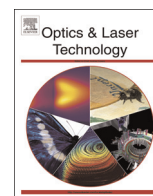




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# Effects of water depth and laser pulse numbers on size properties of colloidal nanoparticles prepared by nanosecond pulsed laser ablation in liquid

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## ABSTRACT

In this paper, pulsed laser ablation method was used for synthesis of colloidal nanoparticles of aluminum and titanium in a distilled water medium. The interaction was performed in a water cell in which the target was placed at different depths of water. The effects of the number of laser pulses and the water depth in which the interaction occurred on average size and size distribution of prepared colloidal nanoparticles were investigated. A UV–vis absorption spectrophotometer and a scanning electron microscope were used for the characterization of the produced nanoparticles. Using image processing techniques and analyzing the SEM images, nanoparticles size properties were achieved. According to the results, position of the target in different water depths has strong effect on size properties of the synthesized nanoparticles. Our results also showed that higher number of laser pulses produces smaller mean size nanoparticles with narrower size distribution.

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## 1. Introduction

There is a growing interest in the fabrication of nanoparticles and their scientific and technological applications. In contrast with bulk materials, nanoparticles have larger surface area and higher density on their surface. Because of such properties, nanoparticles have particular applications in medical technology, biotechnology and energy technology [1,2]. Nanoparticles have special physical and chemical properties that strongly depend on their size and shape. In using nanoparticles for different applications, the size properties (mean size and size distribution) of nanoparticles have significant role in their performance. Colloidal nanoparticles historically were produced by chemical techniques [3,4]. While the chemical techniques have been quite successful for producing a large variety of nanoparticles, they have some limitations in terms of composition and surface contamination. For example, most of the chemical synthesis methods result in large-size particles with wide size variance. Therefore, such produced nanoparticles need further post-processing to enhance the size distribution.

Laser based techniques offer alternative approaches to produce nanomaterials in a clean environment with a good control on size properties of nanoparticles during the fabrication process. Pulsed laser ablation in liquid (PLAL) is a versatile technique for

fabrication of colloidal solutions and can be used to produce nanoparticles for a wide range of applications [5,6]. Furthermore, unlike most chemical techniques, PLAL method does not create any contamination in the nanoparticles, and preserves the material purity. Because of less impurity contamination in colloidal nanoparticles produced by PLAL method, in comparison with chemical techniques, the performance of nanoparticles is much better in different applications. The PLAL technique supports fabrication of various nanomaterials with controlled size distribution and different physical/chemical properties.

Mechanism of nanoparticle formation in PLAL method includes the nucleation during plasma plume, cooling and subsequent growth mechanism followed by nuclei growth and coalescence [7]. In PLAL method, the key issue for obtaining nanoparticles with specific size properties is to control the plasma thermodynamic properties. Practically, interpretation of nanoparticle size properties on the basis of fundamental interaction processes such as formation of cavitation bubble and expansion of plasma plume is very difficult. In this synthesis method, the size properties of prepared nanoparticles strongly depend on the type of material, the laser parameters, and the liquid medium. In most of the experiments, nanoparticle size properties have been investigated as a function of laser fluence, pulse duration and laser wavelength [8–10]. Some other parameters such as liquid depth above target and number of laser pulses also play significant roles in the laser ablation process and therefore size properties of produced nanoparticles.

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In recent years, the importance of aluminum and titanium based nanomaterials have extremely increased due to their noticeable physical and chemical properties in a wide range of applications such as catalysis, coating, thermal protections and solar cell [11–14]. In PLAL process, active metals such as titanium and aluminum usually react with the surrounding liquid molecules and based on the liquid chemical composition form their compound oxide, hydroxide and other molecules on their surface. Consequently, PLAL of these active metal targets generates their compound nanoparticles. For instance, 1064 nm PLAL of titanium and aluminum solid targets in water results in production of their oxide-based nanoparticles [15,16].

In spite of the fact that various metallic colloidal nanoparticles have been synthesized by PLAL, synthesis of aluminum and titanium based nanoparticles have been rarely reported and only limited number of articles have studied their size properties [17–20]. Effects of water depth above target and number of laser pulses on size properties of colloidal nanoparticles have been investigated by a few numbers of authors [21,22]. For example, it was shown that particle size decreases by increasing water depth [21]. Furthermore, the other reports showed that an increase in the number of laser pulses results in finer nanoparticles [23,24].

To our best knowledge, investigation on size properties of the titanium based nanoparticles produced by nanosecond laser ablation in various water depths (in the range of a few millimeters) have not been reported yet. Additionally, the effect of the number of laser pulses (specifically, within the range of a few thousand to tens of thousands) on mean size and size distribution of colloidal titanium and aluminum based nanoparticles have not been studied. In this paper, we extensively investigated the size properties of two colloidal metal nanoparticles produced by PLAL. Particularly, the investigations were focused on mean size and size distribution of colloidal aluminum and titanium based nanoparticles prepared by nanosecond laser ablation with various pulse numbers and different depths of water.

## 2. Experiment and characterization method

### 2.1. Materials and synthesis methods

The experimental setup of PLAL is shown in Fig. 1. Aluminum (Al 5052) and titanium metal plates both with dimensions of  $25 \times 25 \times 2.5 \text{ mm}^3$  were used as targets for PLAL experiments. The measured reflectance (at  $\lambda = 1.06 \mu\text{m}$ ) of the surface of the above mentioned targets at the interface with the water medium was 0.95 and 0.56, respectively. The targets were cleaned with acetone and ethanol before irradiation. A Nd:YAG pulsed laser

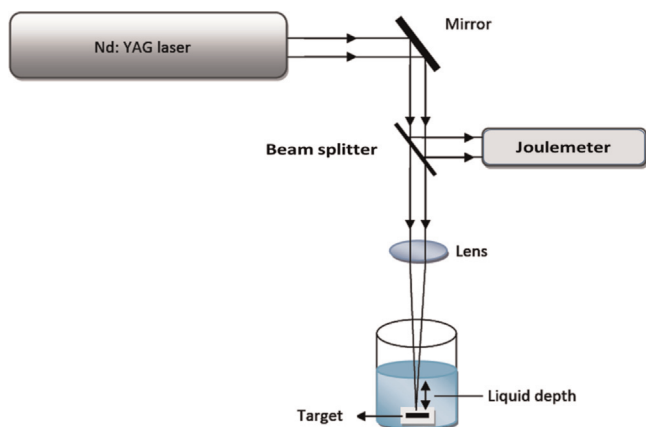


Fig. 1. Schematic experimental setup.

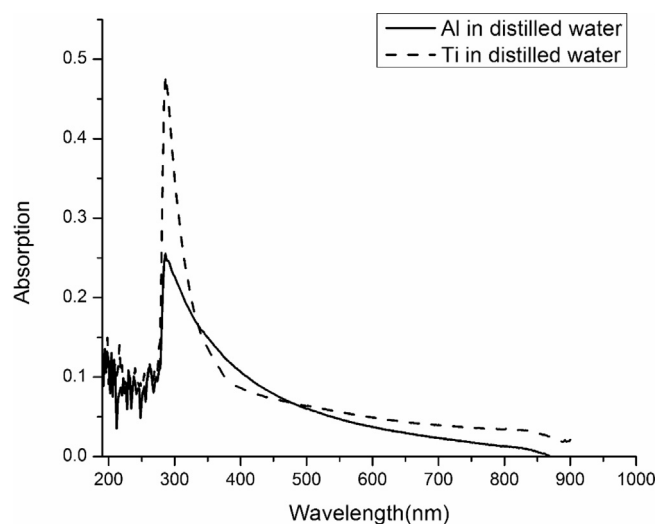


Fig. 2. UV-vis absorption spectra of aluminum and titanium based colloidal nanoparticles prepared by PLAL of aluminum and titanium in distilled water.

(Model EKSPLA Co.,  $\lambda = 1064 \text{ nm}$ , pulse width  $\sim 10 \text{ ns}$ , repetition rate = 10 Hz) was used to irradiate the targets. In each laser shot, a small fraction of the beam was sent to a pyro-electric joulemeter for measuring the pulse energy. The target was placed at the bottom of a glass vessel with dimensions of  $5 \times 3 \times 5 \text{ cm}^3$  filled with 15 ml of distilled water. The vessel was fixed on an  $x$ - $y$  motion stage (micro-positioner) for necessary displacements. Such arrangement provides us the necessary movement of the target for irradiating in a fresh region. It must be noted that in these experiments, after 1500 pulses, the location of laser beam spot on the target was displaced by the micro-positioner. In order to have a specific water depth, appropriate numbers of glass plates were positioned underneath the target to move it up or down in the water cell. For each specific case (that the target was placed at specific water depth) the focusing lens was readjusted to provide focused beam at the target surface. The laser beam was focused onto the target by using a doublet lens with focal length 18 cm. The beam spot diameter at the focus on the target surface was found to be  $\sim 150 \mu\text{m}$ . In these experiments the targets were irradiated with a fix laser fluence of  $46 \text{ J/cm}^2$ . This laser fluence could provide a good concentration of nanoparticles in water which was suitable for the characterization process.

Two separate experiments were performed in order to investigate the effects of two issues on the size properties of the produced nanoparticles: (i) the position of the target with respect to the water surface (water depth above target), and (ii) the number of laser pulses. In the first experiments, aluminum and titanium targets were irradiated with 12,000 laser pulses (total time 20 min) in four different water depths within a range between 4.4 and 10 mm. In fact laser ablation of aluminum and titanium targets was performed in water depths of 4.4–8.4 mm and 6.7–10 mm, respectively. It must be noted that the ranges of water depths chosen for aluminum and titanium targets were different because of the water splashing effect during the irradiation. In fact, for a specific metal target at a specific water depth, the splashing occurs when the laser intensity at the target surface is larger than a certain threshold (we call it threshold intensity). The difference in the ranges of water depths for aluminum and titanium was mainly due to the different splashing thresholds intensities for these two metals. The splashing of liquid at the surface ejects the water liquid towards the focusing lens disturbing the irradiation process. To avoid of water splashing disturbances, a suitable lid with an aperture (for the laser beam entrance) was used on the top of water cell.

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