

RESEARCH PAPER

The Structural Properties of Silver Nanoparticles by The Pulsed Laser Ablation in Liquids Method

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ABSTRACT

This study used the pulsed laser ablation in liquids technique to prepare silver nanoparticles from silver. The initial change in the solution's colour was an indicator of the nanoparticles' production. Two different wavelengths of Nd: YAG laser (1064 nm and 532 nm) were used to sculpt silver nanoparticles in 30 ml of distilled water. The process was carried out using energies ranging from 100 mj to 1000 mj and with pulse counts ranging from 100 to 2000. The colour of the distilled water changed from yellow to dark yellow with an increase in the number of pulses, indicating the production of silver nanoparticles. The effect of laser parameters such as wavelength, energy, and pulses on the size and concentration of nanoparticles prepared by the pulsed laser ablation technique in distilled water was studied. The study showed that the wavelengths (532 nm and 1064 nm) at an energy of 760 mj and 2000 pulse significantly affected the size and concentration of the produced silver nanoparticles. The results indicated that the average size of the prepared silver nanoparticles increases with the increase in laser energy at a wavelength of 532 nm, while the average size decreases with the increase in laser energy at a wavelength of 1064 nm. Furthermore, zeta potential measurements showed the surface charge stability of prepared AgNPs after two months.

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INTRODUCTION

Laser ablation in liquids has revealed significant opportunities and safety for the fabrication of nanostructures, leading to a rapid increase in research on the synthesis of nanostructures using this proper method. By comparing classical physical processes such as vacuum laser evaporation and chemical vapour deposition, the liquid-phase laser ablation process offers advantages, including producing crystalline or semi-crystalline nanoparticles that can be easily obtained without needing subsequent thermal treatment. This is due to the high efficiency of the removed material

with a small size and a large surface area, resulting in pure colloidal solutions of nanoparticles that accumulate in the colloidal solution [1]. The small size, large surface area, tailorability, highly improved solubility, and multifunctionality of NPs open many new research avenues for technology. Pulsed laser ablation of solids in liquids (PLAL) generates nanoparticles of diverse sizes and morphologies, with their dimensions and forms influenced by multiple parameters, including laser strength, pulse duration, frequency, temperature, ablation time, and wavelength [2].

The most eco-friendly, dependable, and

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practically applicable method for creating stable nanoparticles is pulsed laser ablation in liquid (PLAL). This is due to its advantages in terms of easy use without requiring any additional chemical materials, such as stabilizers or surfactants. The characteristics of the nanoparticles can be improved by manipulating the laser parameters (wavelength, pulse duration, repetition rate, energy, and concentration) and the surrounding environment [3].

Nanostructures that are stable, evenly distributed, and exceptionally pure can be more easily synthesized using PLAL (Pulsed Laser Ablation in Liquid) [4]. A vast array of new opportunities for the fabrication of novel nanomaterials has opened up, creating oxide-based nanoparticles [5], metals [6]. By using PLAL, the size of nanoparticles distributed in liquid isopropanol has been significantly reduced, going from 100 nm to 10 nm [7]. Where NPs were synthesized in different ways, chemically and physically [8-12].

Silver nanoparticles (Ag NPs) have gained widespread acceptance in medicine and biology as the gold standard of nanomaterials [13]. Its antibacterial and antifungal properties have made it useful in many contexts, including the cosmetics industry, the medical industry (in the form of wound dressings, ointments, and creams), and water purification [14, 15].

On the other hand, many studies and scientific research have investigated the effect of laser

parameters (laser energy, laser wavelength, and number of laser pulses) on the size of silver nanoparticles, Alyaa Hussein and others prepared silver nanoparticles using the pulsed laser ablation in liquids (PLAL) technique in an aqueous medium, utilising a Nd: YAG laser with an energy of 500 mj and 100 pulses. The prepared particles were characterize by their structural, physical, and optical properties using multiple techniques such as X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), UV-Vis spectroscopy, FTIR measurements, EDX, fluorescence spectroscopy. Their results indicated that the silver nanoparticles are spherical, with dimensions ranging from 34.80 to 46.55 nanometers, and exhibit an absorption spectrum at 534 nanometers. XRD analyses further revealed that the prepared film is polycrystalline and of the cubic type [16, 17]. However, those studies did not examine the stability of AgNPs over time.

Anastasiya E. Tyurnina *at el.* in (2013) prepared silver nanoparticles by the pulsed laser ablation method in water from ion beams using a Q-switched Nd: YAG laser at a wavelength of 1064 nm and a pulse duration of 100 ns. The study showed that the size of the generated silver nanoparticles decreases with the reduction of laser energy and the increase of ablation time [18]. Another study by Mohammed A. Al-Azawi *at el.* in (2015) prepared silver nanoparticles using pulsed laser ablation in deionized water at wavelengths of

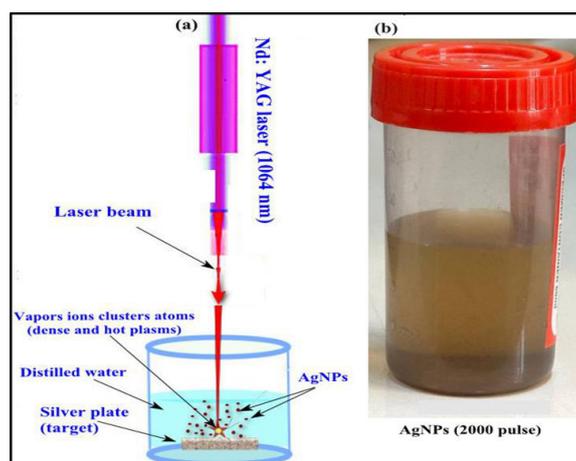


Fig. 1. Schematic diagram of the experimental PLAL setup: (a) the mechanism of ablated silver particles from the bar using Nd: YAC laser 1064 nm [15]. (b) Brownish nanocolloidal solution obtained from silver metal under 2000 P using the PLAL method.

1064 nm and 532 nm. They found that the average size of the silver nanoparticles prepared at a wavelength of 532 nm was smaller than the size of the silver nanoparticles prepared at a wavelength of 1064 nm [19]. Also, other investigations by W. Norsyuhada *W. at el.* (2018) prepared silver nanoparticles were prepared using pulsed laser ablation with a Q-switched Nd: YAG laser in deionized water at a wavelength of 1064 nm, with a pulse duration of 8 nanoseconds. They studied the effect of laser parameters (output laser energy, ablation time) on the size of silver nanoparticles, where they observed that with increasing laser energy and ablation time, the size of the silver nanoparticles increases [20]. Furthermore, using pulsed Nd: YAG and CW diode laser ablation, silver nanoparticles (AgNPs) were produced in de-ionized water and by analyzing the crystallinity, XRD validated the structural qualities. The spherical nanoparticles were found to be 20 nm in CW and 9 nm in pulsed size, according to AFM examination. FTIR verified the little chemical contamination and environmentally friendly nature. There was a 16% increase in thermal conductivity (CW) and a 27% increase (pulsed) in nanofluids [21].

This work aims to study the effect of laser conditions on the structural features of synthesized silver nanoparticles (AgNPs) by pulsed laser ablation (PLA) in distilled water. This technique will reduce the size of the generated silver nanoparticles by fragmenting the newly produced silver nanoparticles. Also, the stability of the prepared AgNPs will be examined through zeta potential measurements.

MATERIALS AND METHODS

AgNPs prepared using the PLAL technique with a pure silver plate of dimensions (1, 2, 2 mm) placed in (30 ml) from the laser source,

where Q-switched Nd: YAG laser, a repetition rate of (10 Hz), wavelengths (532, 1064 nm), and energies (100, 260, 500, 760, 1000 mj) were applied respectively, as illustrated in the schematic diagram in Fig. 1 [15], under number of pulses (100, 500, 1000, and 2000 pulses) for each wavelength. This experiment investigated optical properties previously as the first part of the experiment goal, which is now under the publication process. In this part, structural analyses of the prepared samples were analyzed and studied using FESEM and zeta potential measurements.

The main the main properties of the pulsed laser device used in this experiment is indicated in Table 1.

RESULTS AND DISCUSSION

The colloidal solutions of nanoparticles were prepared, and the effect of laser parameters (laser energy, laser wavelength, and number of laser pulses) on the size and concentration of silver nanoparticles prepared by the pulsed laser ablation method in distilled water was studied. The size of the nanoparticles was measured using a field emission scanning electron microscope FESEM. After a certain period of preparation, the surface charge of the AgNPs was measured using a zeta potential analyser. Among the initial indicators for obtaining nanoparticles is changing the solution's colour gradually as the number of laser pulses increases. Fig. 2 shows the change of the solution colour of the silver particles at one energy and different pulses, where it was noted that the colour of the solution slowly becomes dark towards the yellow colour, indicating the silver nanoparticles.

Structural properties

size of silver nanoparticles

The optical and structural properties of the

Table 1. Characteristics of the pulsed laser device used in this experiment.

Property	Value
Laser Type	Nd: YAG
Wavelength	(532, 1064, 1300) nm
Pulse Duration	10 ns
Laser Power	2000 mj
Frequency Rate	1 – 10 Hz

nanoparticles, such as particle diameter and shape, were analyzed using a field emission scanning electron microscope (FESEM). The obtained images confirmed the formation of nanoparticles measuring less than 100 nanometers, proving that the particle sizes fall inside the designated nanoscale range. The FESEM results revealed that the silver particles are spherical, with diameters ranging from 15.29 to 30.57 nanometers at a wavelength of 1064 nanometers, while at a wavelength of 532 nanometers, the diameters of the silver nanoparticles range between 84.42nm and 14.50nm. However, in a comparable study, the silver particles were spherical, with an average of (46.55 - 34.80) diameter at the wavelength of 1064 nm [16]. Fig. 3 displays the FESEM image of the AgNPs structure synthesized on the glass surface at a wavelength of 1064 nanometers, with a laser energy of 760 mj and a pulse count of 2000.

Furthermore, Fig. 4 demonstrates the size of AgNPs using FESEM at a wavelength of 532 nm under the same conditions from an energy of 760 mj to 2000 pulse. Where the size of the produced silver nanoparticles with 532 nm is smaller than the size under 1064 nm of laser wavelength.

Surface charge

Another remarkable parameter utilized for determining the stability of colloidal silver nanoparticles is the surface charge. Some studies have shown that the colloidal solution is unstable if the surface charge value ranges from (0 – ±10) mV. However, if the surface charge value ranges from (±10 mV) to (±30 mV), the colloidal solution will stabilize moderately. On the other hand, if the surface charge value is (> ±30 mV), the colloidal solution has very high stability [22]. In the current work, surface charge measurements of prepared



Fig. 2. Silver colour solution gradient at different pulse numbers of laser solution obtained from silver metal using the PLAL method.

AgNPs, which were performed using laser pulse ablation in DW methods with 2000 pulses, 760 mj of laser energy, and wavelengths of 532 and 1064 nm, are -35.5 mV and -31 mV, respectively, after two months of preparation as presented in Fig. 5. These findings have demonstrated that the sample

maintained a high level of stability while stored at ambient temperature. This is a significant indicator of AgNPs stability over these experimental conditions. Comparatively, other research findings that assessed the samples' stability just after preparation likewise revealed good stability. This

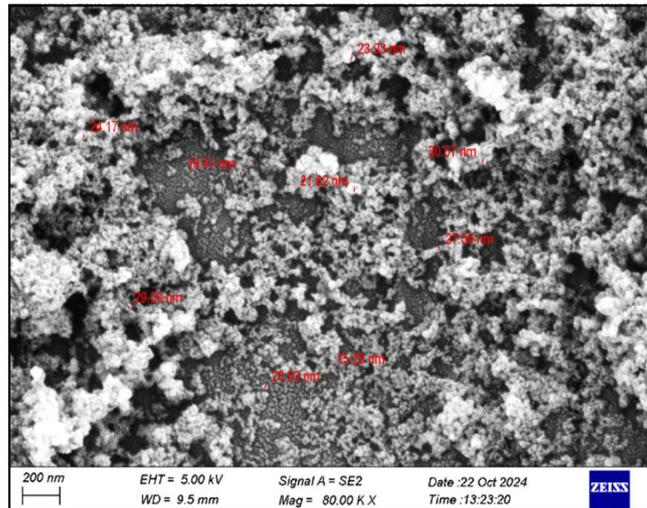


Fig. 3. Scanning electron microscope images with field emission of silver nanoparticles at wavelength of 1064 nm, using an energy of 760 mj per pulse, with a total of 2000 pulse.

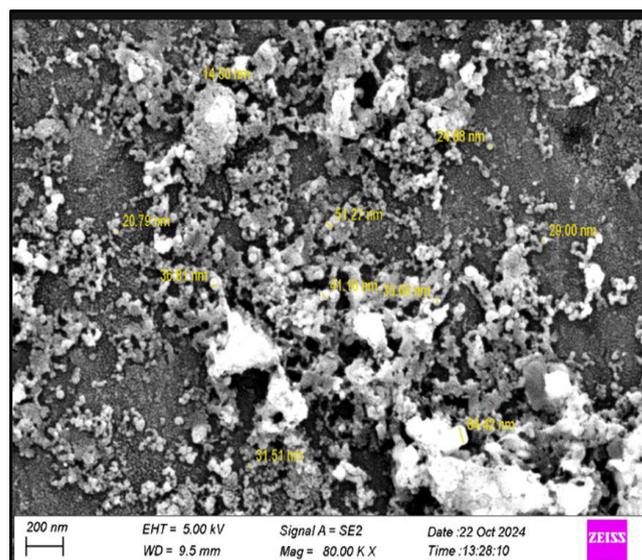


Fig. 4. Scanning electron microscope images with field emission of silver nanoparticles at wavelength of 532 nm, using an energy of 760 mj, and 2000 pulse.

Measurement Results

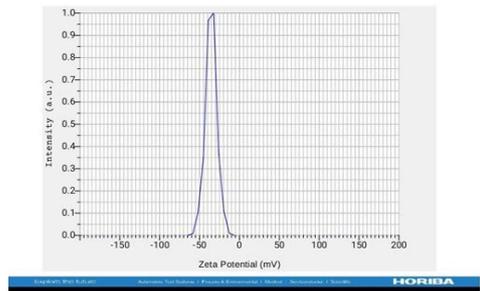
Measurement Results
Date : 21 October 2024 10:46:48
Measurement Type : Zeta Potential
Sample Name : Sample 196
Temperature of the Holder : 25.2 °C
Dispersion Medium Viscosity : 0.891 mPa·s
Conductivity : 0.089 mS/cm
Electrode Voltage : 3.9 V

Calculation Results

Peak No.	Zeta Potential	Electrophoretic Mobility
1	-35.5 mV	-0.000276 cm ² /Vs
2	-- mV	-- cm ² /Vs
3	-- mV	-- cm ² /Vs

Zeta Potential (Mean) : -35.5 mV
Electrophoretic Mobility Mean : -0.000276 cm²/Vs

532 nm



Measurement Results

Measurement Results
Date : 21 October 2024 11:00:11
Measurement Type : Zeta Potential
Sample Name : Sample 195
Temperature of the Holder : 25.3 °C
Dispersion Medium Viscosity : 0.890 mPa·s
Conductivity : 0.090 mS/cm
Electrode Voltage : 3.8 V

Calculation Results

Peak No.	Zeta Potential	Electrophoretic Mobility
1	-31.3 mV	-0.000244 cm ² /Vs
2	-- mV	-- cm ² /Vs
3	-- mV	-- cm ² /Vs

Zeta Potential (Mean) : -31.3 mV
Electrophoretic Mobility Mean : -0.000244 cm²/Vs

1064 nm

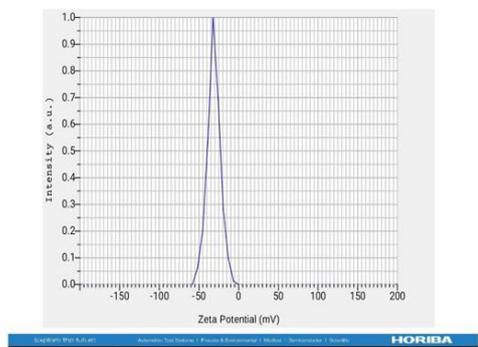


Fig. 5. The findings of surface charge values of the AgNPs prepared by the PLAL method at wavelengths of (532, 1064) nm.

Table 2. Surface charge values of prepared silver nanoparticles using PLAL technique after 2 months.

Laser wavelength	Zeta potential (mV)	Electrophoretic Mobility	Experiment Ref.
532 nm	-35.5	-0.000276 cm^2/Vs	-25.22 [23]
1064 nm	-31	-0.000244 cm^2/Vs	

emphasizes how crucial it is to assess samples over long periods since it provides a fresh perspective on the stability and sustainability of nanostructures while stored at ambient temperature. Table 2 clarifies the surface charge values of silver nanoparticles after a certain preparation period. It was noticed that the stability upon using laser of 532 nm is better than the surface charge value of prepared AgNPs under 1064 nm of laser. All the results are consistent with previous studies as in the two sources [24, 25].

CONCLUSION

The results demonstrated that the pulsed laser ablation approach was effective in safely creating nanoparticles when applied to pure water. Thus, a shift in the hue of the sample-preparation solution is the initial indicator of nanoparticle production. A gradual darkening of the solution's hue is visible to the naked eye. The density of nanoparticles is proportional to the increase in the brownish hue of nano colloidal as the ablation pulse number increases. In addition, the effect of laser parameters (wavelength, laser energy, pulses) on the size and concentration of silver nanoparticles prepared by the pulsed laser ablation method in distilled water was studied. The study proved that the wavelengths used (532, 1064) nm with an energy of 760 mj and 2000 pulses directly impacted the size and concentration of the synthesized silver nanoparticles. The results showed that the average size of the prepared silver nanoparticles increases with the increase in laser energy during the ablation process using the 532 nm wavelength, while the average size of the silver nanoparticles decreases with the increase in laser energy during the ablation process using the 1064 nm wavelength. This is attributed to the fragmentation process, which was enhanced by conducting the

ablation process under constant conditions. The stability of produced AgNPs via PLAL technique is another important goal in this investigation. Therefore, the ranges of surface charge, through Zeta potential measurements, have confirmed the stability of synthesized NPs, which were -35.5 mV and -31 mV, respectively, after two months of preparation.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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