# Green Synthesis of Cobalt Oxide/Gold (Coo/Au) Bimetallic Nanoparticles Using Sinapinic Acid: A Comprehensive Study

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## ABSTRACT

This study reports the successful green synthesis of cobalt oxide/gold (CoO/Au) bimetallic nanoparticles using sinapinic acid as both a reducing and stabilizing agent. The eco-friendly synthesis approach eliminates the need for hazardous chemicals while producing nanoparticles with superior properties. The formation of CoO/Au bimetallic nanoparticles was confirmed through visual observation of color changes from brown to greyish-black upon addition of $HAuCl_4$ to the CoO nanoparticle solution. Comprehensive characterization using UV-Visible spectroscopy revealed distinct surface plasmon resonance peaks at 350 nm and 650 nm corresponding to CoO and Au components, respectively. FTIR analysis confirmed the role of sinapinic acid functional groups in nanoparticle reduction and stabilization, with a characteristic Co-O-Au stretching band at 575 $cm^{-1}$. X-ray diffraction patterns demonstrated the face-centered cubic (FCC) crystalline structure of the bimetallic system, while transmission electron microscopy revealed discrete, spherical nanoparticles with an average diameter of 24.4 nm and single crystalline nature. The synthesized CoO/Au bimetallic nanoparticles exhibit enhanced structural stability and demonstrate significant potential for applications in catalysis, biosensing, and biomedical fields.

**Keywords:** Bimetallic nanoparticles, Green synthesis, Sinapinic acid, Cobalt oxide, Gold nanoparticles, Surface plasmon resonance

## INTRODUCTION

Nanotechnology has emerged as a revolutionary field in materials science and chemistry, offering innovative solutions across diverse applications including catalysis, medicine, environmental remediation, and electronics [1,2]. Metallic nanoparticles have garnered significant attention due to their unique physicochemical properties that arise from their nanoscale dimensions and high surface-to-volume ratios [3]. However, conventional synthesis methods often rely on hazardous chemicals and energy-intensive processes, necessitating the development of environmentally benign alternatives.

Plant-derived phenolic acids, including caffeic acid, ferulic acid, p-coumaric acid, and sinapinic acid, have emerged as promising eco-friendly reducing agents for nanoparticle synthesis. These natural compounds possess inherent antioxidant properties that enable them to function as both reducing agents and stabilizers during nanoparticle formation [4,5]. This dual functionality eliminates the need for additional capping agents while ensuring particle stability and preventing agglomeration.

While single-metal nanoparticles such as cobalt oxide or gold possess valuable properties, their applications are often limited by their individual characteristics. Bimetallic nanoparticles, composed of two distinct metallic components, offer synergistic effects that can significantly enhance optical, electronic, magnetic, and catalytic properties compared to their monometallic counterparts [6,7]. The combination of cobalt oxide with gold creates materials with superior plasmonic responses, enhanced magnetic properties, and improved biocompatibility, making them particularly attractive for biomedical applications [8].

Cobalt oxide nanoparticles exhibit excellent catalytic activity and demonstrate promising applications in antimicrobial treatments, cellular labeling, and biosensor development. Gold nanoparticles are renowned for their unique optical properties, biocompatibility, and applications in drug delivery systems and diagnostic imaging. The integration of these two materials into a bimetallic system is expected to yield nanoparticles with combined advantages and novel functionalities.

The present study aims to develop a green synthesis protocol for CoO/Au bimetallic nanoparticles using sinapinic acid as both the reducing and stabilizing agent. The synthesized nanoparticles were comprehensively characterized using multiple analytical techniques to evaluate their optical, structural, and morphological properties, with particular emphasis on their potential for various technological and biomedical applications.

## MATERIALS AND METHODS

### Materials

High-purity chemicals were employed for the synthesis: sinapinic acid and chloroauric acid ($HAuCl_4$) were procured from Sigma-Aldrich, while ethanol (99% purity), cobalt nitrate [$Co(NO_3)_2$], sodium hydroxide (NaOH), and deionized water were obtained from Nice Chemicals. All chemicals were used as received without further purification.

### Synthesis Protocol

### Preparation of Stock Solutions

A 0.005 N stock solution of sinapinic acid was prepared by dissolving 0.1121 g of sinapinic acid in 10 mL of 99% ethanol, followed by dilution to 100 mL with distilled water to ensure complete dissolution and homogenization. Similarly, a 0.005 N $HAuCl_4$ solution was prepared by dissolving 0.1966 g of chloroauric acid in 100 mL of deionized water under gentle stirring until complete dissolution.

### Synthesis of CoO/Au Bimetallic Nanoparticles

The synthesis was carried out through a sequential reduction process. Initially, 25 mL of 0.005 N $Co(NO_3)_2$ solution was mixed with 5 mL of heated sinapinic acid solution (0.005 N) in the presence of dilute alkali under continuous magnetic stirring at room temperature. The appearance of a light brown coloration indicated the successful formation of CoO nanoparticles through the reduction of cobalt ions by sinapinic acid.

Subsequently, 15 mL of 0.005 N $HAuCl_4$ solution was slowly added dropwise to the CoO nanoparticle suspension while maintaining constant stirring. The immediate color change from light brown to greyish-black confirmed the formation of CoO/Au bimetallic nanoparticles. The reaction mixture was allowed to proceed for 30 minutes to ensure complete reduction and stabilization.

The resulting nanoparticles were collected by centrifugation at 10,000 rpm for 15 minutes, followed by multiple washing cycles with deionized water and ethanol to remove unreacted precursors and byproducts. The purified nanoparticles were then dried at 60°C for characterization studies.

### Characterization Techniques

### UV-Visible Spectroscopy

Optical properties were analyzed using a Cary 5000 UV-Vis-NIR spectrophotometer at the Central Laboratory for Instrumentation and Facilitation (CLIF), University of Kerala. Measurements were conducted in the wavelength range of 400-800 nm using quartz cuvettes with a path length of 1 cm.

### Fourier Transform Infrared (FTIR) Spectroscopy

Functional group analysis was performed using a Thermo Scientific Nicolet iS50 FT-IR spectrometer at CLIF, University of Kerala. Samples were prepared as KBr pellets and analyzed in the range of 400-4000 $cm^{-1}$ with a resolution of 4 $cm^{-1}$.

### X-ray Diffraction (XRD)

Crystallographic analysis was conducted using a Bruker D8 Advance X-ray diffractometer with Cu Kα radiation (λ = 1.5406 Å) operating at 40 kV and 40 mA. Diffraction patterns were recorded in the 2θ range of 20-90° with a step size of 0.02°.

### Transmission Electron Microscopy (TEM)

Morphological characterization was performed using a JEOL JEM-2100 transmission electron microscope at DST-SAIF, Cochin, Kerala. The instrument operated at 200 kV with a point resolution of 0.23 nm and lattice resolution of 0.14 nm. Samples were prepared by dropping diluted nanoparticle suspension onto carbon-coated copper grids and allowing them to dry under ambient conditions.

## RESULTS AND DISCUSSION

### UV-Visible Spectroscopy Analysis

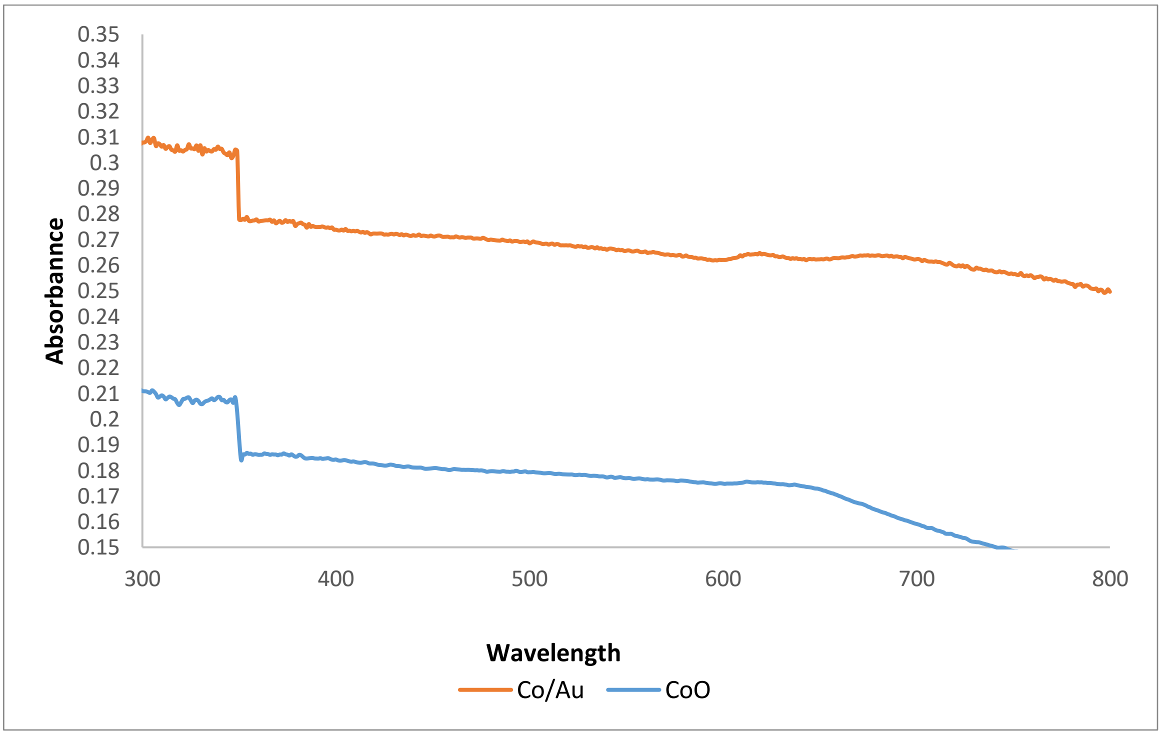

Figure 1: UV-Visible spectrum of CoO and CoO/Au bimetallic nanoparticles showing characteristic absorption peaks at 350 nm (CoO) and 650 nm (Au SPR).

UV-Visible spectroscopy (**Figure 1)** provided crucial evidence for the successful formation of CoO/Au bimetallic nanoparticles. The spectrum of CoO nanoparticles exhibited a sharp absorption peak at 350 nm, characteristic of cobalt oxide nanoparticles. This peak corresponds to the charge transfer transitions within the CoO structure and confirms the successful reduction of $Co^{2+}$ ions to form CoO nanoparticles.

The CoO/Au bimetallic system displayed both the characteristic CoO peak at 350 nm and an additional broad absorption band centered around 650 nm. This second peak is attributed to the surface plasmon resonance (SPR) of gold nanoparticles, which arises from the collective oscillation of conduction electrons when excited by electromagnetic radiation. The coexistence of both peaks in the bimetallic system provides clear evidence for the successful incorporation of gold into the CoO matrix, confirming the formation of CoO/Au bimetallic nanoparticles.

The broad nature of the gold SPR peak suggests the presence of gold nanoparticles with a size distribution, which is typical for chemically synthesized nanoparticles. The slight red-shift of the gold SPR peak compared to pure gold nanoparticles indicates possible electronic interactions between the CoO and Au components in the bimetallic structure.

### FTIR Spectroscopy Results

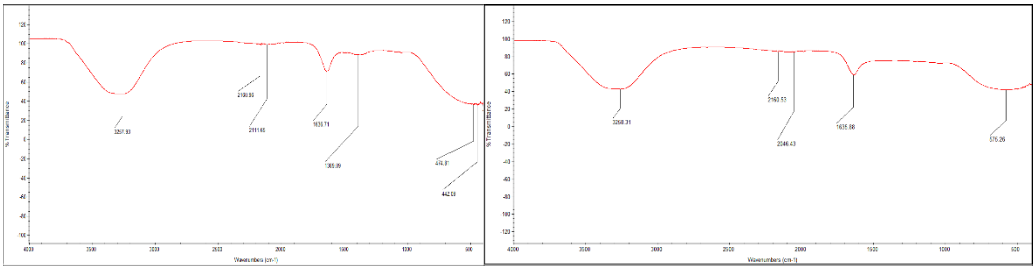

Figure 2: FTIR spectra of (a) CoO nanoparticles and (b) CoO/Au bimetallic nanoparticles showing characteristic functional group vibrations and the appearance of Co-O-Au stretching at 575 cm$^{-1}$.

FTIR analysis (**Figure 2)** provided detailed information about the functional groups involved in the reduction and stabilization processes. The spectrum of CoO nanoparticles revealed several characteristic peaks: a broad peak at 3257 cm$^{-1}$ corresponding to O-H stretching vibrations from sinapinic acid, peaks at 2161 cm$^{-1}$ and 2111 cm$^{-1}$ attributed to C-H stretching modes, a peak at 1636 cm$^{-1}$ indicating C=O stretching, and importantly, a peak at 474 cm$^{-1}$ representing Co-O stretching vibrations.

The FTIR spectrum of CoO/Au bimetallic nanoparticles showed notable changes: the O-H stretching remained at 3258 cm$^{-1}$, C-H stretching was observed at 2046 cm$^{-1}$ (shifted from the CoO spectrum), C=O stretching appeared at 1635 cm$^{-1}$, and most significantly, a new peak emerged at 575 cm$^{-1}$ corresponding to Co-O-Au stretching vibrations. This new peak provides direct evidence for the formation of bimetallic bonds between cobalt oxide and gold components.

The shifts in vibrational frequencies and the appearance of the Co-O-Au stretching mode confirm that sinapinic acid successfully acts as a bridging ligand, facilitating the formation of bimetallic structures while simultaneously serving as a stabilizing agent. The presence of organic functional groups on the nanoparticle surface also contributes to their stability and prevents agglomeration.

### X-ray Diffraction Analysis

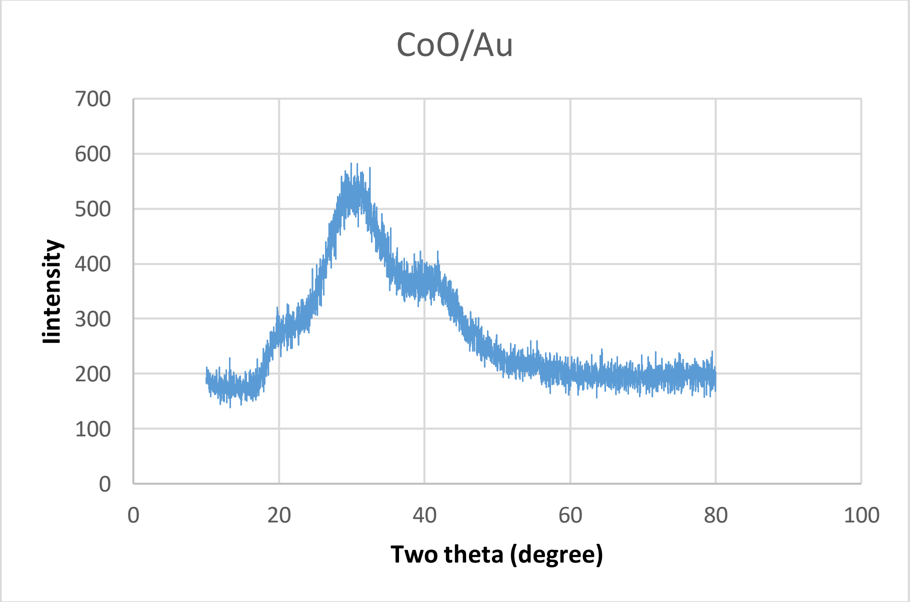

Figure 3: XRD pattern of CoO/Au bimetallic nanoparticles showing characteristic FCC crystal planes at (111), (200), (220), and (311) positions.

XRD analysis (**Figure 3)** revealed the crystalline nature of the CoO/Au bimetallic nanoparticles. The diffraction pattern exhibited peaks at 2θ values of 39.98°, 46.4°, 67.73°, and 81.6°, corresponding to the (111), (200), (220), and (311) crystallographic planes, respectively. These reflections are characteristic of a face-centered cubic (FCC) crystal structure, which is typical for gold nanoparticles and certain cobalt oxide phases. Diffraction Analysis

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The sharp and well-defined diffraction peaks indicate high crystallinity of the nanoparticles. The presence of FCC reflections suggests that the bimetallic system adopts a structure dominated by the gold component, with cobalt oxide either forming a separate phase or being incorporated into the gold lattice structure. The absence of distinct cobalt oxide peaks might indicate either the formation of a solid solution or the presence of very small CoO domains that are below the detection limit of XRD.

The crystallite size calculated using the Scherrer equation from the (111) reflection was found to be consistent with the particle sizes observed in TEM analysis, confirming the single-crystalline nature of individual nanoparticles.

### Transmission Electron Microscopy

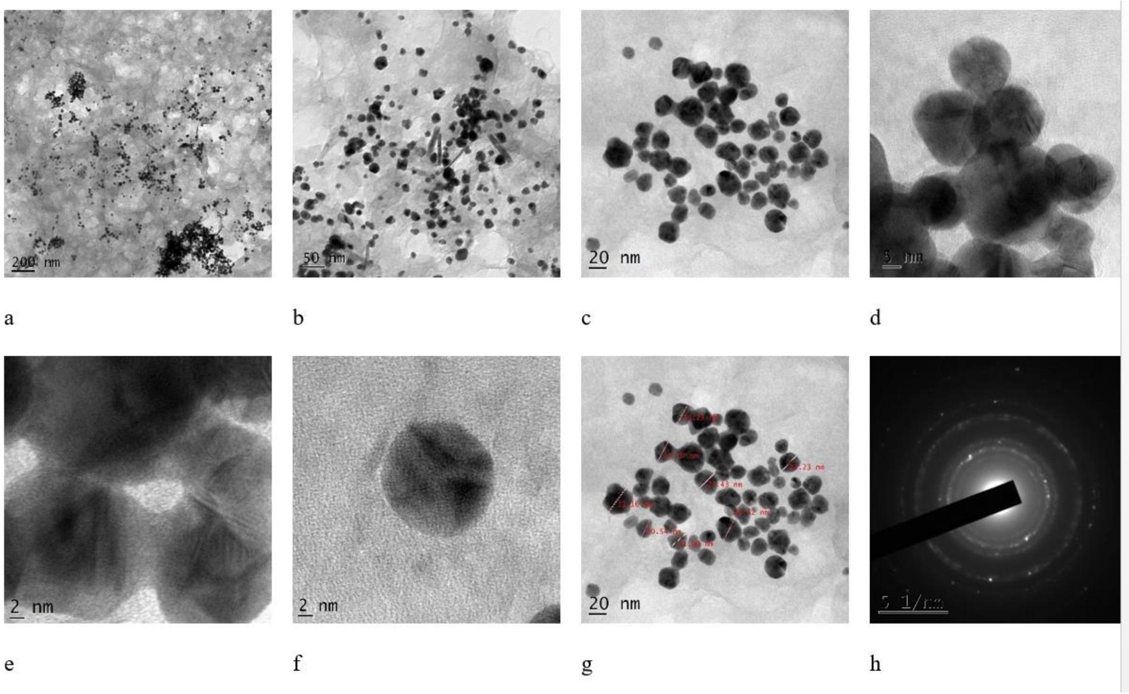

Figure 4: (a-g) TEM images of CoO/Au bimetallic nanoparticles at various magnifications showing discrete, spherical morphology and (h) particle size analysis with SAED pattern confirmation.

TEM analysis (**Figure 4)** provided detailed morphological information about the CoO/Au bimetallic nanoparticles. The micrographs revealed that the nanoparticles are discrete, well-dispersed, and predominantly spherical in shape with minimal aggregation. The particle size distribution analysis showed an average diameter of 24.4 nm, indicating good size control achieved through the green synthesis approach.

High-resolution TEM images demonstrated the crystalline nature of individual nanoparticles, with clearly visible lattice fringes confirming their single-crystalline structure. The selected area electron diffraction (SAED) pattern exhibited concentric rings rather than discrete spots, indicating the polycrystalline nature of the sample ensemble while individual particles remain single crystalline.

The absence of significant particle agglomeration indicates effective stabilization by sinapinic acid, which forms a protective organic layer around the nanoparticles. This stabilization is crucial for maintaining the unique properties of nanoscale materials and preventing their degradation over time.

### Mechanistic Insights

The green synthesis mechanism involves the reduction of metal ions by the phenolic groups present in sinapinic acid. The hydroxyl groups in sinapinic acid donate electrons to reduce $Co^{2+}$ to CoO and $Au^{3+}$ to $Au^0$, while the phenolic acid molecules undergo oxidation. The sequential addition of metal precursors allows for controlled formation of the bimetallic structure, with CoO serving as nucleation sites for gold deposition.

The stabilization mechanism involves the adsorption of sinapinic acid molecules onto the nanoparticle surface through various functional groups, including hydroxyl, carboxyl, and methoxy groups. This organic capping layer prevents particle agglomeration and provides long-term stability to the colloidal system.

### Applications and Future Perspectives

The synthesized CoO/Au bimetallic nanoparticles demonstrate significant potential for various applications:

### Catalytic Applications

The combination of cobalt oxide's catalytic activity with gold's unique electronic properties makes these bimetallic nanoparticles promising candidates for heterogeneous catalysis, particularly in oxidation reactions and environmental remediation processes.

### Biosensing and Biomedical Applications

The enhanced optical properties arising from the SPR of gold, combined with the magnetic properties of cobalt oxide, make these nanoparticles suitable for biosensing applications, medical imaging, and targeted drug delivery systems.

### Electronic and Optoelectronic Devices

The unique electronic properties of the bimetallic system could be exploited in the development of advanced electronic devices, photovoltaic cells, and optical sensors.

## CONCLUSION

This study successfully demonstrates the green synthesis of CoO/Au bimetallic nanoparticles using sinapinic acid as both a reducing and stabilizing agent. The comprehensive characterization confirms the formation of discrete, spherical nanoparticles with an average size of 24.4 nm and single-crystalline FCC structure. The UV-Visible spectroscopy confirms the presence of both CoO and Au components through their characteristic absorption peaks, while FTIR analysis reveals the crucial role of sinapinic acid in nanoparticle stabilization through the formation of Co-O-Au bonds.

The green synthesis approach offers several advantages including environmental friendliness, cost-effectiveness, and the elimination of hazardous chemicals. The resulting bimetallic nanoparticles exhibit enhanced stability and properties compared to their monometallic counterparts, making them attractive for various technological and biomedical applications.

Future research directions should focus on optimizing the synthesis parameters to achieve better size control, investigating the detailed structure-property relationships, and exploring specific applications in catalysis, sensing, and biomedicine. Additionally, scaling up the synthesis process for industrial applications and conducting comprehensive toxicity studies would be valuable for practical implementation.

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