

# Effect of pulse repetition and fluid medium on morphological and optical characteristics of Ag, Au nanoparticles by laser ablation in solutions

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## Abstract

In this research, gold and silver nanoparticles were prepared using pulsed laser ablation in liquids. The effect of pulse repetition rate (1, 3 and 6 Hz) and (1064 , 355 ) nm wavelengths on the ablation efficiency as well as on the optical properties (Absorption Spectra , Refractive Index , Extinction Coefficient , Real and Imaginary Dielectric Constant) of prepared samples were studied using two media ,SDS and Ethanol where the results generally showed an increase in the parameters above with increasing in repetition rate. The results also confirmed an increase in ablation efficiency for wavelength 1064 nm compared to 355 nm . The study also confirmed that there is a clear effect of the medium used on the optical properties and the efficiency of ablation.

**Keywords:** laser ablation, Surface Plasmon, gold, silver, ablation efficiency.

## 1. Introduction

Nano is a Greek word meaning a very small molecule. Particles that have a size in two or more dimensions in the range (1-100) nanometers can be classified as nanoparticles, as described by the American Society for Testing of Materials (ASTM) [1]. Noble metal nanoparticles are the fundamental structures of nanotechnology and have piqued the interest of Due of their size-dependent electrical, catalytic, magnetic, and optical characteristics, researchers are interested in them [2,3]. As the size of the component particles approaches the nanoscale system, almost all physical characteristics of materials change drastically. Because there are fewer atoms per particle, there is a higher surface-to-volume ratio as a result of the majority of atoms being on the particle's surface, and the electronic energy bands that control the majority of the highly modified physical and chemical properties are smaller than those found in bulk materials, these differences in properties are caused[4]. Changes in reactivity, solubility, all properties related to mass and heat transfer, as well as changes in physical properties like melting point and qualitative properties like dielectric constant, are just a few examples. The effect of size also refers to changes in properties brought on by changes in particle measurement. (the effect of size). This is because the bonds of atoms have changed, and that molecules and atoms on the surface are extremely active, which makes it easier to relate it to other materials and give it qualities that are distinct from those of large objects [5]. As particle size decreases, the distance between the electronic levels and the bandgap grows. This is due to the close proximity of electron-

hole pairs and the coulombic interaction between them [6]. With regard to the colloidal silver visible and ultraviolet light spectrum. Experimentally, the blue shift in the absorption spectra shows this increase in band gap. The size-dependent properties of silver nanoparticles (AgNPs) are used in a variety of potential applications, solar energy conversion, and light-emitting devices [7].

Due to the fact that (SPR) energy of silver nanoparticles is placed distant from the interband transition energy, compared to other metal nanoparticles, they have an advantage, such as copper and gold [3].

To acquire a special property, by adjusting the particle size, the silver SPR peak in water may be modulated throughout a wavelength range of 380-500 nm. The successful implementation of this critical component of nanotechnology is dependent, first and foremost, on the regulation of metal nanoparticle shape, content, and particle size during synthesis. Metal NPs can be made using a variety of physical and chemical methods [8].

Because of surface Plasmon oscillation of electrons, gold nanoparticles (AuNPs) have unique spectral properties in the visible region and liberate when light falls on them [9]. Surface plasmon resonance (SPR), which is the resonance in the visible frequency of noble metals such as silver, gold, and copper, and this property is responsible for changing the colors of these elements when they reach nanosize and depends on the size, shape, and medium of the particle, and this property causes an increase in the ambient temperature nanoparticle when light falls on it. Many properties and applications, include using gold particles to kill cancer cells.

The use of gold nanoparticles in the manufacture of

capsules that contain medicine, and this medicine does not come out except when laser beams fall on it, causing the polymer used to make the capsule to melt [10].

Laser ablation is an efficient method for nanoparticle production [11]. One variant of this technique is the pulsed ablation of bulk metals in a liquid environment. When compared with other traditional methods of preparing mineral colloids, one of the advantages of this process is its simplicity [12].

A- down-top (which involves applying an external force that causes the material to disintegrate into small particles, i.e., starting from a large size to end with the nano, as in pulsed laser ablation (PLA), pulsed discharge wire (PWD), thermal solvent decomposition, and mechanical-chemical fabrication); and b- up-top (which involves applying an external force that causes the material to disintegrate into small particles).

B-The second method is top-down accumulation, which begins with atoms and molecules and progresses to nanoscale structures using techniques such as pulsed laser deposition (PLD), vapor deposition (CVD), and electrodeposition.[13]. Fig (1) Top down and bottom-up approaches of nanoparticles production. When employing a high-powered laser, the energy received by the target causes the substance to get heated, which raises the temperature to a point where vaporization or sublimation occurs. When the laser energy falls When a unit of time is high on the target, the substance turns into plasma. Using a pulsed laser, the ablation process is used to remove material. The amount of material eliminated by a single laser pulse depends on the laser's intensity, depth of penetration, optical qualities of the material, wavelength, and pulse duration. [14].

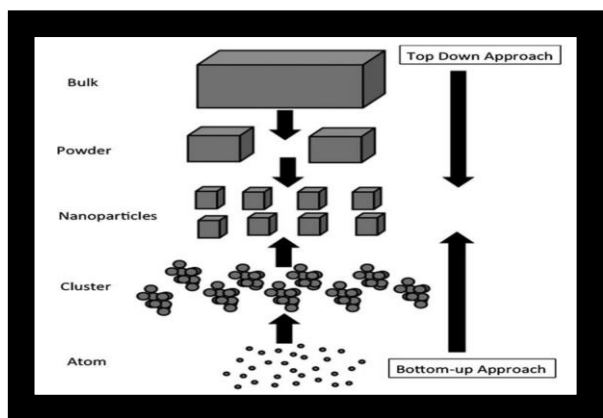


Fig 1 :Top down and bottom-up approaches for creation of nanoparticles.

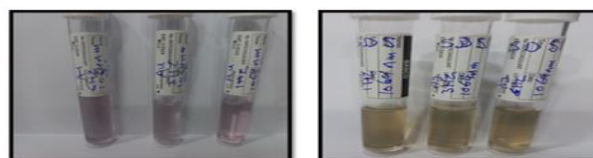
In the paper, we aim to improve the efficiency of ablation of synthesized AgNPs, AuNPs, Au-Ag and Ag-Au alloy by laser ablating of Ag and Au in ethanol and SDS solutions. This study used an empirical approach to investigate the impact of pulse repetition rate. We try to ascertain the experimental circumstances for these variables, which were crucial in the creation of many NPs under intense enough

laser light.

## 2. Experimental Methods

Laser ablation of high-purity silver and gold (PLA) led to the formation of silver and gold nanoparticles Colloidal. The gold piece is 99.99% pure, round in shape, 2 cm in diameter, and 1mm thick. In high-purity ethanol and 10% SDS solution, and with a Q-switched Nd:YAG laser with a wavelength (1064 nm) and a third Harmonic Generation (355 nm) and pulse width (10 ns), the pulse repetition rate (1 , 3 and 6 Hz) and 200 pulses number. The sample is at the bottom of a container containing one of the solutions listed above. A quartz lens with a focal length that concentrates the laser beam onto the sample surface (20 cm). The volume of solution in the ablation container is 4 mL, and the target is excised with laser energy (80 mJ).

Pulsed lasers (Nd:YAG) are used as a power source in many applications. It is popular because it is dependable and simple to use, in addition to being used at second and third harmonic wavelengths (visible 532 nm) and (ultraviolet 355 nm), respectively. These lasers, which may also be employed in continuous wave or Q-switched mode, generate the harmonics by passing a nonlinear optical crystal down the path of the 1064 nm laser beam.



(A) Gold nanoparticles

(B) Silver nanoparticles

Figure 2: Samples of nanoparticles prepared in SDS solution at a concentration of 10% A-Gold Samples of nanoparticles prepared in SDS solution. B-Samples of Ag nanoparticles prepared in SDS solution.

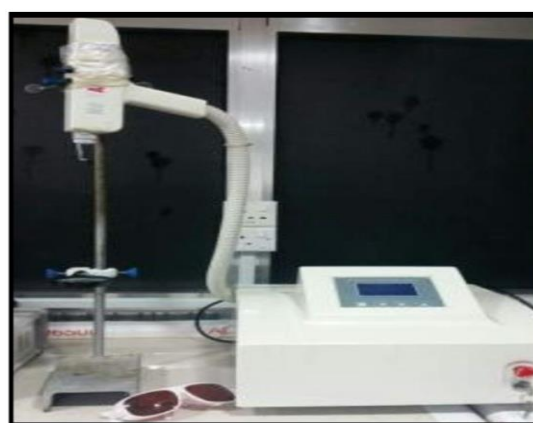


Figure 3: The laser used in ablation.

## 3. Results and Discussion

UV-Visible measurements is an effective method for determining the size and shape of nanoparticles. An Nd:YAG laser operating at (1064 and 355) nm, 80 mJ, and 200 pulses per second was used to analyze the produced gold and silver collides in ethanol and

SDS solutions. Figure (2-A) appear that color of gold colloidal nanoparticles changed with an increase in the pulse repetition rate between (1, 3 and 6) Hz. The figure (2-B) represents the change in the color of the colloidal solution of silver nanoparticles with an increasing pulse repetition rate (1, 3 and 6) Hz, and this is caused by the increase in the concentration of the generated nanoparticles.

The absorption spectra of colloidal gold suspensions are shown in Figure (4). There are significant absorption peaks in the vicinity of (512–524) nm due to surface plasmon resonance, whereas there are minor absorption peaks in the vicinity of (206–248) nm due to interband transitions, which determined the degree of ablation. The presence of strong and isolated peaks reveals the shapes of the particles. TEM pictures confirmed that the gold nanoparticles created were spherical and semi-spherical, as will be detailed later. The modest absorption peak demonstrates the effectiveness of the ablation, since while the thickness of the liquid was fixed at 4 ml, the efficiency of the ablation rose as the thickness of the liquid was lowered [15].

Figure (4-A-B) depicts the absorption spectra of gold nanoparticles at the stability of the wavelength of a Nd: YAG laser at third harmonic (355) nm using two solutions: ethanol and SDS at 10% concentration, respectively, the absorption peak shifted toward shorter wavelengths (blue shift) with the change in the pulse repetition rate. The appearance of these peaks resulted from the small size rate of the resulting nanoparticles with the increase in the repetition rate of the laser pulse used in the ablation process, which led to an increase in absorption values.

Figure (4-C-D) shows the optical absorption spectra of gold colloids at the wavelength of the laser used (1064) nm. Using ethanol and SDS solutions, the characteristic peaks of surface plasmon resonance experienced a red shift with the change in the pulse repetition rate.

Based on the data, we infer that the absorbance in ethanol was greater than the absorbance in the SDS solution at 355 nm, while at 1064 nm, the absorbance was higher in the SDS solution than in ethanol.

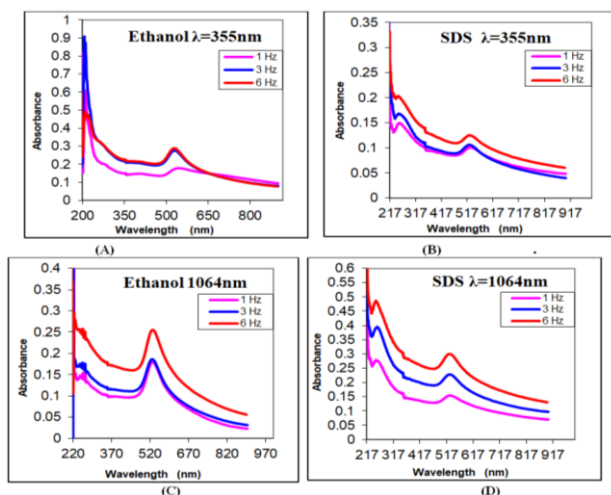


Figure 4: Absorption of the gold (Au) at different repetition rate (1–3–6) Hz and with wavelengths (355 nm, 1064 nm) for ethanol and SDS solution.

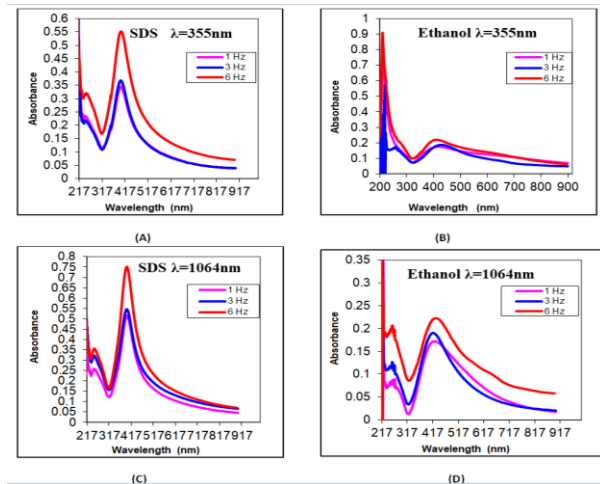


Figure 5: Absorption Spectra of the silver (Ag) for different frequency of the repetition (1–3–6) Hz at a wavelength (355 nm, 1064 nm) for ethanol and SDS solution

After laser ablation of the Ag target in ethanol solution and SDS solution, colloidal Ag solutions was obtained [16]. The absorption spectra of nanoparticle solutions, which were created by ablating a silver target in 10% SDS and ethanol, are shown in Fig. 5. with a laser's (355, 1064) nm wavelength. The laser was set to have a pulse energy of 80 mJ, a pulse width of 10 ns, a pulse repetition rate of 1, 3, and 6 Hz, and a pulse count of 200. There is an absorption peaks around (390–415) nm due to SPR, and weak absorption due to interband transitions in the 200–250 nm region [14,17].

It can be shown from a study of interband transition absorbances at 250 nm that the formation efficiency of colloids rises as the laser wavelength increases. [18,19]

In Figure (5-A) we notice that the absorbance at the shorter wavelength (355 nm) increases with an increase in the pulse repetition rate. As for Figure (5-C) at the wavelength (1064 nm) got a good result through the SPR which represents the absorbance peaks that have a blue shift with the change in the repetition rate. It is clear from the above that there is an increase in absorption with an increase in the laser repetition rate, and the increase in absorbance indicates the efficiency of ablation increases with the increase in repetition rate. [20]

By comparing the above two figures with solution (SDS) and wavelength change, the absorbance values were greater at the longer wavelength than at the shorter wavelength.

The morphologies of the plasmon band were demonstrated in Figure (5-B-D) as being dependent on the laser wavelength. The plasmon bands obtained with 355 laser light are more widely spread in the red spectral area as compared to the spectrums obtained with 1064 nm laser wavelength. Diverse laser wavelengths led to a change in particle size, according to the different Plasmon band shapes. [19].

Figure (5B) The absorption spectra of silver nanoparticles in ethanol solution with a wavelength of 355nm showed that the absorbance increases with increasing pulse repetition rate. As for figure (5D) at the wavelength 1064 nm the SPR which represents the absorbance peaks that have a blue shift with the change in the repetition rate, the increase in absorbance indicates the ablation efficiency increases with the increase in repetition rate. [20] From the above-mentioned results, we conclude by increasing the repetition rate of laser pulses, the maximum wavelength was shifted to a lower wavelength, according to the Mie theory, this blue shift is related to the reduction of particle size [21]. Therefore, we can say that a rise in the number of laser pulse repetitions causes a decrease in particle size. The reason why particle size decreases as laser pulse repetition rates increase is because, at higher repetition rates, more generated NPs from the target

will interact with the laser light. Because of this contact, NPs are fragmented into smaller sizes, resulting in smaller particles being produced at higher repetition rates. [22,23,24].

The experimental results show that there are a difference in optical parameters as in the following tables mentioned.

Table (1) shows a comparison between the refractive index, damping index, real dielectric constant and imaginary dielectric constant for gold and silver nanoparticles in a high-purity ethanol solution with wavelength = 1064nm and the effect of the pulse repetition rate on these parameters. The refractive index is calculated from the equation: -

$$n = c/v \tag{1}$$

Where: -

**n** : Refractive index . **c**: Light speed in a vacuum.  
**v** : Light speed in the medium.

Table 1: Effect of pulse repetition rate on the optical properties of NPs in Ethanol at a wavelength = 1064 nm.

λ(nm)	Au				Ag			
	1064 ( nm )				1064 ( nm )			
	Imaginary Dielectric Constant	Real Dielectric Constant	Extinction Coefficient (K)	Refractive index	Imaginary Dielectric Constant	Real Dielectric Constant	Extinction Coefficient (K)	Refractive index
P.R.R. (Hz)								
1	3.76036*10-7	1.373435	1.60*10-7	1.171936	2.90508*10-7	1.2936409	1.28*10-7	1.1373833
3	4.44545*10-7	1	2.22*10-7	1	2.93916*10-7	1	1.47*10-7	1
6	1.12364*10-6	2.001225	3.97*10-7	1.414646	1.18381*10-6	2.0474373	4.14*10-7	1.4308869

Table (2) shows a comparison between the refractive index, damping index, real dielectric constant and imaginary dielectric constant of gold and silver

nanoparticles in 10% SDS solution with wavelength = 1064 nm and the effect of the pulse repetition rate on these parameters. From the following equation, the Extinction Coefficient was calculated: -

Table (2) : Effect of pulse repetition rate on optical characteristics of NPs in 10% SDS at 1064 nm wavelength

λ(nm)	Au				Ag			
	1064 ( nm )				1064 ( nm )			
	Imaginary Dielectric Constant	Real Dielectric Constant	Extention Coefficient (K)	Refractive index	Imaginary Dielectric Constant	Real Dielectric Constant	Extention Coefficient (K)	Refractive index
P.R.R. (Hz)								
1	1.52717*10-6	2.297476	5.04*10-7	1.5157427	9.0006*10-7	1.821662	3.33*10-7	1.3496896
3	1.3894*10-6	1	6.95*10-7	1	9.44258*10-7	1	4.72*10-7	1
6	3.5135*10-6	3.56621	9.30*10-7	1.888443	1.50316*10-6	2.285006	4.97*10-7	1.5116238

$$K_e = \frac{\alpha \lambda}{4\pi} \tag{2}$$

Where:- **K<sub>e</sub>** : Extinction Coefficient , **λ**: Represents the wavelength , **α** : Absorption coefficient .

Table (3): Effect of pulse repetition rate on the optical characteristics of NPs in ethanol, with a wavelength of 355 nm.

λ(nm)	Ag				Au			
	355 ( nm )				355 ( nm )			
	Imaginary Dielectric Constant	Real Dielectric Constant	Extention Coefficient (K)	Refractive index	Imaginary Dielectric Constant	Real Dielectric Constant	Extention Coefficient (K)	Refractive index
P.R.R. (Hz)								
1	1.22613*10-6	3.324117	3.36*10-7	1.8232161	1.57155*10-6	3.8155697	4.02*10-7	1.9533483
3	9.7339*10-7	2.947842	2.83*10-7	1.7169281	2.67039*10-6	5.0855943	5.92*10-7	2.2551262
6	1.51852*10-6	3.743194	3.92*10-7	1.9347336	2.81907*10-6	5.2323744	6.16*10-7	2.2874384

Table (3) shows the comparison of refractive index, damping index, real dielectric constant and imaginary dielectric constant of gold and silver nanoparticles in high purity ethanol solution with wavelength = 355 nm and the effect of pulse repetition rate on these parameters. We calculated the real dielectric constant from the equation:-

$$\epsilon_r = n^2 - K_e^2 \quad (3)$$

Where:-  $\epsilon_r$  : Real Dielectric Constant.  $n$  : Refractive

index.  $K_e$  : Extinction Coefficient.

Table (4) shows a comparison between the refractive index, damping index, real dielectric constant and imaginary dielectric constant of gold & silver nanoparticles in 10% SDS solution with wavelength = 355 nm and the effect of the pulse repetition rate on these parameters. From the following equation, the Imaginary Dielectric Constant ( $\epsilon_i$ ) was calculated: -

$$\epsilon_i = 2nK_e \quad (4)$$

Table (4) : Effect of pulse repetition rate on the optical properties of NPs in SDS with concentration of 10% and wavelength 355 nm

$\lambda$ (nm)	Ag 355 ( nm )				Au 355 ( nm )			
	Imaginary Dielectric Constant	Real Dielectric Constant	Extention Coefficient (K)	Refractive index	Imaginary Dielectric Constant	Real Dielectric Constant	Extention Coefficient (K)	Refractive index
1	2.2282*10-6	4.627497	5.18*10-7	2.1511618	9.11206*10-7	2.8312737	2.71*10-7	1.6826389
3	2.3651*10-6	4.849880	5.37*10-7	2.2022444	1.00801*10-6	2.9837198	2.92*10-7	1.7273447
6	4.19993*10-6	6.185409	8.44*10-7	2.4870483	1.43124*10-6	3.6216851	3.76*10-7	1.9030725

The morphology of silver and gold nanoparticles was studied using Transmission Electron Microscope (TEM) model (Zeiss- EM10C) operating at an accelerating voltage of 100 kV to analyze the size and shape of the nanoparticles.

Figure (6) shows TEM images and size distribution of NPS of (Au, Ag) prepared by laser ablation, where the wavelength (1064,355) nm and energy (80)mJ, in the SDS .

Figure(6-A-B) shows the particles with size of less than 80nm,150nm for Au NPS colloidal , and note that the form of NPS can be almost spherical .Their absorption spectra were varying depending on size and shape of the nanoparticles.

Figure (6-C-D) shows that the Ag NPs are irregular in shape and dispersed in size, due to the wide and irregular peak and shows formation of particles with size of less than 40nm,80nm for Ag NPS colloidal.

nanoparticles structure of Au &Ag with variable size. Different approaches have been used to study the created nanoparticles materials to reveal the development of their crystallinity, the morphological nanostructure, and changes in their linear optical characteristics. Different scientific methods were used to characterize their physical characteristics. The response of the produced nanostructure was superior to that of both Ag & Au metallic NPs. The Au & Ag nanostructured material's effectiveness therefore supports its applicability for optoelectronic materials. The ablation efficiency of the 1064nm wavelength is also higher than that of the 355nm wavelength, Results indicate that there is a clear effect of the type of medium used.

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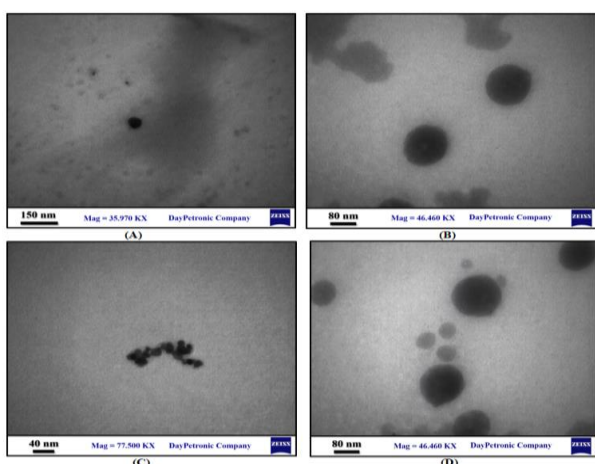


Figure (6) TEM image for spherical. A,B-Gold NPs and C,D-silver NPs

### 4. Conclusion

This research shows the ablation process from the nanosecond laser of the Ag and Au sources immersed in various types of solutions with varied laser pulse repetition effectively synthesized the

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