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Synthesis of Copper-Based Cupric Oxide (CuO) Black Pigment

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ABSTRACT: Copper-based inorganic pigments are widely utilized in industrial applications due to their high thermal stability and color intensity. This study aims to synthesize cupric oxide (CuO) black pigment through a precipitation method using $CuSO_4$ as a precursor and NaOH as a precipitating agent. The synthesis was conducted by reacting $CuSO_4$ and 2 M NaOH solutions at a constant temperature of 60 °C with a stirring duration of 30 minutes, followed by washing to achieve a neutral pH and drying at 80–120 °C. Six experimental trials were performed to evaluate product consistency and mass yield. The results demonstrated that the produced pigment weight ranged from 3.01 to 6.27 grams, corresponding to a mass yield percentage between 18.86 % and 39.29 %. The observed increase in yield across the trials suggests that operational consistency and effective post-synthesis treatments—specifically washing and drying—are decisive factors in determining the physical stability and color quality of the resulting black pigment.

Keywords: Copper, Pigment, Cupric Oxide Black, Precipitation, Mass Yield.

1. INTRODUCTION

Pigment is a substance that gives the impression of color to objects based on its response to light [1]. Historically, natural dyes derived from plants and animals were the primary coloring agents used in various applications. However, these natural pigments were often easily damaged by environmental influences such as UV radiation, temperature fluctuations, and chemical exposure, leading to the development of synthetic alternatives to provide better stability [2].

In modern industrial applications, inorganic pigments are generally preferred over organic ones due to their superior characteristics. These include high thermal stability, high hiding power, and excellent resistance to harsh chemical environments [3]. Transition metal oxides, in particular, have become a cornerstone in the production of high-performance pigments, as they offer a wide range of colors and maintain their structural integrity even under extreme processing conditions [4].

Copper (Cu) is a heavy metal naturally found in the environment, typically characterized by a reddish-yellow or orange color. As a transition metal in group IB with atomic number 29 and a melting point of 1083 °C, copper is highly versatile due to its ability to form various compounds through its Cu^{2+} ions [5,6]. Its unique electronic configuration allows for the development of diverse materials ranging from conductive alloys to functional ceramic pigments.

The determination and recovery of copper content from mineral rocks or industrial waste have been subjects of intensive research in the field of chemical engineering [7]. For instance, metal recovery from electronic waste (e-waste) or low-grade laterite ores often involves complex leaching and hydrometallurgical processes [8,9]. Converting these recovered copper precursors into value-added products, such as pigments, represents a sustainable approach to resource management and waste valorization.

Among the various forms of copper oxides, cupric oxide (CuO) is particularly valued for its deep black color and stability. Unlike cuprous oxide (Cu_2O), which is typically red, CuO is a monoclinic semiconductor that exhibits excellent optical properties suitable for industrial coatings, plastics, and ceramics [10]. The identification of specific pigment types and their potential antioxidant or catalytic properties is a critical step in advanced material synthesis [11].

The synthesis of CuO black pigment is commonly achieved through the chemical precipitation method. This route is favored in large-scale production due to its cost-effectiveness, simplicity, and the ability to control particle morphology through varying reaction conditions [12]. Copper pigments produced from the reaction between copper sulfate ($CuSO_4$) and sodium hydroxide (NaOH) have shown excellent color strength and environmental stability compared to other synthetic routes [13].

However, the quality and consistency of the resulting cupric oxide black pigment are highly dependent on several critical operational factors. Parameters such as reaction pH, temperature, and duration must be precisely controlled to ensure the desired phase purity and particle size distribution [14]. Furthermore, post-reaction treatments, including thorough washing to remove residual salts, drying at specific temperature ranges (typically 80–120 °C), and subsequent grinding, are decisive in determining the final pigment's physical characteristics and dispersibility [15].

The underlying mechanism of the precipitation process involves the formation of copper hydroxide ($Cu(OH)_2$) as an intermediate, which then undergoes dehydration to form CuO. The molarity of the precipitating agent and the stirring speed play vital roles in determining the nucleation and growth rates of the particles [16]. Despite its widespread use, achieving a high theoretical conversion while maintaining a specific yield remains a challenge in many laboratory and industrial setups.

This study aims to systematically evaluate the synthesis and characterization of copper-based black pigment via the precipitation method at a constant temperature of 60 °C. By analyzing six experimental trials, this research provides a detailed investigation into the relationship between precursor mass and the resulting pigment yield and conversion efficiency. The findings are expected to contribute to a better understanding of process stability for producing high-quality inorganic black pigments suitable for industrial-scale applications.

2. MATERIALS AND METHODS

2.1 Materials

The materials used in this research included copper (II) sulfate ($CuSO_4 \cdot 5H_2O$) as the copper precursor and sodium hydroxide (NaOH) as the precipitating agent. Distilled water was utilized for solution preparation and washing processes. Analytical grade filter paper and universal pH indicator strips were employed for separation and pH monitoring, respectively.

The experimental setup consisted of various laboratory glassware, including 200 mL and 500 mL beakers, 100 mL volumetric flasks, Erlenmeyer flasks, glass funnels, and pipettes. A magnetic stirrer equipped with a heating mantle and a magnetic stir bar was used for reaction control. Precise measurements were conducted using an analytical balance, and temperature monitoring was performed with a digital thermometer. A laboratory oven and porcelain crucibles were used for the drying and calcination stages.

2.2 Methodology

The synthesis of cupric oxide black pigment was initiated by preparing two primary solutions. Solution A was prepared by dissolving 15.96 grams of $CuSO_4$ in 100 mL of distilled water using a volumetric flask. Similarly, Solution B was prepared by dissolving 8 grams of NaOH in 100 mL of distilled water to achieve a concentration of 2 M.

The precipitation reaction was carried out in a 500 mL beaker containing 200 mL of distilled water as the reaction medium. Solution A was introduced into the reactor and heated under constant stirring until the temperature reached 60 °C. Subsequently, Solution B was added dropwise into the reactor until a black precipitate was formed. The mixture was maintained at 60 °C and stirred continuously for 30 minutes to ensure a complete reaction.

Upon completion of the reaction, the resulting slurry was filtered using filter paper. The collected precipitate was washed repeatedly with distilled water until the filtrate reached a neutral pH of 7. The neutralized sediment was then placed in a porcelain crucible and dried in an oven at temperatures ranging from 80 °C to 120 °C until a constant weight of pigment powder was obtained. The synthesis process was repeated six times to evaluate the consistency and reproducibility of the results.

Figure 1. Sediment Dry Experiment 1

Figure 2. Sediment Dry Experiment 2

3. RESULTS AND DISCUSSION

The synthesis of copper-based cupric oxide (CuO) black pigment was evaluated through six experimental trials using a consistent precursor mass of 15.96 g of $CuSO_4$. The results focusing on the final pigment weight and the corresponding mass yield are presented in Table 1.

Table 1. Mass yield and physical characteristics of the synthesized CuO pigments

No	Pigment weight (grams)	Yield (%)
1	3.01	18.86
2	4.35	27.26
3	5.29	33.14
4	5.57	34.89
5	6.02	37.71
6	6.27	39.29

The experimental data shows a clear increasing trend in the mass yield, rising from 18.86 % in the first trial to 39.29 % in the final trial. This variation suggests that while the initial precursor concentration remained constant, the efficiency of the precipitation process improved with subsequent trials, likely due to enhanced control over the stirring consistency and the dropwise addition of the NaOH precipitating agent [17].

The formation of CuO via the precipitation method is highly dependent on the reaction temperature and pH. In this study, maintaining a constant temperature of 60 °C was critical to facilitating the nucleation of $Cu(OH)_2$ particles and their subsequent transformation into the stable CuO phase. Previous research indicates that temperatures within the range of 60–80 °C provide sufficient energy to overcome the activation barrier for the dehydration of copper hydroxide intermediates [18].

Furthermore, the use of 2 M NaOH as a strong base ensured that the reaction environment reached an alkaline state. An alkaline pH (typically > 10) is necessary to promote the full precipitation of copper ions. Incomplete pH control often leads to the formation of undesired by-products or intermediate phases, such as basic copper sulfates, which can affect the final pigment color and purity [19].

The quality of the resulting black pigment is significantly influenced by post-reaction steps, particularly washing and drying. The washing process is essential to remove residual sodium sulfate (Na_2SO_4) salts that are formed as by-products. Insufficient washing may lead to an artificial increase in the final mass, which explains why the pigment weight in Trials 5 and 6 reached higher values compared to earlier trials.

Drying the precipitate at 80–120 °C ensures the complete removal of physically adsorbed water and the further dehydration of any remaining $Cu(OH)_2$. The transition to a deep black color confirms the successful formation of CuO, as this metal oxide is known for its high hiding power and chemical stability. Such characteristics make the produced CuO highly suitable for applications in the paint, plastic, and ceramic industries [20].

Beyond its traditional use as a coloring agent, CuO synthesized via precipitation is increasingly relevant in modern energy technologies. For instance, CuO nanostructures are widely explored as electrode materials in lithium-ion batteries and supercapacitors due to their high theoretical capacity and cost-effectiveness. The ability to produce consistent yields of CuO from common precursors like $CuSO_4$ highlights the potential for scaling up this process for sustainable material production on an industrial level [21].

4. CONCLUSION

The synthesis of copper-based cupric oxide (CuO) black pigment using the precipitation method with $CuSO_4$ and NaOH as precursors at a constant temperature of 60 °C was successfully demonstrated. Based on the six experimental trials conducted, the process yielded a stable black pigment with mass percentages ranging from 18.86 % to 39.29 %. The results indicate that the yield tends to increase with trial progression, suggesting that operational consistency and precise control over reaction parameters are vital for product stability.

The study concludes that critical factors, including pH control, reaction temperature, and post-reaction treatments such as washing and drying at 80–120 °C, significantly influence the final pigment's yield and physical characteristics. Furthermore, yields observed in the later trials suggest the presence of residual moisture or byproduct salts, emphasizing the necessity of thorough washing processes. Overall, this precipitation route provides a cost-effective and viable method for producing inorganic black pigments suitable for various industrial applications, particularly in the coatings and ceramic sectors.

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AUTHOR CONTRIBUTION

Conceptualization, A.L. and Z.F.; methodology, A.F.D.; software, A.L. and Z.F.; validation, A.L. and A.F.D.; formal analysis, Z.F.; investigation, A.L.; resources, A.L.; data curation, Z.F. and A.F.D.; writing—original draft preparation, A.L.; writing—review and editing, A.L. and A.F.D.; visualization, Z.F.; supervision, C.S. All authors have read and agreed to the published version of the manuscript.

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