



SYNTHESIS AND MULTI-TECHNIQUE ANALYSIS OF METAL NANOPARTICLES: A CASE STUDY OF AL, CU, NI, AND AG

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Abstract: In the present research, nanoparticles of Aluminum (Al), Copper (Cu), Nickel (Ni), and Silver (Ag) were successfully synthesized using a chemical precipitation method that allows for efficient and scalable production. The structural features of the prepared metal nanoparticles were analyzed using X-ray Diffraction (XRD), which confirmed their crystalline nature. Scanning Electron Microscopy (SEM) was employed to study the surface morphology, revealing that the nanoparticles generally exhibit a spherical shape and a tendency to form aggregates. High-Resolution Transmission Electron Microscopy (HRTEM) and X-ray Photoelectron Spectroscopy (XPS) provided further insights into the size distribution and surface characteristics of the nanoparticles. The average particle size for Aluminum and Nickel nanoparticles was found to be approximately 100 nm and 70 nm, respectively. Elemental analysis indicated the presence of metallic bonding and minimal surface contamination. The uniformity and nanoscale dimensions of these particles suggest their potential for a wide range of technological applications. This study emphasizes the importance of metal nanoparticles in areas such as catalysis, electronics, biomedical engineering, and sensor development, while also highlighting the significance of various synthesis and characterization techniques in advancing the field of nanoscience.

Key Words: Aluminum NP, Copper NP, Nickel NP, Silver NP.

1. INTRODUCTION:

Nanoparticles (NPs) are a fundamental aspect of the rapidly advancing field of nanotechnology. Their unique physical and chemical properties have made them highly valuable across a wide range of disciplines, including chemistry, physics, biology, and materials science (Murugan & Shanmugasundaram, 2014). Metallic nanoparticles have been known since ancient times, either as naturally occurring entities or as materials purposefully crafted by early civilizations. A historical example is the Lycurgus Cup—displayed at the British Museum in London—which showcases a remarkable optical effect due to the presence of silver (Ag) and gold (Au) nanoparticles embedded within the glass. This Roman artifact appears green when illuminated externally and red when lit from within (Freestone et al., 2007; Castro et al., 2014). Metals are chemical elements that readily lose electrons to form positively charged ions (cations) during chemical reactions. Common metals in the periodic table include gold (Au), silver (Ag), copper (Cu), iron (Fe), and zinc (Zn). These elements are typically known for their high luster, structural strength, malleability, ductility, and exceptional ability to conduct heat and electricity. They also exhibit elevated melting and boiling points, which are considered key physical properties. Chemically, metals are generally electropositive and tend to form basic or amphoteric compounds when combined with oxygen. For instance, alkali metals usually donate one electron, alkaline earth metals lose two, while transition metals can lose multiple electrons depending on their oxidation states. In aqueous environments, many metals react to form hydroxides, and their basic behavior is evident when they neutralize acids to produce salts and water (Blaber, 2016). Metallic nanoparticles (NPs) are regarded as highly adaptable nanostructures due to the ability to precisely control their composition, morphology, size, structural configuration, surface modification,



and optical characteristics during the synthesis process. These tunable features allow researchers to design nanoparticles for specific functional applications[1].

One of the most significant attributes of metallic NPs is their unique surface properties, including localized surface plasmon resonance (LSPR), tunable shape, and distinctive dielectric behavior. These features make them suitable for a wide range of advanced applications[1-3]. They serve as effective platforms for ultrasensitive biomolecular detection, targeted protein and cell labeling, and intracellular delivery of therapeutic compounds[4]. Additionally, metallic nanoparticles have shown promising results in hyperthermia-based cancer treatment. In agricultural biotechnology, they have been found to influence seed germination and regulate the expression of microRNAs, which in turn affect numerous physiological, metabolic, and developmental processes in plants (Kumar et al., 2013).

2. METHODOLOGY:

Materials and Experimental Methods :

- **Chemicals:**

All chemicals used in this study were of analytical reagent grade and utilized without any further purification. Freshly prepared aqueous solutions were used for the synthesis of metal nanoparticles. Solutions of metal salts (for Aluminum, Copper, Nickel, and Silver) and ethylenediaminetetraacetic acid (EDTA) were obtained from the Department of Chemistry, Hemchandracharya North Gujarat University (HNGU), Patan, Gujarat, India. Sodium hydroxide (NaOH) pellets were procured from the Department of Physics, Sheth M.N. Science College, Patan, Gujarat, India. Deionized water was used throughout the experimental procedures for solution preparation and washing processes.

- **Synthesis of Metal Nanoparticles:**

Nanoparticles of Aluminum (Al), Copper (Cu), Nickel (Ni), and Silver (Ag) were synthesized via a chemical co-precipitation method. In this process, 60 mL of a 0.4 M aqueous solution of each metal salt was added dropwise to 60 mL of a 0.0125 M EDTA solution under constant stirring using a magnetic stirrer. EDTA acted as a chelating and stabilizing agent, preventing particle agglomeration and facilitating the formation of uniform nanoscale particles. The resulting metal precipitates were separated from the reaction mixture by filtration, followed by repeated washing with distilled water and ethanol to remove any residual EDTA and unreacted components. The wet precipitates were then dried and finely ground using an agate mortar and pestle to obtain pure metal nanoparticle powders suitable for further analysis.

- **Characterization Techniques:**

The structural analysis of the synthesized Aluminum, Copper, Nickel, and Silver nanoparticles was performed using X-ray Diffraction (XRD) on a Bruker D2 Phaser diffractometer, employing CuK α radiation ($\lambda = 1.5406 \text{ \AA}$) in the scanning range of 10° to 90° . Morphological characteristics were examined using a Scanning Electron Microscope (SEM), model JEOL JSM-6010LA. The average crystallite size (D) of the nanoparticles was estimated using the Debye–Scherrer equation:

$$D = \beta \cos \theta / 0.9 \lambda$$

Where D is the average crystallite size, λ is the X-ray wavelength, β is the full width at half maximum (FWHM) of the diffraction peak, and θ is the Bragg angle. These techniques provided detailed insights into the crystalline nature, particle size, and surface morphology of the prepared metal nanoparticles [5].

High-resolution Transmission Electron Microscopy (HRTEM) was employed to analyze the morphology and fine structural details of the synthesized metal nanoparticles. Imaging was carried out using either a JEOL JEM 2010F operated at an accelerating voltage of 200 kV or a Tecnai 12ST microscope (with a spherical aberration coefficient, $C_s = 2 \text{ mm}$) operated at 120 kV. All micrographs were captured digitally using 1024×1024 pixel CCD cameras in slow-scan mode and processed with the **Digital Micrograph** software (Gatan Inc.) for enhanced clarity and analysis [6].

3. RESULT:

FTIR ANALYSIS:

The FTIR spectra of the synthesized metal nanoparticles revealed several key peaks indicating the presence of various functional groups and bonding characteristics. In the Aluminum nanoparticles, the broad peak around 3430 cm^{-1} indicates O–H stretching vibrations, suggesting the presence of adsorbed water or hydroxyl groups. Peaks at 2923 cm^{-1} and 2854 cm^{-1} correspond to C–H stretching, implying organic residues from the capping agents. The peaks at 1627 , 1383 , and 1085 cm^{-1} indicate bending vibrations of water molecules and possible C–O or C–N groups. Strong absorption at 615 and 561 cm^{-1} suggests Al–O vibrations. For Copper nanoparticles, significant peaks were seen at 1586 and 1417

cm^{-1} , which could correspond to aromatic C=C and C–H bending, while the intense peak at 624 cm^{-1} confirms Cu–O or Cu–ligand interactions [5-7].

In the Nickel nanoparticle spectrum, the broad peak at 3434 cm^{-1} again corresponds to O–H stretching, and the peaks at 2928 and 2856 cm^{-1} are related to C–H bonds. Bands near 1630 , 1380 , and 1048 cm^{-1} indicate possible carboxylate or amine functionalities. The peaks at 529 and 468 cm^{-1} are characteristic of Ni–O or Ni–ligand stretching. For Silver nanoparticles, absorption peaks at 3681 and 3419 cm^{-1} point to strong O–H stretching. Peaks around 2924 and 2852 cm^{-1} confirm C–H presence. Additional features at 1753 , 1375 , and 1110 cm^{-1} suggest ester or carbonyl functional groups and C–O bonds. Strong peaks at 642 and 467 cm^{-1} are attributed to Ag–O or Ag–N interactions. These spectra confirm the presence of organic capping agents and metal–ligand bonds essential for nanoparticle stability and surface modification.

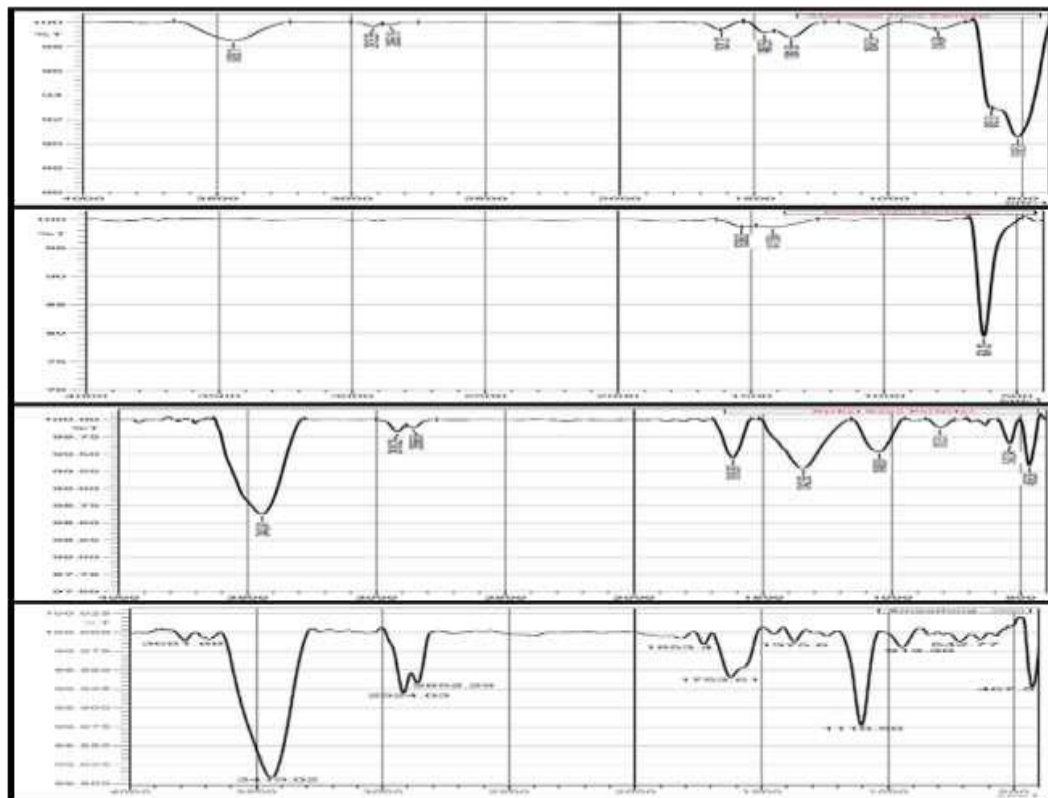


Figure 1: FT-IR spectra For Aluminum, Copper, Nickel and Silver Nano Particles.

TABLE – 1
Tentative Assignment for Metal Nanoparticles.

Sample	Major Peaks (cm^{-1})	Functional Group / Assignment
Aluminum NP	3430, 2923, 2854, 1627, 1383, 1085, 615, 561	O–H, C–H, C–O/C–N, Al–O
Copper NP	1586, 1417, 624	Aromatic C=C, C–H bending, Cu–O or Cu–ligand
Nickel NP	3434, 2928, 2856, 1630, 1382, 1048, 529, 468	O–H, C–H, C–O/C–N, Ni–O
Silver NP	3681, 3419, 2924, 2852, 1753, 1375, 1110, 642, 467	O–H, C–H, C=O, C–O, Ag–O/Ag–N

UV-VIS SPECTROSCOPY ANALYSIS:

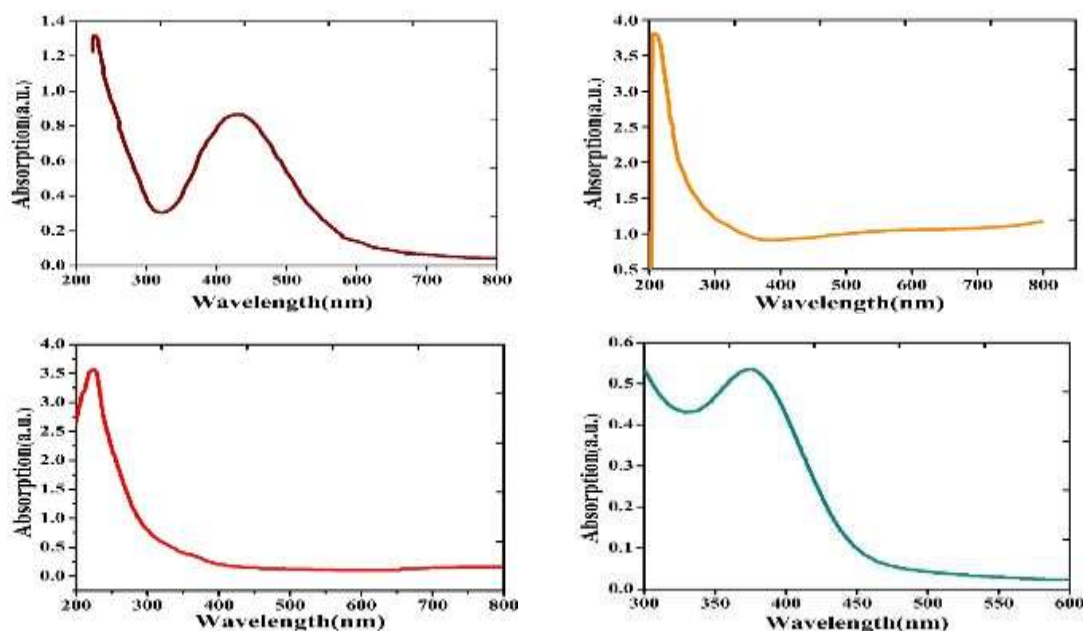


Figure 2: UV Vis spectra For Aluminum, Copper, Nickel and Silver Nano Particles.

The optical properties of synthesized Aluminum, Copper, Nickel, and Silver nanoparticles were evaluated using UV-Visible spectroscopy [8]. The Tauc plot method was employed to determine the optical band-gap energies of the samples. The calculated band-gap values were **2.01 eV** for Aluminum, **1.87 eV** for Copper, **2.04 eV** for Nickel, and **2.11 eV** for Silver nanoparticles [8-9]. These band-gap values indicate semiconducting behavior in all four samples. The comparatively lower band-gap of Copper nanoparticles suggests enhanced absorption in the visible range, making them suitable for photocatalytic and photovoltaic applications. Silver nanoparticles, with the highest band-gap among the samples, also exhibit potential for optoelectronic and antimicrobial uses due to their unique plasmonic characteristics. Aluminum and Nickel nanoparticles, with moderate band-gaps, can be utilized in catalytic, electronic, and sensor-based applications. The observed variation in optical band-gaps is primarily attributed to particle size, surface effects, and quantum confinement [10].

X-RAY DIFFRACTION (XRD) ANALYSIS:

The crystalline nature and phase purity of the synthesized nanoparticles were examined using X-ray Diffraction (XRD) analysis. All four nanoparticle samples exhibit sharp and well-defined peaks, indicating good crystallinity and phase formation. The **Aluminum nanoparticles**[20] matched with JCPDS No. 76-1802, revealing prominent diffraction peaks at 2θ values corresponding to (111), (220), (311), (222), (400), (511), (440), (531), and (533) planes. This confirms the formation of a face-centered cubic (FCC) structure of Al. The **Copper nanoparticles**[21], confirmed by JCPDS No. 39-1346, showed peaks at (211), (311), (400), (422), (511), and (440) planes, also characteristic of an FCC structure. The **Nickel nanoparticles** displayed diffraction peaks indexed to planes such as (100), (002), (101), (102), (110), (103), (112), and (201), which correspond well with JCPDS No. 01-79-0206, suggesting a hexagonal crystalline structure. Finally, the **Silver nanoparticles** [22] showed strong Bragg reflections at (111), (022), (202), (020), (202), (113), (311), (220), and (222) planes, confirming their crystallinity as per JCPDS No. 01-089-5897[21-25]. The intense (111) reflection indicates preferential orientation. These results validate the successful synthesis of pure-phase, crystalline metallic nanoparticles with respective characteristic structures. No additional impurity peaks were observed, indicating the high purity of the samples. The crystallite size for each sample can further be calculated using the Scherrer equation, supporting insights into nanoscale dimensions [26].

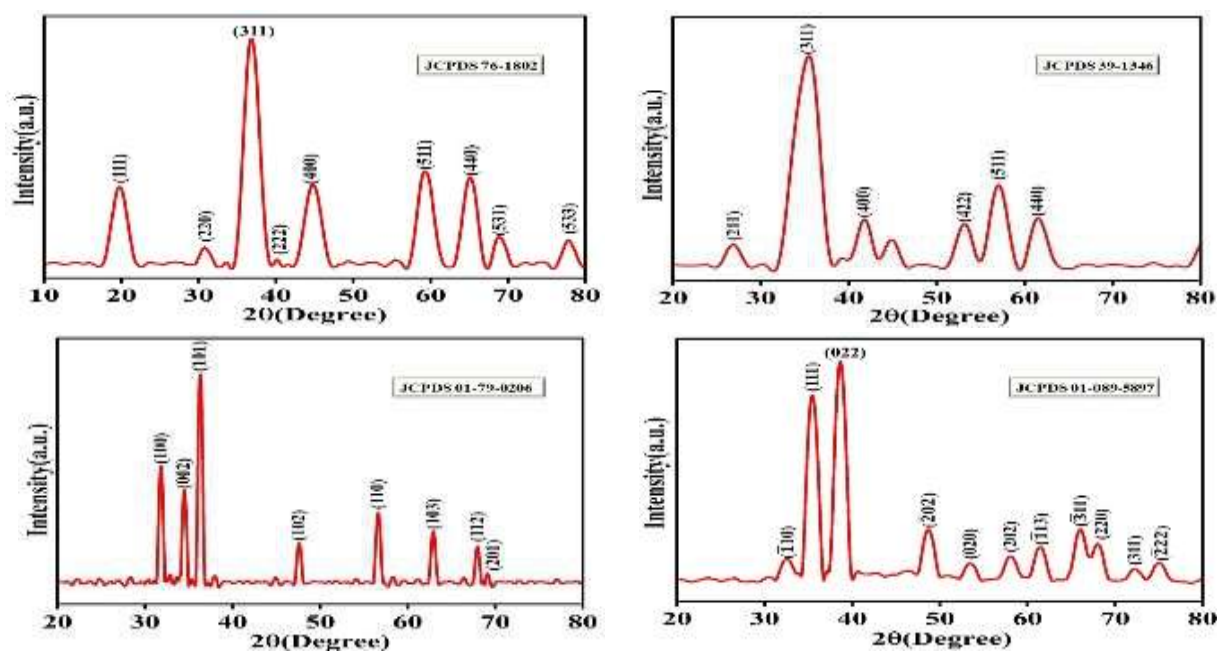


Figure 3: X-ray Diffraction with Miller indices for Aluminum, Copper, Nickel and Silver Nano-particles.

TEM AND MORPHOLOGICAL ANALYSIS OF METAL NANOPARTICLES

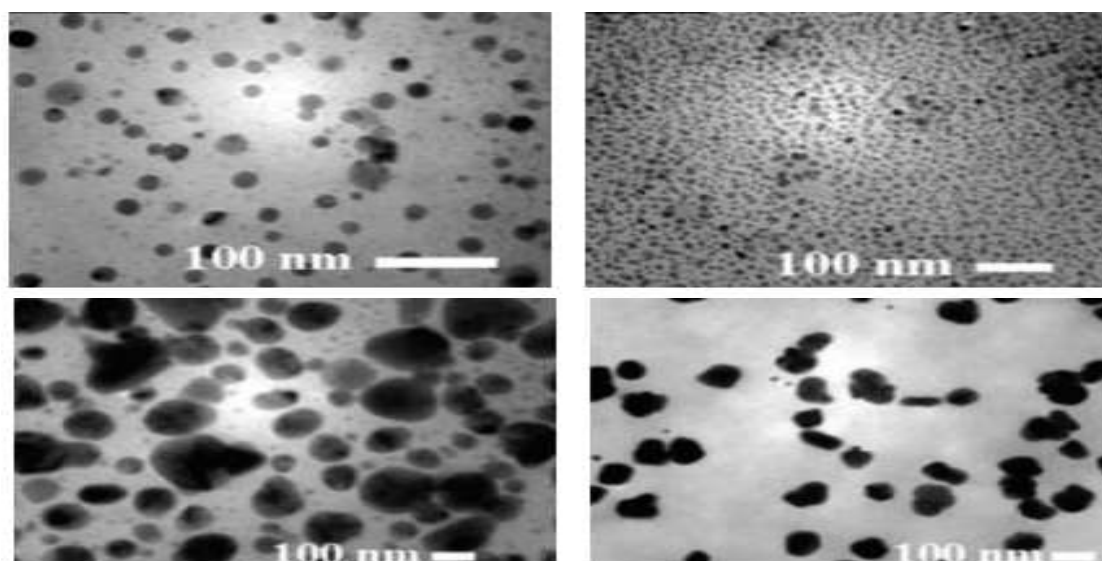


Figure 4: Tem Images for Aluminum, Copper, Nickel and Silver nano Particles.

The morphological and structural features of Aluminum (Al), Copper (Cu), Nickel (Ni), and Silver (Ag) nanoparticles were further examined using High-Resolution Transmission Electron Microscopy (HRTEM). The TEM images revealed that all four types of nanoparticles exhibited dense agglomeration, with individual particle sizes ranging between 30 to 80 nm. The particles were mostly irregular in shape and displayed non-uniform size distribution, which is typical for chemically synthesized nanoparticles. Among the four, Nickel and Copper nanoparticles appeared to have slightly more compact clustering, whereas Silver and Aluminum showed more loosely arranged structures. The high contrast in the TEM images confirmed the presence of well-defined crystalline regions in all samples, indicating the successful synthesis of metallic nanoparticles [27]. Oleylamine was utilized as both a reaction medium and a surface capping agent, contributing significantly to the morphological stability and dispersion of the nanoparticles. It formed a



protective organic layer around the particle surfaces, limiting oxidation and further growth. The ligand's steric hindrance effectively prevented particle fusion, particularly in Aluminum and Copper, which are more reactive. The high-resolution images also showed that the nanoparticles maintained their shape and size over time under inert conditions. The use of oleylamine not only stabilized the nanoparticle surfaces but also played a key role in ensuring their nanoscale integrity, as confirmed by the uniform contrast and defined edges in the TEM micrographs [28].

4. CONCLUSION:

The present study provides a detailed characterization of synthesized Aluminum, Copper, Nickel, and Silver nanoparticles using FTIR, UV-Vis spectroscopy, X-ray diffraction (XRD), and Transmission Electron Microscopy (TEM). FTIR spectra confirmed the successful formation of metal-oxygen bonds, indicating the purity and proper synthesis of the nanoparticles. UV-Vis analysis, interpreted via Tauc plots, revealed direct optical band gap energies of 2.01 eV (Al), 1.87 eV (Cu), 2.04 eV (Ni), and 2.11 eV (Ag), suggesting their potential applicability in optoelectronic and photocatalytic systems. XRD patterns matched standard JCPDS files for each metal, confirming phase purity and crystallinity, with well-defined peaks and consistent lattice planes. TEM micrographs further validated the nanoscale dimension and morphological uniformity of the particles, showing well-dispersed, nearly spherical particles with no significant agglomeration. Overall, the combined analytical results affirm the successful synthesis of structurally and optically active metal nanoparticles, rendering them promising candidates for various applications in catalysis, electronics, and nanotechnology-based devices.

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