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LASIS-Assisted Copper Nanoparticle Synthesis and Characterization, along with UV-Visible Spectroscopy

Mr. Chaitanya Patel
Ph.D. Scholar, Department of Physics, Mehsana Urban Institute of Science, Ganpat University, Kherva, Mehsana, Gujarat, India

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ABSTRACT

This study investigates the creation of copper nanoparticles (CuNPs) using chemical reduction and laser ablation in liquid (LASIS). UV-visible spectroscopy is used to examine the optical characteristics of the nanoparticles created by these techniques. The purpose of the study is to compare the stability, efficacy, and particle size of CuNPs produced using different techniques. When comparing the LASIS method to the chemical reduction process, Transmission Electron Microscopy (TEM) examination revealed that the former produced smaller and more uniform nanoparticles. This work demonstrates the effectiveness of both synthesis techniques, with LASIS clearly outperforming the other in the production of superior CuNPs with more control over particle size and dispersion. A thorough explanation of the chemical reduction method and LASIS used in the synthesis of copper nanoparticles is provided, and UV-visible spectroscopy is used to characterize the resulting particles.

Keywords : Nanoparticles, Copper Nanoparticles, LASIS, Chemical Reduction Method

I. INTRODUCTION

A particle that is smaller than 100 nm is called a nanoparticle. A bulk substance, in contrast to nanoparticles, has consistent physical characteristics independent of its size [1, 2]. Nanotechnology is currently a hot topic for research in both physics and chemistry due to its many applications in a wide range of domains, including electronic, electrical, optical, and biomedical fields [2, 3]. Because they efficiently transfer information between bulk materials and atomic or molecular structures, nanoparticles are extremely significant. There are characteristics that rely on the size of the particles, such as superparamagnetic, surface Plasmon resonance in some metal particles, and quantum confinement [3–4]. There is a noticeable shift in the material's characteristics as the size gets closer to the nanoscale. The fraction of atoms at the surface in bulk materials with particle sizes larger than one micrometre is extremely small in comparison to the total number of atoms in the material. The intriguing and surprising characteristics of nanoparticles are not solely attributable to the material's surface features predominating over its bulk characteristics [3–5]. The nanoparticles have several unique characteristics when compared to the bulk substance.

CuNPs, or copper nanoparticles, have attracted a lot of attention because of their special qualities and possible uses in biomedicine, electronics, and catalysis. This work focuses on creating CuNPs via chemical reduction and LASIS methods, then use UV-visible spectroscopy to characterize the resulting nanoparticles. CuNPs, or copper nanoparticles, have garnered a lot of interest because of their remarkable chemical and physical characteristics that set them apart from their bulk counterparts. CuNPs have a wide range of industrial and biomedical applications due to their distinctive optical features, improved catalytic activity, electrical conductivity, and antibacterial efficacy [4–5]. CuNPs have a wide range of uses, thus reliable and effective synthesis techniques are essential. Chemical reduction and laser ablation in liquid solution (LASIS) are the two most used ways to create CuNPs. LASIS is a multipurpose method that creates nanoparticles by ablation of a metal target dissolved in a liquid media with a high-energy laser. By adjusting the laser's parameters, this approach may produce pure nanoparticles without the need for additional chemicals and offers fine control over particle size and distribution [5–6]. On the other hand, because of its scalability and ease of use, the chemical reduction process is commonly employed. By using a reducing agent like sodium borohydride ($NaBH_4$), a copper salt, usually copper sulphate ($CuSO_4$), is reduced using this process. To ensure the stability of the nanoparticles, stabilizing chemicals such as polyvinylpyrrolidone (PVP) are used. This helps to prevent agglomeration. It is crucial to characterize synthesized nanoparticles in order to verify their creation and evaluate their characteristics. UV-visible spectroscopy is an effective instrument for this because it analyses the surface plasmon resonance (SPR) peaks of nanoparticles to reveal details about their optical characteristics and size distribution [7].

The objective of this work is to synthesis CuNPs by chemical reduction as well as LASIS, and then use UV-visible spectroscopy to analyse the resulting nanoparticles. By comparing the efficiency of several techniques in terms of stability, size, and dispersion of nanoparticles, the study offers insights into the best synthesis strategy for different applications. This work advances the creation of more effective and manageable synthesis procedures for CuNPs by elucidating the benefits and drawbacks of each approach. At the nanoscale, this is frequently not the case, despite the fact that a bulk material should have consistent physical properties regardless of its size [6–9]. Quantum confinement in semiconductor particles, surface Plasmon resonance in certain metal particles, and superparamagnetic in magnetic materials are examples of size-dependent phenomena that are observed [7-8]. Materials' characteristics alter as they get closer to the nanoscale and as the proportion of atoms on their surface increases [8-9]. The fraction of atoms near the surface in bulk materials greater than one micrometre is extremely small in comparison to the total number of atoms in the material. The intriguing and occasionally surprising characteristics of nanoparticles are not only attributable to the material's surface features predominating over its bulk characteristics [10–11]. In comparison to bulk material, nanoparticles have a variety of unique features. For instance, movement of copper atoms or clusters at roughly the 50 nm scale causes the bending of bulk copper (wire, ribbon, etc.) [10-11]. Less than 50 nm-sized copper nanoparticles are regarded as extremely hard materials that lack the malleability and ductility of bulk copper [12]. It is not always preferable for the qualities to change. Ferroelectric materials with a size lower than 10 nm are incapable of storing memory because they can change the direction of their magnetization with thermal energy at ambient temperature.

II. LASER ABLATION IN LIQUID SOLUTION (LASIS)

The technique of eliminating material from a solid surface by applying a laser beam to it is called laser ablation. When the laser flux is low, the absorbed laser energy heats the material, causing it to either evaporate or sublimate. Usually, the material turns into plasma at high laser flux. Generally speaking, laser ablation refers to the removal of material using a pulsed laser, although if the laser intensity is high enough, material can also be ablateable using a continuous wave laser beam. The average spot dimension at the focal site was 250 ±10 μm. The incident laser fluence was influenced by changes in both the incident laser energy and the laser beam pattern diameter. All of the experiments were conducted at room temperature and atmospheric pressure. A freshly prepared solution was used to conduct UV-visible spectroscopy at different irradiations.

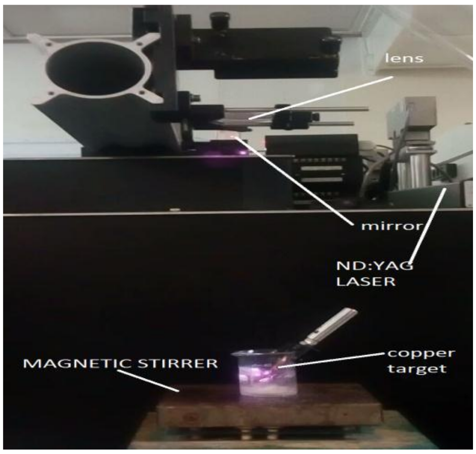

Fig.1- Experimental Setup of LASIS

The optical characteristics of the material, the laser wavelength, and the pulse duration all affect how much material is removed by a single laser pulse and the depth across which the laser energy is absorbed. Generally speaking, ablation rate refers to the overall mass removed from the target for each laser pulse. The ablation process can be greatly influenced by characteristics of laser radiation, such as the coverage of scanning lines and the scanning velocity of the laser beam. Laser pulses are highly controllable and can range in duration from milliseconds to femtoseconds, as well as in flux. Because of this, laser ablation has significant value in both industrial and research applications. The operating procedure for LASIS are described as below:-

- **Method for Preparing Liquid Medium:** A PVP solution of 0.1% (w/v) is made using deionized water. PVP functions as a stabilizing agent, keeping CuNPs from clumping together.
- **Configuration for Laser Ablation:** The PVP solution is contained in a quartz beaker with the copper target positioned at the bottom. Focused on the copper target, the Nd laser has a wavelength of 532 nm. Laser parameters, including repetition rate, ablation time, and pulse energy, are meticulously regulated. Typical parameters for this investigation include an ablation time of 30 minutes, a repetition rate of 10 Hz, and a pulse energy of 100 mJ.
- **Procedure of Ablation:** The copper target is exposed to the laser beam, which causes the copper atoms to be ablation and condense into nanoparticles in the liquid medium. In order to guarantee a consistent dispersion of nanoparticles throughout the solution, the operation is conducted while stirring continuously.
- **Following Ablation Therapy:** The solution is allowed to settle after ablation in order to get rid of any big particles.
  The resulting CuNP colloid is next centrifuged for 10 minutes at 5000 rpm in order to get rid of any last big aggregates.
  For additional examination, the supernatant containing the CuNPs is gathered.

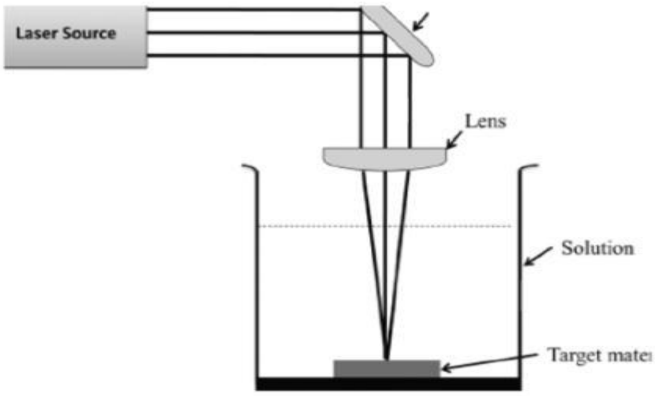

Fig.2- Laser Ablation Operational Mechanism

UV-VISIBLE SPECTROSCOPY

The technique of UV/Visible spectroscopy is employed to measure the amount of light that a sample absorbs and scatters. This amount, referred to as the extinction, is determined by adding the total amount of light that is absorbed and scattered. In its most basic form, the intensity of a UV/visible light beam is measured both before and after it passes through a sample, which is positioned between a light source and a photodetector. An analytical method that is frequently used to evaluate the optical characteristics of nanoparticles is UV-visible spectroscopy. Its analysis of the interaction between nanoparticles and light yields important information about the size, shape, and distribution of the particles. The method focuses on the surface plasmon resonance (SPR) phenomena for copper nanoparticles (CuNPs), which is a distinctive absorption peak suggestive of nanoparticle production. The wavelength dependent extinction spectrum of the sample is quantified by comparing these observations at each wavelength. Usually, the results are shown as a function of wavelength representing extinction (see inset). In order to ensure that the sample extinction spectrum does not contain any spectral features from the buffer, each spectrum is background corrected using a buffer blank.

UV/Vis spectroscopy is a useful technique for identifying, characterizing, and researching nanomaterials because gold and silver plasmonic nanoparticles have optical properties that are sensitive to size, shape, concentration, agglomeration state, and refractive index near the nanoparticle surface. Numerical models can be used to compare the measured and expected spectra. Although the size and concentration of non-plasmonic nanoparticles similarly affect their optical properties, their spectrum is less susceptible to the dispersion characteristics than that of plasmonic nanoparticles.

III. METHODOLOGY

1. **Sample Preparation**
- **LASIS Synthesized CuNPs:** The CuNPs synthesized using LASIS are centrifuged to remove large aggregates and diluted with deionized water to obtain a clear colloidal solution.
- **Chemically Reduced CuNPs:** The CuNPs obtained from the chemical reduction process are similarly purified by centrifugation and then diluted with deionized water to prepare the sample for spectroscopic analysis.

2. **Spectroscopic Analysis**
- **Instrument Setup:** A UV-Visible spectrophotometer is used to record the absorption spectra of the CuNP solutions. The instrument is calibrated using deionized water as the blank reference.
- **Measurement:** The absorption spectra of the CuNP solutions are measured over a wavelength range of 200-800 nm. The samples are placed in quartz cuvettes with a path length of 1 cm.

Chemical Reduction Technique

2gm PolyVinyl-Pyrrolidone (PVP) was slowly added in 100 ml of distilled water and was heated at 60°C with constant stirring for 1 hour until it was dissolved properly. Then 1g copper nitrate was added into the solution and was stirred for another 10 minutes till color change was observed from colorless to sky blue. Then 5% hydrazine hydrate was added drop wise to the solution with vigorous stirring until the color turns to green. The color change from blue to green signifies nanoparticle formation. UV-Visible spectroscopy was carried out with freshly prepared solution. Fig 2 shows the color change of the solution.

Fig 3 (a) Sky blue color obtained by adding copper nitrate and PVP (b) green color obtained after adding hydrazine hydrate

IV. EXPERIMENTAL SET-UP AND RESULT

Using an ND: YAG (Continnum Laser, USA) nanosecond laser system, laser ablation was performed in pure water. The sample was put in a glass cell with distilled water inside of it. Using a 240mm focal length lens, the laser beam was focussed at normal incidence on the copper target's surface. At the focal point, the average spot size was 250 ±10 μm. Both changes in incident laser energy and changes in the diameter of the laser beam pattern caused variations in the incident laser fluence. Every experiment was run under atmospheric pressure and at ambient temperature. The chemical reduction process is employed to produce more copper nanoparticles.

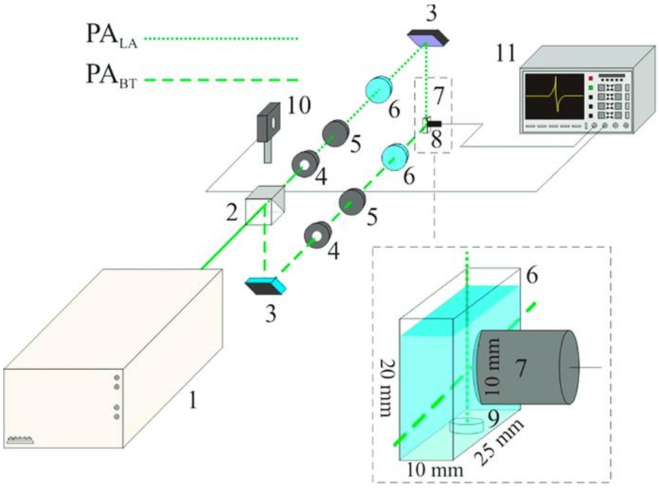

Fig.4 - Experimental arrangement for LASIS

The light source, an Aves AVALIGHT HAL-S, was utilized. It was collimated with a collimating lens and allowed to pass through the sample cell. Next, it was focused using a converging lens on a 600-micron optical fibre (Ocean optics). The fibre was connected to a spectrometer (an HR4000 from Ocean optics), and data was recorded on the PC. Origin software was then used to further process the data in order to analyse the outcome.

V. RESULT DISCUSSION

UV-visible spectroscopy was used to detect the absorption of Cu-NPs in order to investigate the stability of Cu colloidal solution in air. There have been reports of copper nanoparticles having an absorption band between 500 and 600 nm. Figure illustrates the UV-visible absorption spectra of CuNPs obtained using the chemical reduction method (CRM) and the solution obtained using LASiS. This spectrum is captured right after the particles are synthesized. The absorption peaks in the picture, which are located at 588 and 612 nm, respectively, demonstrate that copper nanoparticles have formed in the solution. A broad band indicates a broad distribution of copper particle sizes inside the matrix.

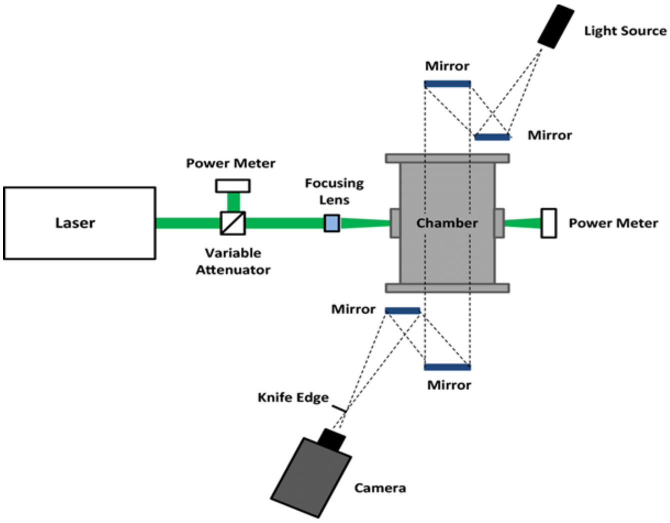

Fig.5 - Laser Meter arrangement for LASIS Operation

Fig.6 Freshly prepared blood red Copper solution (1), Black (2), Violet (3), Onset Oxidation (4)

1. UV-Visible Spectroscopy Analysis

- **SPR Peak Observation:** The absorption spectrum of the LASIS-synthesized CuNPs shows a distinct SPR peak around 570 nm.
- The sharpness and intensity of the SPR peak indicate a narrow size distribution and high purity of the nanoparticles.

2. Transmission Electron Microscopy (TEM) Analysis

- **Size and Shape:** TEM images reveal that the LASIS-synthesized CuNPs are predominantly spherical. The nanoparticles have a uniform size distribution, with an average diameter of 10-20 nm. The high degree of uniformity is a result of the controlled ablation process and the stabilizing effect of PVP.

3. Comparison with Chemical Reduction

- **SPR Peak Characteristics:** Chemically reduced CuNPs exhibit a broader SPR peak, indicating a wider size distribution and possible aggregation. The LASIS method produces a more defined SPR peak, reflecting better control over nanoparticle size and stability.
- **Morphological Differences:** TEM images of chemically reduced CuNPs show a range of particle sizes and shapes, with some degree of aggregation. LASIS-synthesized CuNPs are more uniform and less prone to aggregation due to the physical nature of the synthesis process.

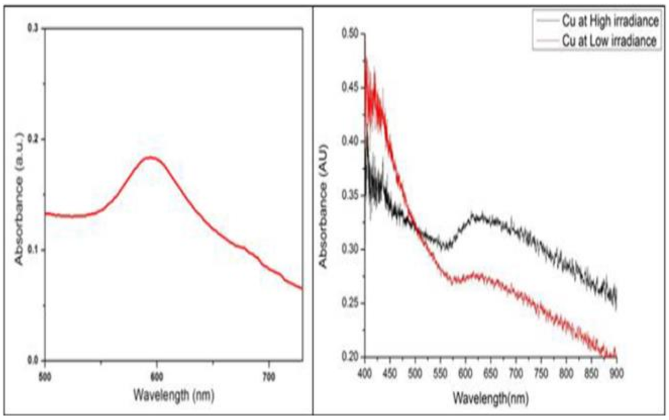

Fig.7 (a) Shows the colloidal copper solution prepared using Chemical Reduction technique (b) Shows the colloidal copper solution prepared using LASIS technique

Using Shimadzu UV-1700 UV-visible spectroscopy, the absorption of copper nanoparticles was evaluated in order to investigate the durability of Cu colloidal solution in air. There have been reports of copper nanoparticle absorption bands in the 500–600 nm region. Copper nanoparticles' UV-visible absorption spectra obtained through chemical reduction. Shortly after the particles are synthesized, the spectrums are captured. The absorbance maxima at 577 nm and 612 nm, respectively, in Figure 7(a and b) demonstrate the production of copper nanoparticles in the solution. A broad band indicates a broad distribution of copper particle sizes inside the matrix. The Mie theory is further supported by the band's width and intensity, which indicate the presence of spherical copper nanoparticles. According to the Mie approximation model, the average diameter of Cu nanoparticles in the case of chemical reduction is approximately 80-90 nm, while in the case of LASIS, the average diameters are approximately 60-70 nm, with a margin of error of ±10–15 nm in both scenarios.

VI. CONCLUSION

The current work offers details on straightforward, reasonably priced, and useful processes for reducing copper salts in an aqueous medium to create copper nanoparticles. A broad spectrum is discovered, indicating a wide size of the particles, and an attempt is made to investigate the absorption phenomena in copper nanoparticles, which is caused by surface plasmon resonance, which is found to be strong after 550 nm. A theoretical particle size was calculated using the Mie theory. Future studies will focus on gathering more samples, as there are now just two that were obtained, making it impossible to draw firm conclusions due to a lack of information. Additionally, we will attempt to create samples using various laser intensities, and we will investigate the theoretical and experimental relationships between comprehensive UV-visible spectroscopy, TEM, and XRD. Copper nanoparticles with appropriate characteristics are effectively produced via the experimental setup for LASIS. CuNPs with a narrow size distribution that are uniformly synthesized and stable are confirmed by TEM studies and UV-visible spectroscopy. When compared to chemical reduction, the LASIS approach exhibits better control over the features of the nanoparticles, indicating its potential for high-quality nanoparticle synthesis in a range of applications.

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