

The Effects of Concentration, Stir Rate, and Processing Temperature on the Iridescence of  
Polymethyl Methacrylate Nanoparticles

David Baruela

Materials Engineering Department

California Polytechnic State University, San Luis Obispo

Advised by Dr. Katherine C. Chen

June 8, 2012

# Approval Page

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Author: David Baruela

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CAL POLY STATE UNIVERSITY

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Prof. Katherine Chen

Faculty Advisor

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Signature

Prof. Trevor Harding

Department Chair

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Signature

## Abstract

Synthetic opals were synthesized by creating polymethyl methacrylate (PMMA) nanospheres in order to determine which conditions would create the best iridescent samples. The factors affecting the iridescence were nanosphere concentration, stir rate, and processing temperature. PMMA solutions were made by adding 17 mg of granular azobis to a solution of 16 mL of distilled water with 3 mL of methyl methacrylate (MMA). The solution was stirred at different rates, slow and fast, and different temperatures, 70 °C and 90 °C, under a constant flow of nitrogen gas for 40 minutes until the polymerization reaction was complete. Glass substrates were prepared by being cleaned with isopropyl alcohol for 10 minutes before being soaked in distilled water for 5 minutes. The glass was then suspended in a diluted solution consisting of 20 mL of distilled water and different concentrations of the PMMA nanosphere solution, 50, 100, and 150  $\mu$ L. The samples were dried at 50 °C for 36 hours to evaporate the solution and grow a thin film of self-assembled PMMA nanospheres on the glass substrates. Light spectrometry was used to determine the maximum absorbance wavelength of light when light was being transmitted perpendicular or at an angle to the samples. The Bragg equation was used to estimate to the size of the PMMA spheres from the wavelength of maximum absorbance. To verify the size of the nanospheres, samples were sputtered in gold and examined using the scanning electron microscope (SEM) and measured using the imaging software ImageJ. Nanosphere diameters ranged from 290 nm to 370 nm. Using a nanosphere concentration of 150  $\mu$ L, a fast stir rate, and a processing temperature of 90 °C produced the most consistently iridescent samples.

Keywords: materials engineering, polymethyl methacrylate, nanoparticles, iridescence, opal, photonic crystal

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## Introduction

### Broader Impacts

In 2009, a study testing the reading, mathematics, and science literacy of 15 year olds in 65 countries ranked the US as 19<sup>th</sup> in science literacy<sup>1</sup>. Last year, only 30% of high school graduates who took the ACT were considered ready for college level science<sup>2</sup>. There is an increasing need to not only educate students about engineering, but to introduce them to a subject that might soon be commonplace in their lives. Fortunately, \$3B of the 2013 federal budget is expected for educating children in the fields of science, technology, engineering, and mathematics<sup>3</sup>. In addition, about \$1.8B of the 2013 federal budget is expected for the National Nanotechnology Initiative. Unfortunately, there are few chances to create nanoparticles, unless in a university setting. The goal of this senior project was to refine a process that can be used for teaching younger students about nanotechnology.

One of the largest obstacles to overcome when trying to use nanoparticles as a teaching tool for students is the issue of safety. Obviously the safety of the students is paramount. Unfortunately, research is still ongoing about the long term effects of nanoparticle exposure. Because nanoparticles are so small, many fear that nanoparticles might be absorbed through the skin and harm vital systems or organs within the body. In addition to the fear of the nanoparticle products themselves, there are many laws and safety regulations that need to be followed when dealing with the chemicals used to fabricate the nanoparticles. If proper disposal methods are not taken, drinking water could be contaminated by hazardous chemicals. But in an educational setting, especially one for small children, separating hazardous chemicals from harmless chemicals can be difficult when multiple chemicals look similar to one another. Safe experiences with nanotechnology is imperative to ensure that today's youth will be comfortable with nanotechnology in order to help push the boundaries of what is technologically possible.

### Iridescence

The problem with teaching any topic to students is making something new understandable to students. Often the first step in explaining nanotechnology is trying to explain how small features are on the nanoscale. Instead, the focus should be on the effects that something on the nanoscale can have on the macroscopic scale. Iridescence can be an excellent tool for teaching about nanotechnology and the nanoscale. Iridescence is the ability for an object to appear different colors depending on the angle of light or viewing angle.

Light is both a wave and a particle. The wave nature of light is what helps create iridescence. When light strikes a surface, the incident wave is either transmitted, absorbed, or both. Objects appear to be a certain color when light of a certain wavelength is transmitted, while all other light is absorbed. If two waves combine, the resulting light can become stronger if the two waves are in phase, weaker if the two waves are out of phase, or no resulting light if the two waves are perfectly half a wavelength out of phase and cancel one another out (Fig. 1). The first result is called constructive interference, and can lead to bright colors. The second result is a weaker form

of constructive interference. The third result is called destructive interference, which can lead to less darkness<sup>4</sup>.

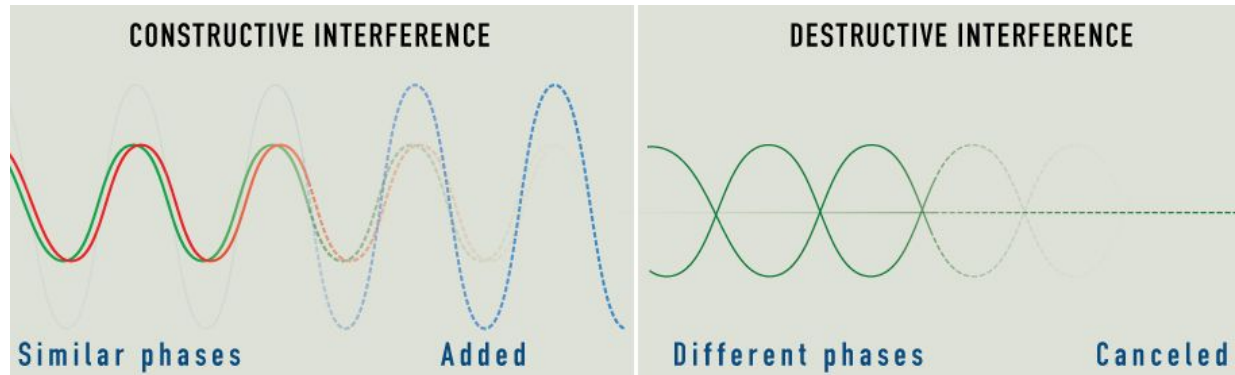


Figure 1: Constructive interference occurs when two waves of light are in phase and result in a more intense color. Destructive interference occurs when two waves are out of phase and cancel one another, resulting in darkness<sup>5</sup>.

There are many common objects that display iridescence. For example, the iridescence of bubbles is caused by thin film interference (Fig. 2). The walls of the soap bubble vary from one area to another. As light hits the walls of the soap bubble, it will bend at different angles due to the different thicknesses of the films, and reflect different wavelengths of light. Bubbles that are brightest are thicker than the wavelengths of light they are reflecting, so the thicker the bubble, the brighter the colors, <sup>6</sup>.

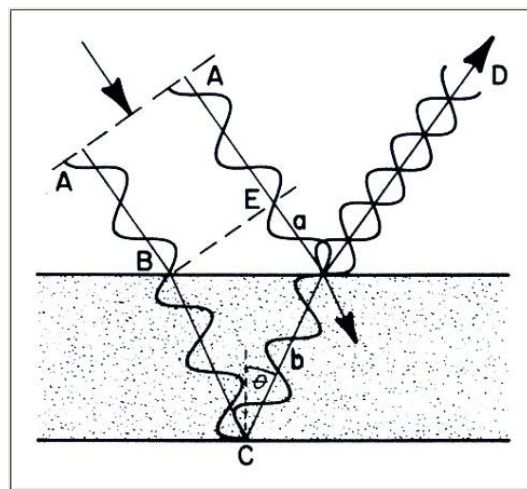


Figure 2: Thin film iridescence is caused by light being reflected from one part of a thin material interferes with light being reflected from a different part of the material<sup>5</sup>.

The brilliant iridescent colors of the Morphos butterfly are caused not only by pigments in the wing, but the structure of the wing itself (Fig. 3). There are repeated structures on the wing of the butterfly that are spaced regularly 200 nm apart<sup>7</sup>. This regularity acts as a diffraction grating, causing interference. Because the structures are on the same order of magnitude as the light itself, the light can become separated and different wavelengths of light will interfere differently depending on the structure. In the case of the Morphos butterfly, only blue light will be in phase and undergo constructive interference.

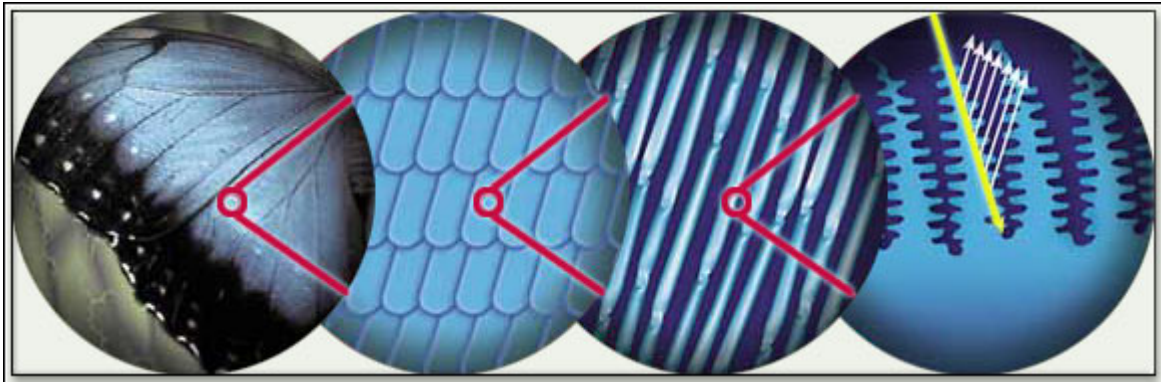


Figure 3: The structures on the scales of the Morphos butterfly are ordered and small enough to act as a diffraction grating, only allowing blue light to be reflected through constructive interference<sup>5</sup>.

## Opals

The natural gemstone opal has iridescent qualities like bubbles, but it is caused by their repeating nanostructure, similar to the Morphos butterfly. Opals are made up of nanospheres of silica that are close packed, with a void space of air (Fig. 4). The repeated, ordered structure of silica nanospheres and air qualify it as a special type of material called a photonic crystal. Photonic crystals have the ability to transmit light of only certain frequencies; this is called a photonic band gap. Photonic band gaps can be used to improve the speed of computers by replacing optical fibers with photonic crystal fibers<sup>8</sup>. Photonic crystals can also be used to improve the efficiency of solar cells by lowering the reflectivity of a solar cell, allowing more light to be absorbed<sup>9</sup>.

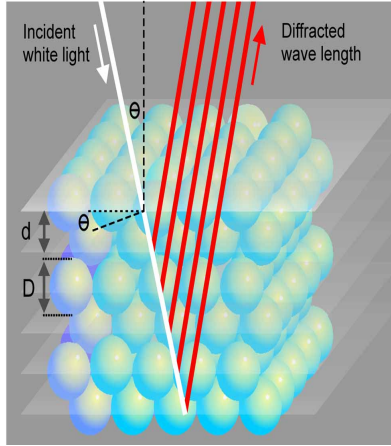


Figure 4: The structure of an opal consists of highly ordered silica nanospheres and the void space between them, this structure can allow light of specific frequencies to be transmitted<sup>10</sup>.

An opal's photonic band gap can be tuned depending on 3 things: the angle of incident light, the size of the nanospheres, and the differences in refractive indices. The angle of incident light will affect the diffracted light based on Bragg's Law (Eq. 1). The size of the nanospheres and the differences in refractive indices will determine what light is absorbed by the opal from combining Bragg's law and Snell's law,<sup>11</sup> this equation will be called Bragg's equation for simplicity (Eq 2).

$$\lambda = 2d\sin\theta \quad (\text{Equation 1})$$

$$\lambda_p = 1.633D \sqrt{n_s^2 f + n_v^2 (1 - f)} \quad (\text{Equation 2})$$

The focus of this project was to try and mimic the structure and iridescent quality of opals by creating nanospheres of polymethyl methacrylate (PMMA). The concentration of nanospheres, the temperature of the polymerization, and the stir rate were varied to determine the effect on the iridescent quality of the PMMA nanospheres.

### Polymerization of PMMA

PMMA is made through free radical chain-growth polymerization<sup>12</sup>. Chain growth polymerization occurs through the steps initiation, propagation, and termination (Fig. 5). During initiation, a granular form of 2,2'-azobis(2-methylpropionamidine) is broken down at low heat to create two radicals. Once the MMA is added, the radicals bond with the MMA monomer. Next is the propagation stage, MMA monomers continue to latch onto the free radical end of the growing polymer chain. Although one free radical is being consumed, another free radical is

being formed at the new end of the polymer chain. Termination is the end of the polymerization reaction and occurs when two free radicals join together, prohibiting more MMA monomers from growing the existing polymer chain. Once polymerized, the PMMA should form a colloidal suspension of solid nanospheres.

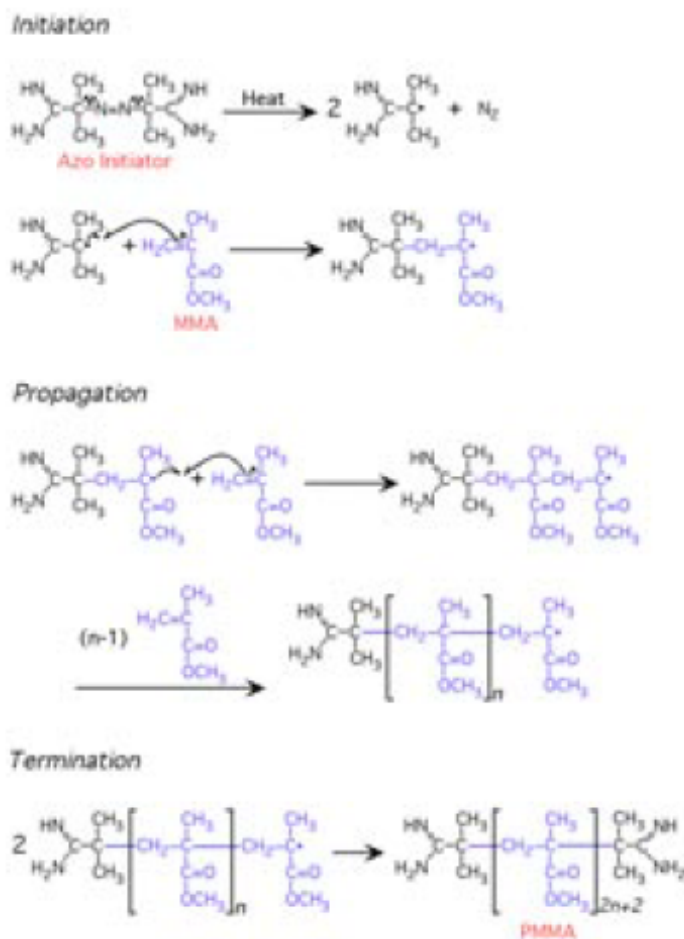


Figure 5: Polymerization of MMA into PMMA occurs in three stages: initiation, propagation, and termination<sup>12</sup>.



## Experimental Procedure

### Creating the PMMA Nanoparticles

To create the PMMA nanospheres, 16 mL of distilled water in a small round bottom flask was heated to 70 or 90 °C, and before 3mL of MMA were added (Fig. 6). When the temperature stabilized, 17 mg of granular azobis were added to initiate the reaction. This whole reaction was stirring at either a slow or fast rate under a slow flow of nitrogen for 40 minutes<sup>13</sup>. Cold water running through the condenser was used to force any solution that was evaporating to condensate and drip back into the reaction flask (Fig. 7).

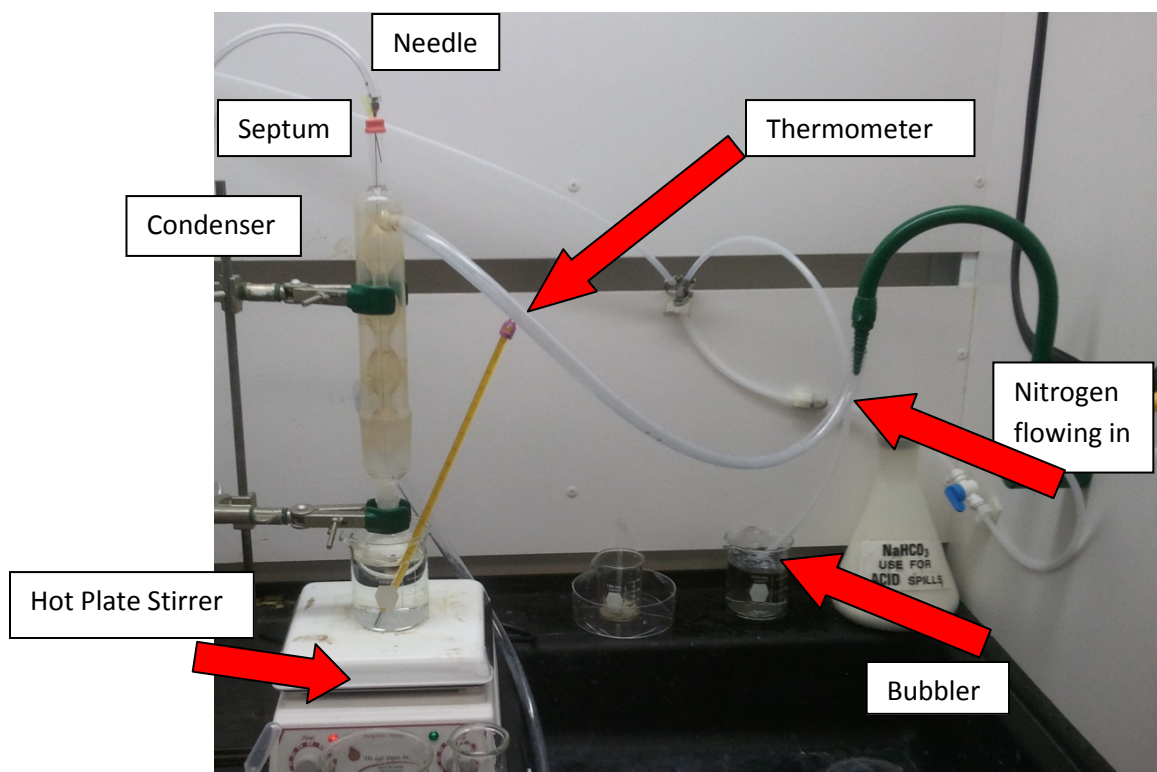


Figure 6: Set up for synthesizing nanoparticles.



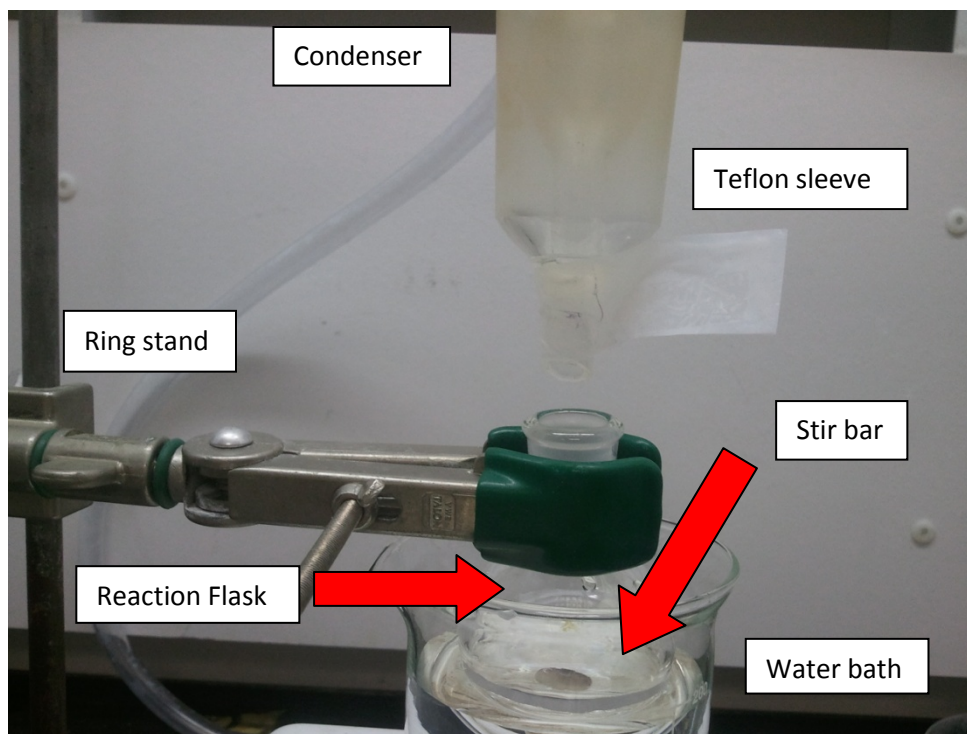


Figure 7: The MMA and azobis is added to the water by lifting the condenser and quickly pouring into the round bottom flask.

### Evaporating the PMMA Solution

In order to test the iridescence, the nanoparticles needed to be separated from their solution. Glass cover slips were chosen as substrates for the nanospheres. The glass substrates were cleaned in isopropyl alcohol by being suspended from a toothpick via tape (Fig. 8). The substrates were cleaned for 10 minutes before being cleaned in distilled water for 5 minutes.



Figure 8: Cleaning the glass substrate with isopropyl alcohol before suspending substrate in the diluted solution.

20 mL of distilled water were placed into three beakers for each trial. 50, 100, or 150  $\mu\text{L}$  of the nanosphere solution was added to each beaker to vary the nanosphere concentration between samples. Two glass substrates were suspended in each beaker in case one sample was lost or broken during testing. The diluted solution was dried for 36 hours at 50  $^{\circ}\text{C}$ <sup>14</sup>. After drying, the tape was cut from the samples and each sample was visually tested to determine any macroscopic trends. Specifically, the iridescence of each sample was tested.

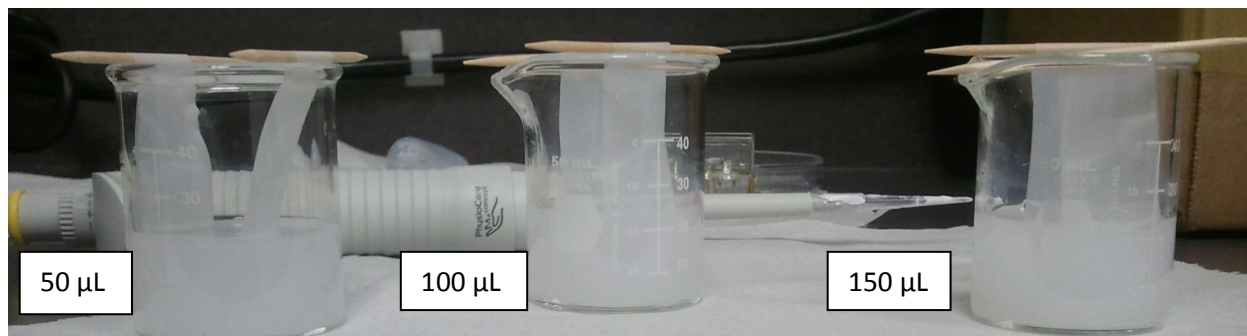


Figure 9: Distilled solutions before drying.

### Instrumental Analysis Methods

Each sample was then tested in the Ocean Optics USB4000 Fiber Optic Spectrometer to determine a trend between the experimental conditions and the iridescence of the samples. The iridescence of the samples would correlate to which wavelength of light was absorbed the most and the average nanoparticle diameter (Eq. 2).

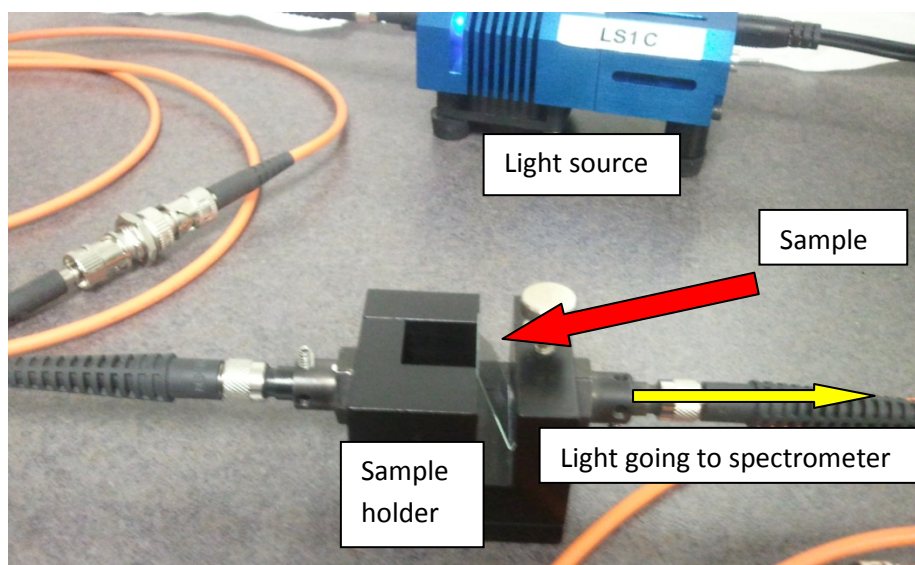


Figure 10: Set up for spectrometer testing while the sample is tilted.

One sample under each condition was broken and placed onto a conductive taped stage and sputtered with gold. The Scanning Electron Microscope (SEM) was used to examine each sputtered sample at various magnifications. Pictures were taken at 50,000 and 40,000x to examine the sample and determine whether or not nanospheres had been produced and self-ordered. Imaging software ImageJ was used to determine the size of the created particles. When applicable, 30 random measurements were taken of each picture.

### **Safety and Clean-up**

Since this project was meant to be duplicated many times as an educational experiment, cleaning after the project and disposing of any waste in a safe manner was vital. Specifically, the MMA and azobis required a special hazardous waste container made of high density polyethylene. Any glassware or instrument that touched these chemicals needed to be thrown away or washed. If washing was required, water would suffice for most cases, but multiple rinses would need to be done and the liquid would need to be captured in the waste container. The reaction flask and stir bar and diluted solution beakers in particular needed to be washed with acetone as well as water to remove any leftover MMA or PMMA. Again, any waste was not permitted in the sinks but instead collected into the waste container.

## Results

By looking at the samples visually, there was a noticeable difference between samples based on their nanosphere concentration. Regardless of the stir rate or temperature used, increasing the latex concentration led to a more uniform film of PMMA on the sample. There was also an observable difference between samples that were made at 70 °C (Fig. 11) and those made at 90 °C (Fig. 12). The samples made at higher temperatures were almost completely covered in PMMA, whereas the samples made at lower temperatures had bands of PMMA rather than an even coating of PMMA. The iridescent samples were those processed at 90 °C and had a nanosphere concentration of 100 or 150  $\mu\text{L}$ . The samples that were made at 90 °C and had a nanosphere concentration of 50  $\mu\text{L}$  had certain parts that were a shade of blue.

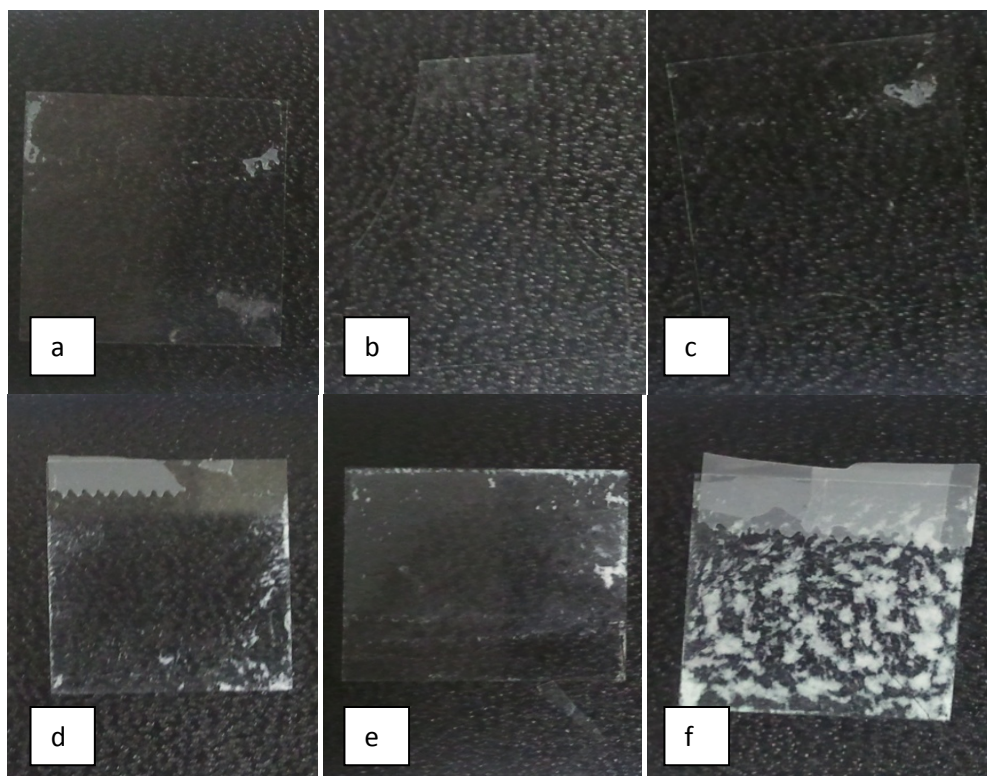


Figure 11: Samples made at 70 °C. Concentration of the latex solution in each beaker was 50, 100, and 150  $\mu\text{L}$  respectively in parts a, b, and c then d, e, and f. Samples in a-c were processed at the low stir rate and samples in d-f were processed at the high stir rate.



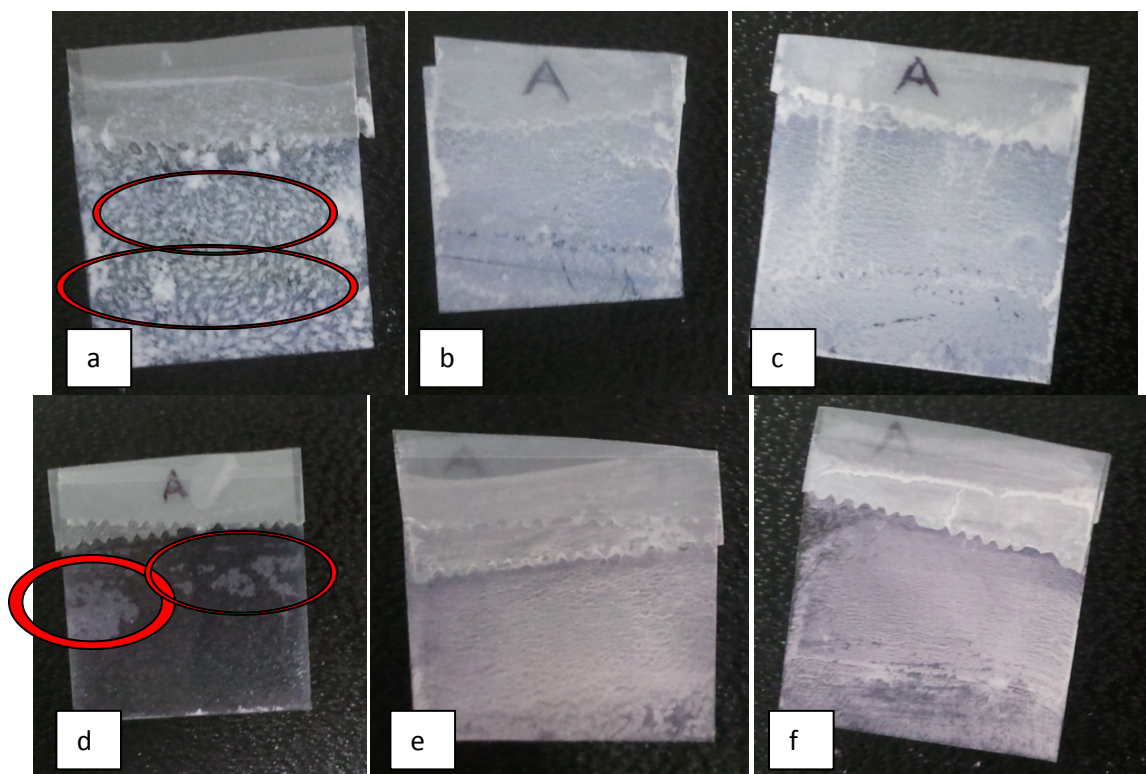


Figure 12: Samples made at 90 °C. Concentration of the latex solution in each beaker was 50, 100, and 150  $\mu\text{L}$  respectively in parts a, b, and c, then d, e, and f. Samples in a-c were processed at the low stir rate and samples in d-f were processed at the high stir rate. Except for the sample in d, all samples had a fairly uniform film of PMMA. The noted areas are where the samples appeared to be blue.

A spectrometer was used to scan which wavelengths of light were most for each sample. However, the same wavelengths of light were still absorbed in the same amounts despite changing the scanned area and angle of the sample. All samples absorbed light of about 400 nm the most, regardless of the conditions used in making the sample. The samples made at 70 °C mainly absorbed light around 400 nm (Fig. 13). The samples made at 90 °C absorbed light of around 400, 650, 790, and 862 nm which lead to different nanoparticle diameters (Fig. 14).

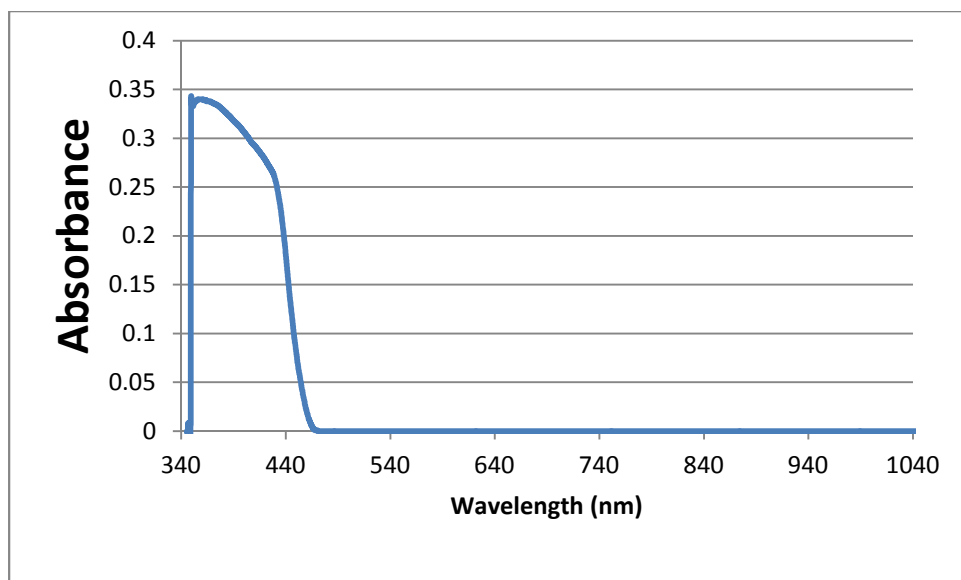


Figure 13: A typical spectrometer reading for samples processed at 70 °C, light with a wavelength lower than 450 nm is absorbed the most. This sample had a nanosphere concentration of 100  $\mu$ L and was made with the fast stir rate.

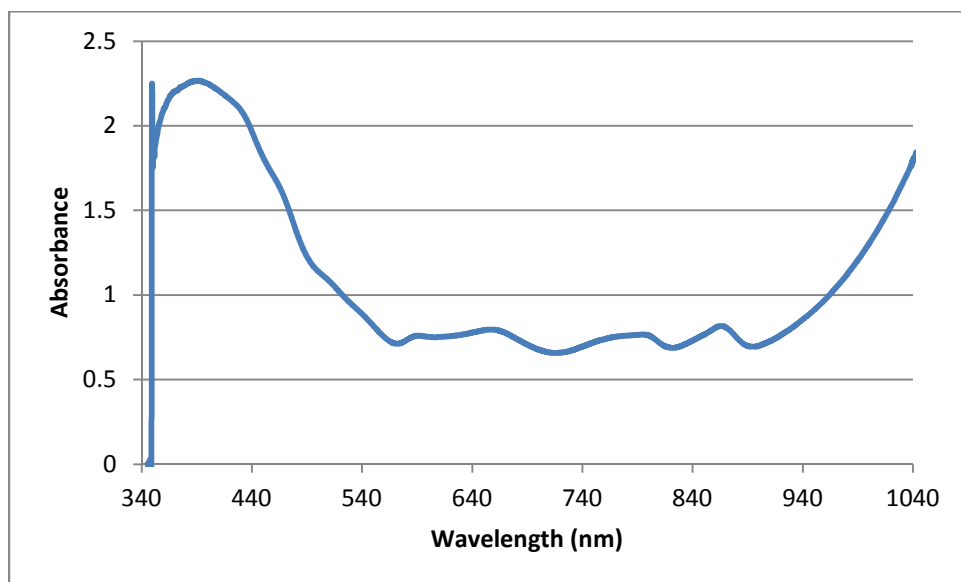


Figure 14: This spectrometer reading is of a sample processed at 90 °C, a slow stir rate, and a nanosphere concentration of 150  $\mu$ L. Several peaks are shown, predicting nanospheres of many different diameters.

Bragg's equation was then used to estimate the standard size of the PMMA nanospheres. According to Bragg's equation and the absorbance scans, all samples made at 70 °C should have a relatively similar average nanosphere diameter of 175 nm, regardless of the concentration or stir rate. All samples made at 90 °C appear have nanospheres of 175, 290, 345, and 380 nm.

Pictures were taken of every sample around 50,000x using the SEM and the diameter of 30 nanospheres was measured on each sample using imaging software ImageJ. The nanospheres did not have nanospheres of several discrete sizes, as estimated from Bragg's equation. Instead, the nanospheres had only one or two discrete sizes. The sample made at the lower temperature and the slower stir rate did not have nanospheres, instead the PMMA had formed into clumps that were much larger than the nanoparticle size. For example, the sample that had the medium nanosphere concentration had PMMA clumps were around 2.4  $\mu\text{m}$  in length (Fig. 15). Most of the samples had nanospheres, but though the nanospheres were not ordered as expected from literature.

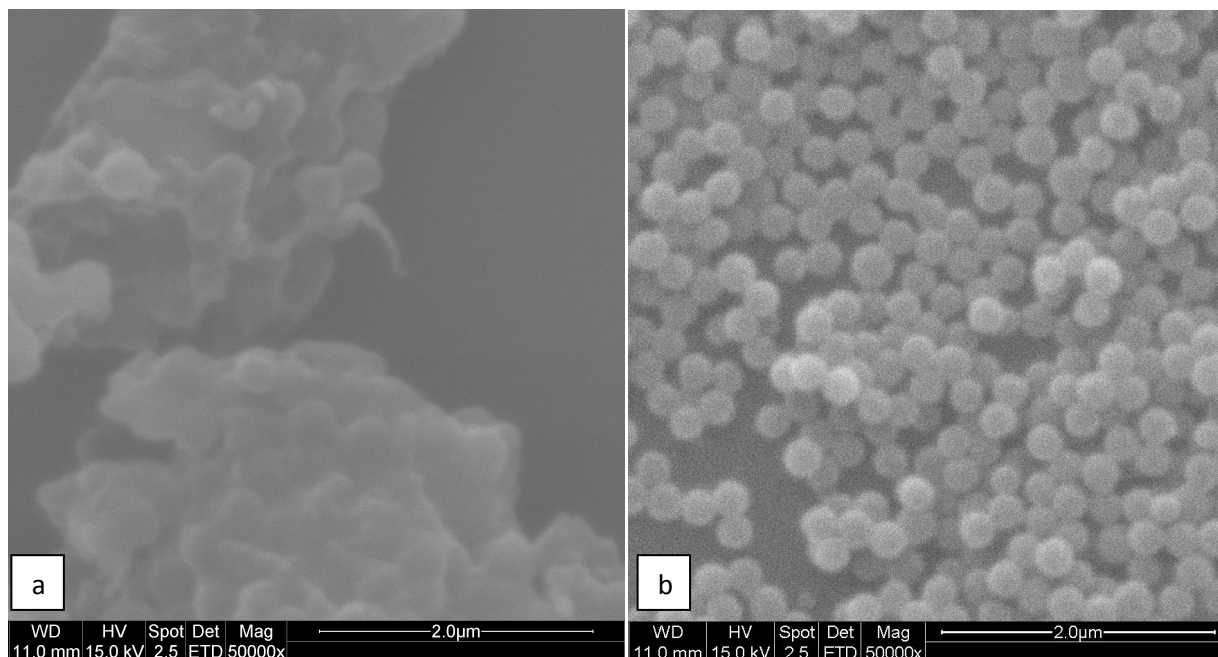


Figure 15: Each SEM picture was taken under the same settings, nanosphere concentration, and processing temperature. A shows a sample made using a slow stir rate and b shows a sample using a fast stir rate.

## Discussion

From visual inspection, the observable trend was that nanosphere concentrations of 100 and 150  $\mu\text{L}$  resulted in a more uniform distribution of the PMMA film. Also, the samples that were processed at 90  $^{\circ}\text{C}$  had a more uniform distribution of the PMMA film than samples processed at 70  $^{\circ}\text{C}$ . Samples made at 90  $^{\circ}\text{C}$  had a more even distribution of PMMA likely due to the higher temperature quickening the rate of the reaction, allowing free radicals to join sooner than if the nanospheres were made at 70  $^{\circ}\text{C}$ .

Finding the wavelengths of maximum absorbance for each slide was an incorrect way of testing the iridescence of the samples for two reasons. One way that finding the absorbance of the sample was incorrect was because the goal of this project was to test the iridescence of the nanospheres. On a macroscopic scale, the iridescence could be observed by seeing different colors when the sample was tilted at different angles. A more accurate way of characterizing the sample would have been to measure the reflected light rather than the absorbed light.

Another reason why finding the absorbed wavelengths is the wrong way to characterize the samples, is because the assumptions used for Bragg's equation are incorrect. According to Bragg's equation, the diameter of the nanosphere can be estimated from the wavelengths of maximum absorbance. However, Bragg's equation assumes that there is a certain degree of order in the sample (Fig. 16), which was not an accurate assumption (Fig. 17).

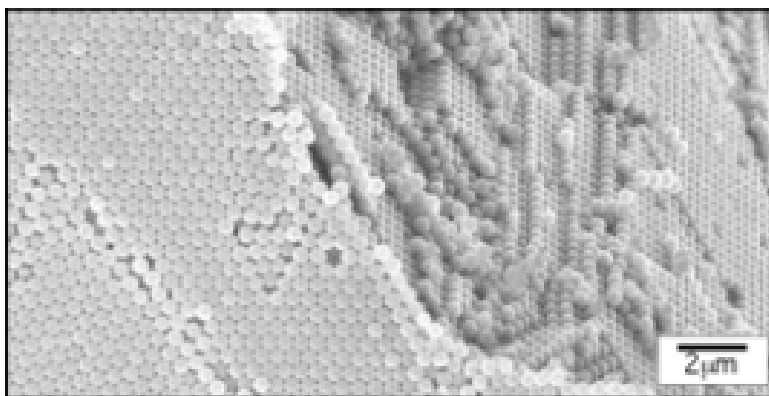


Figure 16: An ideal opal structure<sup>12</sup>.



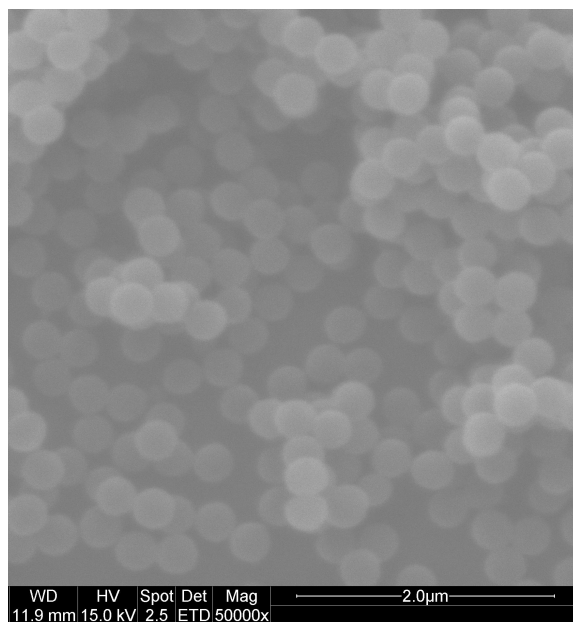


Figure 17: The nanospheres never had the close-packed and ordered structure expected. Even in the most ideal samples. This sample was made at 90 °C, slow stir rate, and 150 μL nanosphere concentration.

Bragg's equation assumes that the nanospheres are perfectly ordered, leading to a certain percentage of the film to consist of PMMA and the other percentage of the film to be air. In order to estimate the size of the nanospheres, a filling fraction of 0.76 was used. However, the SEM images show that the PMMA nanospheres did not form an ordered structure. Although most of the samples had an opalescent structure, even the iridescent samples did not have nanoparticle sizes that were predicted by Bragg's equation (Table I).

Table I: Summary of predicted nanosphere size using Bragg's equation and the measured diameters using SEM.

Sample	Average Diameter From Bragg's Equation	Measured Diameter From SEM
T3C50	176 nm	12 $\mu\text{m}$
T3C100	176 nm	1.8 $\mu\text{m}$
T3C150	176 nm	2.4 $\mu\text{m}$
T4C50	176 nm	290.1 nm
T4C100	176 nm	308.2 nm
T4C150	176 nm	298 nm
T6C50	176 nm, 292 nm, 344 nm, 378 nm	356.6 nm
T6C100	176 nm, 288 nm, 348 nm, 374 nm	367.4 nm
T6C150	179 nm, 283 nm, 344 nm, 377 nm	336.1 nm
T7C50	176 nm, 355 nm, 378 nm	323.2 nm
T7C100	176 nm, 289 nm, 347 nm, 380 nm	311.6 nm
T7C150	160 nm, 285 nm, 347 nm, 384 nm	298.3 nm

All of the samples had a peak in their absorbance reading around 400 nm that led to an expected nanosphere diameter of 176 nm. This is because the glass used as a substrate for the nanospheres naturally absorbs light of about 400 nm the most (Fig. 18). In the samples where PMMA had clumped together, the absorbance was virtually identical to the control spectrograph because the absorbance of the scanned area of the sample was essentially a clean substrate. The samples that had a uniform film of PMMA had absorbance peaks, but these could still not be used to predict nanoparticle size because there was no order to the samples.

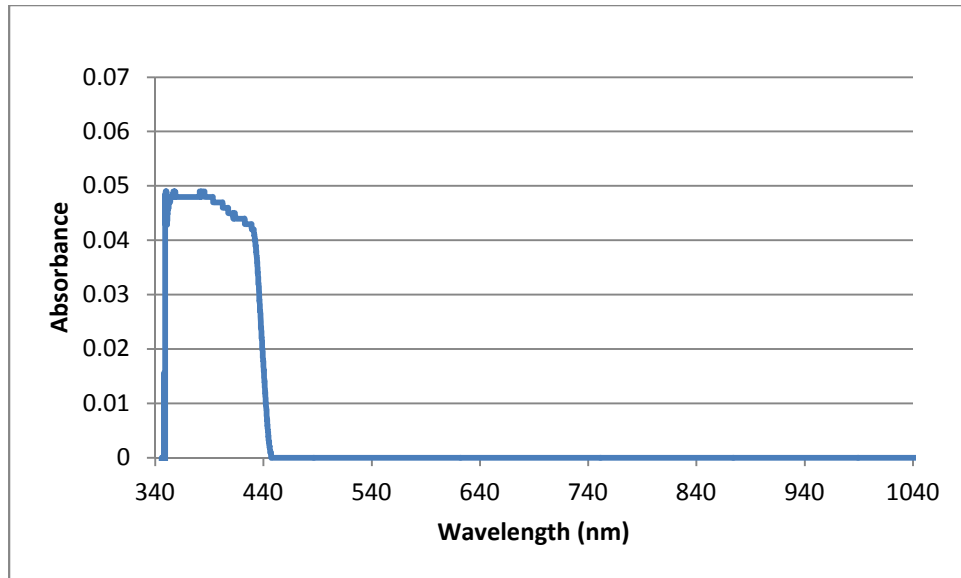


Figure 18: A glass cover slip with no nanospheres absorbs light in a similar way to the samples. This explains why all of the samples absorb light of around 400 nm the most, regardless of the PMMA film.

## Conclusion

Iridescent films were created by synthesizing PMMA nanospheres by mixing 16 mL of distilled water, 17 mg of granular azobis, and 3 mL of MMA at 90 °C with either a slow or fast stir rate. Using a nanosphere concentration of 100 or 150  $\mu\text{L}$  and 20 mL of distilled water, the solution could be dried onto a glass substrate by evaporating for 36 hours at 50°C.

1. Temperature was the most important factor in creating iridescent samples, at 90 °C there was an increased likelihood of creating iridescent samples.
2. Samples with a 100 or 150  $\mu\text{L}$  concentration of PMMA nanospheres were more likely to be iridescent due to a thicker and more even coating of PMMA on the samples.
3. Nanoparticle size could best be tested using a SEM. Testing samples for iridescence might be possible using a spectrometer to test for transmitted wavelengths.
4. This process would not be suitable as an outreach event due to the lack of portability of the equipment set-up and safety precautions needed for the Azobis and MMA.
5. However, this process could fit as a future lab for Materials Engineering students. All of the equipment needed is either already owned by the Materials Engineering department or inexpensive to obtain. Once the system is set up, little maintenance is required. However, since the polymerization reaction takes place in a fumehood, class sizes would need to be limited or additional fumehoods would be required.

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