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Gold nanoparticle production by continuous-wave laser ablation in liquid media Producción de nanopartículas de oro mediante láser de onda continua en medio líquido

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KEYWORDS

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Nanopartículas de oro; ablación láser; nanofabricación; láser de onda continua; nanomateriales

ABSTRACT: Over the past two decades, gold nanoparticles have gained much attention because of their unique qualities, such as optical effects, catalytic response, high surface area, and low toxicity. Chemical synthesis and pulsed laser ablation are the primary methods to create these nanoparticles. However, pulsed laser ablation can be expensive because it requires specialized lasers. Using low-cost, continuous-wave lasers is suggested to create gold nanoparticles with diameters of approximately 100 nm. Two different gold target thicknesses were used to observe particle production at different irradiation times and pulse durations. Also, a simple approximation was used to estimate the on/off duration based on target thickness, which yielded good results. This technique can be implemented without incurring high costs, increasing the global ability to produce and use nanoparticles for various purposes.

RESUMEN: Durante las dos últimas décadas, las nanopartículas de oro han ganado interés debido a sus características únicas, como los efectos ópticos, la respuesta catalítica, la alta área superficial y la baja toxicidad. La síntesis química y ablación láser han sido las técnicas más utilizadas en la fabricación de estas partículas. Sin embargo, la ablación láser es una técnica que resulta costosa debido a la alta inversión necesaria en láseres especializados. En este trabajo, sugerimos la utilización de láseres de onda continua, los cuales representan una técnica de bajo costo en la fabricación de nanopartículas de oro que alcanzan diámetros de aproximadamente 100 nm. Dos diferentes espesores de blancos de oro fueron probados para observar la producción de partículas a diferentes tiempos de irradiación y duraciones de pulsos. También se usó una aproximación simple para estimar la duración de encendido/apagado con base en el espesor del blanco utilizado, en la que se obtuvieron buenos resultados. La técnica presentada puede ser implementada sin incurrir en altos costos de inversión, lo que puede incrementar la posibilidad a nivel global de obtener nanopartículas para diferentes aplicaciones.



1. Introduction

Nanomaterials are a fascinating group of materials that have captured the attention of researchers and industry alike. This is due to their incredible properties at the nano level, which are noticeably different from what we see on a macro scale [1,2]. One of the most intriguing subgroups of nanomaterials is nanoparticles (NPs). These tiny particles have received much attention for their potential applications due to their small size and large surface area [3]. Gold NPs, in particular, have gained widespread interest in fields such as medicine, where they are being used for drug delivery because of their low toxicity and small size (less than 100 nm). Furthermore, NPs have a unique behavior when interacting with light, causing them to scatter or reflect light depending on their size and the wavelength of the incoming light. This has led to their increasing use in energy applications like solar panels [4,5] and as co-catalysts to produce green hydrogen [6,7].

There are two primary approaches for making gold nanoparticles: bottom-up, where they are growing from raw materials, and top-down, where they are produced by the elimination of material from a target. The application of these approaches depends on the type of raw materials, intended application, and level of cleanliness required [6]. One method is chemical synthesis, which involves using chemical gold precursors, like salts such as sodium tetrachloroaurate and the sol-gel process. However, this approach can be expensive due to the cost of the precursors and the requirement of technical expertise to achieve the desired sizes and quantities of nanoparticles. As an alternative, physical transformation approaches like micro-mill or laser ablation have become popular in the last decade [8, 9].

Laser ablation is a prevalent technique for generating nanoparticles from metals through physical transformation. This involves creating a plasma state using a laser to detach material from the surface of the metal, causing it to evaporate. However, this method requires high-power pulsed lasers that operate at 250W or higher, necessitating large spaces and specialized infrastructures. These factors contribute to increased nanoparticle production costs [10-12]. Neodymium lasers, with wavelengths near 1000 nm, are a common example of equipment used in this process, as they provide the necessary energy to ablate most metals.

When utilizing a laser to irradiate a solid substance, it produces elastic propagation waves. The power of the laser determines the type of elastic waves produced, namely those that modify the surface (ablation regime) and those that do not (thermoplastic expansion regime).

During laser ablation, particles in an excited state are emitted from the surface. This leads to the creation of a luminous plasma plume that is highly energetic, dense, and non-equilibrium, with temperatures reaching about 10,000 K, speeds of roughly 10^6 cm/s, and pressures of a few tenths of GPa [13]. As the plume expands, it cools rapidly, compressing the surrounding medium within nanoseconds. During this expansion, particles at the interface with the medium intermittently lose energy for a few milliseconds after the initial laser impact on the material surface, slowing down the plume particles at the interface. The cooling plume triggers condensation, nucleation, and clustering of the plume species in the gas phase, eventually leading to the formation of nanoparticles (NPs). The plume eventually decelerates creating a concentration gradient at the interface, which promotes the diffusion of plume species and NPs into the medium. When a liquid medium is used, the strong confinement limits the quenching time of the



expanding plasma plume, resulting in a smaller average diameter distribution of NPs compared to when air, vacuum, or gas mediums are used [14].

Laser ablation is linked to two particle size increase processes. One process involves cavitation phenomena during nanosecond ablation, which occurs when the energy per pulse is high. The other process occurs during short-second ablation, where the energy is lower, and cavitation is prevented.

During the laser ablation process in liquids, newly formed nanoparticles are positioned in the trajectory of the laser beam and undergo a series of interactions with it. These interactions can be classified into two types: intra-pulse, where the particles interact with the same laser pulse that generated them (typical in long-pulse ablation), and interpulse, where the particles interact with subsequent pulses (typical in fs/ps pulse ablation). Such interactions have been shown to reduce particle dispersion [14].

There are limited procedures available for producing gold nanoparticles using low-cost Continuous Wave (CW) lasers, despite well-established methods for producing them via high-power pulsed lasers through laser ablation. Therefore, we propose the use of low-cost CW lasers for this purpose. In this regard, we will present a simple approximation for calculating the laser and material characteristics required for ablation. Additionally, we will describe an economic analysis of different techniques available for producing gold nanoparticles, demonstrating the significant advantage of using CW-lasers. This approach will enable the implementation of this technique without incurring high expenses, thereby making it more accessible worldwide for various purposes.

2. Materials and Methods

A low-power blue light CW laser named LASER-PUR-500MW was used to produce gold nanoparticles. The laser boasts a theoretical wavelength of 450 nm, a maximum power of 6 W, and a spot diameter of 3 mm. To regulate the laser's on/off function, we designed an Arduino system to emulate a pulse with a maximum frequency of 43 Hz. The diameter of the point of incidence was measured on a piece of wood to determine the focal length.

To determine the specific wavelength and bandwidth of the laser, we used a minispectrometer Newport 78356. By irradiating the center of the sensor, we were able to register the counts of photon energy in the Y-axis at specific wavelength values ranging from 100 to 800 nm in the X-axis.

In order to carry out the ablation process, two gold films were utilized. Both films were of 99.99% purity and had a 500 nm and 300 μm thickness, respectively, with the former being used to analyze the formation of NPs. The latter was used to validate the calculations made to predict the amount of laser energy required for successful ablation.

The experiment utilized clean glass vessels to hold each gold target, employing deionized water and a surfactant solution 0.005 M of Sodium lauryl ether sulfate ($\text{CH}_3(\text{CH}_2)_{10}\text{CH}_2(\text{OCH}_2\text{CH}_2)_n\text{OSO}_3\text{Na}$), and the laser was positioned 30 mm away from the targets. The targets were then exposed to the laser for 10



and 20 minutes at a pulse frequency of 43 Hz in the case of the 500 nm film. For the 300 nm film, exposure times of 20, 40 and 60 minutes were used at 1 Hz (as shown in Figure 1). The experiment aimed to evaluate the effect of single-point ablation on two different thicknesses of gold targets, where a higher thickness did not show a response in less time (10 minutes).

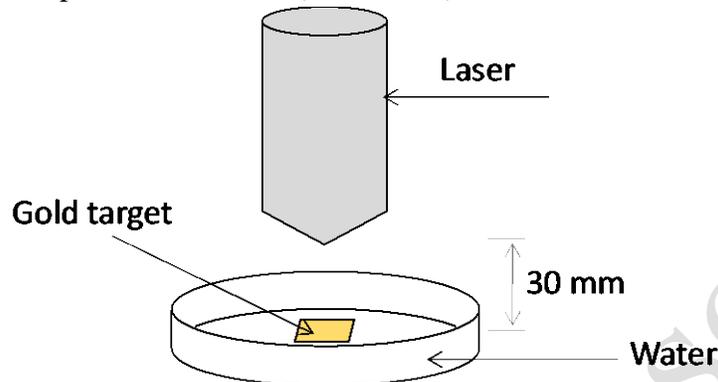


Figure 1. Schematics of the experimental setup.

This dispersant is frequently utilized in pulsed laser gold nanoparticle creation as it facilitates uniform distribution of solid particles in the solvent and ensures optimal interparticle separation [16, 17]. The experiments were performed with both water alone and with the inclusion of the dispersant.

The gold target was removed, and the particle dispersion was dried on a hotplate at 100 °C for 15 minutes. Subsequently, the gold nanoparticles were re-suspended in 1 mL of deionized water and carefully placed onto a cover-glass surface located within a desiccant. The water was allowed to evaporate completely, ensuring the NPs were affixed to the glass surface before microscopic analysis.

To analyze the size distribution of gold nanoparticles (NPs), we captured ten images of 2x2 μm analysis areas from various locations using a Park Systems XE7 atomic force microscope (AFM) with an Acla-10 cantilever in non-contact mode. In initial tests, we opted for this mode as the particles moved even with low cantilever stiffness. After obtaining measurements for each irradiation time and target thickness condition, we employed a threshold tool to scrutinize the nanoparticles and determine their size distribution through statistical analysis using box and whisker diagrams. To determine significant differences between the outcomes, we utilized a one-way analysis of variance (ANOVA).

Electron Dispersive X-ray Spectroscopy (EDS) and Scanning Electron Microscopy (SEM) were used to confirm the chemical composition of the particles obtained.

3. Results and discussion

Laser characterization was performed to pinpoint the exact wavelength and bandwidth of the laser. The findings indicated that the highest point of the wavelength was roughly 455 nm, with a 10 nm range between 452 nm and 462 nm. By directing the laser toward the sensor's center and recording the data displayed on the digital screen, we determined that the maximum potential value was 2.8 W. Similar studies were cross-checked and are presented in Table 1.

Table 1. Laser parameters, operation assembly, and average particle diameter were determined in this study and compared with those of other works.

Laser type	Laser Parameters	Average particle diameter (nm)	Reference
Continuous	Frequency (Hz): 43 Irradiation time (min): 10 Laser Power P (W): 2.8 Laser wavelength (nm): 455	60- 180	This work
Continuous	Laser Power P (W): 1-200 Laser wavelength (nm):1064	8-25 nm	[18]
Pulsed	Frequency (Hz): 15 Irradiation time (min): 5 Laser Power P (mJ): 25 Laser wavelength (nm): 532	10-50	[19]
Pulsed	Frequency (Hz): 5 Irradiation time (min): NA Laser Power P (J/cm^2): 4.3 Laser wavelength (nm): 1064	1-226	[20]

For a target with a thickness of 500 nm, the optimal frequency of laser application to achieve laser ablation is around 43 Hz. Frequencies lower than that can be harmful to the film's integrity and reduce the number of particles produced. Our initial tests at a frequency of 1 Hz on a 500 nm gold target resulted in holes instead of the intended nanoparticles, indicating that the energy input was too high. Conversely, when we used a 300 μ m target at 43 Hz, the energy input was too low to produce ablation or surface modification. However, when we reduced the pulse frequency to 1 Hz, we were able to achieve ablation at a depth of approximately 25 μ m, which resulted in an increased number of particles after subsequent laser pulses.



Selected AFM images of NPs formed using the 500 nm gold target at a frequency of 43 Hz for 10 and 20 minutes are depicted in Figures 2a and 2b. It is confirmed that gold NPs are formed using a CW low-power laser, as evidenced by the results obtained using the two different target thicknesses.

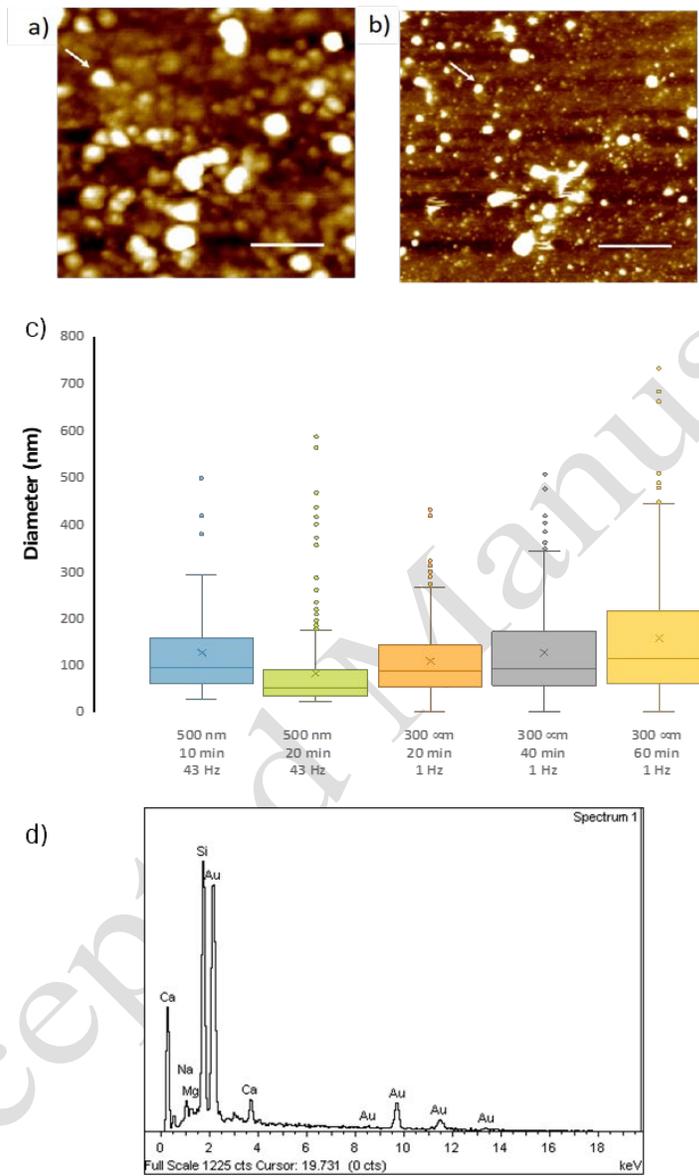


Figure 2. Gold nanoparticles produced by laser ablation with blue light laser in water on the 500 nm gold film at a) 10 minutes of irradiation at 43 Hz, b) 20 minutes of irradiation at 43 Hz. Scale bar 250 nm and c) Particle size distribution for the different times of ablation and gold targets d) EDS spectrum.

The chemical composition of the particles visualized was verified using EDS (Figure 2d), where it was found that there were no other chemical elements different from gold, which also indicated the purity of the particles produced.

According to Figure 2c, the gold NPs generated using both gold targets had a size ranging from 70-140 nm. Figure 2b shows that the 500 nm target and 20 minutes of ablation produced smaller particles. The longer laser pulses (with an on/off cycle of $t=23$ ms) applied to the target are responsible for the reduction in the particle dispersion with extended irradiation periods. This is due to the intra-pulse interaction between the newly formed particles and the same pulse that generated them, as explained in [15]. The data presented in Figure 2c shows a positive correlation between the on/off cycle and particle size. Interestingly, this contradicts the earlier findings. Moreover, as the target thickness rises, NP dispersion has a notable uptick. However, when the pulses are prolonged ($t=1$ s), the newly formed particles tend to coalesce with the existing ones, leading to larger particle sizes.

Various studies have yielded nanoparticles with sizes ranging from a few nanometers to several hundreds of nanometers. For instance, researchers created particles with an average diameter of 10 nm in a liquid medium using high-power pulsed lasers, dispersants, and varying laser fluences [12]. While other group controlled the laser intensity to ensure uniformity of irradiation over the gold target, resulting in NPs with approximately 50 nm diameter [21]. Another group of researchers produced NPs nearly 100 nm in size through a high-power pulsed laser using a target made by sputtering gold in a quartz substrate [20]. It has been observed that similar ranges of gold and silver nanoparticles can be obtained through different methods. However, traditional methods like sol-gel synthesis, biochemistry, or wet chemistry may lead to impurities that can affect the outcome. Therefore, laser ablation is considered a more suitable method of fabrication with fewer impurity issues that may affect the outcome [9].

Using CW lasers can produce nanoparticles of similar size to those achieved through chemical synthesis or pulsed lasers. The crucial element in this technique is regulating both the ablation time and thickness of the gold target to prevent surpassing the ablation threshold. It was demonstrated that using low thicknesses and frequencies that reach the ablation threshold leads to NPs under 100 nm, while greater thicknesses and lower frequencies produce larger particle sizes.

The size of nanoparticles is primarily affected by the energy generated from the cavitation process within the solid. Comparing ablation processes using pulsed and continuous lasers, it was found that continuous lasers rely on the characteristics of liquid and solid materials for the formation of nanoparticles. The lack of cavitation bubbles during this process results in a longer and gentler heating process. It was also noted that localized absorbent sites can only be created by making multiple laser passes. The use of continuous lasers requires greater care in handling reagents and equipment to avoid possible contamination [22].

When utilizing a continuous wave (CW) laser for material ablation, the addition of dispersants may have adverse effects. The surfactant causes an excessive dispersion of particles, preventing their concentration in a single area. However, employing a CW laser with longer pulse durations can produce larger particles through intra-pulse interaction. Unfortunately, the dispersant moves particles away from the laser pulse, diminishing interaction and particle size. [12] determined that the concentration of the dispersant is critical to achieve smaller particles, with a 10^{-2} M concentration of sodium dodecyl sulfate resulting in 6 nm particles. The use of dispersants has made it difficult to study the colloids that are produced as a result



of gold laser ablation [23]. Figure 3 outlines the correlation between fabrication cost and particle size when utilizing chemical synthesis and laser ablation techniques, including both continuous wave and pulsed lasers.

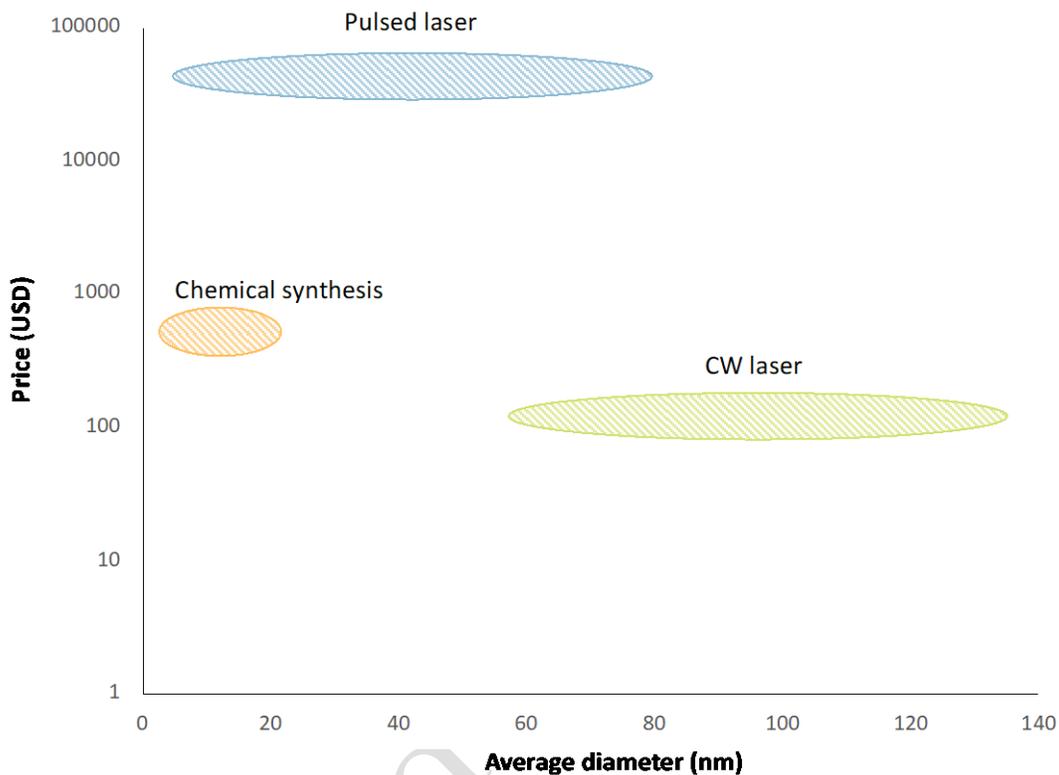


Figure 3. Cost comparison for producing gold nanoparticles using chemical synthesis and laser ablation with pulsed and continuous wave (CW) lasers.

The preparation of gold nanoparticles typically involves the use of tetrachloroauric acid ($H[AuCl_4].3H_2O$) as a starting material and standard laboratory equipment, such as beakers and stirrers. As a result, the primary cost associated with this process is related to the chemical reagents [24]. In contrast, conventional laser ablation methods that use high-power pulsed lasers allocate most of their expenses toward the lasers and their corresponding pulse control systems [21, 25]. By using continuous wave (CW) lasers to generate gold nanoparticles, the costs of equipment and chemical reactants can be significantly reduced while still achieving particle sizes within the range of more expensive techniques. Additionally, the technical expertise and infrastructure required to produce nanoparticles via the method outlined in this study are minimal, providing a significant advantage for producing and utilizing these nanomaterials. However, it should be noted that the number of nanoparticles formed is currently limited and represents an area for improvement.

4. Concluding remarks

Gold nanoparticles with an average diameter of 120 ± 40 nm were produced using a low-cost continuous wave laser. Two different gold target thicknesses of 500 nm and 300 nm were used to produce ablation

at the required pulse frequencies (on/off cycles) and ablation times. Low target thicknesses were shown to require shorter pulses and long irradiation periods to obtain particles with smaller average diameters. In comparison, thicker targets require longer pulses and irradiation periods to reach the ablation threshold required to produce the nanoparticles. The production of nanoparticles using deionized water as a medium showed better results than using a dispersant. Although it is necessary to improve the number of gold NPs created, the use of low-cost lasers to produce nanoparticles is one attempt to increase the use of nanomanufacturing techniques, allowing the entry of this kind of technology to different researchers and countries. So far, access to nanotechnology has been restricted to a limited community concentrated in the richest countries. Broadening access to technology will increase the applications and will further improve the quality of life for communities in need, due to the increased use of gold nanoparticles in areas like medicine and clean energy production.

Declaration of competing interest

We declare that we have no significant competing interests including financial or non-financial, professional, or personal interests interfering with the full and objective presentation of the work described in this manuscript.

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Author contributions

Laura Carmona-Saldarriaga: Visualization, investigation, formal analysis, data curation, conceptualization. Alex Ossa: Writing – methodology, formal analysis, project administration, funding acquisition, formal analysis, conceptualization.

Data availability statement

Data will be made available on request.

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