

Alternative textile dyes: Synthesizing and applying PMMA nanoparticles to create structural coloration

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SUMMARY

Synthetic textile dyes are one of the most significant contributors to global pollution, releasing toxic wastewater and lowering dissolved oxygen levels in bodies of water near textile factories. A solution is to develop structural coloration onto textiles, which depends solely on the shape of an object and does not contain any hazardous chemical components that might enter wastewater. We colorized textiles by synthesizing and spray-coating nanospheres that create structural color on fabrics. We hypothesized that various structural colors of polymethyl methacrylate nanospheres (PMMA) can be produced. PMMA nanospheres have not been explored in their ability to reflect color but would be a less toxic alternative to researched materials such as poly(styrene-methyl methacrylate-acrylic acid). We synthesized PMMA nanospheres by stirring distilled water, methyl methacrylate, and 2,2'-Azobis(2-methylpropionamide dihydrochloride) initiator under an inert atmosphere. The optimal method of preparing and spray-coating the PMMA involved centrifugation, mixing a 100:1 ratio of PMMA/water solution and carbon black, and then spray-coating the resulting PMMA particles onto fabric in five layers. The PMMA particles showed high durability on the fabric. By changing the amounts of distilled water, methyl methacrylate, and initiator, the resulting colors included red-violet, violet, blue, turquoise, green, and yellow-green. We successfully produced structural coloration by synthesizing and applying nanospheres in various colors to fabrics and optimized the method of applying nanospheres onto fabric for the most saturated color. In addition, we also synthesized a potentially more sustainable alternative to PMMA as it is biodegradable. Our results suggest that structural coloration with PMMA particles is a promising non-polluting alternative to dyes for the future.

INTRODUCTION

Dyes are the most commonly used colorants on textiles, but they have negative impacts on the environment and on the people living near textile factories, particularly due to the wastewater that is released after the dyeing process (1). Each year, the global production of synthetic dyes totals approximately 7×10^7 tons, with textile industries alone accounting for over 10,000 tons of these dyes. However, due to inefficient dyeing processes, a significant portion

of azo dyes—ranging from 15% to 50%—fail to bind to fibers and are consequently released into wastewater. This wastewater is laden with various hazardous substances, including heavy metals like mercury, chromium, cadmium, lead, and arsenic. These pollutants travel vast distances with the wastewater, persisting in water and soil for extended periods. Their presence poses grave health risks to organisms and detrimentally affects soil fertility and the photosynthetic activities of aquatic plants. This disruption leads to the creation of anoxic conditions for aquatic flora and fauna. Moreover, textile dyes exacerbate water pollution by increasing biochemical and chemical oxygen demand, hindering photosynthesis, impeding plant growth, entering the food chain, and fostering resistance and accumulation, thereby potentially inducing toxicity, mutagenicity, and carcinogenicity (2).

In the past, there has been research in finding treatment methods for wastewater released from the dyeing process of textiles, such as adsorption and nanofiltration, but a shift to nontoxic production must be made (3). Structural color is an alternative to dye that mimics the way nature reflects light to produce color and has the potential to be more sustainable. Unlike dyes or pigments that depend on chemicals, structural color depends solely on the shape of the object. The shapes are often micro- or nano-structures that when light is shined on them, wavelengths in the visible spectrum are reflected to produce color (2). This method of creating colors has inspired biomimetic research, particularly in new textile dyeing technology. A previous study has synthesized structures that produce structural coloration (2).

Nanospheres that can reflect color are first synthesized through the polymerization of monomer methyl methacrylate, then spray-coated on black cotton fabric (2, 4). Previous research utilized a toxic form of styrene to produce structural coloration onto fabrics (2). Being exposed to high levels of styrene can cause physical problems such as imbalance, changes in color vision, tiredness, and slowed reaction time. Polymethyl methacrylate (PMMA) nanospheres are a safer substance. A closed-loop recycling method – the ability to indefinitely recycle a material – of PMMA also exists, making it less hazardous to the environment (5). Once synthesized, the PMMA is prepared in a solution with distilled water (DW) and carbon black (CB) to be spray-coated onto black cotton fabric (4).

In addition, starch acetate (SA) is a more sustainable alternative as they are biodegradable. SA synthesis reaction involves acetylating the starch by reacting it with acetic anhydride and a sodium hydroxide catalyst while stirring under heat (6). The replacement of the hydrogen atom of a hydroxyl group with an acetyl group yields SA. The SA is then mixed

with a nonsolvent and then put into a hydrothermal autoclave that can aggregate particles into larger nanospheres in the size range that can reflect color (7). To become nanospheres that can reflect light, the synthesized SA must have a high degree of substitution across all particles, meaning the many hydrogen atoms were substituted with hydroxyl groups to yield SA.

For both PMMA and SA, the nanospheres must be monodisperse to create structural color, which means they are uniform and have the same molecular mass or degree of polymerization (**Figure 1**). On the other hand, if the nanostructures made are polydisperse, color cannot be reflected. To obtain consistent color, the nanospheres must be angle-independent. To do this, the nanospheres require short-range order, which is the tendency for the particles to form small, ordered regions that do not link together at long distances (**Figure 1**). This way, the viewing angle will not matter as light is scattered in every direction, eliminating the need for an optimal angle to view it. Establishing an amorphous state of short-range nanoparticles results in vivid, non-iridescent structural colors (4).

Changing the size or spacing of the repeating nanostructures changes the color that is reflected. Nanoparticles with diameters of 310 nm, 375 nm, and 425 nm appear blue-violet, green, and orange-pink, respectively, due to the diffraction of light of successively longer wavelengths (4). Currently, structural colors that can be produced are limited to the blue and purple range of colors, which can be explained by the natural prevalence at which they appear in nature. Colors in the red and orange regions are more difficult to produce (8).

For structural coloration to become potentially commercialized, the ability for nanoparticles to adhere to fabrics is crucial. Since PMMA has hydrophobic properties, we hypothesized that PMMA will endure well against water, but not abrasion. We tested the durability of PMMA on black cotton fabric and performed three tests. Overall, for all three tests, the saturation was mostly maintained. Furthermore, we found that the saturation of the structural colors was maintained as the nanoparticles adhered well to the fabrics.

In this study, we combined two separate methods of using an established method of PMMA synthesis and a novel method of spray-coating application (4, 9). We hypothesized that it was possible to synthesize PMMA particles as a more efficient, environmentally-friendly method to produce a range of structural colors. Given that the spray-coating

method has been used for poly(styrene-methyl methacrylate-acrylic acid) (P(St-MMA-AA)), but has not been used with PMMA particle technology, we hypothesized that the spray coating method would work (4). P(St-MMA-AA), in contrast to PMMA, includes styrene, which is classified as a mutagen and potentially carcinogenic. Exposure to styrene can result in memory loss, difficulties in concentration and learning, brain and liver damage, and cancer (10). To avoid such dangerous effects while still obtaining essential qualities such as monodispersity and short-range order, PMMA was the best alternative. We hypothesized that PMMA particles could produce distinct colors by altering the amounts of DW, methyl methacrylate (MMA), or 2,2'-Azobis(2-methylpropionamide dihydrochloride) initiator.

The final goal of this research was to discuss sustainable alternatives of nanospheres for future research. Since PMMA is a nanoplastic and nanoplastic particles are harmful to the environment, it is not sustainable for large-scale use; we synthesized PMMA since it was a reliable method. The ultimate goal of this research was to create sustainable nanospheres, and we hypothesized that starch acetate nanospheres (SANs) would be a more sustainable alternative to PMMA nanospheres. SANs are most suitable as an alternative to PMMA because they are in the size range that reflects light in the visible spectrum and are also monodisperse and short-range (6). In addition, SANs are biodegradable, which is a sustainable aspect PMMA does not have. To analyze the SANs, we used a UV-visible spectrophotometer to determine if the substance made was SA.

The application of nanospheres through a spray-coating technique could bring to light a new approach to making textile coloration in the fashion industry more sustainable.

RESULTS

The purpose of this research was to produce various colors from structural coloration to replace toxic dyes. We hypothesized that PMMA particles could produce distinct colors by altering the amounts of reagents, which would change the size of nano-spheres and thereby reflect different colors. Our goal was to discover the optimal combination of reagents (i.e., MMA, DW, initiator, CB) and spin speed of centrifugation to yield different colors.

PMMA synthesis consisted of stirring DW, MMA, and 2,2'-Azobis(2-methylpropionamide dihydrochloride) initiator under heat and an inert atmosphere. A PMMA synthesis reaction ends with a milky white substance that produces color when dried on a surface (**Figure 2a-c**). Colors ranging from red-violet, violet, blue, turquoise, green, and yellow-green were successfully produced (**Figure 3**). There is a general trend between each substance for creating colors across the visible spectrum (**Table 1**). As the amount of MMA and initiator increased, colors with shorter visible wavelengths were created. For example, by increasing the amount of MMA used by approximately 0.02 g, colors changed from blue (~470 nm) to violet (~380 nm). On the other hand, more water created colors with longer wavelengths, such as green (~570). To create red-violet, 45.0 mL of DW, 7.5 mL of MMA, and an average of 0.0347 g of initiator, or 50.0 mL DW, 6.0 mL MMA, and 0.0277 g of initiator were used; both methods appear to the eye as the same color. To create violet, 32.0 mL of DW, 6.0 mL of MMA, and an average of 0.0530 g of initiator were used. To create blue, 32.0 mL of DW, 6.0 mL of MMA,

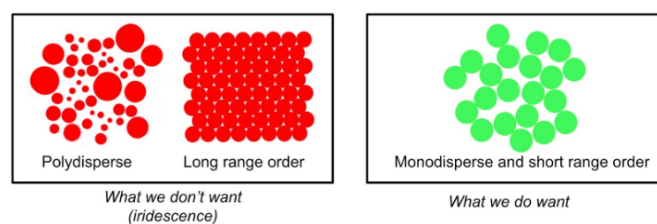


Figure 1. Monodisperse and short-range nanospheres are required to produce angle-independent structural coloration. Monodisperse spheres are uniform in size and density, and short-range ordered particles form small, ordered regions that do not link together at long distances. The polydispersity of particles will not reflect light to create structural coloration, and long-range order will cause iridescence of the color.

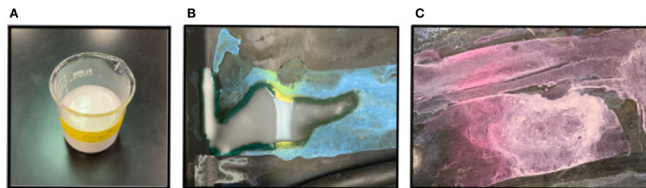


Figure 2. Successful PMMA synthesis reaction. (A) A milky white substance is produced following successful PMMA synthesis. PMMA particles were spray-coated on a black surface and dried to a (B) turquoise or (C) red-violet color, indicating successful reactions.

and an average of 0.0394 g of initiator were used. To create turquoise, 32.5 mL of DW, 7.0 mL of MMA, and an average of 0.0185 g of initiator were used. To create green, 60.0 mL of DW, 5.0 mL of MMA, and an average of 0.0207 g of initiator were used. To create yellow-green, 50.0 mL of DW, 6.0 mL of MMA, and an average of 0.0304 g of initiator were used (Table 1). There was a total of 44 successful trials and 21 failed trials.

We optimized the method of applying nanospheres onto fabric for the most saturated color. After testing different ratios of PMMA/DW solution to CB, the spray-coated color with 1% CB and five layers of spray-coating was the optimal measurement for the most saturated color (Figure 4). Using more than five layers caused uneven coatings and accumulation of particles that gave the fabric a rough appearance. Using more than 1% of CB made the color too dark, especially on black fabric since the CB overpowers the strength of the reflected color from the PMMA. However, too little CB meant that the CB did not support the reflected color. The 1% ratio of CB created the most optimal relationship with to the color reflected. The saturation of the structural coloration is also dependent on the ability of the spray-coating to stay on the fabric. We tested the durability of spray coatings by doing three tests, which involved soaking, rubbing, and exposure to soap (Figure 5). Soaking simulated exposure to rainwater, rubbing to abrasion caused during everyday occurrences, and soap to washing machines. Since PMMA appeared to have hydrophobic qualities, we observed that for all three tests, saturation was maintained.

Our final goal was to discuss SANs as a more sustainable alternative to PMMA nanospheres. To synthesize SA, we pregelatinized cornstarch by stirring 30 g in water at 95°C.

Color	Distilled Water (mL)	MMA (mL)	Average amount of initiator (g)	Number of successful trials
Red-Violet	50.0	6.0	0.0227	6
Red-Violet	45.0	7.5	0.0346	3
Violet	32.0	6.0	0.0571	8
Blue	32.0	6.0	0.0394	14
Turquoise	32.5	7.0	0.0185	2
Turquoise	28.0	7.5	0.0455	1
Green	60.0	5.0	0.0207	6
Yellow-green	50.0	6.0	0.0304	4

Table 1. The amount of DW, MMA, average amount of initiator, and number of successful trials to create a color. The number of successful trials indicates the number of times the color was produced under their specified conditions. Changing the amount of water, MMA, or initiator all resulted in a change of color, but the water and initiator had the strongest impact. Two rows of red-violet and turquoise exist due to the vast difference in reagents that produced red-violet and turquoise. The colors are in order from smallest to largest wavelengths.

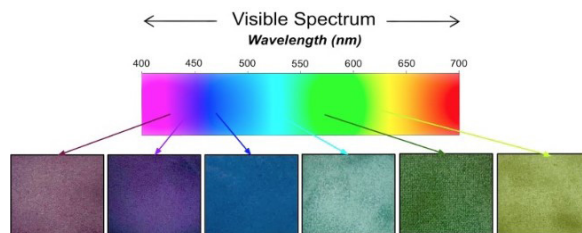


Figure 3. Colors produced by PMMA particles and spray-coating. Colors ranging from red-violet, violet, blue, turquoise, green, and yellow-green were produced. The colors correspond to the approximate wavelength that it is reflecting.

Pregelatinized starch reacts with acetic anhydride quicker compared to ungelatinized starch, and, therefore, there is less water in the reaction (6).

We used a UV-visible spectrophotometer to determine if the substance made was SA. The SA product and cornstarch were tested from 400 nm to 700 nm wavelength. We mixed Lugol's solution, a starch indicator, with plain cornstarch or the synthesized SA product. When the iodine in Lugol's solution binds to alpha-1,4 glucans found in starches, it turns into a blue color. In theory, if SA was successfully produced since the curves for SA and cornstarch were distinct, with only starch taking on long wavelengths. The blue line is the SA, which differed greatly from the other curves that only included starch, indicating that we successfully produced SA (Figure 6).

DISCUSSION

We discovered how to produce various colors from structural coloration and determined the characteristics of the particles such as their durability on fabric. In addition, we optimized the method of applying nanospheres onto fabric for the most saturated color. We hypothesized that PMMA particles could produce distinct colors by altering the amounts of DW, methyl methacrylate (MMA), or 2,2'-Azobis(2-methylpropionamide dihydrochloride) initiator. We synthesized PMMA nanospheres that are monodisperse and in short-range order and applied them to fabrics in different colors of nanosphere coatings. We optimized the method of applying nanospheres onto fabric for the most saturated color, determined the ratio of reactants needed to create certain colors, and determined the durability of the nanospheres to be quite strong. In addition, we hypothesized that starch acetate nanospheres (SANs) would be a more sustainable alternative to PMMA nanospheres. We successfully synthesized starch acetate.

Each successful trial had overarching characteristics and discrete amounts of reagents. A successful PMMA chemical reaction was indicated by a milky white substance (Figure 2a) that often appeared as solidified clumps on the thermometer. Whenever the resulting product was clear like fresh MMA, it was confirmed as a failed reaction as it indicates that the MMA did not react with the initiator. We also assessed a successful trial by letting drops of the solution dry on a black surface immediately after the reaction (Figure 2b-c). The black surface was the surface of the fume hood; the surface does not have to be black, but a darker shade helps emphasize the structural color the most. If successful, the PMMA will dry into a color, which is also the color that appears when the PMMA

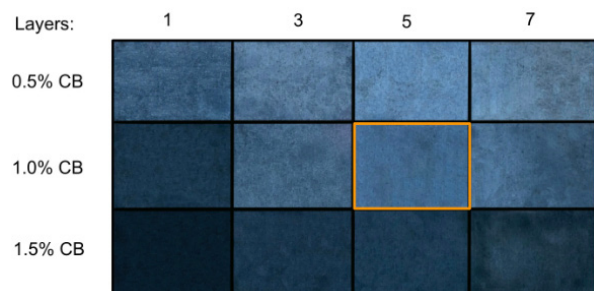


Figure 4. A 1% ratio of CB and 5 layers of spray-coating create the most saturated color. By varying the amounts of CB and layers of spray-coating, the optimal amount of each factor was determined. Three concentrations of CB (0.5%, 1.0%, and 1.5%) were tested along with 1, 3, and 5 layers for each amount. The highlighted box indicates the fabric spray-coated using the most optimal ratio of CB and layers of spray-coating.

is spray-coated onto fabric. To create structural coloration on textiles to replace dyes, we hypothesized that PMMA particles would be able to produce several colors by altering the amounts of reactants. We used DW ranging from 28.0-50.0 mL, MMA from 5.0-7.5 mL, and initiator approximately 0.020-0.050 g (Table 1). There is no strict relationship between the amount of each substance; thus, the discovery of set measurements to produce each color is dependent on further experimentation. All parameters were changed because changing just one did not sufficiently change color to another. Changing only one reactant produced different shades of the same color.

Warmer colors such as yellow, orange, and red are more difficult to make as particles tend to scatter blue wavelengths more strongly (8). Zeng et al. also appeared to only make colors of blue and purple. In this research, red was not produced but we successfully made a warmer red-violet. The majority of angle-independent colors in nature are blue, as seen in the morpho butterfly and the sky. Individual particles backscatter light more in the blue wavelength range while suppressing the structural red color due to modes and their interaction with the structural resonances (8). Other sources of error for the lack of successful trials could include failure to insert enough inert atmosphere into the reaction or the lack of experimentation in changing the amounts of reactants.

Instead of spray-coating poly(styrene-methyl methacrylate-acrylic acid), we spray-coated PMMA for the first time onto fabric to produce structural coloration. PMMA nanospheres are a safer substance, which can be synthesized easily and attached to fabric. We centrifuged PMMA and then used a 100:1 ratio of PMMA/DW solution and carbon black. Using the centrifuged PMMA immediately after being centrifuged prevents the spray-coated color from fading with white speckles. Dried PMMA are often solids that are difficult to put back into smaller particles, so they are not evenly distributed enough to optimally reflect light when spray-coated. Although using dried PMMA does create color, using it without drying is best as the particles are in their simplest form compared to their dried clumped form. The CB is used to emphasize the reflected color more by absorbing the rest of the wavelengths. The black cotton cloth was chosen as a control because the color would appear best on a black surface, although

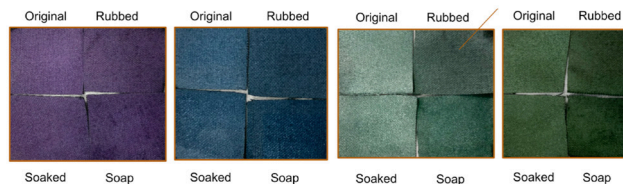


Figure 5. PMMA particles have strong durability and adherence to the fabric. Violet, blue, turquoise, and green (left to right) underwent three tests to test the durability of the particles on the fabric. Compared to the original color, the soaked fabric showed the least change in color saturation. The rubbed and washed fabrics exhibited a more faded appearance compared to the original.

spray-coating nanospheres onto sustainable fabrics such as hemp, bamboo, or recycled polyester fabrics, would be ideal. Changing the type of fabric may cause nanospheres to adhere differently but would be the most eco-friendly option.

We hypothesized that because PMMA has hydrophobic properties, it will adhere well to fabric against water but not abrasion. For all three tests, the saturation was mostly maintained (Figure 5), indicating that PMMA has considerably high durability. The first test in particular, which soaked the spray-coated fabric in water, appeared to have no change in saturation. In Zeng et al., polyacrylate glue was used to stick the nanospheres onto the fabric, but it was found in this research that the spheres maintained their adhesiveness without any glue. In addition, we found that adding glue increased the durability of the particles staying on the fabric but caused the particles to be unevenly distributed as they aggregated together in glue.

The main goal of this research was to create sustainable structural coloration, and SANs were considered to be more sustainable due to their biodegradable nature. In this research, SANs were synthesized, but they did not produce structural coloration and we are unsure if they are in nanoparticle form. Further research is necessary to synthesize nano-sized SA particles. However, SANs still have the potential to create sustainable structural coloration and replace toxic chemical dyes in the future.

We synthesized monodisperse and short-order ranged PMMA nanospheres that reflected various wavelengths of light to produce structural coloration. We determined the general durability of the nanospheres to stay on cotton fabric, and the best ratio of CB to produce the best saturation. Finally, we synthesized SA as a future biodegradable material for structural coloration on fabrics.

MATERIALS AND METHODS

PMMA particle synthesis

PMMA particle synthesis required an intricate setup in the fume hood. We placed a crystallizing dish on a hot plate and clamped right above it a two-neck round bottom flask. In the first neck, we inserted a condenser wrapped with Teflon tape, and the condenser was also connected at the top to a bubbler with a plastic tube and rubber tubing. In the second neck, a thermometer and a glass pipette were inserted along with a stopper. The thermometer measures the internal temperature of the reaction, and we used the pipette to spray inert gas to maintain an inert atmosphere throughout the reaction. The inert gas consisted of pure nitrogen, carbon dioxide, and argon. The nitrogen prevents the 2,2'-Azobis(2-

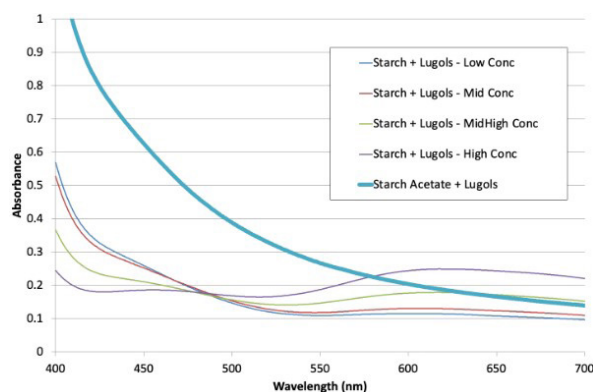


Figure 6. UV-visible absorbance of SA or starch with Lugol's solution. Starch was tested at low, mid, mid-high, and high concentrations. SA (blue line) differs greatly from the different concentrations of starch, indicating that SA was most likely successfully produced.

methylpropionamide dihydrochloride) initiator from oxidizing. There was another thermometer to measure the water temperature in the crystallizing dish. Inside the round bottom flask was an oval-shaped magnetic stirrer.

To synthesize monodisperse and short-range ordered PMMA nanospheres, DW, MMA, and 2,2'-Azobis(2-methylpropionamide dihydrochloride) (**Table 1**) were stirred together under heat (70-75°C) (4). The nonpolar MMA starting monomer is immiscible with water, and rapid stirring of a mixture caused small spheres in the form of a mini-emulsion before heat-induced polymerization.

Water in the crystallizing dish was heated to 80-90°C and the stir setting on the hot plate. DW and MMA were first stirred together and heated to 70-80°C in the round bottom flask before the initiator was added. To ensure that an inert atmosphere was maintained inside the round bottom flask, the wine preserver was sprayed every ten minutes throughout the reaction. The bubbler indicated if the gas had successfully entered the flask. The reaction took an hour to complete.

To spray-coat fabric with PMMA particles, an airbrush with a 0.3 mm nozzle was used. CB was used to prepare the PMMA for spray-coating.

Centrifugation

To separate nanoparticles from the water, the PMMA particles were centrifuged at 9,000 rpm for 15 minutes. A pipette was used to discard the water after centrifugation. Some iridescence can be observed after centrifuging.

Once the PMMA nanospheres were synthesized, centrifuging the substance at 9,000 rpm proved the best among other rates. It was initially predicted that a higher rate would cause the spheres to deform. However, a higher rate of 9,000 rpm was deemed to show the same color as PMMA centrifuged at 4,000 with a higher yield, so 9,000 rpm was used for all spray-coated fabrics.

Using the centrifuged PMMA immediately after being centrifuged also prevents the spray-coated color from being faded with white speckles. Dried PMMA solidifies into hardened forms that are difficult to put back into smaller particles that are easier to spray-coat.

Testing for the best saturation of structural coloration on fabric and durability of spray-coating

To spray-coat the PMMA nanospheres, Zeng et al. 2017's method was used as a reference with some modifications (2). The centrifuged nanospheres were used in a 100:1 of PMMA/DW solution and CB. In the PMMA/DW solution, PMMA had a 10% weight percentage. We sprayed the solution onto 2-inch x 2-inch squares of black cotton. Each fabric was sprayed five times with drying time after each layer.

The optimal number of spray coatings and CB ratio were found by testing varying amounts of carbon black used and the number of spray coatings (**Figure 4**). Four different colors and their appearances were tested using 0.5, 1, and 1.5% CB along with one, three, and five layers for each ratio. Each layer was equivalent to spraying a 3-inch x 3-inch piece of fabric for five seconds.

The durability of spray coatings was tested by doing 3 tests (**Figure 5**). First, the spray-coated fabric was soaked completely in water and dried. Since PMMA has hydrophobic properties, pouring water over the fabric did not work and it had to be fully immersed into the water. Second, the spray-coated fabric was rubbed 16 times with a paper towel; blue was rubbed 100 times to show how the fabric responds to excessive abrasion. Third, a miniature washing machine was simulated by mixing a drop of Dawn soap into 40 mL of water in a beaker. The beaker was placed on a hot plate at a stirring setting of 2, and the spray-coated fabric was stirred in it for 10 seconds. Then, it was rinsed with 40 mL of water for 10 seconds and dried.

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