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Synthesis of Cu_2O Nanoparticles and Their Oxidative Effects on Organic Pollutants

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Abstract

Copper (I) oxide has become a promising target in the development of nanoparticle technology aimed towards catalyzing environmentally friendly reactions. The purpose of this experiment was to synthesize an effective catalyst that will degrade environmental pollutants while leaving a negligible trace on the surroundings. By varying levels of PVP added, cubic and octahedral nanoparticles were utilized due to their low production costs, decreased energy threshold, and surface area reactivity. Both methyl orange and methyl blue dye were tested with copper (I) oxide particles under an enclosed dual light apparatus to determine absorption spectra via spectrophotometry. SEM analysis was conducted to ensure particle shape accuracy levels. The results indicate successful catalysis of organic pollutants by Cu_2O catalysts in both cubic and octahedral synthesized form. The novel synthesis and differing absorbance levels among particle shapes highlight the promising nature of copper oxide catalysts, along with the need for additional testing in specific reactions on environmental pollutants under varying pH, light, and time characteristics.

Keywords: Copper Oxide Catalyst; Environmental Pollutants; nanoparticles; Spectrophotometry; SEM; PVP

1. Introduction

In recent years, there have been multiple approaches proposedfor synthesizing nanomaterials (such as nanoparticles) for various functions. In particular, attention has been given to the ability to obtain nanoparticles of specific sizes and shapes for use in targeted reactions. Multiple studies have reported the potential application of nanomaterials towards environmental remediation.[1] This consideration yields could offer much value due to the current rise in environmental pollutant levels year over year.[2]

Copper (I) oxide (Cu_2O) has emerged as a promising oxidative catalyst, capable of absorbing organic pollutants with limited environmental trace. Cu_2O is a known effective catalyst in solar energy conversion and is currently undergoing experimental trials commercially.[3]Sui et al. have reported the synthesis of cubic copper (I) oxide particles characterized by SEM analysis.[4] This particular study employs both cubic and octahedral shaped particles, testing each under the influence of light with positively and negatively charged dyes to quantify each facet's absorption spectra and relative effectiveness in known reactions.

2. Materials and Methods

2.1 Synthesis. All reagents were used as received. In a typical procedure, an aqueous solution was prepared by mixing 17 mL of DI water, 1 mL of 0.68 M copper sulfate, and 0.31 g or2.74 g of polyvinylpyrrolidone (PVP) depending on the desired shape. The former was calculated to produce cubes, while the latter was configured to produce octahedrons. The resulting solution was mixed on a VWR stir plate for 15-20 min. 1 mL of 0.74 M sodium citrate and 1 mL of 1.2 M anhydrous sodium carbonate were then added dropwise. The resulting solution was then stirred on the plate for 10 min. Next, 1.4 M glucose was added dropwise until the solution reached saturation, followed by another 10 min on the stir plate for proper dissolving. The precipitate was collected via centrifugation at 2500 rpm for 5 min. The tubes were then poured out, washed with 25 mL ethanol, and placed back in the centrifuge a total of three times. A small amount of nanoparticle solution was then injected into aluminum stubs for SEM characterization. Finally, the tubes were allowed to dry at room temperature for at least 12 h.

2.2 Characterization. To conduct size, shape, and atomic mass percentage analysis, the plugs were inserted into an FEI XL-30 Field Emission Scanning Electron Microscope (ESEM/SEM) with energy-dispersive X-ray spectroscopy (EDS) capability, previously described by Murphy et al.[5]

2.3 Photocatalytic Degradation. 5 mg of Copper (I) Oxide particles was transferred from the dried centrifuge tubes to labeled glass vials using a balance and metal stir rod. 30 separate microcentrifuge tubes were labeled using an alpha-numeric system to represent each glass vial at desired time intervals. 10 mL of Methyl Orange or Methylene Blue Dye was then added to each reaction vessel using a 10 mL graduated cylinder. Magnetic stir bars were utilized to facilitate absorption. The light apparatus consisting of two bulbs at a downward angle was constructed under the cover of an aluminum sheet to enclose the vessels under the beam. 200 μLof solution was transferred from each vessel to its respective micro-centrifuge tube at fixed time intervals for degradation analysis. The overall reaction time under the light was 60 min, excluding transfer periods. The interval-marked tubes were subsequently placed in a centrifuge for 2 min at 13,000 rpm. Following centrifugation, 90 μL of solution from each tube was placed into a weld plate for spectrophotometry using a pipette.

3. Results and Discussion

3.1 Materials Synthesis and Characterization. SEM analysis showed moderately precise cubes and octahedrons when analyzed under the electron beam [**Figures 1 & 2**]. The mean edge length for the synthesized cubic particles was approximately 1.6± 0.5μm, while the mean edge length for octahedrons was slightly larger at approximately 2.8± 0.7μm. It was noted that a limited number of cubic nanoparticles in each view appeared to have a hole protruding through the base, suggesting possible premature formation. The octahedrons were well-formed, however, a limited number in each slide were observed to be somewhat truncated. Mass spectrometry revealed an atomic mass percentage makeup of 24% Aluminum and 76% Copper. This ratio serves to support the makeup of the particles and minimalize the effects of any miscellaneous reagent remnants.

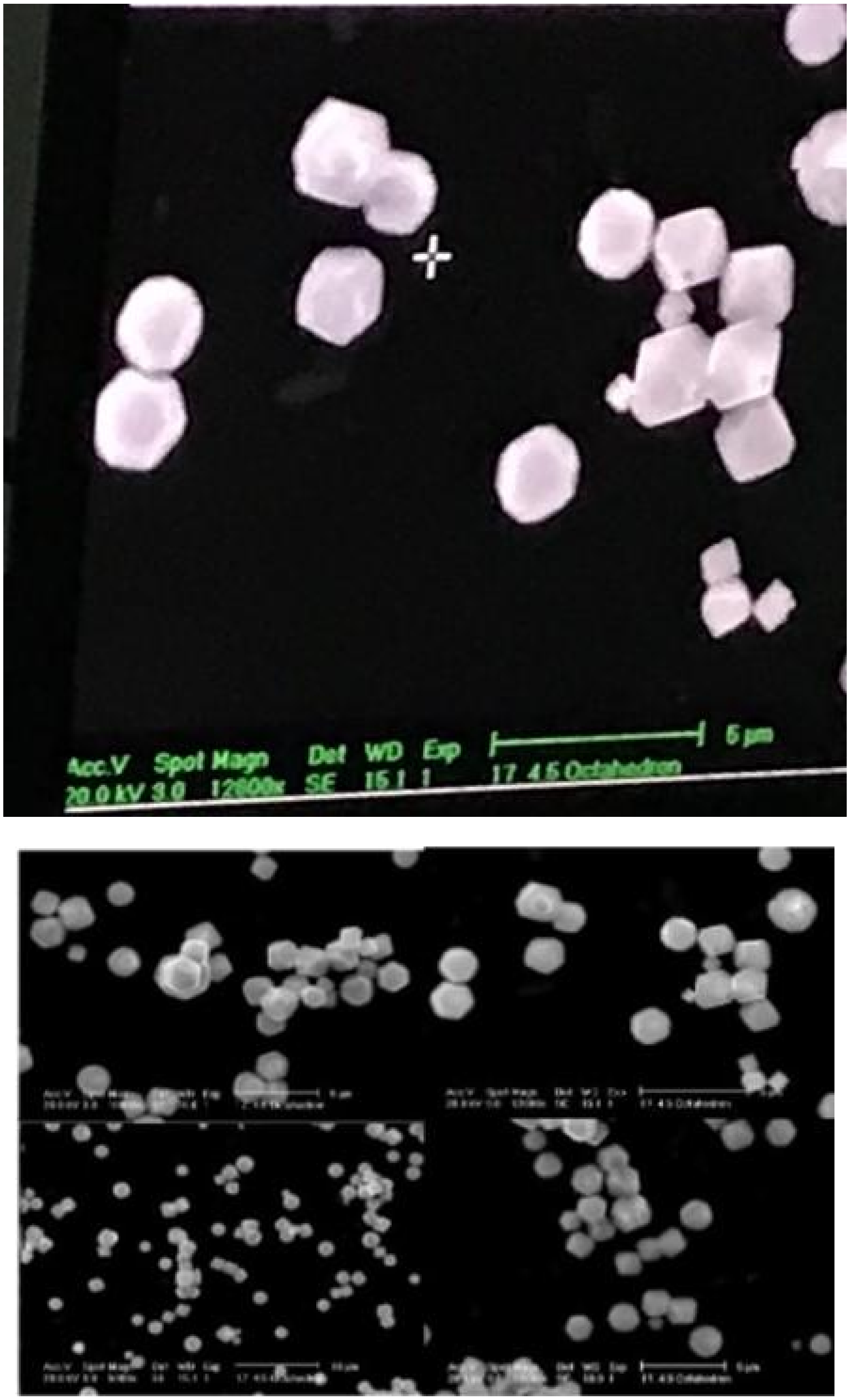

Figure 1. SEM Images of the Cu_2O Octahedrons Synthesized

Figure 2. SEM Images of the Cu_2O Cubes Synthesized.

3.2 Photocatalytic Reactivity. The weight measurement of dried Cu_2O nanoparticles showed slight variations within 0.5 mg, which were documented. These were accounted for in dye volume through ratio recalculation. In the photocatalytic degradation of methyl orange dye by the octahedral particles (4.5 mM), a third reaction vessel was unable to be used due to excessive moisture observed in the particles. As a result, 20 tubes were collected from two tubes to keep drying intervals constant. In the photocatalytic degradation of methyl blue dye by octahedral particles, one of the light bulbs lost power between the t=25 and t=30-min collection.

Results from the degradation absorbance graphs show consistent absorbance of the dye pollutants by the copper (I) oxide catalysts in octahedron particles on both dyes and cubic particles on the methyl orange dye. This can be seen in the negative slopes of the respective graphs, as well as similar absorption spectra y-intercepts.[4,6] The effect of octahedral particles on methyl orange dye was significantly greater than on the methylene blue dye, with the orange dye reaction producing a k-value of 0.0002 min^{-1}in magnitude greater than the methylene blue degradation [**Figures 3 & 4**]. The cubic nanoparticles had an irresolute effect on the methyleneblue dye, attributed to the scattered error bars and little precision displayed relative to the trendline [**Figure 5**]. This is highly indicative of no reaction taking place, resulting in uncorrelated data across the diagram. In this manner, the cubic nanoparticles proved more refined in degrading the methyl orange dye, with an absorbance of 0.0003 min^{-1} [**Figure 6**]. The dispersed results of the cubic methylene blue degradation are highly consistent with the expected electrostatics of the respective facets, and the SEM analysis may explain the observed differences in cubic degradation reactions between the dyes. This could be due to the holes observed in the(100) facets, which would be susceptible to higher or lower absorption to polar molecules such as the dyes depending on surface facet makeup. Even in the presence of similar defects, the (111) facets of the octahedrons showed attractive properties in degrading the negatively charged methyl orange dye. The smaller absorbance depicted by the octahedrons in the presence of the positively charged blue dye was predicted due to the shared charge among the(111) facet and dye molecules accordingly.

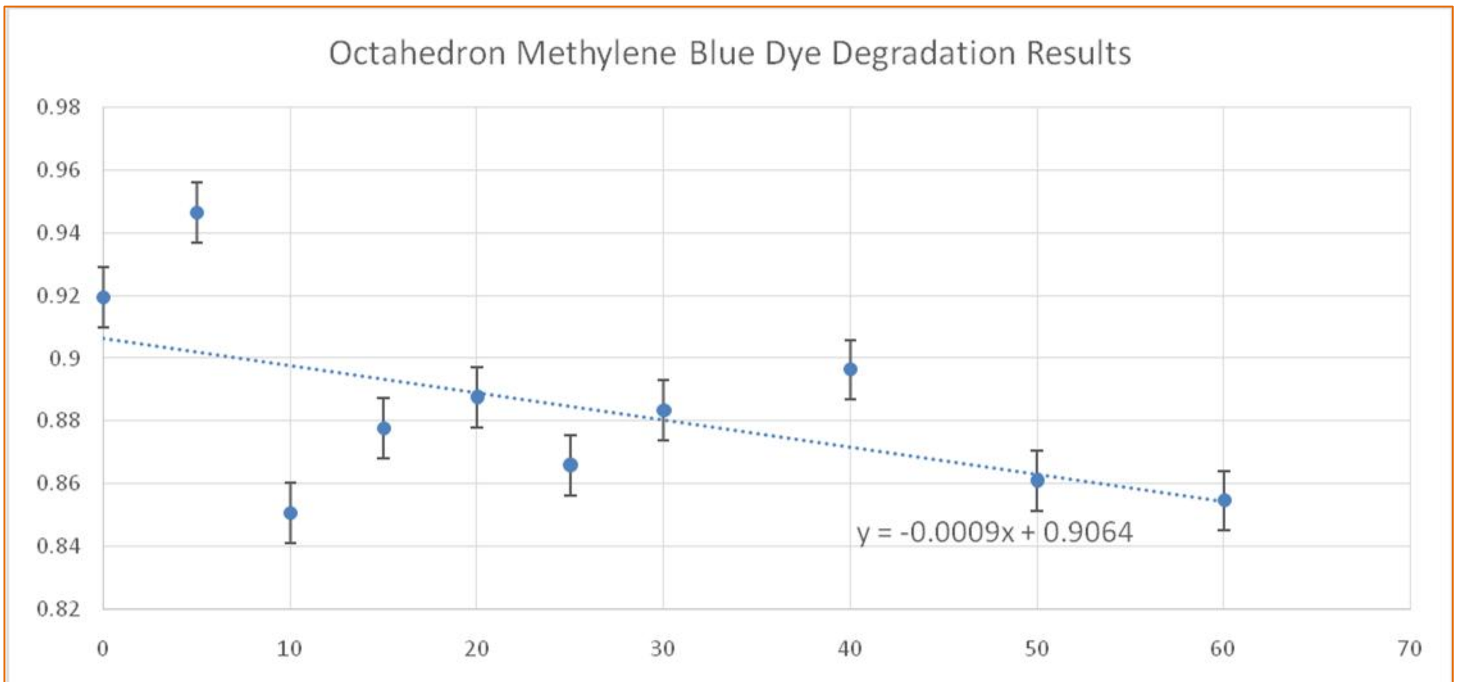

Figure 3. Graphical Representation of Methylene Blue Dye Degradation Results by Octahedral Copper (I) Oxide Nanoparticles.

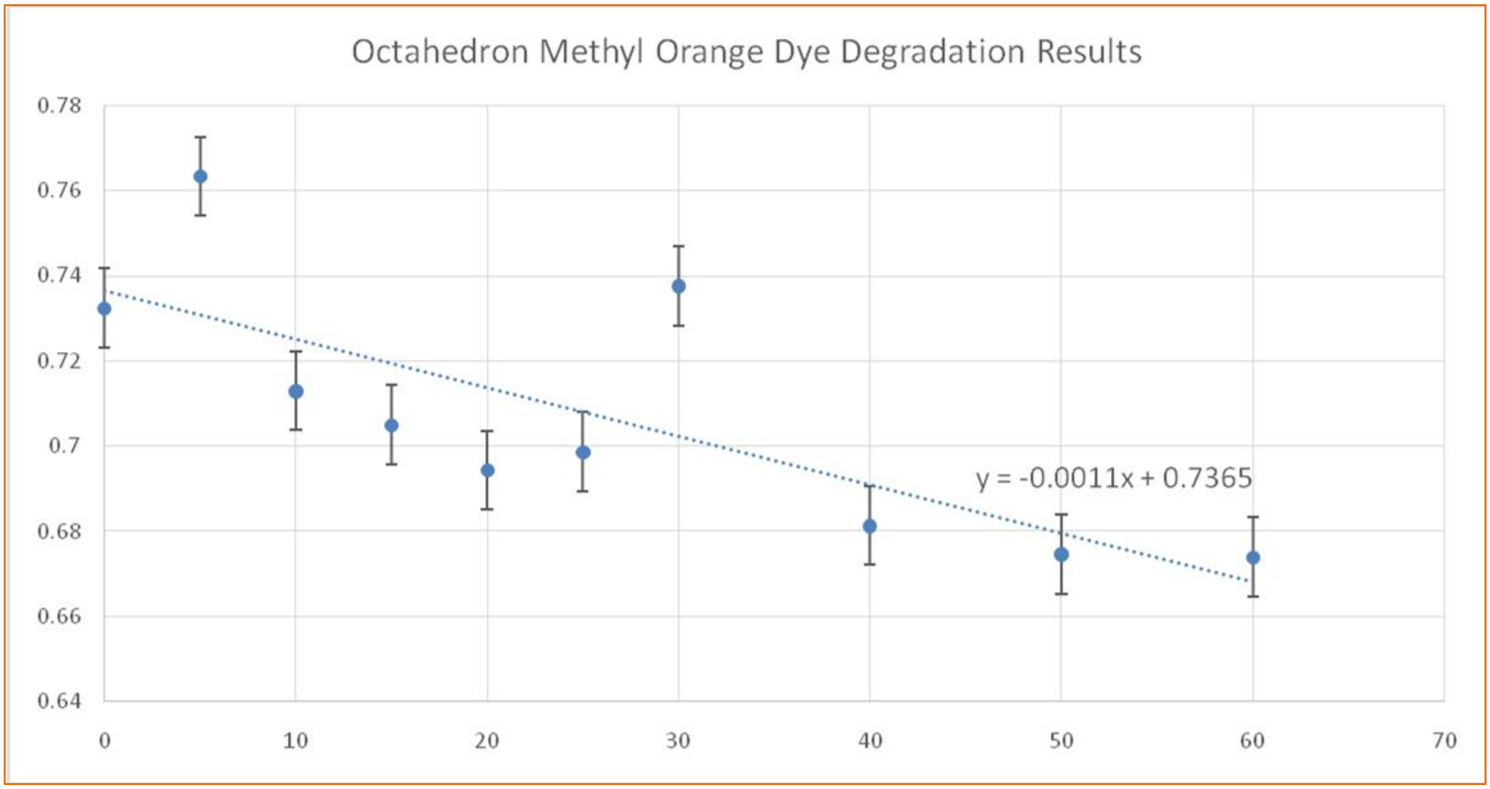

Figure 4. Graphical representation of Methyl Orange Dye Degradation Results by Octahedral Copper (I) Oxide Nanoparticles.

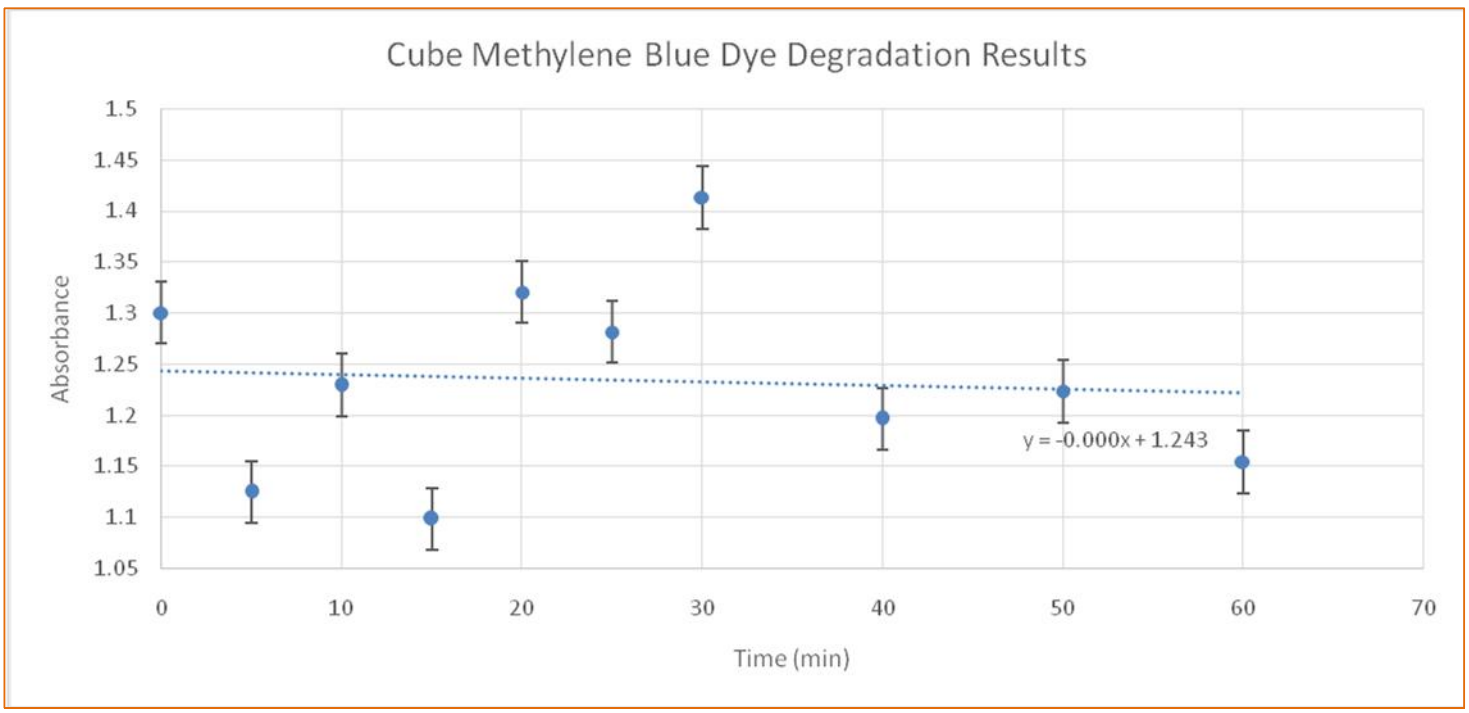

Figure 5. Graphical Representation of Methylene Blue Dye Degradation Results by Cubic Copper (I) Oxide Nanoparticles.

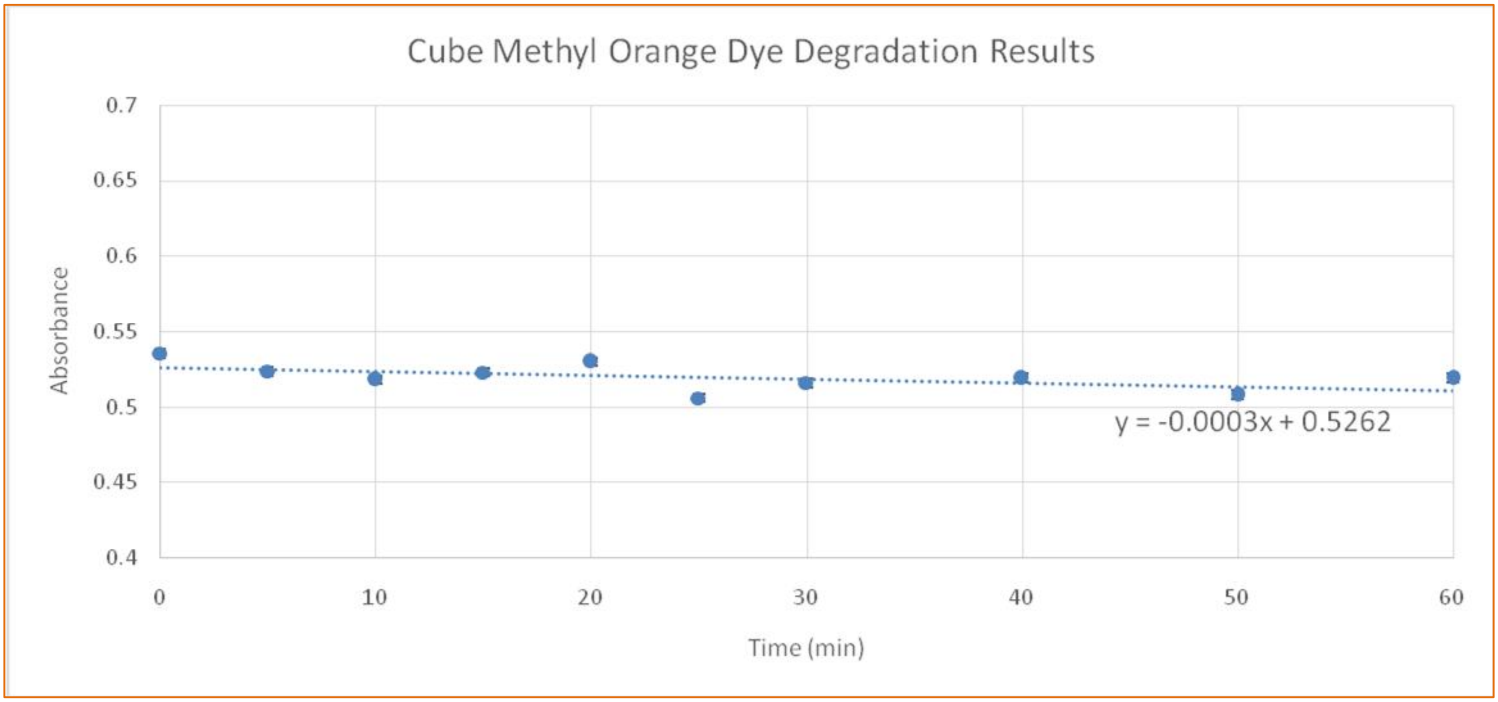

Figure 6. Graphical Representation* of Methyl Orange Dye Degradation Results by Cubic Copper (I) Oxide Nanoparticles.
*Error Bars included but hardly visible.

4. Conclusion

In this paper, we highlight a mechanism by which Cu_2O nanoparticles can be synthesized according to the desired shape based solely on the PVP levels used. The experimental viability of these particles can be analyzed in terms of both shape accuracy via qualitative inspection as well as atomic percentage through mass spectrometry. Degradation of polar dyes under the influence of light provided replicable circumstances for use in commercial byproduct environments. Our results echo the need for further research under additional variables such as pH, light, temperature, and pressure levels.[6] Copper (I) oxide proved to be a competent oxidative agent in the degradation of organic pollutants, as depicted in the graphical analysis. These results supported the hypothesis that the (111) facets of the Copper (I) Oxide octahedrons would degrade anionic methyl orange dye more rapidly than the methylene blue dye due to favorable electrostatic attractions. Similarly, the cubic nanoparticle results supported the prediction that methylene blue dye degradation would produce no evidence of a reaction taking place. However, the cubic nanoparticle reaction with anionicmethyl orange dye did reveal a correlated slope. This points to the need for further testing under extended time intervals. Unverified shapes in this experiment, such as spheres and truncated octahedrons, should also be studied to properly critique the elemental results of the slight malformations found under SEM.[6]

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Conflicts of Interest

The author declares no conflict of interest.

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