© 2022 ІМФ (Інститут металофізики ім. Г. В. Курдюмова НАН України) Надруковано в Україні.

PACS numbers: 79.20.Eb, 81.16.Mk, 87.19.xb, 87.64.Cc, 87.64.Dz, 87.64.km, 87.85.Rs

Nanoparticle Preparation and Antibacterial-Activity Analysis Using Pulsed Ablation at 1064 and 532 nm

Ruaa A. Mohammed¹, Falah A-H. Mutlak¹, and Ghada Mohammed Saleh²

¹College of Science Department of Physics, University of Baghdad, Baghdad, Iraq ²College of Science Department of Biology, University of Baghdad, Baghdad, Iraq

Q-switched Nd:YAG laser with wavelengths of 1064 nm and 532 nm, with energy in the range from 200 mJ to 1000 mJ, and with 1 Hz repetition rate is employed to synthesis of Ag nanoparticles (NPs) using pulse laser ablation in a water. In this synthesis, at first, silver nanocolloid is prepared by ablation target at fundamental wavelength of 1064 nm, and then, it is followed by ablation of Ag target at second harmonic generation of 532 nm for different energies to form Ag NPs. Surface plasmon resonance (SPR), surface morphology and average particle size are characterized by UV-Vis spectrophotometer and transmission electron microscope (TEM). The absorbance spectra of Ag NPs show sharp and single peaks around 400 nm and 410 nm, respectively. The average diameters of silver nanoparticles are achieved as 18 nm and 21 nm corresponding to 1064 nm and 532 nm, respectively. As far as the consequences of toxicity are concerned with, silver nanoparticles with a diameter of 8 nanometers are shown to kill both Escherichia coli (E. coli) and Staphylococcus germs. These results can be used in biomedical applications.

Лазер Nd:YAG з модуляцією добротности та з довжинами хвиль у 1064 нм і 532 нм, з енергією в діяпазоні від 200 мДж до 1000 мДж і зі швидкістю повторення у 1 Гц використовується для синтези наночастинок Ag (HЧ) за допомогою імпульсної лазерної абляції у воді. У цій синтезі спочатку наноколоїд срібла одержують абляційною ціллю на фундаментальній довжині хвилі у 1064 нм, а потім за нею слідує абляція цілі Ag на другій гармонічній ґенерації 532 нм для різних енергій з утворенням Ag-HЧ. Поверхневий плазмонний резонанс, поверхнева морфологія та середній розмір частинок характеризуються спектрофотометром у видимій і ультрафіолетовій областях світла та трансмісійним

829

електронним мікроскопом. Спектри поглинання Ag-HЧ показують різкі та поодинокі піки близько 400 нм і 410 нм відповідно. Середні діяметри наночастинок срібла досягаються як 18 нм і 21 нм, що відповідають 1064 нм і 532 нм відповідно. Що стосується наслідків токсичности, то наночастинки срібла діяметром у 8 нанометрів, як показано, вбивають як кишкову паличку (*E. coli*), так і мікроби стафілокока. Ці результати можуть бути використані в біомедичних застосуваннях.

Key words: laser, ablation, nanoparticles, silver, antibacterial agent.

Ключові слова: лазер, абляція, наночастинки, срібло, антибактерійний засіб.

(Received 22 November, 2021; in revised form, 9 September, 2022)

1. INTRODUCTION

Nanoparticles are the essential materials structure of nanotechnology and have many implementations in a variety of fields, such as biosensors and electronic nanodevices [1, 2]. Mainly nanoparticles of metals are more attractive this is due to the physical and chemical properties it possesses, which depend entirely on its size. The creation of metal nanoparticles in a liquid circumference by laser ablation of solids is an alternative to the well-known chemical process and is characterized comparatively simple laboratory setup. Laser ablation creates metal nanoparticles similar to chemical approaches, although the surface does not contaminate with residual ions derived from reactants [3]. Surface plasmon resonance (SPR) [4] occupies the optical absorption spectrum of mineral nanoparticles as they pass to longer wavelengths by increasing the scale of these nanoparticles. However, the echo of noble metallic nanoparticles will be evident in this region.

The aspect of the reciprocal oscillation of electrons contributes to the power of the plasmon resonance assigned to them [5]; they are usually thought to be adaptive to size, shape and environment [6]. Therefore, by the next UV/Vis absorption, the plasmon resonance would allow the transmission of both shape and size clearly by this form of absorption. Since, the frequency of transfer between these ranges is far from the surface plasmon resonance energy of Ag, it is also noted that these silver nanoparticles, in particular, have an advantage over the majority of other metallic nanoparticles, such as gold and copper. It is well known that silver nanoparticles have a plasmon resonance; the wavelength depends on the shape and size of the nanoparticles at 400 nm [7]. Although it is significant to control the allocation of particles in size, the process of the formation of colloids by laser ablation was not thoroughly investigated. Therefore, the effects of status of ablation on ablation effectiveness and particle size have been studied by several researchers [8, 9]. We focus on the synthesis of silver nanoparticles by laser removal of mineral components in pure water without any chemical additives. In this work, using Q-switched Nd:YAG pulses at 1064 nm and wavelength of 532 nm, Ag NPs are synthesized in a water, and separate laser energy is used to irradiate silver. These nanoparticles may provide open avenues for biomedical applications for the use of these particles.

2. EXPERIMENTAL SETUP

Figure 1 shows the laboratory experimental scheme of the formation Ag colloids through laser ablation. Deionized and redistilled at the bottom of an ultrapure water filled glass container, the silver plate (>99.99%) was mounted. The silver foil was coated with a film of water that was about three-millimetre thick. Slowly, the solvent is stirred during the ablation process and the dissolution of the colloid particles. Preparations were rendered in an open-air (aerobic) state. The working Q-switched Nd:YAG laser system was used with pulse energy of 200-1000 mJ/pulse, a pulse output of 10ns and a repeat rate of 1 Hz. A convex lens with a focal length of 12 cm was used; the laser beams are focused at 2.1 mm (for 532 nm) and 2.4 mm (for 1064 nm) in diameter and by selecting the centre section of the laser beam to observe the intensity of the unfocused spot of the laser light. When the laser lamp has a discharge voltage, the light energy is controlled and tested by an optical power detector. As an irradiation source, the main (1064 nm) and second harmonic (532 nm) outputs of the Nd:YAG laser were used.

3. CHARACTERIZATION OF Ag COLLOIDS

There was 3 ml of the colloid solution after each ablation-

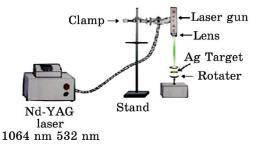


Fig. 1. The experimental framework for the preparation of Ag colloids using laser ablation.

fragmentation. For the UV/Vis optical spectral characterization, a 1 cm wide quartz cell was transferred. The Hitachi U-3410 spectrophotometer from Japan has been used to record the colloid absorption spectrum. To take the micrographs of the electron of resulting nanoparticles, a TEM (JEM-200CX) at 200 kV was used to observe colloidal particles. Two tubes were used to collect a blood sample of 2 mL: the first tube containing Ag NPs and the second tube containing whole blood. Blood samples were incubated for 1 hour at 37° C. A haematological autoanalyser (Orphee Mythic 22 Hematological Analyzer; Diamond Diagnostic, USA) was used to test various blood parameters, such as red blood cells (RBCS), white blood cells (WBCS) and hemoglobinine (HB).

4. RESULTS AND DISCUSSION

Figure 2 showing the normal colloidal solution absorption spectra formed by ablation and targeting Ag in distilled water with focusing laser radiation. The laser light size of spot and energy were 2.1 mm (for 532 nm, 1000 mJ/pulse) and 2.4 mm (for 1064 nm, 1000 mJ/pulse), sequentially, and the ablation period was 15 minutes. Spectrum is characterized by the characteristics of Ag colloidal solutions [10] with a small, symmetrical peak at 401 nm and a large tail dilating to the range of UV wavelength. The absorption of electronics bandwidth is approximately 70 nm (FWHM), which is significantly lower than the chemically formed colloid [4]. At 400 nm, the apex and the tail portion are extracted from the particles. Transition of the excitation of plasmon and interband, sequentially. The apparent solo peak at 401 nm is attributed to the certainty that nanoparticles existent in solution are spherical.

The colloidal solution absorption for the energy used was intend-

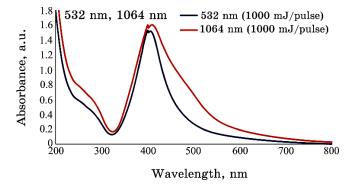


Fig. 2. UV/Vis absorption spectrum of silver-water colloidal particles by laser (532 nm, 1064 nm).

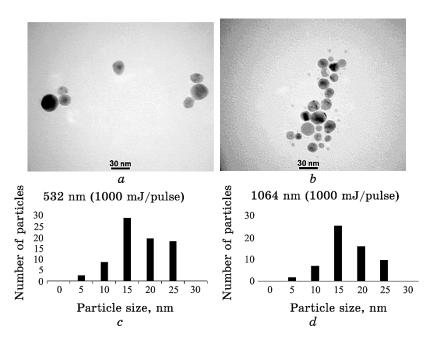


Fig. 3. (a, b) TEM images of Ag NPs produced by laser ablation with wavelengths of 532 nm and 1064 nm, respectively; (c, d) size distribution of Ag NPs.

ed to analyse the constancy of the formed Ag nanoparticles. As shown in Figure 3, there is a slight change towards a longer wavelength in the direction of the surface plasmon peak, with a small decrease in absorption. Hence, over these energies, silver nanoparticles do not curd and have stability. In comparison, the size and form of the Ag particles tend to the plasmon band was substantially influenced [11]; the ablation performance of the 250 nm inter band absorption absorbance was measured. The variance of the efficacy of the ablation depends primarily on the laser effect. To approximate the relationship, experiments of persistent spot size should be carried out between the quality of fluence and ablation efficiency. Absorption spectrum of silver colloidal solutions primed various values of laser fluence in Fig. 4 showing that. The scale of the spot was of 2.1 mm (for 532 nm), and the ablation was performed for 15 minutes. As the laser fluence increased, ablation and absorption at the plasmon resonance peak increase. Minimal average of particles' size also shows the blue shift change for 200, 400, 600, 800 and 1000 mJ/pulse.

Figure 5 indicates the absorption spectrum of silver colloids as silver foil for varying energies for both crystals is ablated by continuous fluence.

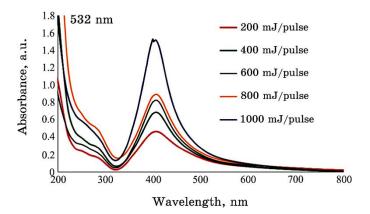


Fig. 4. Silver-colloid spectral absorption through laser fluence diverse for 532 nm and crystals.

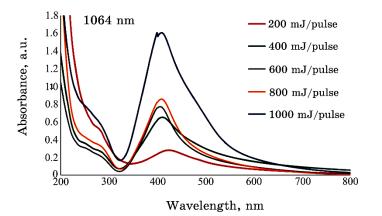


Fig. 5. Silver colloid absorption spectrum primed at 1064 nm by distinct laser fluence.

The scale of the spot was 2.4 mm (for 1064 nm), and the ablation was performed for 15 minutes. Ablation performance and absorbance have been increased and converted to higher energies by increasing the ablation energy from 200 to 1000 mJ/pulse. This shows an augmentation in the volume of absorbed crumbs in the spectral domain of UV/Vis and a small decrease in the average size. Ablation adequacy and absorption were decreased to 400 nm after 15 minutes of ablation. Follow up of ablation, capability greater than 1000 mJ/pulse again leads to an increase in absorption. This can be clarified by the approach of self-absorption, since many particles of metal are formed on the pathway of incidental laser beam light. Thus, blue transition is associated with the disintegration of massive particles. Absorption bandwidth of the peaks reduced to 69 nm. Directly, this refers to a cramped dispensation of particles. In various previous studies [8, 12], the performance of 532 nm of colloid formation was higher than that of 1064 nm $< 1 \text{ J/cm}^2$ lasers.

The above subject demonstrates that self-absorption of the light of laser beam by colloidal particles has a major effect on the effectiveness of ablation. Strength of self-absorption via Ag particles at 532 nm should to greater than at 1064 nm due to plasmon bands about 401 nm.

Chen and Yeh [13] statement matching enlargement of the plasmon bands because of silver particle aggregation. This segment discusses the improvement of the laser ablation procedure for nanosize of the silver colloids. These pulses are suppressed by the 1064 nm laser pulses with energy of 1000 mJ/pulse in 15 minutes by the colloids, which are distinguished by a maximum extinction of SP at 401 nm and a bandwidth of 84 nm as the broad extinction tail extending to the NIR spectral district. The frittering of the 1064 nm laser pulse formed a colloid marked through the evanescence narrow of the SPR (74 nm), while the overall extinction path of the SP band stayed unchanged. Preparation of 532 nm laser pulses resulted in moreover becomes narrower of the extinction band, and the absorption was increased by more than 1064 nm laser pulses in this case. In general, it can be concluded that only large-scale nanoparticles with comparatively strong extinction in the NIR spectral region have been impacted through friability of 1064 nm pulses due to the fact that the wavelength will exceed the absorption range within the SPR during preparation of 532 nm, that the majority of these nanoparticles are redistributed and distributed into the colloid. Increase in absorption and decrease in bandwidth is less than prepared by 532 nm for preparation through of subsequent handling with two wavelengths, but the constancy of the silver colloid formed by this is greater than that of the other cases, as seen in Fig. 4.

5. APPLICATIONS

The aim of this experiment was to study nanoparticles in choosing these particles as antibacterial agents or in some other biological application in terms of the efficiency of these particles by reducing the sizes of the diameters based on the use of wavelength and with different energies.

5.1. Ag NPs ANTIMICROBIAL ACTIVITY

The ability to suppress bacterial growth may be assessed thanks to

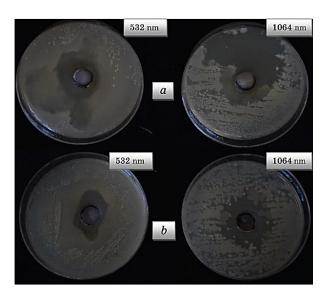


Fig. 6. Presence of *E. coli* Ag NPs treated at applied laser wavelengths of (a) 532 nm and (b) 1064 nm.

the creation of Ag NPs. Produced samples have been examined for the presence of E. coli and Staphylococcus aureus (E). Incubation was taking place for 24 hours on each of the samples. The absence of bacterial cell growth near Ag NP drops shows that Ag NPs have antibacterial properties. To test the Ag antibacterial potency, measurements were taken near where it diffuses. Increases in the inhibition zone breadth have resulted in increased Ag NP activity, which can be seen in the images of the inhibition zone (Fig. 6).

E. coli was more resistant to Ag NPs than *Staphylococcus*. A growing number of microbicidal treatments employ silver nanoparticles' interactions with biosystems, which are just now becoming evident. Silver nanoparticles with the same surface area, but different active facets will have different effective surfaces. Silver nanoparticles' capacity to kill germs is linked to their effective surface areas and size, both of which are critical.

6. CONCLUSION

Laser ablation of pure silver sheets in a liquid may be used to create silver nanoparticles, if the proper laser characteristics are employed. A shorter laser wavelength produced silver nanoparticles with lower concentrations that were smaller. While the longer laser wavelength may be used to make larger sizes with higher concentrations, low laser energy also leads to larger elliptical-shaped sizes. NANOPARTICLE PREPARATION AND ANTIBACTERIAL-ACTIVITY ANALYSIS 837

These nanoparticles have antibacterial action against microorganisms including *Escherichia coli* and *Staphylococcus aureus*. Silver nanoparticles may be synthesized successfully using only an Nd:YAG laser. Using 355 nm laser settings, researchers were able to synthesize 10 nm size nanoparticles with high energy density and high density per square centimetre of 25 J/cm². Laser ablation particle size may be changed by changing the number of laser pulses. If the laser is changed from 1064 to 355 nm, the particle size of colloids generated by laser ablation may be controlled that we observed. This technique may be used to make the appropriate size colloids in solutions using a solution. As the laser wavelength is reduced, the mean particle size decreases from 27 to 8 nm, as seen by TEM.

ACKNOWLEDGEMENTS

We would like to acknowledge the Iraqi Ministry of Higher Education and Scientific Research and Laboratory of laser, Department of physics, University of Baghdad for their generous support.

REFERENCES

- 1. Y. Ying, R. M. Rioux, C. K. Erdonmenz, S. Hughes, G. A. Somorjai, and A. P. Alivisatos, *Science*, **304**: 711 (2004).
- 2. X. Y. Kong, Y. Ding, R. Yang, and Z. L. Wang, Science, 303: 1348 (2004).
- J. Pfleger, P. Smejkal, B. Vlckova, and M. Slouf, *Proc. SPIE5122*, p. 198 (2003).
- 4. R. Baruse, H. Moltgem, and K. Kleinermanns, *Appl. Phys. B*, **75**: 711 (2002).
- 5. A. Henglein, J. Phys. Chem., 97: 5457 (1993).
- 6. A. Kawabata and R. Kubo, J. Phys. Soc. Jpn., 21: 1765 (1966).
- 7. S. Eustis, G. Krylova, A. Eremenva, A. W. Schill, and M. EL-Sayed, *Photochem. Photobiol. Sci.*, 4: 154 (2005).
- 8. T. Tsuji, K. Jryo, Y. Nishimura, and M. Tsuji, J. Photochem. Photobiol. A, 145: 201 (2001).
- S. Link, M. B. Mohamed, B. Nikoobakht, and M. A. EL-Sayed, J. Phys. Chem., 103: 1165 (1999).
- 10. M. Kerker, J. Colloid Interf. Sci., 105: 297 (1985).
- F. Mafune, J. Kohno, Y. Takeda, T. Kondow, and H. Sawabe, J. Phys. Chem. B, 104: 9111 (2000).
- 12. T. Tsuji, K. Iryo, H. Ohta, and Y. Nishimura, *Jpn. J. Appl. Phys.*, **39**, Pt. 2: 981 (2000).
- 13. Y. H. Chen and C. H. Yeh, Colloids Surf. A, 197: 133 (2002).