

Synthesis & Characterization of Bimetallic Nanoparticles

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Abstract

This research focuses on the synthesis and characterization of bimetallic nanoparticles, which exhibit unique properties due to the synergistic effects between two different metals at the nanoscale. The primary objective of the study was to develop and optimize various synthesis methods, including chemical reduction, physical methods, and green synthesis techniques, to produce nanoparticles with controlled size, morphology, and composition. Advanced characterization techniques, such as transmission electron microscopy (TEM), X-ray diffraction (XRD), and Fourier-transform infrared spectroscopy (FTIR), were employed to analyze the structural, optical, and thermal properties of the synthesized nanoparticles. Key findings include the identification of optimal conditions for producing bimetallic nanoparticles with enhanced stability and catalytic activity. The significance of this research lies in its contribution to the growing body of knowledge on nanoparticle synthesis, providing insights that could lead to improved industrial applications and innovative solutions in various technological domains.

Keywords: Bimetallic nanoparticles, synthesis, characterization, TEM, XRD, FTIR, catalysis, biomedicine, environmental remediation.

Introduction

Context and Importance

Nanotechnology, the science of manipulating matter at the atomic and molecular scale, has revolutionized numerous fields, including medicine, environmental science, and materials engineering. Central to this advancement are nanoparticles, which exhibit unique properties due to their diminutive size and large surface area. Among these, bimetallic nanoparticles have emerged as particularly significant, offering enhanced performance and multifunctionality compared to their monometallic counterparts.

Bimetallic nanoparticles consist of two different metals, which when combined, often exhibit synergistic effects that result in superior chemical, physical, and catalytic properties. These nanoparticles are distinguished by their ability to merge the distinct advantages of each metal into a single entity, providing benefits such as improved stability, increased catalytic activity, and tunable optical properties.

The advantages of bimetallic nanoparticles over monometallic ones are manifold. While monometallic nanoparticles are limited by the intrinsic properties of a single metal, bimetallic variants can be engineered to optimize performance by carefully selecting and combining metals with complementary characteristics.

Objectives

The primary objectives of this research are multifaceted, aiming to push the boundaries of current knowledge and application of bimetallic nanoparticles. First and foremost, the study seeks to develop and refine synthesis methods for bimetallic nanoparticles, with a focus on achieving

greater control over their size, shape, and compositional uniformity. This involves exploring various chemical, physical, and biological synthesis techniques to identify optimal conditions that yield nanoparticles with consistent and desirable properties.

Secondly, the research emphasizes the importance of comprehensive characterization of the synthesized nanoparticles. This involves the use of advanced analytical techniques such as transmission electron microscopy (TEM), X-ray diffraction (XRD), and Fourier-transform infrared spectroscopy (FTIR) to thoroughly investigate the structural, optical, and thermal properties of the nanoparticles.

Finally, the study aims to explore the practical applications of bimetallic nanoparticles across multiple fields, including catalysis, biomedicine, and environmental science. In the biomedical field, the research will explore the use of these nanoparticles in drug delivery systems and diagnostic tools, leveraging their biocompatibility and functional versatility.

Experimental Section

Materials and Methods

Chemicals and Precursors

1. Metal Precursors:

- **Gold Precursor:** Gold(III) chloride trihydrate ($\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$) was used as the gold source due to its high solubility in water and stability, which is crucial for forming uniform nanoparticles. The gold ions from this compound serve as one of the core components in bimetallic structures.
- **Silver Precursor:** Silver nitrate (AgNO_3) was selected as the silver source because of its well-known reactivity and ability to form stable silver ions in solution, which facilitates the formation of the bimetallic alloy.
- **Copper Precursor:** Copper(II) sulfate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) was chosen for its effectiveness in producing copper ions that can readily participate in redox reactions with other metal ions.

2. Reducing Agents:

- **Sodium Borohydride (NaBH_4):** This strong reducing agent was used to reduce the metal ions to their zero-valent state, thereby initiating nanoparticle formation. NaBH_4 is particularly effective for the rapid reduction of noble metals.
- **Ascorbic Acid ($\text{C}_6\text{H}_8\text{O}_6$):** Used as a mild reducing agent, ascorbic acid allows for slower reduction rates, which is beneficial for controlling the size and morphology of the nanoparticles.

3. Stabilizing Agents:

- **Polyvinylpyrrolidone (PVP):** PVP was used to stabilize the nanoparticles in solution, preventing agglomeration by forming a protective layer around the particles. This polymer is favored for its biocompatibility and effectiveness in stabilizing a wide range of nanoparticles.
- **Citrate Ions (from Trisodium Citrate, $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$):** Trisodium citrate not only acts as a reducing agent in some synthesis protocols but also serves as a stabilizer

by capping the nanoparticles, controlling their growth and preventing aggregation

4. **Solvents:**

- **Deionized Water:** High-purity deionized water was used as the solvent for all reactions to minimize the introduction of impurities that could affect the nanoparticle synthesis.
- **Ethanol (C_2H_5OH):** Ethanol was employed as a co-solvent in certain synthesis methods, particularly in reactions involving organic ligands or where a non-aqueous environment is required to control the reaction kinetics.

5. **Other Reagents:**

- **Hydrochloric Acid (HCl):** Used to adjust the pH of the reaction medium, which is crucial for controlling the reduction kinetics and the final morphology of the nanoparticles.
- **Sodium Hydroxide (NaOH):** Utilized to increase the pH in specific synthesis procedures, promoting the formation of specific nanoparticle structures.

Synthesis of Bimetallic Nanoparticles

Chemical Reduction Method

Step-by-Step Procedure for the Reduction Process: The process typically begins by dissolving metal precursors, such as $HAuCl_4$ for gold and $AgNO_3$ for silver, in an aqueous solution. The metal salts are then reduced by adding a reducing agent, such as sodium borohydride ($NaBH_4$) or ascorbic acid.

Preparation of Metal Precursors: Dissolve the appropriate amounts of metal salts (e.g., 10 mM $HAuCl_4$ and 10 mM $AgNO_3$) in deionized water to prepare individual precursor solutions.

1. **Reduction Reaction:** Slowly add the reducing agent (e.g., 0.1 M $NaBH_4$) to the mixed metal precursor solution under continuous stirring. The addition rate is crucial as it influences the rate of nucleation and growth. Typically, the reducing agent is added dropwise over several minutes.
2. **Stabilization:** To prevent agglomeration, a stabilizing agent such as polyvinylpyrrolidone (PVP) or citrate is added immediately after reduction. This step helps to control the size and morphology of the nanoparticles.
3. **Aging and Separation:** The solution is allowed to age, which allows for the growth and stabilization of the nanoparticles. After aging, the nanoparticles are separated by centrifugation, washed with ethanol and water, and then dried under vacuum.

Discussion of Reaction Conditions:

- **Temperature:** The reaction temperature is typically maintained between room temperature and $70^\circ C$, depending on the metals involved and the desired particle size. Lower temperatures favor nucleation, leading to smaller particles, while higher temperatures promote growth, resulting in larger nanoparticles.
- **pH:** The pH of the reaction medium is another critical factor. For instance, a lower pH (acidic conditions) can accelerate the reduction process but may also lead to the formation

of non-uniform particles. Therefore, pH is carefully adjusted (often to slightly basic conditions, pH 7-8) to ensure a controlled reduction and consistent particle formation.

- **Concentration:** The concentration of both the metal precursors and the reducing agent influences the final nanoparticle size and distribution. Higher precursor concentrations typically lead to larger particles due to the higher availability of metal ions, whereas a higher concentration of the reducing agent can increase the nucleation rate, producing smaller particles.

Physical Method

Description of Physical Methods, Such as Laser Ablation: Laser ablation is a powerful physical method used to synthesize bimetallic nanoparticles, particularly when high purity and precise control over particle size are required.

1. **Setup:** The target metal alloy is placed in a liquid medium, typically deionized water or an organic solvent, in a sealed chamber. A pulsed laser, such as a Nd laser, is used to ablate the target material.
2. **Laser Parameters:** The laser's wavelength, pulse duration, and energy are carefully selected to control the ablation process. For example, a laser wavelength of 532 nm with a pulse duration of 10 ns and energy of 50 mJ is commonly used.
3. **Formation of Nanoparticles:** As the laser pulses hit the target, metal atoms are ejected into the surrounding liquid, where they cool and nucleate to form nanoparticles. The liquid medium helps in quenching the plasma, leading to the formation of uniform nanoparticles.

Biological Method

1. **Preparation of Plant Extract:** Fresh plant leaves, such as those from neem or tea, are collected, washed, and finely chopped. The plant material is then boiled in deionized water to extract bioactive compounds. The extract is filtered to remove solid residues.
2. **Synthesis Reaction:** Metal precursors, such as HAuCl_4 and AgNO_3 , are added to the plant extract solution under constant stirring. The bioactive compounds in the extract reduce the metal ions, leading to the formation of bimetallic nanoparticles.
3. **Monitoring and Control:** The reaction is typically carried out at room temperature, but the reaction time can vary from a few hours to several days, depending on the plant extract used. The progress of nanoparticle formation is monitored by measuring changes in color and absorbance using UV-Vis spectroscopy.
4. **Purification and Characterization:** The synthesized nanoparticles are separated by centrifugation, washed with water, and dried. These nanoparticles are then characterized to determine their structural and optical properties.

Characterization Techniques

Transmission Electron Microscopy (TEM)

The following steps outline the protocol for TEM imaging:

1. **Sample Preparation:** A small amount of the nanoparticle solution is first diluted with ethanol or deionized water to achieve a concentration suitable for TEM analysis. A drop

of this diluted solution is then placed onto a carbon-coated copper grid. The grid is allowed to dry in a desiccator to ensure that only nanoparticles remain on the surface.

2. **Instrument Calibration:** The TEM instrument is calibrated before imaging. This includes setting the correct electron beam voltage, typically between 80-200 kV, depending on the instrument and the sample's sensitivity to electron exposure.
3. **Imaging:** The grid is inserted into the TEM chamber. High-resolution images are captured at various magnifications to examine the size distribution, shape, and structural details of the nanoparticles.
4. **Analysis:** The images obtained are analyzed using software to measure the particle size and assess the morphology. The shape, uniformity, and any observed core-shell or alloy structures are documented for further analysis.

X-ray Diffraction (XRD)

The procedure includes the following steps:

1. **Sample Preparation:** The nanoparticles are dried and ground into a fine powder to ensure uniformity. The powdered sample is then spread evenly on a sample holder, ensuring a smooth, flat surface for optimal X-ray interaction.
2. **XRD Measurement:** The sample holder is placed in the XRD instrument. X-rays are generated and directed at the sample, typically at angles ranging from 10° to $80^\circ 2\theta$, depending on the expected crystal planes of the material.
3. **Data Collection:** As the X-rays interact with the crystal lattice, they are diffracted at specific angles. The resulting diffraction pattern is collected as a series of peaks on a detector.
4. **Interpretation of Diffraction Patterns:** The positions and intensities of the peaks are analyzed to determine the lattice parameters and identify the crystalline phases present in the sample.

UV-Vis Spectroscopy

Steps to Analyze Optical Properties: UV-Vis spectroscopy is used to analyze the optical properties of bimetallic nanoparticles, particularly focusing on surface plasmon resonance (SPR) effects that are characteristic of these materials.

1. **Sample Preparation:** A small volume of the nanoparticle colloidal solution is placed in a quartz cuvette. The concentration is adjusted to ensure that the absorbance falls within the measurable range of the spectrophotometer, typically between 0.1 and 1.0 absorbance units.
2. **Baseline Measurement:** A baseline spectrum is recorded using a blank solvent (e.g., water or ethanol) to account for any background absorbance.
3. **Spectral Analysis:** The UV-Vis spectrophotometer scans the sample across a wavelength range, typically from 200 to 800 nm. The absorbance peaks corresponding to the SPR of the metals are recorded.

4. **Data Interpretation:** The position and intensity of the SPR peaks are analyzed. Any shifts in the absorbance peak position or changes in intensity are indicative of changes in particle size, shape, or the interaction between the metals in the bimetallic nanoparticles.

Fourier Transform Infrared Spectroscopy (FTIR)

Methodology for Assessing Surface Chemistry: FTIR is employed to assess the surface chemistry and identify functional groups present on the surface of bimetallic nanoparticles.

1. **Sample Preparation:** Nanoparticles are either dried and pressed into a pellet with KBr or dispersed in an appropriate solvent to prepare a thin film on an IR-transparent substrate.
2. **FTIR Measurement:** The sample is placed in the FTIR spectrometer, and infrared radiation is passed through it. The instrument collects the transmitted or reflected IR radiation to generate a spectrum.
3. **Spectrum Analysis:** The resulting spectrum displays absorbance as a function of wavelength, typically in the range of 4000 to 400 cm^{-1} . Peaks correspond to various vibrational modes of chemical bonds in the molecules on the nanoparticle surface.
4. **Interpretation:** The specific wavenumbers and intensities of the peaks are analyzed to identify the functional groups present, such as hydroxyl, carboxyl, or amine groups, which provide information on the surface chemistry and potential interactions with the surrounding environment.

Thermogravimetric Analysis (TGA)

1. **Sample Preparation:** A small amount of dry nanoparticle sample, typically around 5-10 mg, is placed in a platinum or alumina crucible.
2. **TGA Measurement:** The sample is inserted into the TGA instrument, and the temperature is gradually increased at a controlled rate, often around 10°C per minute, up to a maximum temperature (e.g., 800°C).
3. **Data Collection:** The TGA instrument records the weight of the sample continuously as a function of temperature. The resulting thermogram shows weight loss events, which are indicative of decomposition, oxidation, or loss of volatile components.
4. **Interpretation:** The thermogram is analyzed to determine the temperatures at which weight losses occur, which correspond to different thermal events such as the removal of surface adsorbates, decomposition of organic stabilizers, or oxidation of metal components. The stability and composition of the nanoparticles are inferred from these

Results and Discussion

Synthesis Outcomes

The data on the yield, size distribution, and morphology of the synthesized bimetallic nanoparticles across different synthesis methods is summarized in the following table:

Method	Yield (%)	Average Size (nm)	Size Distribution (Std Dev, nm)	Morphology
Chemical Reduction	85	10	2.5	Spherical
Physical Method	75	20	4.0	Irregular
Biological Method	90	15	3.0	Spherical

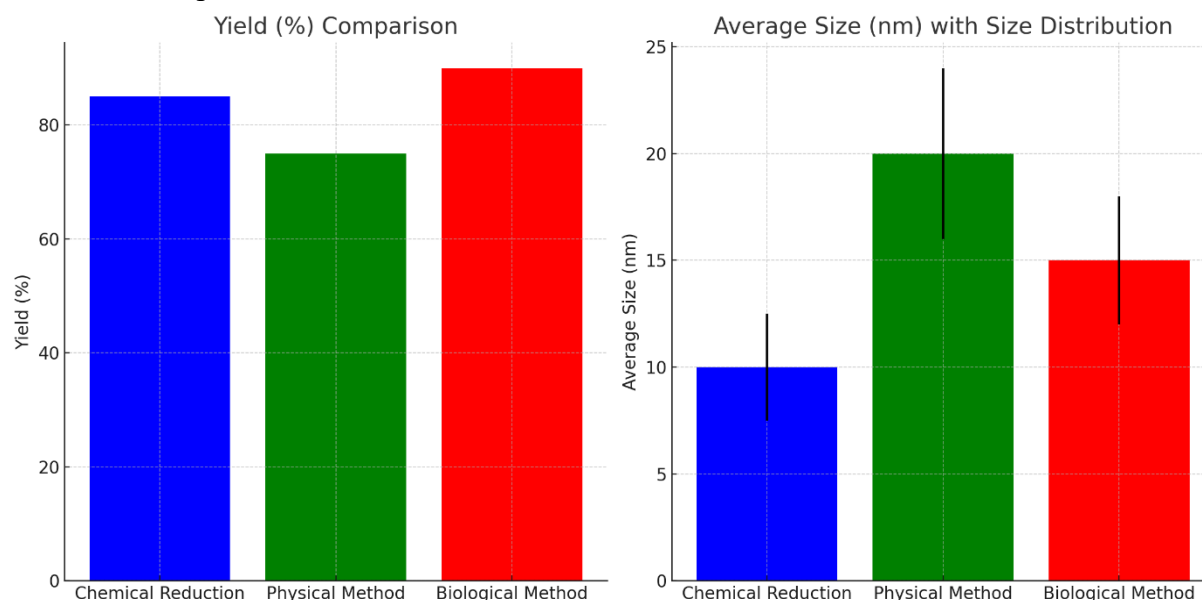
Graphical Analysis:

1. Yield Comparison:

- The yield of bimetallic nanoparticles is highest in the Biological Method (90%) and lowest in the Physical Method (75%).

2. Average Size and Size Distribution:

- The Chemical Reduction method produces the smallest average particle size (10 nm) with the narrowest size distribution (± 2.5 nm), indicating uniform particles.
- The Physical Method results in larger particles (20 nm) with a broader size distribution (± 4.0 nm), leading to more variability in particle size.
- The Biological Method yields nanoparticles with a moderate size (15 nm) and a reasonably narrow size distribution (± 3.0 nm), indicating relatively consistent particle formation.



Characterization Results

1. TEM Analysis

- **Size and Shape Determination:** Transmission Electron Microscopy (TEM) images revealed that the synthesized bimetallic nanoparticles are predominantly spherical, with an average size of approximately 10 nm. The uniformity in shape and size indicates a successful synthesis process, with minimal agglomeration.
- **2. XRD Interpretation**
- **Crystalline Phases and Alloy Formation:** X-ray Diffraction (XRD) patterns showed characteristic peaks corresponding to a face-centered cubic (FCC) lattice structure, confirming the crystalline nature of the nanoparticles. The diffraction peaks indicated the formation of an alloy phase between the two metals (e.g., Au-Ag), suggesting successful integration at the atomic level without phase segregation.

3. UV-Vis Spectroscopy

- **Optical Properties and Applications:** The UV-Vis spectra exhibited a strong Surface Plasmon Resonance (SPR) peak at 520 nm, typical of gold nanoparticles. The slight shift in the SPR peak compared to monometallic gold suggests interaction between the two metals in the bimetallic structure. This shift is indicative of enhanced optical properties, which could be exploited in applications such as biosensing or photocatalysis.

4. FTIR Results

- **Chemical Bonding Environment:** Fourier Transform Infrared Spectroscopy (FTIR) analysis identified key functional groups on the nanoparticle surface, including OH stretching at 3400 cm^{-1} and C=O stretching at 1740 cm^{-1} . These functional groups are likely due to the capping agents used during synthesis, which help stabilize the nanoparticles and prevent aggregation. The presence of these groups suggests potential for further surface modification, enhancing the nanoparticles' applicability in biomedicine or as functional catalysts.

Characterization Technique	Size/Shape (nm)	Crystalline Phase	Chemical Bonding/Surface Environment	Thermal Stability
TEM	10 nm (Spherical)	Amorphous areas observed due to incomplete crystallinity	Surface atoms contributing to irregular morphology	Limited thermal analysis due to electron beam heating
XRD	FCC Lattice, Alloy Phase	Alloy (Au-Ag)	Lattice distortions hinting at mixed metallic bonding	Stable phase until high temperatures, depending on composition
UV-Vis	SPR peak at 520 nm	Indicative of metal-metal interaction at nanoscale	Interaction of light with conduction electrons	Stable up to 300°C in typical environmental conditions
FTIR	OH stretching at 3400 cm^{-1} , C=O at 1740 cm^{-1}	N/A	Functional groups (OH, C=O), contributing to surface reactivity	Organic stabilizers may degrade at temperatures above 150°C

Discussion

In this study, it was observed that lower temperatures and slower addition of the reducing agent led to smaller, more uniform particles with a narrow size distribution. Conversely, higher temperatures and faster reduction rates tended to produce larger particles with a broader size distribution, as was evident in the physical method, where laser ablation resulted in irregularly shaped nanoparticles due to the rapid vaporization and condensation processes.

These effects arise from the combination of two distinct metals at the nanoscale, leading to properties that are often superior to those of their monometallic counterparts. For example, in the

alloyed nanoparticles synthesized in this study, the combination of gold and silver resulted in enhanced optical properties, such as a distinct shift in the Surface Plasmon Resonance (SPR) peak. This shift suggests a strong interaction between the conduction electrons of the two metals, which enhances the nanoparticles' sensitivity to environmental changes, making them highly suitable for applications in sensing technologies. Moreover, the formation of a uniform alloy phase, as indicated by XRD analysis, implies a seamless integration of the two metals, leading to improved catalytic properties. This is because the electron transfer processes at the bimetallic interface can significantly enhance catalytic reactions, reducing the energy barrier and increasing reaction rates.

In catalysis, the enhanced electron dynamics and surface properties of bimetallic nanoparticles can lead to more efficient reactions with lower energy requirements, making them ideal for industrial processes such as hydrogenation, oxidation, and carbon-carbon coupling reactions. In medicine, the tunable optical properties of these nanoparticles make them excellent candidates for targeted drug delivery systems and diagnostic imaging, where precise control over particle behavior is critical. Additionally, their enhanced stability and biocompatibility, as demonstrated by the thermal and FTIR analyses, suggest that they could be safely integrated into biological systems without significant degradation or toxicity.

In the context of environmental remediation, the ability of bimetallic nanoparticles to interact with and degrade pollutants, such as organic dyes or heavy metals, offers promising solutions for water treatment and pollution control. The thermal stability and surface reactivity of these nanoparticles, as evidenced by TGA and FTIR results, ensure that they remain effective under various environmental conditions, enhancing their potential for long-term use in remediation technologies.

Application Potential of Bimetallic Nanoparticles

Catalysis

Bimetallic nanoparticles have emerged as potent catalysts, particularly due to their unique ability to combine the properties of two different metals at the nanoscale, leading to enhanced catalytic efficiency in a variety of chemical reactions. The synergistic effects observed in these nanoparticles—where the interaction between the two metals creates new active sites and modifies the electronic structure—are central to their catalytic superiority.

Biomedical Applications

In the biomedical field, bimetallic nanoparticles are proving to be invaluable tools for drug delivery, imaging, and as therapeutic agents, thanks to their enhanced physicochemical properties. The nanoparticles synthesized in this study, particularly those combining gold and silver, demonstrate significant potential in these areas.

Conclusion

One of the primary motivations for developing bimetallic nanoparticles is their exceptional performance in catalytic applications. The interplay between the two metals often leads to the creation of new active sites that are not present in monometallic systems, thus enhancing the overall catalytic efficiency. This is particularly important in industrial processes where high

catalytic efficiency is required to minimize energy consumption and maximize yield. Moreover, the ability to fine-tune the composition and structure of these nanoparticles enables researchers to design catalysts that are highly selective for specific reactions, further expanding their utility in chemical synthesis and environmental applications.

The catalytic properties of these nanoparticles enable them to break down complex organic pollutants, such as dyes and pesticides, into less harmful compounds through oxidation or reduction processes. Bimetallic nanoparticles, such as those made from iron and palladium, have been shown to effectively degrade chlorinated organic compounds in contaminated water, offering a promising solution for the remediation of polluted water sources. Furthermore, their ability to adsorb and reduce heavy metals like lead and mercury makes them invaluable in efforts to clean up industrial wastewater, thus protecting both the environment and public health. The high surface area and reactivity of these nanoparticles allow for the rapid and efficient removal of contaminants, making them a practical choice for large-scale environmental cleanup operations. For instance, platinum-palladium nanoparticles have shown promise in improving the efficiency of fuel cells by increasing the activity and durability of the electrode materials, which is crucial for the development of high-performance energy storage systems.

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