4.1 Overview**

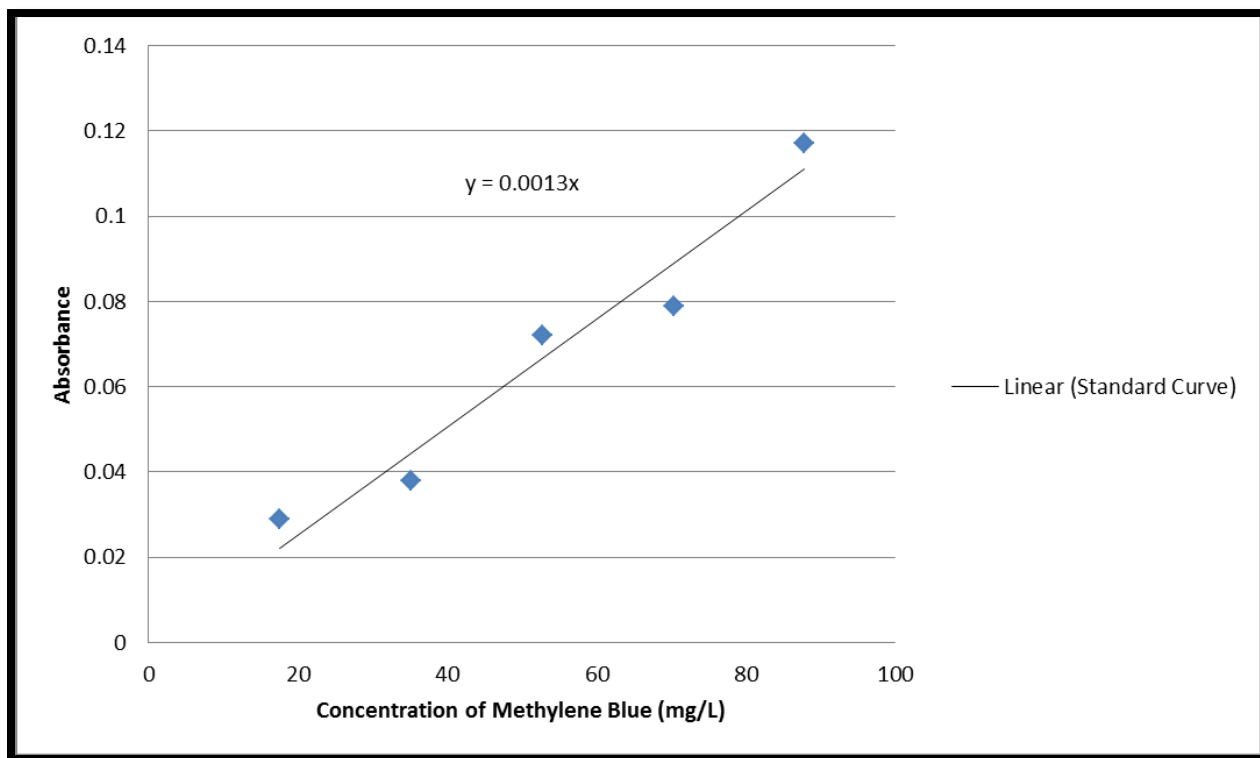
Before conducting any treatability experiments, it was necessary to create a methylene blue standard curve. The experiments were conducted in small petri dishes, Ti adhered with adhesives, and subjected to UV light over a span of 30 minutes to 24 hours. The results gathered from this portion of the project permitted a better understanding and knowledge about the TiO<sub>2</sub>/UV process and laid out the basis for future research for the degradation of organic contaminants in runoff water. It also provided a solid groundwork to test the feasibility of coating run-off collection basins with titania in order to reduce the concentration of harmful organics in water.

### **4.2 General Findings**

Throughout the experiments, some methods proved to be more successful than others. The most successful method involved uncovering the top of the petri dish, followed by sample filtration before being put into the spectrophotometer. All experimental runs included a control. The following sections provide more detailed descriptions of the results along with discussion of the resulting outcomes.

### 4.3 Standard Curve

The methylene blue standard curve created for this experiment showed light absorption of methylene blue in a spectrophotometer versus concentration. In Figure 3 the standard curve is shown.



**Figure 3: Methylene blue standard curve at 395 nm**

The standard curve is actually a linear regression as shown above in the figure. The wavelength of methylene blue absorption is 395 nm. The standard curve was utilized throughout our experiments. The standard curve was used during every test to find the concentration of the organic contaminant, methylene blue, in the solution at certain points in time. This helped our data show the effect of  $\text{TiO}_2/\text{UV}$  on the organic contaminants.

## 4.4 Main Results

### 4.4.1 Covered Samples with no Syringe filter

The first trial run, as described in the methodology section, was in covered petri dishes. Three different samples were run: a control, control with glue, and the titanium dioxide sample. The control sample was run to show how much methylene blue was degraded normally just by the ultraviolet radiation (no  $\text{TiO}_2$ ). The control with glue showed any affect the glue that was used had on the methylene blue and the last sample showed the results of the titanium dioxide on the methylene blue. The trial was run for 24 hours and the results for the  $\text{TiO}_2/\text{UV}$  treatment are shown in Figure 4. It was concluded from the results of the first trial that the titanium dioxide removed the methylene blue from the water. From Figure 4 we can see that within the first 10 hours the lowest concentration was reached. From that time on the concentration stayed fairly constant. This could be due to many different factors. The first trial was run using four different samples of titanium dioxide; so each was taken at a different hour to collect data. Each sample can react differently depending on any differences from human error or positioning under the ultraviolet light. From this discrepancy the next run would be using just one sample that would be replaced after each sample was drawn and put through the spectrophotometer. From our data, we concluded that the plastic cover was too thick and prevented some of the ultraviolet radiation from reaching the titanium dioxide. This was concluded due to the small decrease in concentration shown in Figure 4. The starting concentration was 80 mg/L, as shown in Figure 4.

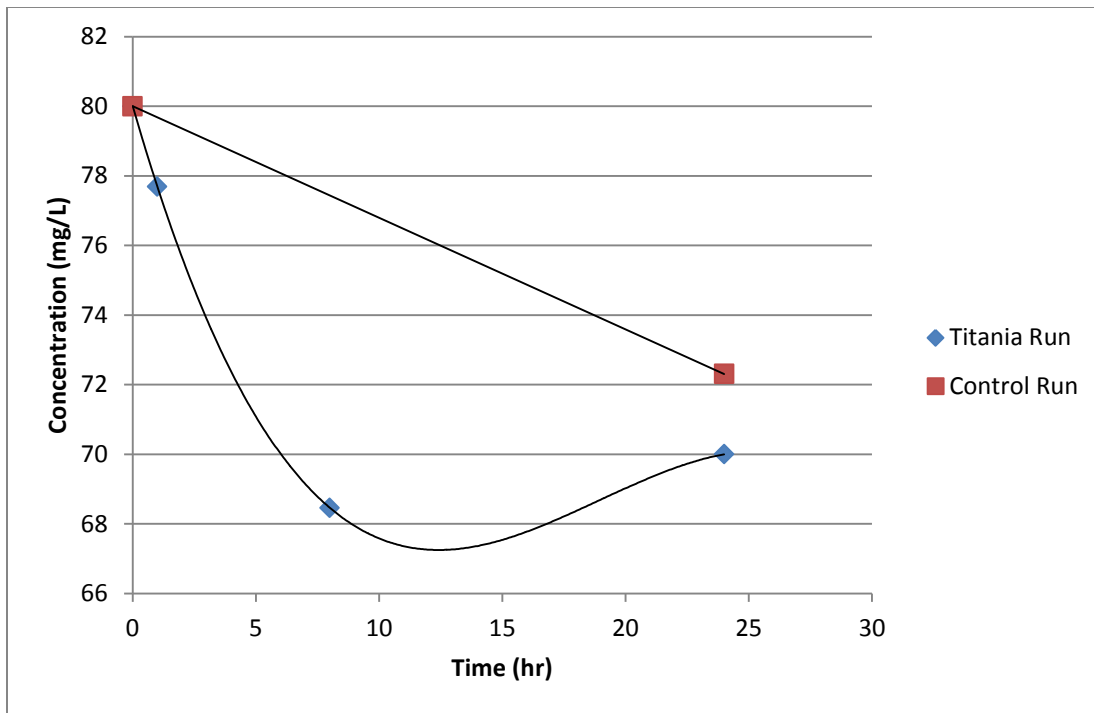


Figure 4: First Trial – Concentration Change for TiO<sub>2</sub>/UV treated Sample vs. Control

In Figure 4 the control run is compared to the titanium dioxide run. We can see that the final concentrations are similar the control being 72.3 mg/L and the TiO<sub>2</sub>/UV treated being 70 mg/L. This data showed that at a time of 8 hours in this experiment there was a difference between the titania and control samples of 8.5 mg/L showing that the titania did affect the methylene blue concentration and removed a large amount faster than just ultraviolet light. Also from the data it can be seen that during the TiO<sub>2</sub>/UV run there was very little change after the eight hour mark, which could be due to the fact that the titania was used up, or that there was not enough surface area for the titania to come in contact with the methylene blue. In the next trial a smaller amount of stock methylene blue solution was added to try and let titania remove all of the organic material in the petri dish. Overall our data from the first trial showed that the titania was a feasible product to remove organic material from wastewater and that the glue used to bind titania to the petri dish was strong enough and did not affect the data.



#### 4.4.2 Uncovered Samples with no Syringe filter

Through discussions and data observed in the first trials a change in the methodology was made. The samples were left uncovered during testing to allow the UV to reach the  $\text{TiO}_2$ . The first experiment shows that the UV/ $\text{TiO}_2$  is a viable process to remove organic material from water supplies.

As described in the covered sample section there were thoughts that the ultraviolet light was not passing through the plastic cover and the methylene blue solution. In our second experiment both observations were tested. The amount of methylene blue was lowered from 5ml to 3ml and the covers were removed for three of the samples. The graph in Figure 5 shows the 5 ml run and the 3 ml run on the same graph.

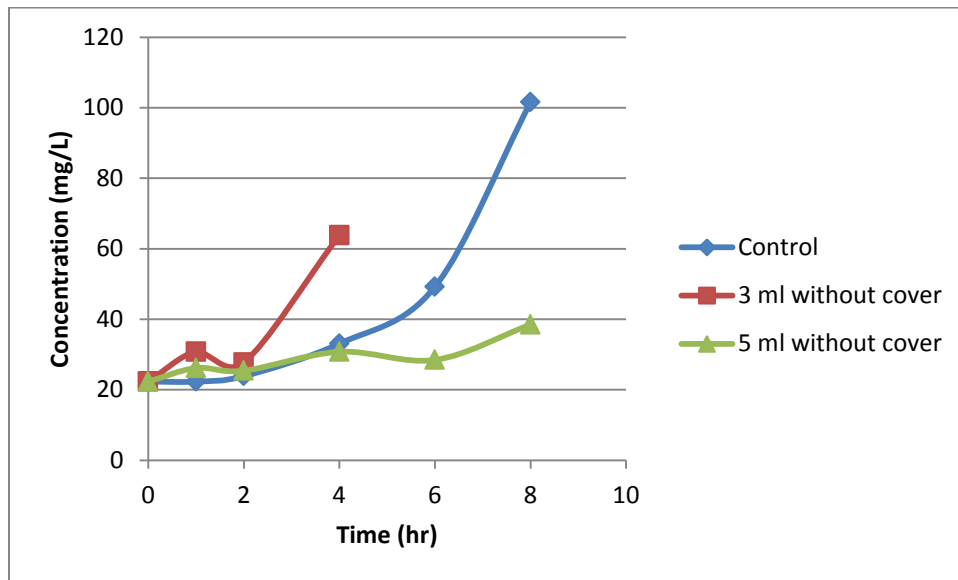


Figure 5: Second Trial –  $\text{TiO}_2$ /UV treated runs for initial MB concentration of 22.3 mg/L

Both runs were uncovered. Although the concentration increased, there was an evident color difference. The 3 ml run, referring to the amount of methylene blue solution in the petri dish,

turned clear after only two hours while it took the 5 ml run four hours to reach that same color. Although the 3 ml run in color seemed to react much quicker to the TiO<sub>2</sub>/UV it evaporated after only 3 hours not giving a large run time for the experiment. The conclusion from this test is that the smaller the amount of methylene blue solution in the petri dishes, the faster are the reactions. The only drawback is that there is not a large run time to obtain during the test due to evaporation. Taking these two factors into account we decided that the most effective method was to keep 5ml of the methylene blue solution in the petri dishes. Figure 6 shows the two runs of 5ml of methylene blue solution but one with the petri dish cover on and the other with the cover off.

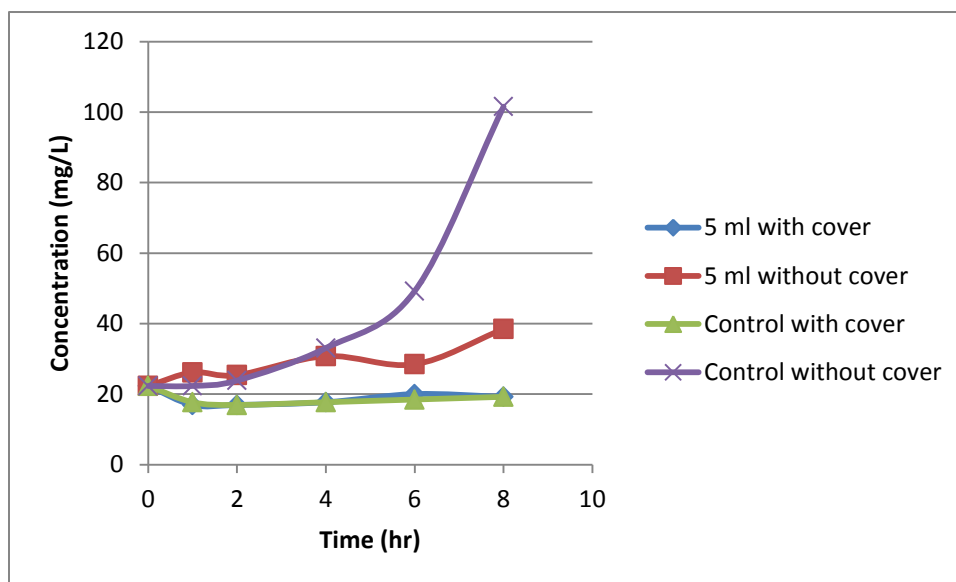


Figure 6: Second Trial – Titania treated runs for covered and uncovered 5 mL samples vs. Controls

The data from the graph supports the 5 ml run with the cover as the more effective run although through observation we found this not to be true. Along with the 5ml and 3ml samples we found that the color decreased quicker with the 5ml run. The 5ml run without the cover turned to a clear color after 4 hours while the covered run never completely got to that clear color through the entire eight hours of testing. Although our data show some discrepancy the decision made

from the experiment was to remove all the covers instead of keeping the covers on the petri dishes. The outside contamination was a problem during our experiments but a solution to this problem is to use a syringe filter which is discussed in the following section,

Outside contamination is a possibility when the solutions are uncovered during testing. In the lab, although it is clean, there can be dust and other particles floating around. Particles were in fact found in our solution during our experiment. This was concluded by the results given from the spectrophotometer. Although the samples were turning a clear color, the problem was that some of the particles would pass through the syringe and be floating in the solution when placed into the spectrophotometer. This affected the results because spectrophotometer measures the absorbance of light through the solution, so these particles caused interference. Therefore, the spectrophotometer was reading a higher concentration than the actual concentration of the solution. Figure 7 below displays this interference.

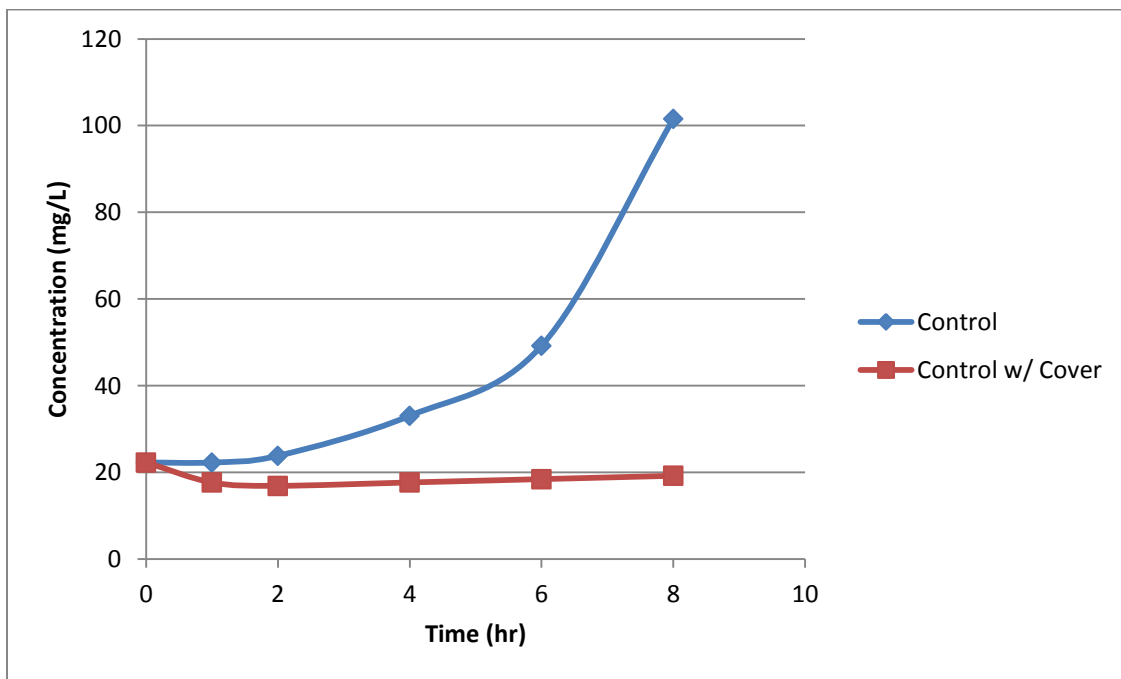


Figure 7: Second Trial - Control vs. Control with cover

A control run is a sample of just methylene blue. From our first experiments in the covered samples section of the paper, we found that there is some decrease in methylene blue concentration without any additional chemicals in the solution. In the graph in Figure 7 we can see that the control with the cover shows this slight decrease in concentration. The control solution on the other hand shows a large increase in concentration and this is due to interference. Although the solutions in Figures 5 through 7 show that with a covered solution better results are found, the color difference is not great showing that the petri dish cover limits the amount of ultraviolet light reaching the titanium dioxide. In order to fix this problem a solution was found to use syringe filters. The filters would be used when transferring the solution from the petri dish to the spectrometer cells. The solution would go through the filter removing the particles and keeping just the methylene blue in the solution.

One of the largest effects of removing the petri dish cover is evaporation. This creates a limited time for our experiments to run as well as removes the methylene blue solution. Shown in figure 5 the 3ml solution completely evaporates in four hours, leaving the titanium dioxide on the bottom of the petri dish with an amount of methylene blue that did not evaporate with the water in the solution. When this occurs in the environment it leaves the organic materials in the soil which is why this experiment is trying to remove the organic materials before it reaches this point. The result from our experiment has the organic material on the titanium dioxide in the retention pond instead of out in the environment. Although this is the result that we would like from our experiment the results are not conducive to show how effective  $\text{TiO}_2/\text{UV}$  oxidation is for removing organic material. To counteract this problem the amount of methylene blue solution was kept at 5ml which takes hours to evaporate. It has been shown that the methylene blue concentration has been greatly decreased before that amount of time has elapsed. Another

technique is to add deionized water to the petri dish after the solution has evaporated to dissolve the methylene blue back into the water so the titanium dioxide can react with the methylene blue. After testing this technique, the results showed that there was very little methylene blue left that the solution got to a certain concentration where the spectrophotometer could not detect light absorption any lower.

The results from these experiments showed that the elimination of the petri dish cover is a necessary step in our methods. This allows ultraviolet light to come into contact with the titanium dioxide. Outside interference is allowed showing how the environment will react with the solution in the retention pond. Finally, showing that the biggest variable that our experiment has when removing the cover is evaporation, this variable is inevitable when the solution is interacted with the environment and in order to minimize the effects of evaporation the process must be changed to find the quickest and most effective way to remove the organic materials from the solution in order for no contaminant to evaporate into the environment. Overall the positive factors of removing the petri dish cover outweigh the negative factors and for the rest of the experiments the petri dish cover was removed and a syringe filter used to remove any interference caused by particles settling in our solution.

#### **4.4.3 Uncovered Samples with Syringe filter**

The most successful method for our testing was using samples that were not covered by the top of the petri dish and when all samples were filtered before being put into the spectrophotometer. For the three groups of testing done with this method, we saw averages of 57.1%, 87.1% and 57.9% decrease in the amount of MB in our solution. This is a significant decrease compared to what our control samples showed for concentration decay. This test

produced an accurate small scale model for how a detention pond would respond to the same TiO<sub>2</sub>/UV treatment.

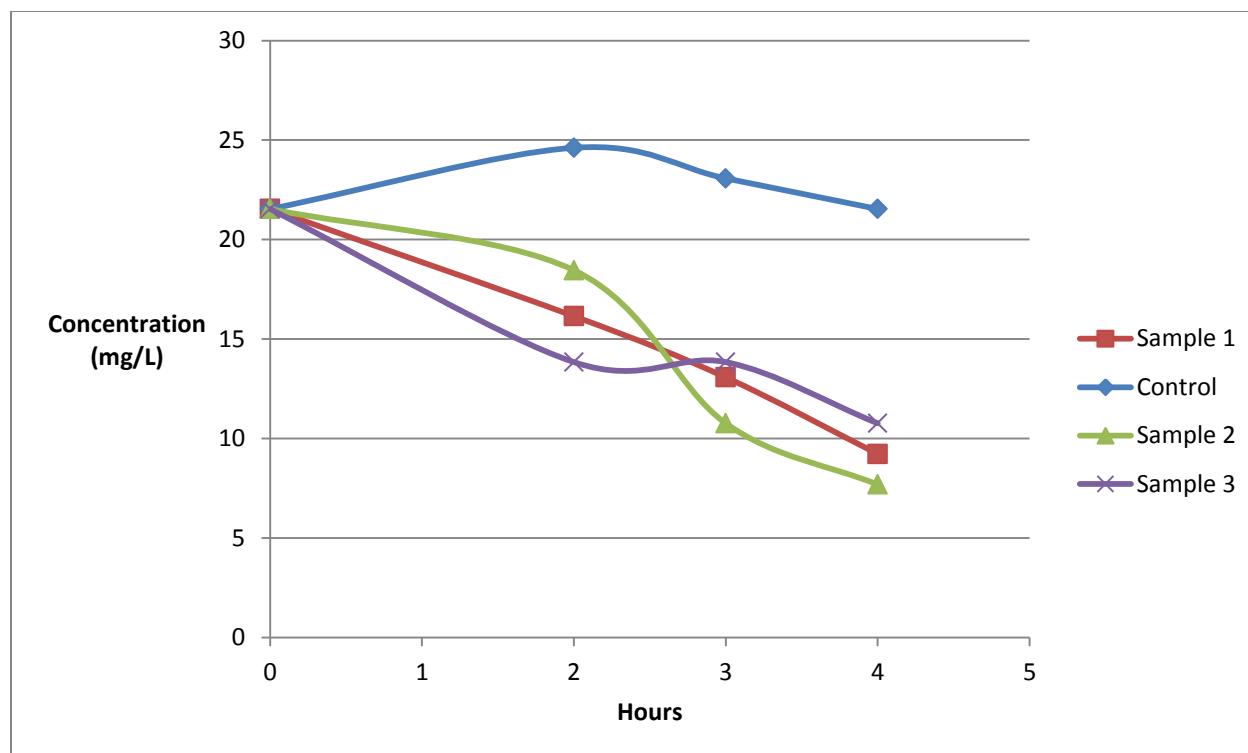
#### **4.4.3.1 Third Trial**

The first group of samples to use this methodology was the third trial. This trial had 3 samples with the normal titania coating on the petri dish and one dish without it as a control. All three test samples saw a large decrease in the amount of MB in solution, with the maximum being a 64.2% decrease in concentration. Table 1 shows the drop in light absorbance and therefore the concentration of the sample after our 4 hour testing time frame.

Table 1: Third Trial Data

	<b>Change in Absorption</b>	<b>Change in Concentration</b>	<b>% Change in Concentration</b>
<b>Control</b>	0.0	0.0	0.0 %
<b>Sample 1</b>	0.16	12.31	57.1 %
<b>Sample 2</b>	0.18	13.85	64.3 %
<b>Sample 3</b>	0.14	10.77	50.0 %

As one can see, the samples with the titania coating that were subjected to the UV light had a considerable drop in the concentration of the organic compound, MB. This is consistent with what we had predicted would happen with the proper TiO<sub>2</sub>/UV light treatment. Within the first hour all of the samples saw a decrease in MB concentration which indicated that the chemical mechanism that degrades the organic compound happens fairly quickly. The data can be seen in the Figure 8.



**Figure 8: Third Trial - Uncovered/Filtered Samples vs. Control**

The average decrease in concentration for these three separate samples was 57.1 %. This drastically deviates from the control sample that stayed at the same concentration after the 4 hours of testing. With the amount of organic compound in solution more than cut in half, we concluded that the TiO<sub>2</sub>/UV light combination can realistically degrade organic compounds in solution. Through observation we were able to see the titania/UV working when samples were left out they lost their blue coloring very quickly. This confirms the data we gathered from the spectrophotometer, which also indicated a significant drop in organic compound concentration in our solution. This fits with our initial hypothesis that this is a feasible technique to lower the concentration of organic compounds in a solution, such as detention ponds.



#### 4.4.3.2 Fourth Trial

This group of samples also had pure water added to them to maintain a constant volume during testing (Further details are in the Methodology). The trial had 4 samples with the normal titania coating on the petri dish and one dish without it as a control. The 4 test samples exhibited a significant decrease in MB concentration, with the maximum being a 94.3% decrease. This trial was the most effective in reducing the concentration of organic compound in solution.

Table 2 shows the drop in absorbance and therefore the concentration of the sample after our 8 hour testing time frame.

Table 2: Fourth Trial Data

	<b>Change in Absorption</b>	<b>Change in Concentration</b>	<b>% Change in Concentration</b>
<b>Control</b>	(0.45)	(34.6)	(128.6 %)
<b>Sample 1</b>	0.032	24.6	91.4 %
<b>Sample 2</b>	0.034	19.2	71.4 %
<b>Sample 3</b>	0.0347	24.6	91.4 %
<b>Sample 4</b>	0.0348	25.4	94.3 %

It's apparent that the samples with the titania coating that were subjected to the UV light had a considerable decrease in the concentration of the organic compound, MB. This behavior is different from what was observed from the control sample, which actually showed an increase in concentration over the 8 hour testing period. The decrease in organic compound concentration in the 4 testing samples is consistent with what we had predicted would happen with the proper

TiO<sub>2</sub>/UV light treatment. Within the first hour all of the samples saw a decrease in MB concentration which indicates that the chemical mechanism that degrades the organic compound happens fairly instantly. Also, the 8 hour testing period resulted in almost all of the organic material being degraded to the point of almost disappearing. For example, the initial concentration of 26.9 mg/L for samples 3 and 4 was reduced to final concentrations of 2.3 mg/L and 1.5 mg/L, respectively. The data can be seen in Figure 9.

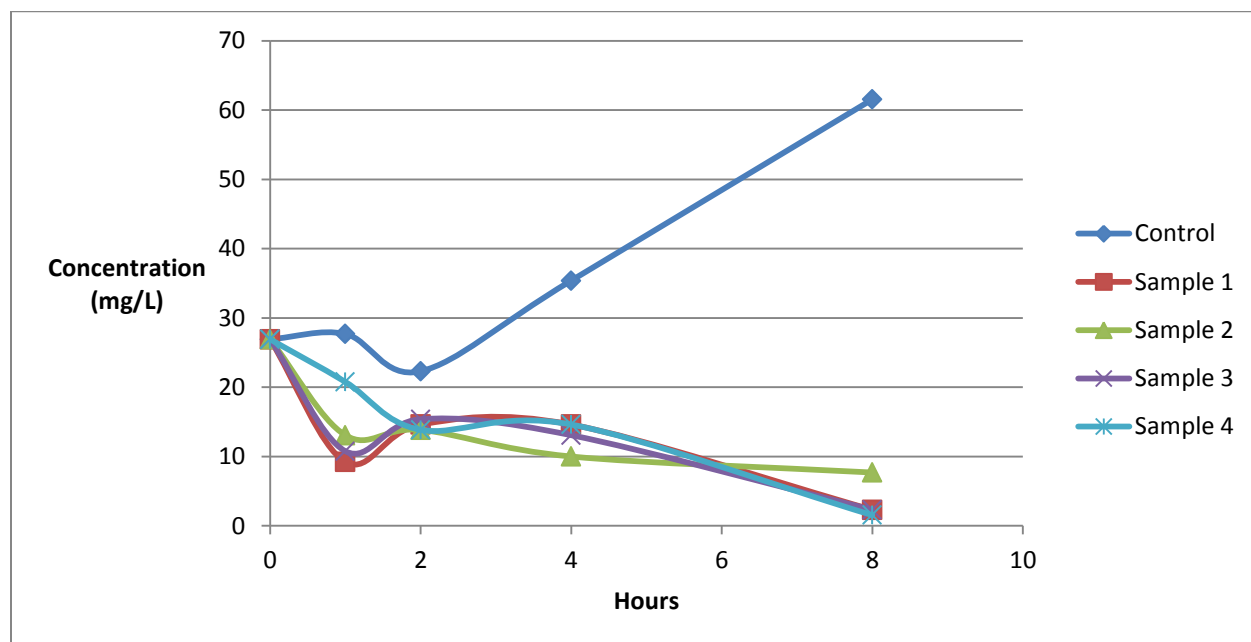


Figure 9: Fourth Trial - Uncovered/Filtered Samples vs. Control

The average decrease in concentration for these 4 separate samples was 87.1 %. This drastically deviates from the control sample that almost doubled in concentration, possibly due to evaporation of the water in the solution. With the amount of organic compound in solution reduced to nearly zero, again we concluded that the TiO<sub>2</sub>/UV light combination can realistically degrade organic compounds in solution. Also, through visual observation we were able to see the titania/UV working. When samples were left out they lost their blue coloring very quickly, while the control stayed a consistent shade of royal blue. This confirmed the data we gathered

from the spectrophotometer, which also indicated a significant drop in organic compound in our solution. Once again, this fit with our initial hypothesis that this is a feasible technique to lower the concentration of organic compounds in a solution, such as detention ponds.

#### 4.4.3.3 Fifth Trial

The last group of samples to use this methodology was the Fifth Trial. This trial had 2 samples with the normal titania coating on the petri dish and one dish without it as a control. A syringe filter was used on one of the titania treated samples when a sample was taken. This was to show how important the syringe filter was in eliminating all particles that would interfere with the spectrophotometer results. The sample that used the syringe filter saw an expected 57.9 % decrease in MB concentration, while the unfiltered sample had essentially no readable change in concentration (-2.63 %). Table 3 shows the drop in absorbance and therefore the concentration of the sample after our 4 hour testing time frame.

**Table 3: Fifth Trial Data**

	<b>Change in Absorption</b>	<b>Change in Concentration</b>	<b>% Change in Concentration</b>
<b>Control</b>	0.012	9.23	31.6 %
<b>Sample 1 – Filtered</b>	0.022	16.9	57.9 %
<b>Sample 2 – No filter</b>	(0.001)	(0.77)	(2.63 %)

As expected, the sample with the titania coating that was subjected to the UV light and sample filtering had a considerable drop in the concentration of the organic compound, MB. This is consistent with what was predicted would happen with the proper TiO<sub>2</sub>/UV light treatment and use of the syringe filter to deter any interference from particles in the sample. The

data for the unfiltered sample clearly shows that something is hindering the spectrophotometer's ability to measure the decrease in light absorption at a wavelength of 395 nm. The absorbance remains constant, and even stays higher than the control sample. The data can be seen in the graph in Figure 10.

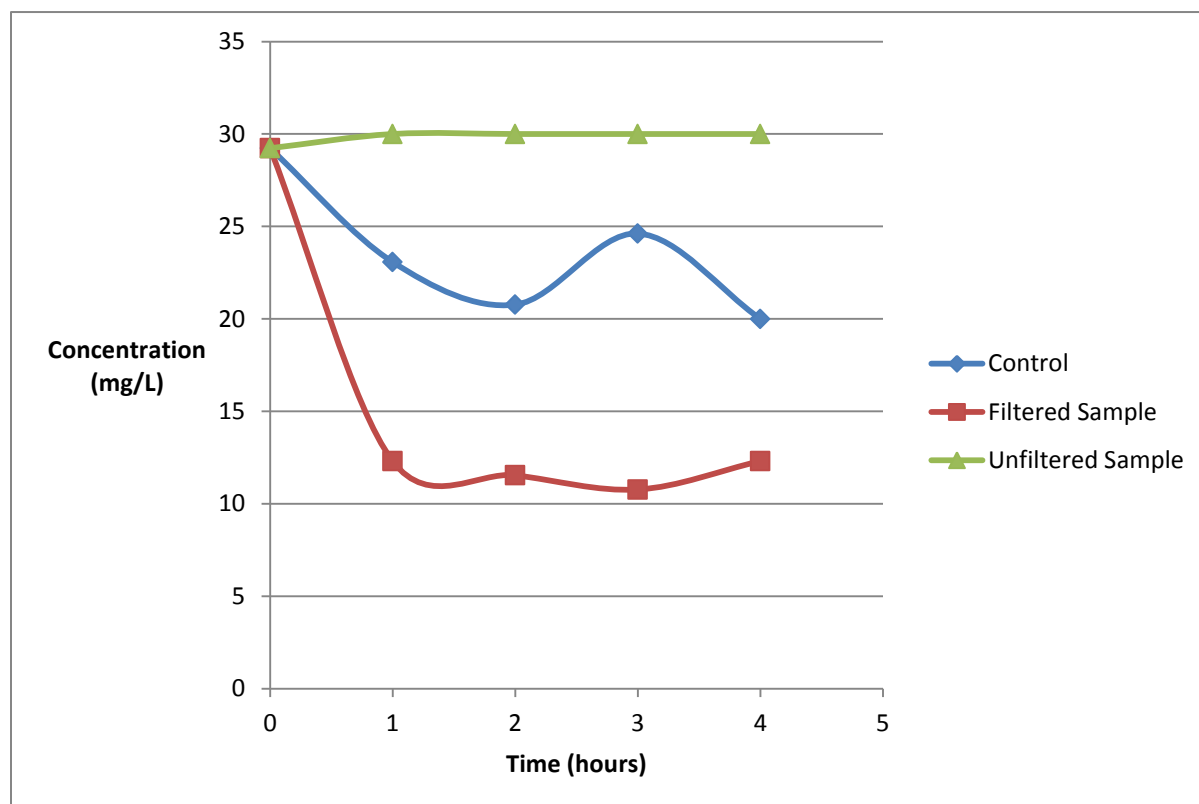
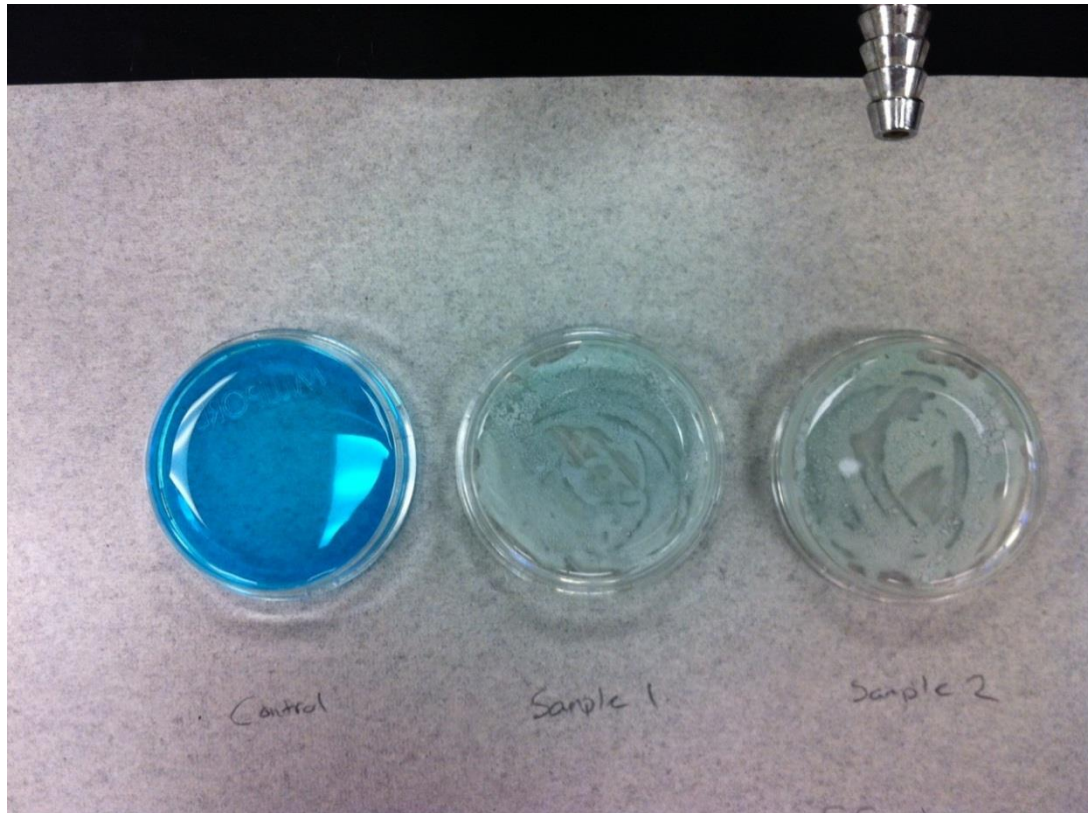


Figure 10: Fifth Trial - Filtered vs. Unfiltered Samples

Although the concentration of methylene blue in the control sample decreased in a similar pattern as the filtered sample, there was still a difference of 26.3 % between their total decreases in MB concentration. This indicates that the titania/UV technique when used in combination with a syringe filter is an effective way to diminish the concentration of organic compounds in solution. Also, through visual observation we were able to see the titania/UV working when samples were left out they lost their blue coloring very quickly, while the control stayed a consistent shade of royal blue. As can be seen below, this was the case for both the

titania treated samples, indicating that the only issue with the unfiltered sample was particle interference.



**Figure 11: Fifth Trial - Control, Sample 1 & Sample 2 (Hour 3)**

This confirmed the data we gathered from the spectrophotometer for our filtered sample, which also indicated a significant drop in organic compound in our solution. Also, the color change in both samples indicates an obvious reduction in the concentration of MB, despite what the spectrophotometer read. Once again, this fits with our initial hypothesis that this is a feasible technique to lower the concentration of organic compounds in a solution, such as detention ponds.

## 5.0 Conclusions and Recommendations

The environment suffers from different toxins from manmade products. Toxic organic compounds such as gasoline and oil are two large contributors to the detriment of our environment. These two compounds are found in vehicles which are one of the largest contributors to pollution. The area of concern of our project is the collection of these organic compounds off of parking lots and other areas where these contaminants can leak from vehicles. In order to remove these compounds before they evaporate or run off into the environment around the lots, a basin will be created to collect the water runoff after a storm with the contaminants. From there titanium dioxide along with UV radiation from sunlight will remove the organic compounds from the water before they are released into the environment.

Titanium dioxide with UV radiation was tested in our experiments to observe the effect on organic compounds and to discover ways to combine it a concrete basin. From the data collected about this process and organic compounds it can be concluded that the titanium dioxide/UV process removes the organic contaminants at high rates. One test revealed that when an initial concentration of 30 mg/L of methylene blue in solution was used, the titanium dioxide/UV removed 20mg/L in the first three hours when it came in contact with methylene blue. This compares to the 5 mg/L removed in the untreated control sample after four hours. Our experiments showed that titanium dioxide/UV is a viable process to remove toxic organic materials such as gasoline and oil from water before it is released into the atmosphere and environment. The second part of our experiment found an effective way to bind titanium dioxide to a petri dish and to find possible ways to bind it to a concrete basin. Through our experiment we found that Superglue held titanium dioxide very well to petri dishes also showing that titanium dioxide will combine with cement as long as it has the proper time to dry.

The next step for this project is to go to full scale testing. Through small scale testing we proved that the titanium dioxide worked effectively in removing the target organic compound and found possible ways to bind titanium dioxide. Large scale testing should provide more data on how the basin should be created to improve contact between the titanium dioxide and toxic organic materials. Another experiment which should be looked into is how long titania is effective. Due to the limits of our experiments we could not test large amounts of methylene blue and titanium dioxide because of our limited budget. Additionally, tests should be conducted for longer periods of time so more precise data can be obtained. This was a successful project and showed the capability of titanium dioxide photocatalysis. From the scope of our results and recommendation there is room for this project to grow and become an even more important to the reduction of pollution in the environment. From our research we hope that the effect of titanium dioxide becomes a widely used material to help eliminate pollution.

After the completion of small scale testing, large scale testing should be considered. We suggest the following procedure for future work. We recommend a testing reservoir be created to accomplish this objective using a concrete basin created with a pavement lining. The process to create the reservoir is shown below:

1. Calculate the area needed to create a basin large enough and at the correct depth to recreate the most effective method found in small scale testing.
2. Find an area where the basin can be stored and creating depending on the size of the basin.
3. Create the reservoir using concrete and pavement
  - a. To create the reservoir a mold must be created to hold the concrete while it is drying.

- b. After the concrete mixture dries the titanium dioxide is adhered to the concrete using the methods described in the earlier methods.
4. Once this mold has dried, the reservoir can be filled with a mixture of methylene blue dye and water at a known concentration. The height of this mixture should be adjusted to match the most effective height in small scale testing.
5. The reservoir can then be introduced to natural sunlight or different UV wavelengths to show the effect of titanium dioxide on organic materials.
6. After the mixture has been affected by the UV light for an amount of time described in the small scale experimentation above the remaining mixture will be collected and removed and using the same methods described above tested to see the remaining organic contaminant left in the mixture
7. The final test completed will be a real world application using organic compounds such as gasoline and oil in the water mixture. The same procedure will be followed to show the effect of titanium dioxide on these two organic contaminants.



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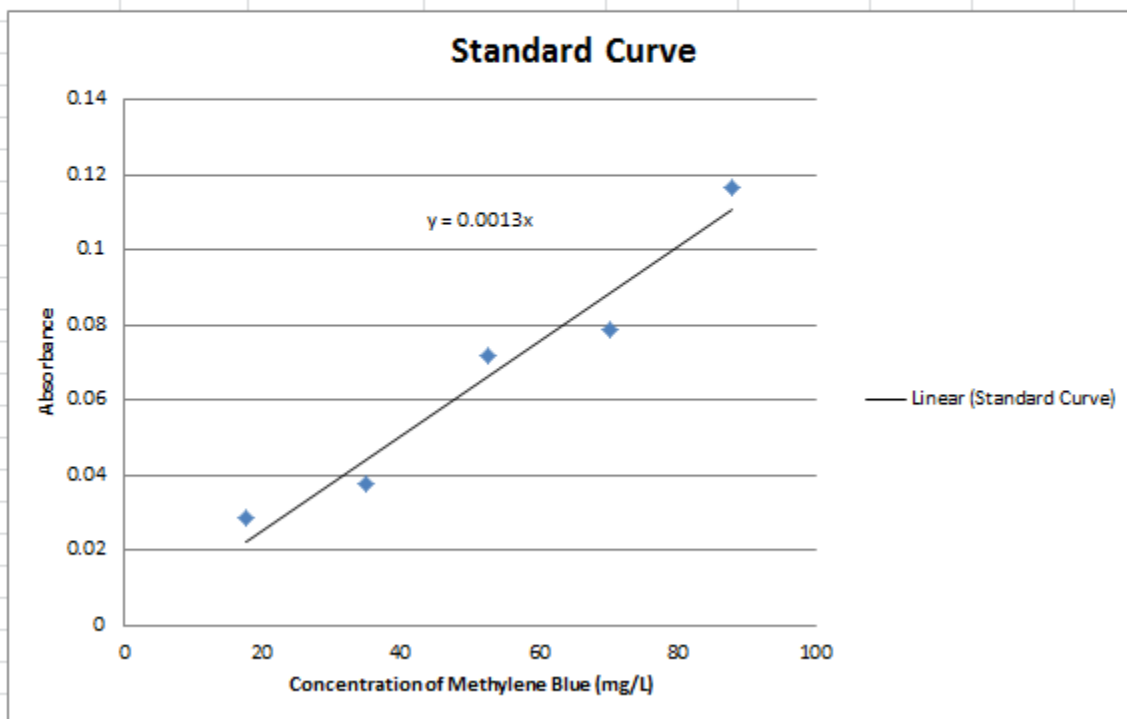
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## 7.0 Appendices

### 7.1 Standard Curve

	A	B	C	D	E	F	G	H	I
1	MB wt %	mg/L	Mass of MB (g)	Vol of H2O (mL)	Absorbance			Wavelength	
2	0.001%	17.57	0.001757	100	0.029			395	
3	0.002%	35.14	0.003514	100	0.038				
4	0.003%	52.71	0.005271	100	0.072				
5	0.004%	70.28	0.007028	100	0.079				
6	0.005%	87.85	0.008785	100	0.117				



Slope of the Standard Curve line:

$$y = 0.0013x \text{ (at 395 nm)}$$

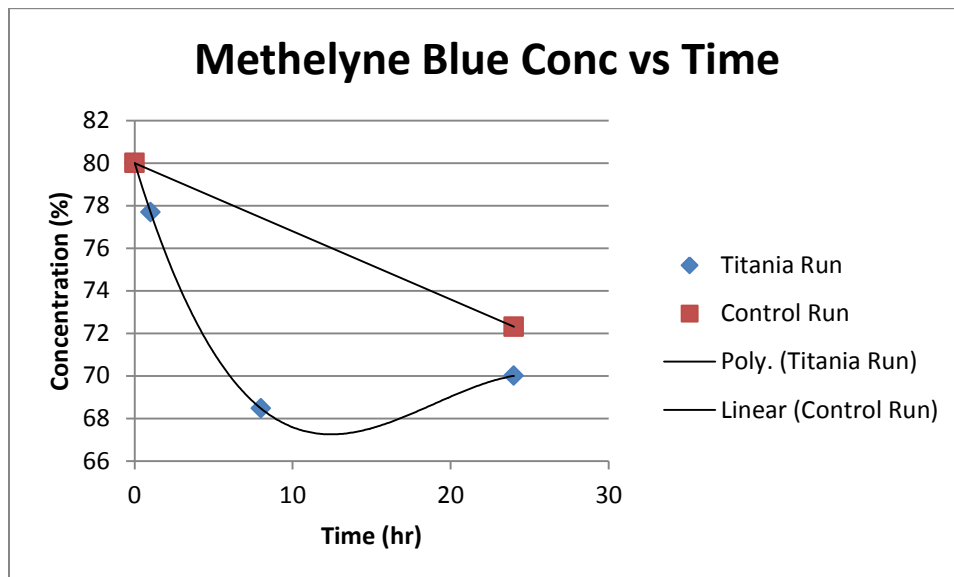
So,

$$\frac{\text{Absorbance}}{0.0013} = \text{Concentration}$$

## 7.2 First Trial

	A	B	C
1	Stock Solution		
2	Methelyne Blue	Water	Absorbance
3	.0093 g	100 ml	0.104
4			

6	7	Starting Absorbance	Color (visual)	Concentration Start (mg/L)	Amount of Stock Solution (ml)	Amount of Titinia (g)	Time (hr)	Ending Absorbance	Ending Color (visual)	Concentration Final
8	Control	0.104	Darker Blue	80	5	0	0	0.104	Darker Blue	80
9	Control	0.104	Darker Blue	80	5	0	24	0.094	dark blue	72.30769231
10	Control w/ Glue	0.104	Darker Blue	80	5	0	24	0.095	dark blue	73.07692308
11	0.5 Hour	0.104	Darker Blue	80	5	0.2	0.5	0.092		70.76923077
12	1 Hour	0.104	Darker Blue	80	5	0.2	1	0.101	Darker Blue	77.69230769
13	8 Hour	0.104	Darker Blue	80	5	0.2	8	0.089	dark blue	68.46153846
14	24 Hour	0.104	Darker Blue	80	5	0.2	24	0.091	dark blue	70
15	0 Hour	0.104	Darker Blue	80	5	0.2	0	0.104	Darker Blue	80
16										



### 7.3 Second Trial

	B	C	D	E
	Stock Solution			
	Methelyne Blue	Water	Absorbance	Concentration
		100 ml	0.029	22.30769231

	A	B	C	D
6				
7		Run	Absorbance	Concentration
8	<b>Control w/ cover</b>	<b>0</b>	0.029	22.30769231
9		1	0.023	17.69230769
10		2	0.022	16.92307692
11		4	0.023	17.69230769
12		6	0.024	18.46153846
13		8	0.025	19.23076923
14	<b>Control</b>	<b>0</b>	0.029	22.30769231
15		1	0.029	22.30769231
16		2	0.031	23.84615385
17		4	0.043	33.07692308
18		6	0.064	49.23076923
19		8	0.132	101.5384615
20	<b>5mL w/ cover</b>	<b>0</b>	0.029	22.30769231
21		1	0.022	16.92307692
22		2	0.022	16.92307692
23		4	0.023	17.69230769
24		6	0.026	20
25		8	0.025	19.23076923
26	<b>5 mL</b>	<b>0</b>	0.029	22.30769231
27		1	0.034	26.15384615
28		2	0.033	25.38461538
29		4	0.04	30.76923077
30		6	0.037	28.46153846
31		8	0.05	38.46153846
32	<b>3 mL</b>	<b>0</b>	0.029	22.30769231
33		1	0.04	30.76923077
34		2	0.036	27.69230769
35		4	0.083	63.84615385
36		6		0
37		8		0

## 7.4 Third Trial

Water                      Absorbance      Concentration  
 100 ml                      0.028              21.53846154

7		Absorbance	Color (visual)	Concentration
8	<b>Control - 0</b>	0.028	Royal	21.53846154
9	2	0.032	Royal Blue	24.61538462
10	3	0.03	Royal Blue	23.07692308
11	4	0.028		21.53846154
12	<b>Sample 1 - 0</b>	0.028	Royal	21.53846154
13	2	0.021	Almost clear	16.15384615
14	3	0.017	Almost clear	13.07692308
15	4	0.012		9.230769231
16	<b>Sample 2 - 0</b>	0.028	Royal	21.53846154
17	2	0.024	Almost clear	18.46153846
18	3	0.014	Almost clear	10.76923077
19	4	0.01		7.692307692
20	<b>Sample 3 - 0</b>	0.028	Royal	21.53846154
21	2	0.018	Almost clear	13.84615385
22	3	0.018	Almost clear	13.84615385
23	4	0.014		10.76923077

## 7.5 Fourth Trial

Methelyne  
 Blue                      Water                      Absorbance                      Concentration  
    100 ml                                      0.035                      26.92307692

	Run	Absorbance	Color (visual)	Concentration	Water Added to Sample (mL)
<b>Control</b>	<b>0</b>	0.035	Royal	26.92307692	
	1	0.036	Royal	27.69230769	0
	2	0.029	Royal	22.30769231	0
	4	0.046	Royal	35.38461538	0
	8	0.08	Royal	61.53846154	0
<b>Sample 1</b>	<b>0</b>	0.035	Royal	26.92307692	
	1	0.012	Turquoise	9.230769231	0
	2	0.019	Almost Clear	14.61538462	0
	4	0.019	Almost Clear	14.61538462	0
	8	0.003	Clear	2.307692308	3
<b>Sample 2</b>	<b>0</b>	0.035	Royal	26.92307692	
	1	0.017	Turquoise	13.07692308	0
	2	0.018	Almost Clear	13.84615385	0
	4	0.013	Almost Clear	10	0
	8	0.01	Clear	7.692307692	3
<b>Sample 3</b>	<b>0</b>	0.035	Royal	26.92307692	
	1	0.014	Turquoise	10.76923077	0.4
	2	0.02	Almost Clear	15.38461538	0
	4	0.017	Almost Clear	13.07692308	1.4
	8	0.003	Clear	2.307692308	1
<b>Sample 4</b>	<b>0</b>	0.035	Royal	26.92307692	
	1	0.027	Light Blue	20.76923077	0.4
	2	0.018	Almost Clear	13.84615385	0
	4	0.019	Almost Clear	14.61538462	1.2
	8	0.002	Clear	1.538461538	1

## 7.6 Fifth Trial

7		Run	Absorbance	Color (visual)	Concentration
8	<b>Control</b>	<b>0</b>	0.038	Royal	29.23076923
9	No Titania	1	0.03	Royal	23.07692308
10		2	0.027	Royal	20.76923077
11		3	0.032	Royal	24.61538462
12		4	0.026	Royal	20
13	<b>Sample 1</b>	<b>0</b>	0.038	Royal	29.23076923
14	Filter	1	0.016	Turquoise	12.30769231
15		2	0.015	Almost Clear	11.53846154
16		3	0.014	Almost Clear	10.76923077
17		4	0.016	Almost Clear	12.30769231
18	<b>Sample 2</b>	<b>0</b>	0.038	Royal	29.23076923
19	No filter	1	0.039	Turquoise	30
20		2	0.039	Almost Clear	30
21		3	0.039	Almost Clear	30
22		4	0.039	Almost Clear	30