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Original Article

The effect of laser pulse energy on ZnO nanoparticles formation by liquid phase pulsed laser ablation

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ABSTRACT

Zinc Oxide nanoparticles were prepared using pulsed laser ablation process from a pure zinc metal placed inside a liquid environment. The latter is composed of acetyltrimethylammonium bromide (CTAB) of 10^{-3} molarity and distilled water. A Ti:Sapphire laser of 800 nm wavelength, 1 kHz pulse repetition rate, 130 fs pulse duration is used at three values of pulse energies of 0.05 mJ, 1.11 mJ and 1.15 mJ. The evaluation of the optical properties for the obtained suspension was applied through ultraviolet–visible absorption spectroscopy test (UV/VIS). The result showed peak wavelengths at 210 nm, 211 nm and 213 nm for the three used pulse energies 0.05 mJ, 1.11 mJ and 1.15 mJ respectively. This indicates a blue shift, which means smaller sizes of prepared nanoparticles, correlated with the decrease in laser energy. The blue shift in the absorption edge refers to the quantum confinement property for the produced nanoparticles. In addition, Fourier Transform Infrared Spectroscopy (FTIR) analysis was utilized to confirm zinc oxide nanoparticles formation represented by the absorption values at 435–445 cm^{-1}. The shape and morphology of ZnO nanoparticles were characterized with scanning electron microscope (SEM).

1. Introduction

Due to their size-dependent features, nanoparticles preparations and their technological applications have promising fields in science and technology in term of unpredictable properties and behavior [1–4]. Peculiar and magnificent changes occur in the physical, chemical, optical, electrical, as well as magnetic properties when materials sizes are reduced to the nanoscale [5–7]. Many methods have been concerning the synthesis of nanoparticles such as chemical and physical vapor deposition, wet chemistry, sol-gel, and flame synthesis [8–11]. These methods were developed to meet the desired composition, size distribution, and form for generated nanoparticles. Recently, laser ablation of materials has shown itself as the most common and efficient method for that purpose. This is related to its simplicity, effectiveness, effortless experimental setup, high purity represented by freeness from surface

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contaminations, and minimum required chemical species for production [12,13]. The process of generating nanoparticles inside an aqueous environment, through laser ablation, is called "Liquid phase pulsed laser ablation (LP-PLA)". This route is one of the most efficient methods where the nanoparticles generated through laser ablation when the laser beam is focused on a solid target surface immersed in a liquid environment [14].

In LP-PLA, many naturally occurred phases are essential to produce novel nanoparticles. As a rule, it is correlated with choosing the suitable laser beam, laser working parameters, the solid targets, and the liquid environment. Focusing a high power laser beam on a fully immersed solid target material in a very short time scale results instantaneous and rapid increase in the surface temperature and pressure [15,16]. As a result, part of the metal surface vaporizes and expands forming a plume. Many species such as atoms, ions and clusters rush out towards the liquid in a high kinetic energy reacting with the surrounding liquid molecules forming new compounds [14,17].

Metallic oxides nanoparticles have been employed in plenty of applications in the fields of biomedical engineering, optoelectronics, nonlinear optics and chemical catalysts. Zinc oxide nanostructures are nanomaterials found its way in many optical and electronic applications and development of many optoelectronic devices due to their broadband gap of 3.37 eV and uncommon exaction binding energy at normal ambient temperature [18–21]. Furthermore, due to their unique optical, thermal and electrical properties, zinc oxide nanostructures are also utilized in many advanced applications such as bio sensing, bio imaging and as drug delivery [22].

According to literature, a number of researchers have proposed the mechanism of nanoparticles preparation using pulsed laser in aqueous solutions [23]. Sasaki et al. [24] have produced titanium oxide in purified water and SDS solution, they found that the crystallinity of nanoparticles is highly dependent on the SDS concentration in the solution. Liu et al. [25] generate Ti oxide and Ni oxide nanoparticles using a high power CW laser through ablation of solid targets inside aqueous environment, producing a material which is characterized in term of chemical composition, particles size and distribution as well as morphology. Ganjali et al. [26] have employed a pulsed fiber laser in a LP-PLA process to synthesize Ni nanoparticles from Ni powder inside a 30 mL of acetone, yielding spherical shapes of 10 nm diameter with a narrow size distribution. Darwish et al. [27] used a Q-switched nanosecond laser to produce different shapes of cadmium sulfide nanostructures in liquid of sulfur immersed in cadmium chloride, which was carried out by two modes, the top-down, and bottom-up. Al-Nassar et al. [28] produced zinc oxide nanoparticles by focusing a pulsed laser on a Zn solid target immersed in aqueous solution of distilled water and CTAB. In their experiment, they studied the effect of the depth of the target immersed in the liquid.

This work is devoted for the preparation of zinc oxide (ZnO) nanoparticles from a zinc solid plate immersed in an aqueous solution of CTAB using a femtosecond Ti:sapphire laser. The objective is to study the effect of pulse energy variation on the formed nanoparticles in term of size and stability.

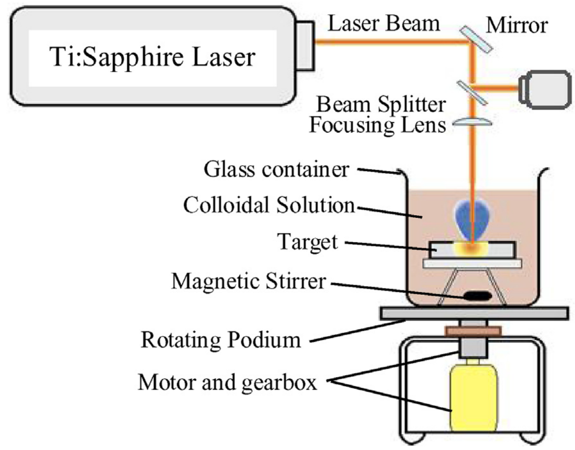

Fig. 1 – Schematic diagram for the experimental setup.

2. Experimental procedure

The typical setup of LP-PLA process is illustrated in Fig. 1. Zinc oxide nanoparticles were prepared by pulsed laser ablation from a high purity zinc plate (Fello Co., Inc.; 99.9%) in a liquid environment. The zinc plate is immersed beneath the liquid surface (8 mm) inside a cylindrical glass container filled with 10 mL solution of distilled water and CTAB. The role of CTBA is to prevent nanoparticles agglomeration and control the particles size [29]. To remove the organic impurities and remnants, the solid zinc substrate is cleaned with acetone followed with ethanol to remove the acetone with its redeposits contaminants. Later, the solid zinc is cleaned by ultrasonic cleaning (ultrasonic apparatus type EMAG 50 HC).

Laser ablation was carried out with Ti:Sapphire pulsed laser (type Quadronix IntenC) of 800 nm wavelength. A series of intense short pulses focused via a convex lens of 100 mm focal length on a small spot of about 13 μm on the solid target for a period of 10 min. The process was accomplished with operating parameters of 130 fs pulse duration, 2.5 mJ/pulse laser energy and 1 kHz pulse repetition rate. To investigate the effects of pulse energy on the product varieties, the ablation process was carried out with three different energies 0.05 mJ, 1.11 mJ and 1.15 mJ. The laser output was measured with a power meter type Newport 841-PE. The glass container is placed over a rotating podium to avert laser trapping inside the produced cavities due to target ablation. The generated plasma and the produced nanoparticle over the interaction zone on the target may partially prevent the laser beam from the reaching the target and alter the interaction [29]. Therefore, a magnetic stirrer, which rotates at 600 rpm, was placed inside the container. This ensures a steady interaction of laser with the metal target during the process through circulating and homogenizing the colloidal solution.

Characterizing the produced zinc oxide nanoparticles passes through some suitable tests, studying the optical absorption/transmission properties of the dispersed nanoparticle in the colloidal solution was done via a spectrophotometer type Varian Cary-50 with a device of 1 cm optical path cell. Through this test the UV/VIS extinction spectrum for the colloidal solution was obtained. FTIR spec-

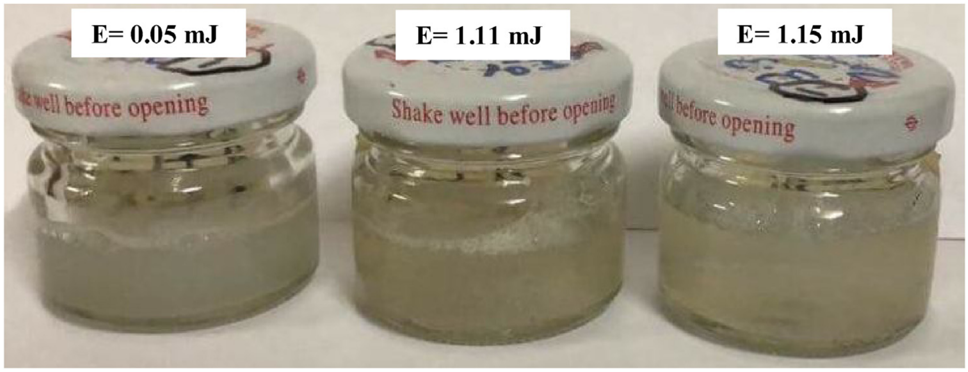

Fig. 2 – Color change in the solution of ZnO nanoparticles due to pulse energy variation.

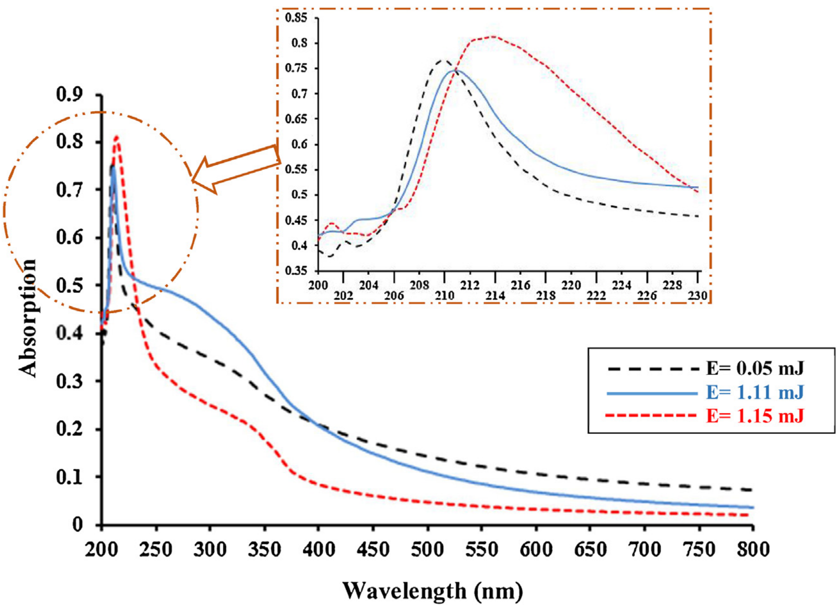

Fig. 3 – UV-Visible absorption spectra of ZnO nanoparticles prepared in different laser energy (λ = 800 nm, τ = 130 fs and PRR = 1 kHz).

troscopy device (type PerkinElmer Spectrum 100 Series FT-IR spectrometer) was used to obtain the adsorption of organic species on the ZnO nanoparticles. The FTIR spectra is measured at room temperature with the spectrometer using the KBr Pellet technique in the wavenumbers range 400–4000 cm^{-1} [30]. Inferring the chemical bonding of the produced nanoparticles is done through the processes of lyophilizing, gently mixing with KBr powder (300 mg) and compressing with a pressure of 40 MPa for a duration of 5 min in form of discs. Moreover, taken SEM images were aided to clarify the shape and morphology of the produced nanoparticles.

3. Results and discussion

The interaction of intense laser pulses with the solid zinc target in the CTBA environment causes target ablation and plasma plume expanding. The liquid suppresses plume expansion resulting in high pressure and temperature which is indicated by the liquid splashing during the process [31]. Vaporization of the solid target and liquid leads to grey colloidal solution formation which is clearly visible to the naked eye as seen in Fig. 2. Color changes give an indication for the stuck nanoparticles with the liquid. The shapes, sizes and distribution of zinc oxide nanoparticles were characterized through the UV/VIS, FTIR and SEM analyses.

Materials may have peculiar electronic and optical properties when their nanoparticles sizes are near or below their exciting Bohr radius, this is related to the quantum confinement phenomena [32]. Observation of the optical properties of quantum size produced nanoparticles can be done efficiently with the UV/VIS absorption spectroscopy [33]. Fig. 3 displays the absorption spectra of the produced nanoparticles for the three values of pulse energies. As can be seen, the absorption spectra have peaks centered at 210 nm, 211 nm and 213 nm for the pulse energies 0.05 mJ, 1.11 mJ and 1.15 Mj, respectively. This indicates the production of zinc oxide nanoparticles inside the solution.

The Plasmon band of the solution moves towards shorter wavelengths (blue shift) with decreasing the pulse energy and that reveals more nanoparticles formation. Shifting towards

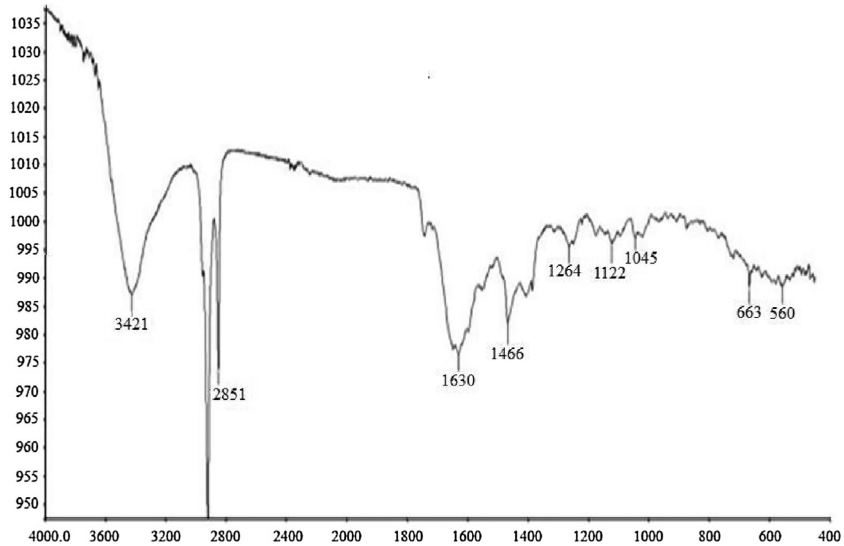

Fig. 4 – FTIR spectra of ZnO nanoparticles prepared at different laser energies (λ = 800 nm, τ = 130 fs and PRR = 1 kHz).

Table 1 – Laser pulse energy versus absorption peak.

Sample	Pulse energy(mJ)	Absorption peak wavelength (nm)
1	0.05	210
2	1.11	211
3	1.15	213

shorter wavelength gives an indication for smaller nanoparticles formation due to the quantum confinement effect [34–37]. The absorption values rely on the concentration of produced nanoparticles, the higher concentration results in a greater absorption peak [32,38]. The results show a higher absorption peak amplitude for greater pulse energy due to the higher concentration of the produced nanoparticles inside the solution.

The effect of pulse energy variation on the absorption peaks is obtained from the UV-VIS absorption test for three samples as clarified in Table 1. As shown, the higher the laser pulse energy the more shifting towards the blue in the absorption peaks which indicates producing tinier sizes of nanoparticles.

Fourier transform infrared (FTIR) spectra of produced ZnO nanoparticles were recorded at room temperature on a FTIR spectrometer with KBr reference in the range of 4000–400 cm^{-1}. FTIR measurement is essential to confirm the formation of crystalline ZnO nanocrystals, the adsorbed other particles on the surface of nanoparticles as well as the bonding between Zn-O atoms or molecules. Fig. 4 presents the FTIR spectra for the produced nanoparticles in the absence and presence of surfactants. The analysis reveals the formation of different species and bonding for different absorption bands or single values.

The absorption bands and values indicate the formation of zinc oxide nanoparticles, arising O—O bond, O—H stretching vibration from ZnOH species and O—H stretching vibration from C—H species as listed in Table 2. The reaction of zinc oxide nanoparticles and hydroxyl group results the free O—H stretching bond, which revealed from 3408 cm^{-1} absorption peak value, these results are consistent with those reported in the literature [19,22].

Fig. 5 shows the SEM images for ZnO based nanoparticles produced by ablation of zinc in CTAB of 10^{-3} molarity and distilled water at pulse energies 0.05 mJ, 1.11 mJ and 1.15 mJ. The size of particles varies in a range between few tens of nanometers to hundreds of nanometer. The images reveal that the produced nanoparticles are almost homogeneous and spherical in shape. The interaction of the laser pulses with the solid zinc in different pulse energies may change the particles sizes, shapes and distribution. When a low pulse energy of 0.05 mJ is used the particle size tends to decrease due to the ablation accompanied with light removal of the upper surface layer without any indication of melting effects as seen clearly in Fig. 5a. As the pulse energy increases, the volume of particle increases due to higher amount of optical energy deposition on the solid surface.

The extreme increase in temperature, plasma density as well as the exposed area of the solid metal causes the increase the removed particle size as well as population. The continuous heating for the plasma plume by the series of pulses

Table 2 – Absorption Bands or Peaks values for the FTIR analysis.

Absorption rangeor value (cm^{-1})	Absorption Indication
435 to 445	Presence of zinc oxides nanoparticles
1040 to 1070	The probability of O-O bonds arising
3408	Attributed to O—H stretching vibration from ZnOH species
2924	Attributed to O—H stretching vibration from C—H species

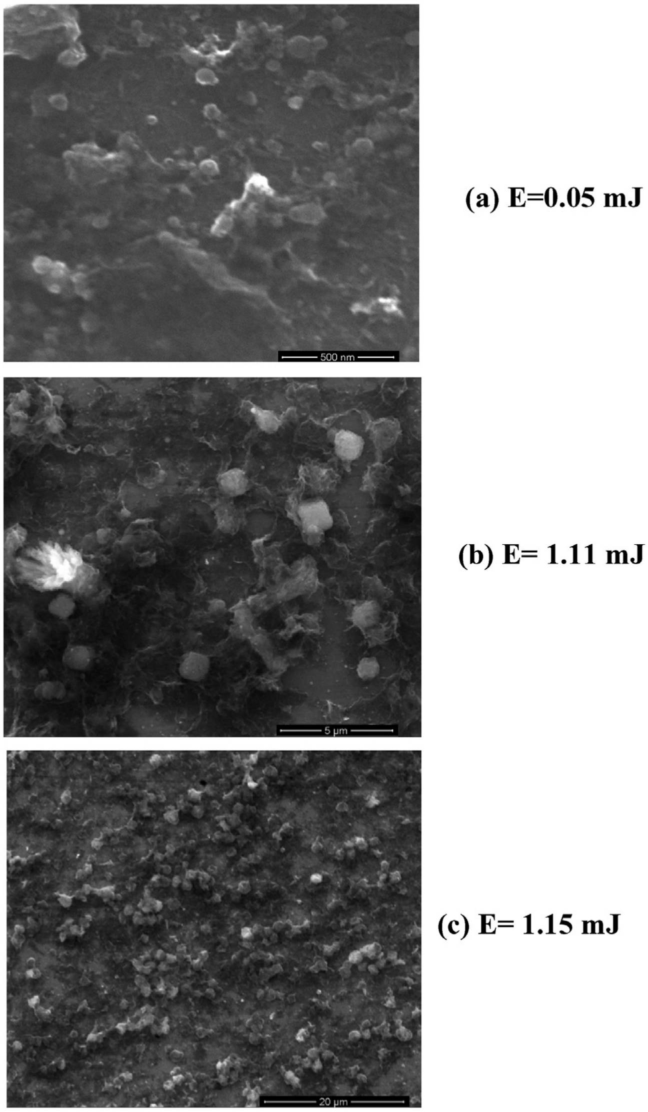

Fig. 5 – SEM images for the prepared ZnO nanoparticles in different laser energies ($\lambda = 800$ nm, $\tau = 130$ fs and PRR = 1 kHz).

maintain its extreme temperature which in turn contribute in ablation process. Fig. 5b and c show larger particles whose walls contain traces of molten material formation when higher pulse energies used, it is clear that a high percent of ZnO based nanoparticles are agglomerated [39].

4. Conclusions

Synthesis of Zinc oxide nanoparticles by pulsed laser ablation method in liquid environment was investigated. According to the obtained results, the following remarks were concluded:

1 In the presence of CTAB, the decrease in the laser pulse energy yielded smaller size of nanoparticles with narrow size distributions which is indicated by the blue shift indicated through UV-Visible absorption spectra test.
2 Increasing the value of pulse energy leads formation of larger particle sizes, more spherical, homogeneous and broad size distribution.
3 Confirmation of zinc oxide nanoparticles formation can be represented by the absorption values at 435–445 cm^{-1} as the FTIR test showed.
4 Employing higher pulse energy in ZnO nanoparticles synthesis leaded to an increase in the particle size and concentration inside the solution. This may encourage the aggregation and agglomeration resulted in spindle-like ZnO aggregates composed of many well-defined nanoparticles.

Conflicts of interest

The authors declare no conflicts of interest.

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