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Production of Zirconium Oxide (ZrO_2) Micro and Nanoparticles by Pulsed Laser Ablation in Liquid

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Abstract

Pulsed laser ablation in liquid (PLAL) has become a technique of growing importance for the processing of micro and nano particles of metals and metal oxides. In comparison with different physical and chemical techniques, this approach has many advantages. This work was dedicated to the production of zirconium oxide (ZrO_2) micro and nano particles from a solid zirconium target submerged in distilled water using PLAL technique to study the effect of number of pulses on the structural and optical properties of the produced colloidal solution. Many kinds of tests such as Fourier transform infrared (FT-IR), UV-visible (UV-Vis.) and field emission scanning electron microscopy (FE-SEM) have been used to characterize the products. The (UV-Vis.) absorption spectra of all colloidal samples have shown broad peak at wavelength of 289 nm. The functional groups of (ZrO_2) in liquid media were also determined by Fourier transform infrared (FT-IR) analysis. Field emission scanning electron microscope (FE-SEM) images verified the micro and nanostructure of produced materials.

Keywords: Zirconium oxide, Pulsed Laser Ablation in Liquid, Nanoparticles, Microparticles.

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إنتاج الجسيمات المايكروية والنانوية لأوكسيد الزركونيوم بطريقة الاستئصال بالليزر النبضي في السائل

هاله جاسم فيحان، جاسم محمد منصور و عمار عايش حبيب

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الخلاصة

أصبحت تقنية الاستئصال بالليزر النبضي في السوائل (PLAL) ذات أهمية متزايدة لمعالجة الجسيمات المايكروية والنانوية للمعادن وأكاسيدها. تعد هذه التقنية ذات ميزات مهمة بالمقارنة مع التقنيات الكيميائية والفيزيائية الأخرى. تم تخصيص هذا العمل لإنتاج اوكسيد الزركونيوم (ZrO_2) كجسيمات مايكروية ونانوية التركيب وباستعمال الزركونيوم الصلب داخل الماء المقطر، إذ تم دراسة تأثير عدد النبضات على الخصائص التركيبية والبصرية للمحلول الغروي الناتج. استخدمت عدد من التقنيات للتشخيص مثل تحويلات فورير للأشعة تحت الحمراء (FT-IR)، ومطيافية الاشعة المرئية-فوق بنفسجية (UV-Vis)، وفحوصات المجهر الالكتروني الماسح الباعث للمجال (FE-SEM) أظهرت نتائج طيف الامتصاصية للعينات وجود ذروة عريضة عند الطول الموجي (289 nm). تم تحديد المجاميع الوظيفية لأوكسيد الزركونيوم في الوسط السائل باستعمال تقنية (FT-IR) وكذلك بينت صور المجهر الالكتروني الماسح الباعث للمجال وجود الجسيمات النانوية والمايكروية.

كلمات مفتاحية: أوكسيد الزركونيوم، الاستئصال بالليزر النبضي في السائل، الجسيمات النانوية، الجسيمات المايكروية.

Introduction

ZrO_2 is a white polymorphic powder whose crystal can be monoclinic, tetragonal or cubic. The monoclinic zirconia structure phase is stable at room temperature and it is formed at the temperature range of (400–1170 °C). It transforms above (1170 °C) into a tetragonal phase. The stability of tetragonal phase is up to (2370 °C), and the cubic phase of zirconia is formed at the temperature range between 2370 and 2600 °C [1]. This zirconia phase transformation affects its density as well as the physical characteristics; tetragonal and cubic structures of Zirconia, for example, have a higher densities and a higher crystallization temperatures compared to the monoclinic form [1,2]. ZrO_2 has a broad band gap of approximately (5.0 - 5.5 eV) depending on its phase (cubic, tetragonal, monoclinic or amorphous) and preparation process [3]. Micro and nano ZrO2 particles have drawn great interest of many scientists and researchers, due to their unique properties such as low thermal conductivity, efficient dielectric characteristics, strong thermal stability, chemical stability, high refractive index, high oxygen ion and conductivity, high fracture toughness, high thermal shock resistance, high hardness and mechanical strength [4-8], laser mirrors, broad band interference filters, ionic conductors, photo catalysis, sensors, coatings, waste water treatment, fuel cells and memory devices [9-16]. These fascinating properties of ZrO_2 and its various applications in different areas led to develop a specific technique (PLAL) for synthesizing this oxide and investigating which aqueous medium is ideally suited for its synthesis.

PLAL technique is a simple and efficient process for the preparation of metal micro and nanoparticles, semiconductors and oxide isolators. Without the need for high vacuum chambers, high purity and stable particles can be created. In the PLAL process, by optimizing the laser parameters, material size can be controlled [17]. It is also possible to synthesize various types of particles free of surface-active substances and counter ions using this technique [18-20]. The plasma is created to produce particles through PLAL technique as a result of very high temperatures that occur when high power laser beam irradiates the target. The resulting plasma containing the target metal vapor expands adiabatically, resulting in rapid plasma plume cooling and thus forming particles [21, 22].

The aim of this study is to prepare zirconium oxide solutions using the pulsed laser ablation technique in liquids and to study the optical and structural properties of the resulted nano/micro structured zirconium oxide solution and to investigate the surface nature of the prepared particles.

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Experimental Details

Figure (1) shows the schematic diagram of the PLAL system setup for the production of (ZrO_2) colloidal solution. Q-Switched Nd:YAG nanosecond laser is used in this work. It operates at a wavelength of (1064/ 532 nm), repetition rate of (1-6 Hz) pulse, pulse duration of (10 ns) and energy of (500-1000 mJ). This laser beam is focused on a rigid ZrO_2 target of 99.99% purity which is submerged in the ablation liquid (D.W.) inside a rotating beaker to ensure uniform irradiation on target and the movement of water that can enhance the ablated particle diffusion also to disperse the produced NPs. Ultrasonic cleaning system was used to clean the zirconium oxide target before starting the experiment then washed with ethanol and acetone. The experiments of zirconium oxide particles production were done at (500, 1000, 1500, and 2000) pulses, (1 Hz) repetition rate, (10 ns) pulse duration and (500 mJ) energy. In the experiments of this research work, laser ablation of the zirconium target in D.W. is accompanied with the production of a plasma plume visible to the eye near the target surface and a milky colloidal solution of micro and nano ZrO_2 is obtained after laser irradiation.

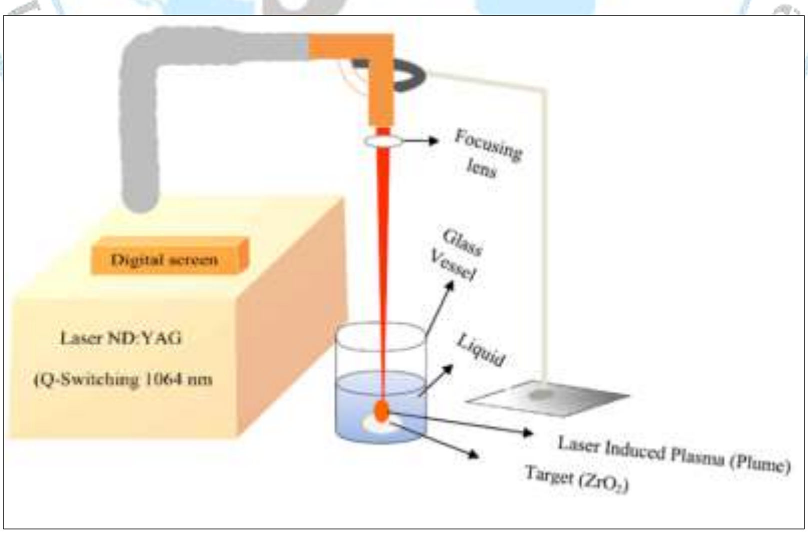

Figure 1: Schematic diagram of the Pulsed Laser Ablation in Liquid

Results and Discussion

PLAL technique was applied to produce ultrafine particles of zirconium oxide in distilled water. The structure and morphology of the particles obtained by pulsed laser ablation technique were studied using FE-SEM and FT-IR, and the optical properties were investigated by using UV-Vis. spectroscopy.

Results and Discussion

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UV-Vis. Spectroscopic Analysis

The absorption spectra of ZrO_2colloidal are shown in Figure (2). Clear peaks for all samples can be observed at wavelength of 289 nm which is due to the electronic transition from valance to conduction bands, which is recorded previously for metal oxide nanoparticles [23]. The produced colloidal solutions seem to be stable, and even after two weeks no precipitations were found at the bottom of the containers as recorded by other reports [24]. Several studies have shown that oxygen vacancies play a significant role in the stability of nano-crystalline cubic and tetragonal zirconium [25]. The spectra also show a broadband with a long tail towards higher wavelengths, suggesting that the produced particles are not homogeneous in size [25, 26]. The results of the absorption spectra of zirconium oxide particles suspended in the liquid showed that with the increase in the number of laser pulses the absorbance increases where the lowest absorbance is obtained for the prepared sample with 250 pulses and the highest absorbance is obtained for the prepared sample with 2000 pulses.

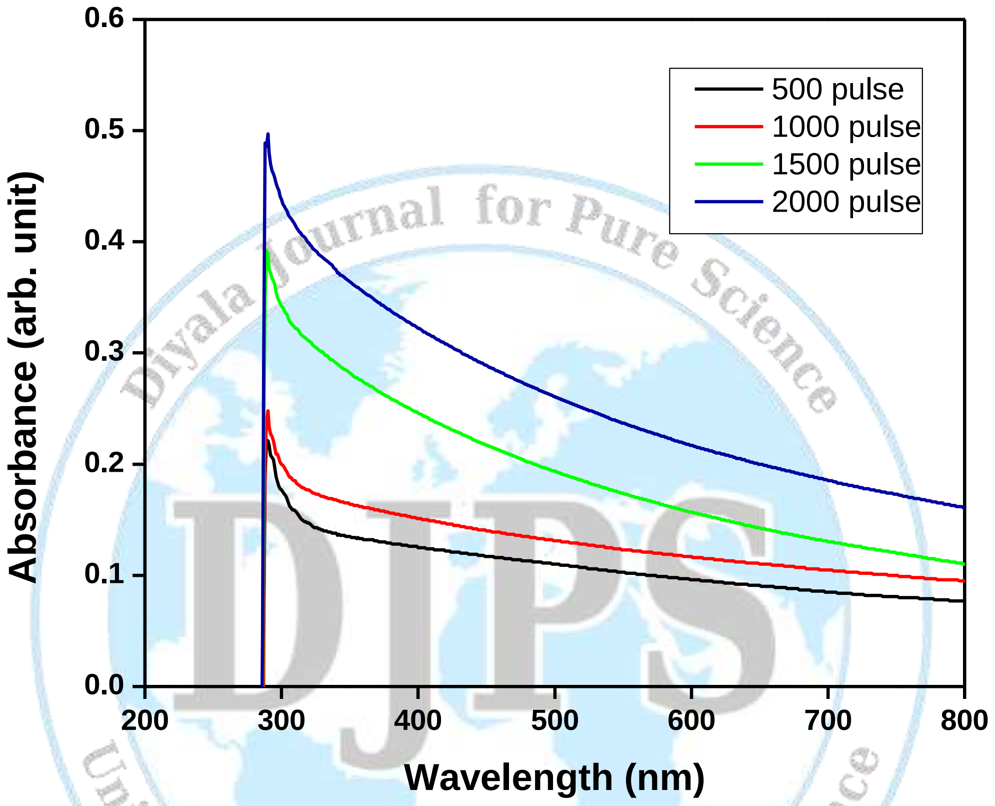

Figure 2: UV-Vis absorption spectra of ZrO_2 particles prepared in D.W. (λ = 1064 nm) (E = 500 mJ) and with different number of pulses

FT-IR Spectroscopic Analysis

Figure (3) displays the FT-IR spectra of ZrO_2 samples that were performed to approve the molecular bonding and the existence of functional groups. It can be noticed that the samples displayed transmittance in the (400-800 cm^{-1}) region showing (Zr-O) stretching band and thus assuring the production of ZrO_2 particles [27, 28]. The broad absorption band observed in the (3000-3600 cm^{-1}) region and some well-defined bands between (1300-1750 cm^{-1}) correspond to the vibration of (O-H) bond mode as a result of that the synthesized (ZrO_2) particles adsorbed water molecules or hydroxyl groups on the surface [29-31]. The hydroxylic group of moisture can also be assigned to these absorption peaks around (3000-3600 cm^{-1}). The band appeared at (~1000 cm^{-1}) is a characteristic of peroxide (O – O) groups [27, 28].

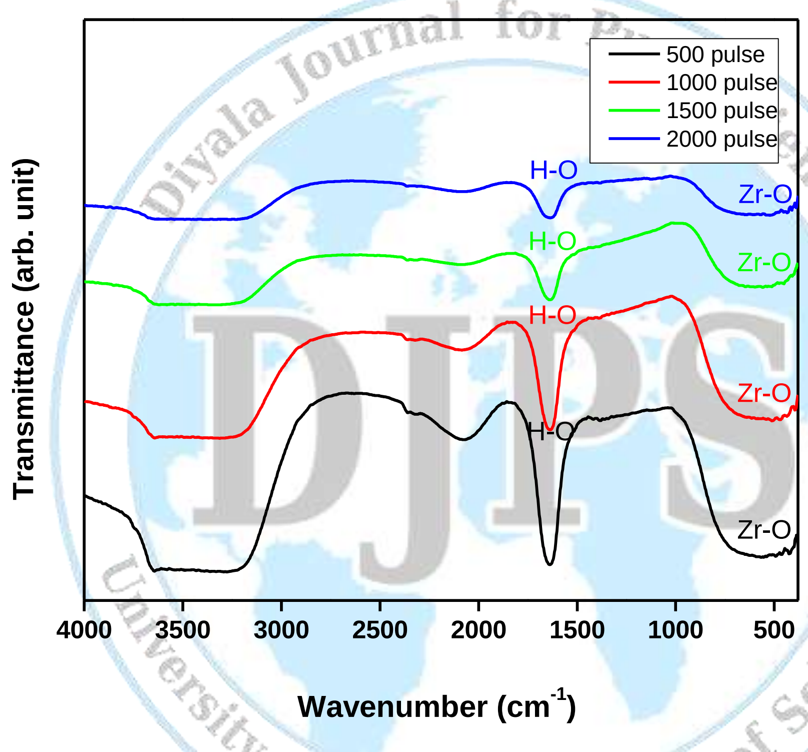

Figure 3: FT-IR spectra of ZrO_2 particles prepared in D.W. (λ = 1064 nm) (E = 500 mJ) and with different number of pulses

Morphological studies

The morphological structure of the zirconium oxide solution was observed with field emission scanning electron microscope (FE-SEM). Figures (4) and (5) show the FE-SEM images of zirconium oxide ZrO_2 which confirm that our produced material is in micro and nano-scale. The FE-SEM image and size distribution of ZrO_2 nanoparticles show a mixture of different sizes and shapes where the particles are spherical or irregular. The calculated average size diameters of ZrO_2 nanoparticles are about (53.47-57.78 nm). Figures (4 and 5) show the (FE-SEM) images and volume distribution of the resulting zirconium oxide solution using the pulsed laser ablation technique in distilled water with 1064 nm wavelength and 500 mJ energy using 1000 and 2000 pulses respectively with a repetition rate of 1Hz.

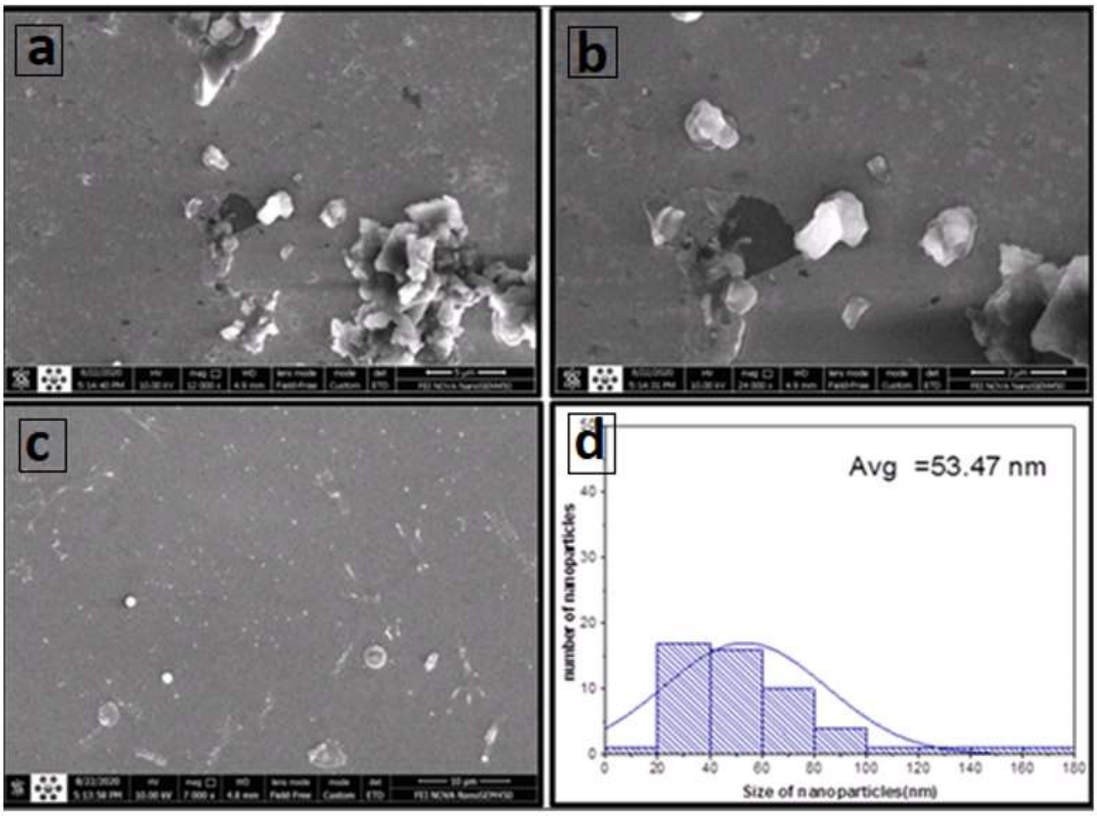

Figure (4): FE- SEM images and volume distribution of ZrO_2 particles prepared in D.W. (λ = 1064 nm) (E = 500 mJ), (Number of Pulses =1000 pulses), (a) scale (1 µm), mag. (12K x), (b) scale (3 µm), mag. (24K x), (c) scale (10 µm), mag. (7K x), (d) Avg. (53.47 nm).

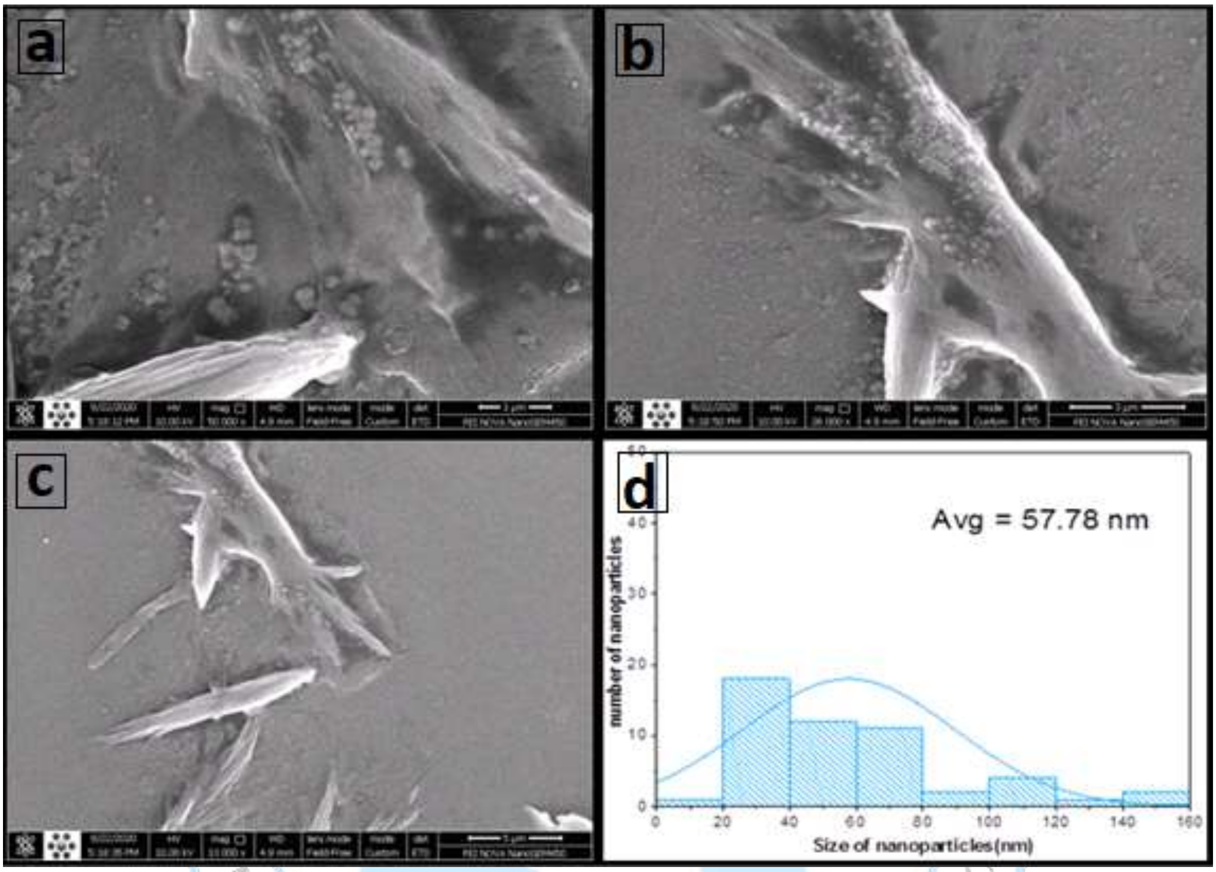

Figure 5: FE- SEM images and volume distribution of ZrO_2 particles prepared in D.W. (λ = 1064 nm) (E = 500 mJ), (Number of Pulses = 2000).), (a) scale (1 µm), mag. (50K x), (b) scale (3 µm), mag. (26K x), (c) scale (5 µm), mag. (13K x), (d) Avg. (57.78 nm).

Conclusion

This research work has successfully produced pure zirconium oxide particles (ZrO_2) by using a simple method of nanosecond PLAL technique.

The optical absorption of colloidal ZrO_2 in D.W. solution reveals a broad band with a long tail toward the (271 nm) wavelength indicating the formation of inhomogeneous sizes and a particle coagulation shift in wavelength of maximum optical extinction. The FT-IR spectra confirm the formation of micro and nanoparticles (ZrO_2) as well as the molecular nature and functional groups for micro and nanoparticles of (ZrO_2). The morphology of the synthesized NPs was studied using FE-SEM analysis which confirms that the ZrO_2 micro and nanoparticles produced in distilled water have spherical and irregular shapes and their size is in the range of ~10-160 nm.

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