**Facile Synthesis of Reduced Graphene Oxide and Metal Nanoparticles using Beta Carotene as Reducing Agent**

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(Use Times New Roman Font: 14 pt, Centered) (Names should be written in First Name Surname order)

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**Abstract.** The synthesis of reduced graphene oxide (rGO) and copper nanoparticles (CuNPs) was successfully carried out using β-carotene as a natural reducing and stabilizing agent. The reduction of graphene oxide was indicated by a visual color change from brown to blackish and confirmed by UV-Vis and FTIR spectroscopy. The UV-Vis spectra showed a red-shift from 232 nm (GO) to 298 nm (rGO), while FTIR spectra indicated a significant decrease in oxygen-containing functional groups, confirming effective reduction. Copper nanoparticles were synthesized by reacting CuCl₂ with β-carotene in the presence of ammonia, showing a characteristic surface plasmon resonance peak at 574 nm in the UV-Vis spectrum. Optimization studies revealed that a CuCl₂ to β-carotene volume ratio of 10:3 produced the highest absorbance (0.939) at 566 nm, indicating optimal nanoparticle formation. These findings demonstrate that β-carotene serves as an effective reductor for synthesizing both rGO and CuNPs, offering a sustainable approach to nanomaterial production.

*Keyword: β-carotene, copper nanoparticles, reduced graphene oxide, synthesis, redusing agent*

# INTRODUCTION

In the manufacturing industry, the reduction of metal oxides or other oxide materials is a crucial process that continues to be a major focus of research and development. This is due to the significant role that reduced oxides play across a wide range of applications, including catalysts, nanoparticles, electronics, energy storage (such as batteries), and energy conversion technologies [1]. Among these, Reduced Graphene Oxide (rGO) has emerged as a highly promising material for enhancing the electrochemical performance of lithium-ion batteries (LIBs), particularly in electric vehicles. According to Hao et al. (2020), the integration of rGO with SnS2 as an anode material in LIBs significantly improves battery performance—yielding a specific capacity of 1198 mA·h·g-1, an energy density of 149.5 W·h·kg⁻¹, a power density of 35 kW·kg-1, and a capacity retention of 90% after 10,000 charge-discharge cycles. This marks a performance increase of up to 50% compared to using pristine graphene oxide [2]. In recent years, various methods have been developed to reduce oxide materials, such as photoreduction, electrochemical reduction, the Hummers method, and chemical reduction using compounds such as sodium borohydride (NaBH4), hydrazine, ethylene glycol, or sodium citrate. While effective, these conventional methods often require stringent conditions such as high pressure, specific pH, or elevated temperatures, which drive up costs. Moreover, they may be ineffective at addressing surface defects in precursor materials and are generally unsuitable for reducing more stable oxides [3]. Furthermore, these chemical processes often produce toxic residues, posing serious environmental concerns [4,5]. To address these limitations, the use of environmentally friendly reducing agents, known as bioreductors, has gained increasing attention. One such promising compound is β-carotene, a naturally occurring pigment with strong antioxidant properties.

The use of β-carotene as a bioreductor is particularly compelling due to its dual function: not only does it effectively reduce metal and non-metal oxides into more functional reduced forms, but it also provides excellent oxidative stability to the resulting materials [6]. This enhances product longevity and preserves functionality. In addition, the functional groups present in β-carotene can interact with the surface of metal oxides, potentially modifying the resulting material’s physical and chemical characteristics—such as increasing its electrical conductivity or catalytic activity [7]. The novelty of this study lies in the synthesis of Reduced Graphene Oxide (rGO) and metal nanoparticles using β-carotene as both a reducing agent and a natural stabilizer. While many natural compounds have been explored for reduction processes, the specific application of β-carotene in this dual role remains underexplored. This work not only contributes to the development of sustainable nanomaterial synthesis methods but also opens new pathways for tailoring the properties of reduced materials approaches. Therefore, this research aims to develop a synthesis method for producing rGO and metal nanoparticles by employing β-carotene.

## EXPERIMENTAL

## Materials

The materials that used in this study were β-carotene, distilled water, N-Methyl-2-pyrrolidone (Merck, p.a), ammonia (NH4Cl, <25%, Merck, p.a), acetone (C3H6O, Merck, p.a), and copper chloride (CuCl2, >99%, Sigma-Aldirch).

## Synthesis of Reduced Graphene Oxide (rGO) using β-carotene

Reduced graphene oxide (rGO) was synthesized using β-carotene as a reducing agent. Initially, 0.5 g of graphene oxide was dispersed in 30 mL of N-Methyl-2-pyrrolidone (NMP) and subjected to ultrasonication for 30 minutes to achieve a homogeneous dispersion. Subsequently, 0.96 g of β-carotene extract was added to the GO dispersion, maintaining a weight ratio of 1:3 (GO:β-carotene). The resulting mixture was transferred into a reflux system and heated at 90 °C for 24 hours under continuous stirring. During this process, the color of the mixture changed gradually from brown to blackish-brown, indicating the reduction of graphene oxide. After the reaction, the mixture was cooled to room temperature, filtered, and washed repeatedly with distilled water to remove residual impurities and solvents. The final product was then dried in an oven at 70 °C for 24 hours. The synthesized rGO was characterized to confirm the reduction process and identify functional group changes. UV-Visible spectroscopy (UV-Vis) was used to monitor electronic transitions, while Fourier-transform infrared spectroscopy (FTIR) was performed to analyze functional group modifications associated with the reduction of GO.

## Synthesis of Reduced Graphene Oxide (rGO) using β-carotene

A copper chloride solution was prepared by dissolving the salt in acetone to obtain a clear and homogeneous solution. Separately, β-carotene was dissolved in acetone and added gradually to the copper chloride solution to initiate the reduction of copper ions. The reduction process was carried out at varying volume ratios of copper to β-carotene solutions: 10:1, 10:2, 10:3, and 10:4 (v/v). To facilitate the reduction process of nanoparticles, 2 mL of ammonia solution was added dropwise into each mixture under continuous stirring. The reaction was allowed to proceed for 7 hours at room temperature. After completion, the resulting solution was evaporated in an oven at 80 °C to remove the solvent and isolate the copper oxide nanoparticles (CuNPs). The obtained nanoparticles were then characterized using UV-Visible spectroscopy (UV-Vis) to confirm the formation of CuNPs and evaluate their optical properties.

# RESULT AND DISCUSSION

## Reduction of Graphene Oxide (rGO) using β-carotene

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**FIGURE 1.** Visual observation of the reduction process of graphene oxide (GO) using β-carotene.

The reduction of graphene oxide (GO) using β-carotene was conducted under reflux conditions at 90 °C for 24 hours. A visual indicator of successful reduction was the change in the color of the solution from brown to blackish, as illustrated in **Figure 1**. This color change is consistent with the restoration of the conjugated graphene structure, indicating the transformation of GO into reduced graphene oxide (rGO). To confirm the success of the reduction process, characterization was performed using UV-Vis and FTIR spectroscopy. According to previous studies, reduced graphene oxide typically exhibits a characteristic absorption peak in the range of 280–300 nm, which corresponds to the π → π\* transition of C=C bonds in the aromatic ring structure [8]. The UV-Vis spectra presented in **Figure 2** support these findings. GO exhibited a peak at 232 nm, which is associated with the π → π\* transition of the C=C bond in the disrupted sp2 domains of GO.

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**FIGURE 2**. UV-Vis absorption spectra of graphite, graphene oxide (GO), and reduced graphene oxide (rGO).

In contrast, rGO showed a red-shifted absorption peak at 298 nm, indicating the restoration of the extended π-conjugation in the graphene sheets. For comparison, graphite exhibited an absorption peak at 268 nm. These results confirm the partial recovery of the aromatic structure in rGO following the reduction process. Further analysis using FTIR spectroscopy, as shown in **Figure 3**, provides additional evidence of successful reduction. The FTIR spectrum of pristine GO shows a broad O–H stretching vibration around 3400 cm⁻1, a strong C=O stretching vibration around 1600 cm⁻1, and C–O stretching bands near 1050–1220 cm⁻1. These peaks are characteristic of oxygen-containing functional groups in GO [9]. After reduction with β-carotene, the FTIR spectrum of rGO reveals a significant decrease in the intensity of oxygen-related functional groups. The broad O–H stretching band is still present around 3500 cm⁻1 but with lower transmittance, and the C=O and C–O peaks are markedly reduced. Additionally, a peak at ~1600 cm⁻1 is still observed, attributed to C=C skeletal vibrations in the graphene structure, confirming partial restoration of the graphitic network. A comparative analysis of the FTIR spectra of graphite, GO, and rGO confirms the successful removal of oxygen functionalities and the recovery of conjugated C=C bonds, which is consistent with the structure of reduced graphene oxide [10]. These results validate that β-carotene can effectively serve as a reducing agent for the synthesis of rGO with enhanced structural and optical properties.

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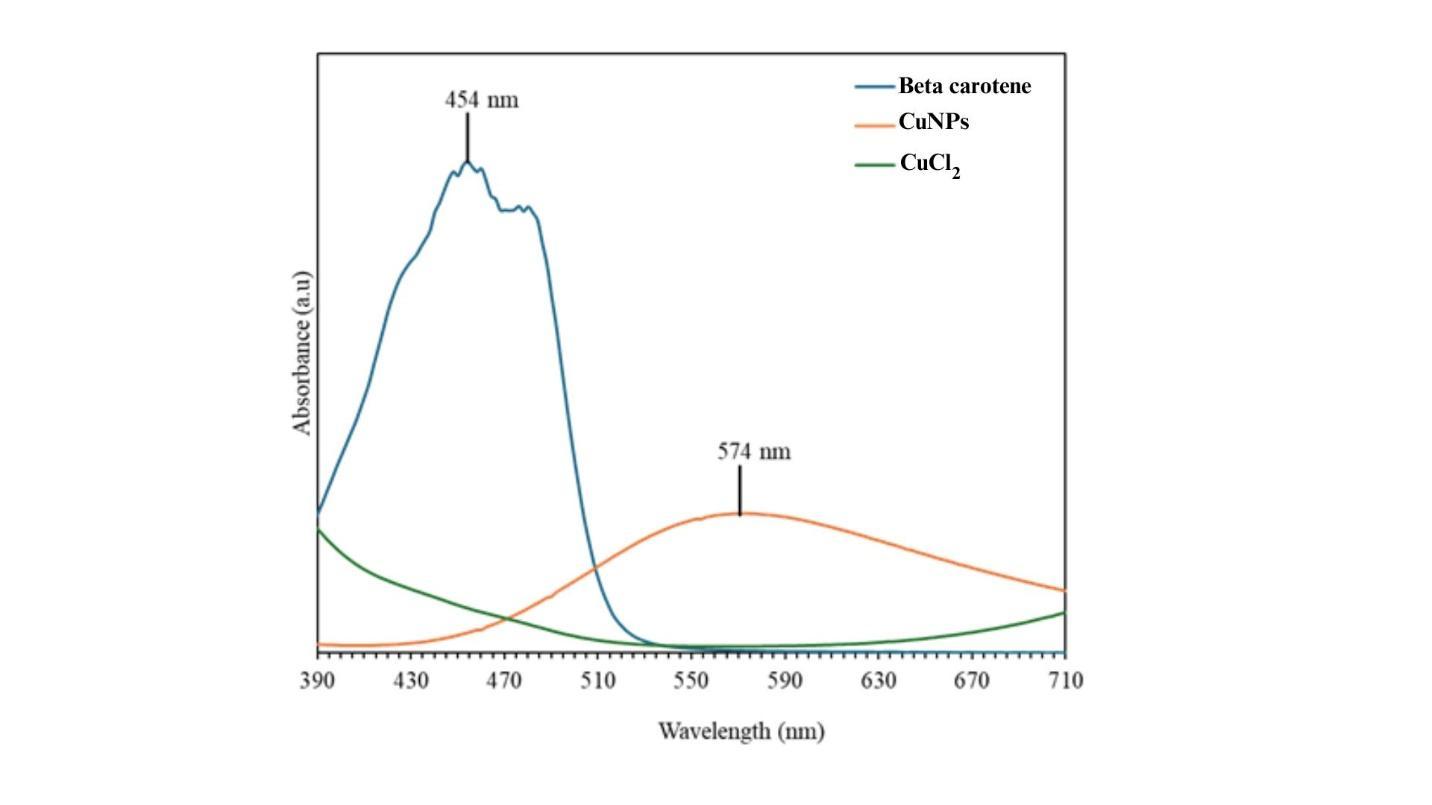
**FIGURE 3**. FTIR spectra of graphene oxide (GO), reduced graphene oxide (rGO), and pristine graphene.

## Formation of Copper Nanoparticles (CuNPs) using β-carotene

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**FIGURE 4.** Visual illustration of the synthesis process of copper nanoparticles (CuNPs) using β-carotene as a reducing agent.

A noticeable change in the color of the reaction solution—from light green to blue, and eventually to blackish brown—was observed during the synthesis of copper nanoparticles, as shown in **Figure 4**. The initial shift from green to blue upon the addition of ammonia is attributed to the formation of the coordination complex [Cu(NH3)3]2+ [11], indicating the initial activation of copper ions prior to reduction. The mechanism of nanoparticle formation involves the reduction of Cu2+ ions by β-carotene, a natural bioreductant. In this reaction, Cu2+ ions are reduced to metallic copper (Cu0), which then undergo nucleation. These nuclei aggregate and stabilize to form copper nanoparticles (CuNPs) [12]. The UV-Vis spectral analysis, as presented in **Figure 5**, further confirms the formation of CuNPs. β-carotene alone exhibits absorption peaks in the range of 430–480 nm due to its π → π\* transitions. In the presence of CuNPs, a new, broad absorption peak emerges at 574 nm, corresponding to the surface plasmon resonance (SPR) of metallic copper nanoparticles [13]. This SPR band is typical of CuNPs and usually falls within the 500–600 nm range, depending on particle size, shape, and surrounding environment [14].



**FIGURE 5**. UV-Vis absorption spectra of CuCl2, β-carotene, and copper nanoparticles (CuNPs).

To optimize the synthesis process, various volume ratios of CuCl2 to β-carotene were tested. As shown in **Figure 6**, the absorbance of the colloidal solution increased with β-carotene concentration up to a certain point. Higher absorbance indicates a greater quantity of nanoparticles formed in suspension. The optimum condition was achieved at a volume ratio of 10:3 (CuCl2:β-carotene), which yielded the highest absorbance value of 0.939 and a peak wavelength of 566 nm. This suggests efficient reduction and favorable nanoparticle formation at this ratio. The detailed influence of β-carotene volume on absorbance and peak wavelength is summarized in **Table 1**.

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**FIGURE 6.** UV-Vis absorption spectra of copper nanoparticles (CuNPs) synthesized at various volume ratios of CuCl₂ to β-carotene: 10:1, 10:2, 10:3, and 10:4.

**Table 1.** Effect of β-carotene volume variation on the wavelength and absorbance of copper nanoparticle (CuNP) formation.

| Variation | Wavelenght (λ) | Absorbance (a.u) |
| --- | --- | --- |
| 10:1 | 574 nm | 0.348 |
| 10:2 | 569 nm | 0.696 |
| 10:3 | 566 nm | 0.939 |
| 10:4 | 566 nm | 0.870 |

# CONCLUSION

This study demonstrated the successful synthesis of reduced graphene oxide (rGO) and copper nanoparticles (CuNPs) using β-carotene as a biocompatible reducing and stabilizing agent. The reduction of GO was evidenced by a red-shift in the UV-Vis absorption peak and a decrease in oxygen functional groups observed in FTIR spectra, confirming the partial restoration of the graphene conjugated structure. For CuNPs synthesis, the reaction process involved the formation of a [Cu(NH₃)₃]²⁺ complex followed by reduction with β-carotene. The resulting nanoparticles displayed a distinct surface plasmon resonance peak at 574 nm, validating successful nanoparticle formation. Optimization experiments identified a CuCl₂:β-carotene volume ratio of 10:3 as the most effective, yielding the highest absorbance and suitable wavelength, indicating efficient particle formation.

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