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Spectroscopic Study of MgO Nanoparticles Prepared by Pulse Laser Ablation in Liquid and Chemical Methods

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ABSTRACT

MgO NPs were synthesized by pulse laser ablation in liquid and chemical methods and analysis by X-ray diffraction (XRD), scanning electron microscopy (SEM) and atomic force microscopy (AFM) techniques. Absorbance and Fluorescence spectra of the produced nanoparticles was measured by U.V.-Vis. and Fluorescence spectrophotometers

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INTRODUCTION

The most efficient physical methods for nanofabrication is laser ablation, it is a typical example of top-down approach in fabrication of nanoparticles, laser ablation is the process of removing material from a solid surface by irradiating it with laser beam (S.Barcikowski *et al*, 2007)

The laser ablation of material from target leads to form nanoclusters either in vacuum by deposition on substrate yielding to the formation of nanostructure film or in liquid when the nanoclusters can be released into the liquid forming a colloidal nanoparticles solution, also the difference occurs between these processes when plasma begins to expand ,which occurs freely in vacuum but is confined by liquid layer, the liquid delays the expansions of the plasma leading to high plasma pressure and temperature, which allows to formation of novel materials (R. M.Tilaki *et al*, 2007).

The more effective collection of synthesized particles can be achieved by laser ablation in liquid environment. The liquid not only confines the ablated species at the liquid-solid interface, but also it acts as a mediator for chemical reaction at the liquid-solid interfaces, therefore the liquid media surrounding the target plays an important role on affecting the shape, mean size, size distribution and composition of the particles (B., Geetika *et al*, 2009).

In the solid-state chemistry, materials with high specific surface area, high porosity and individual particle sizes in the 1–100 nm size range are becoming more available. Ultra-fine metal oxides have found uses as bactericides, adsorbents and specifically catalysts. Magnesium oxide is an interesting basic oxide that has many applications. For example, magnesium oxide with ultrafine, nanoscale particles and high specific surfaces area has shown great promise as destructive adsorbent for toxic chemical agents (J.V. Stark *et al*, 1969).

Magnesium oxide is obtained mainly by thermal decomposition of magnesium hydroxide or carbonate (M.A. Aramendia, *et al* ,1969, B.Q. Xu, *et al*, 2001) and recently by the sol–gel process (H.S. Choi, 2000, T. Lopez, *et al*, 1998). The oxide morphology and particle size depend on the preparation conditions (pH, gelling agent, calcinations rate and temperature).

In this paper, the (Calcination) method will be used for the purpose of obtaining MgO nanoparticles.

In spectroscopy, the fluorescence depends on the radiative transitions that are resulted by the transitions of the excited molecules from upper state to lower state, these transitions are, absorption, fluorescence and phosphorescence. The radiation life time (τFM) can be calculated using Bowen &Wokes relation as follow (Yarive A., 1975)

$$\frac{1}{\tau_{FM}} = 2.88X10^{-9} n^2(\nu'^2)\int \varepsilon(\nu')d\nu' \text{.....1}$$

where:

n: refractive index of a medium,

ύ: wave number at the maximum absorption,

∫ (ύ) dύ : the area under the absorption spectrum curve as a function of the wave number ύ.

The radiation life time relates to the fluorescence lifetime (τF) as follow (Yarive A, *et al*, 1975)

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$$\tau_{F=}\phi_F \tau_{FM} \text{ ....} 2$$

Where

ϕF: quantum yield

which can be calculated by the ratio of fluorescence spectrum area to the area of absorption spectrum as follow: (Yarive A, *et al* ,1975)

$$\phi_F = \frac{\int F(\nu')d\nu'}{\int \varepsilon(\nu')d\nu'} \text{..... ....} 3$$

Where

∫ F(ύ)d ύ:the area under the fluorescence spectrum curve as a function of the wave number ύ.

2. Method for Produce MgO Nanoparticles:

Pulsed Laser Ablation in Liquid medium (PLAL) Method:

Pulsed laser ablation in liquid media (PLAL) is a promising technique for controlling the fabrication of nanomaterial via rapid reactive quenching of ablated species at the interface between plasma and liquid. Also this technique is a versatile technique for preparing various kinds of nanoparticles materials such as noble metals, alloys, oxides and semiconductors (T. Sasaki *et al* 2006),(Wasan Mubdir Khilkala *et al* 2014).

The mechanism of the laser-induced plasma and ablation of particles from the target using a pulsed laser starts when the laser beam irradiates at the interface between the solid and the liquid through the liquid, a plasma plume is formed at the interface, and confined by the surrounding liquid during each pulse.(Maha Al-Kinany *et al.* 2014)

The confined plasma expands adiabatically at a supersonic velocity, creating a shock front that in turn induces an elevated pressure and increase of plasma temperature. Such transient pressure in front of the plasma plume impinges the ablation species of metallic ions, atoms and clusters into the confined liquid, and chemical reactions between the ablated species and the liquid occur, forming nuclei of oxides and/or hydroxides, or other compounds depending on the type of liquid. It is believed that in the case of PLAL the shock wave plays a self-limiting role for the generation of NPs and alters the efficiency of the process (D. Kim, M. Ye *et al*.1989, D. Kim, B. Oh *et al*, 2004).(Wasan Mubdir Khilkal *et al*.2014)

PLAL has been demonstrated that size of synthesized material can be controlled by changing different laser parameters such as: laser fluence, wavelength, and pulse laser duration or by changing the type of surfactant solution. The parameters of laser and the type of surfactant effect on the stability and size of produced NPs.

Calcination Method:

Calcination method can be defined as “Heating to high temperatures in air or oxygen”. Calcination is used to mean a thermal treatment process in the absence or limited supply of air or oxygen applied to ores and other solid materials to bring about a phase transition, thermal decomposition, or removal of a volatile fraction[A. D. McNaught *et al*, 1997).

3. Experimental:

Pulse Laser Ablation in Liquid Medium (PLAL) Method:

Laser ablation of magnesium plate (Mg) in aqueous media was carried out with a nanosecond Q-Switch Nd-YAG pulsed laser. The laser wavelength λ operates at ;1064nm, 6Hz repetition rate with a number of pulse (200)p and with energy of (100) mJ. Metal plate placed on the bottom of glass vessel containing 3ml SDS solution with concentration (0.01)M.

Calcination:

(12.30)g of Magnesium nitrate Mg(NO3)2.6H2O were dissolved in ethylene glycol solution (25 mL) and were put on magnetic stirrer for 1 h. (2.70)g of Na2CO3(2.70 g) was dissolved in 100mL D.W, after that take 12.5 mL from then added into the above mixture under sonication. After sonication that lasted for 15 min, the solution obtained was kept at rest for about 5 hrs. Then, it was filtered, washed using water and dried at 50 °C. Finally, the samples were obtained through calcination. The code of the sample obtained under calcination conditions was (410 °C, 5 h, 3 °C/min) (C. Vijila *et al*, 2001).

The structural measurements such as, morphological features by X-ray diffraction atomic force microscope (AFM), scanning electron microscope (SEM) and atomic force microscope (AFM) as well as optical properties by using UV-visible and Fluorescence measurements.

RESULT AND DISCUSSION

X-ray diffraction:

XRD peaks of MgO NPs prepared by PLAL and chemical methods, are shown in fig.(1.a), (1.b). Peaks at 2θ values of 42.78 and 62.07 degree corresponding to (200) and (220) planes of MgO have been observed and compared with the JCPDS, powder diffraction card MgO file No. 43–1022.The results different when MgO prepared by chemical method because different in intensity due to the different in the density of the material MgO. Increase the broadening of the FWHM in the XRD spectrum when prepared MgO NPs by PLAL because decrease grain size of MgO.

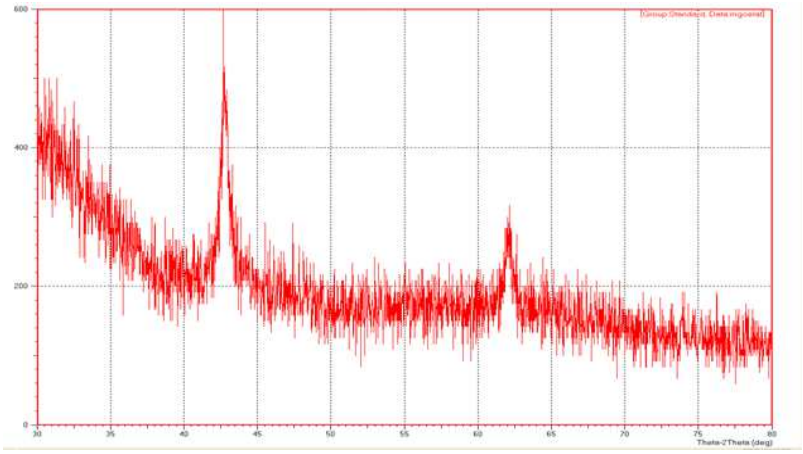


Fig. 1.a XRD of MgO NPs prepared by PLAL method

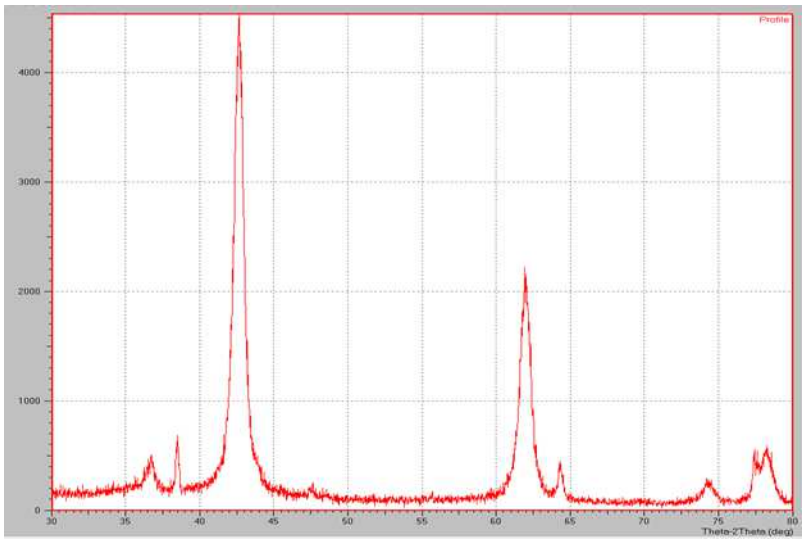


Fig. 1.b: XRD of MgO NPs prepared by chemical method

Scanning Electron Microscopy (SEM):

Fig.(2.a) shows SEM image for MgO NPs prepared by PLAL. Nanoparticles resulting in Fig.(2.a) close to the tube form. While the SEM image of the MgO NPs prepared by chemical method is small crystallites, spherical in shape within a big mass of planar aggregates, shown fig.(2.b)

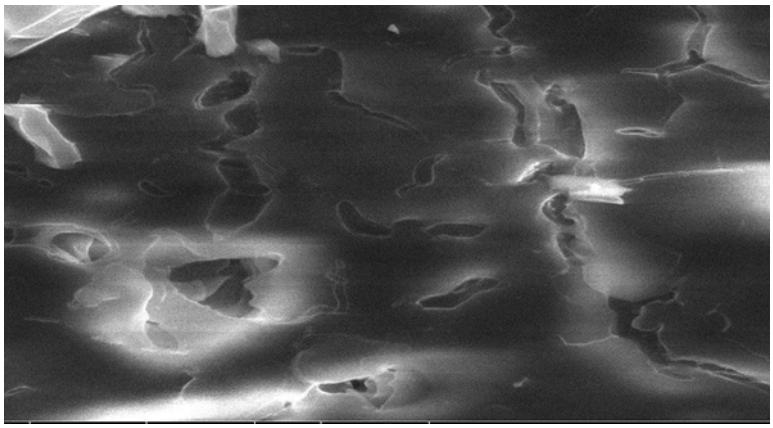


Fig. 2.a: SEM of MgO NPs prepared by PLAL method

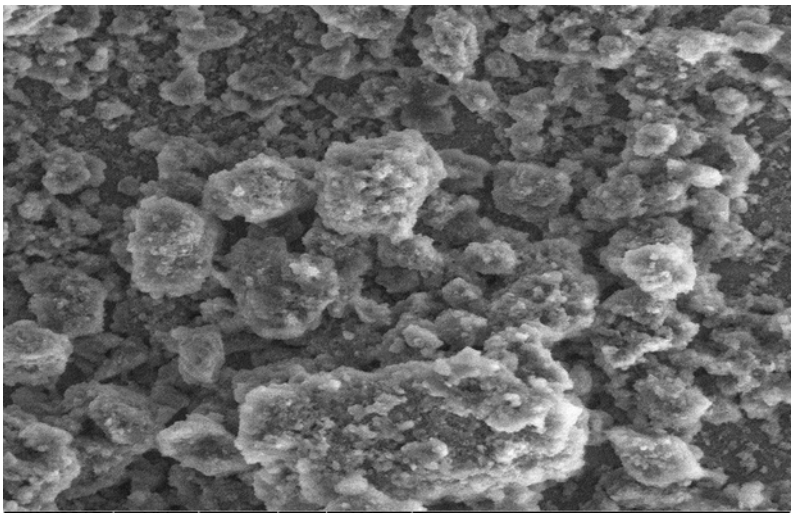


Fig. 2.b: SEM of MgO NPs prepared by chemical method

Atomic Force Microscopy (AFM):

Fig.(3.a) shows AFM images of the MgO NPs prepared by PLAL. The grain size was (50.52) nm. While the AFM image of the MgO NPs prepared by chemical method is shown in fig.(3.b).The grain size was (92.99) nm.

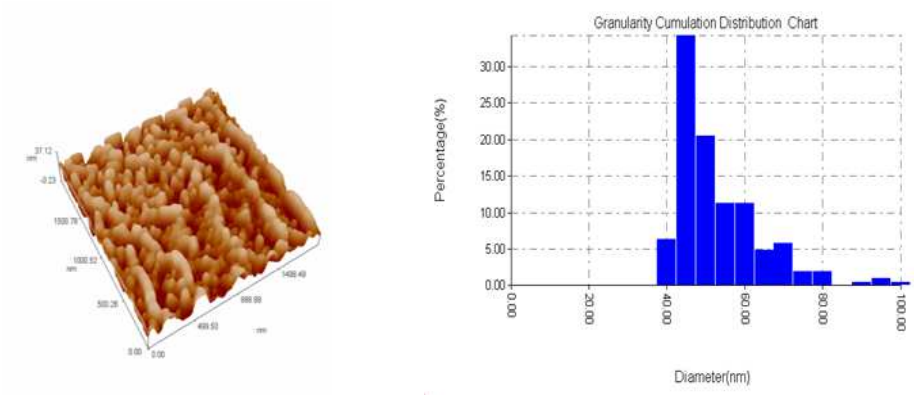


Fig. 3.a: AFM of MgO NPs prepared by PLAL method

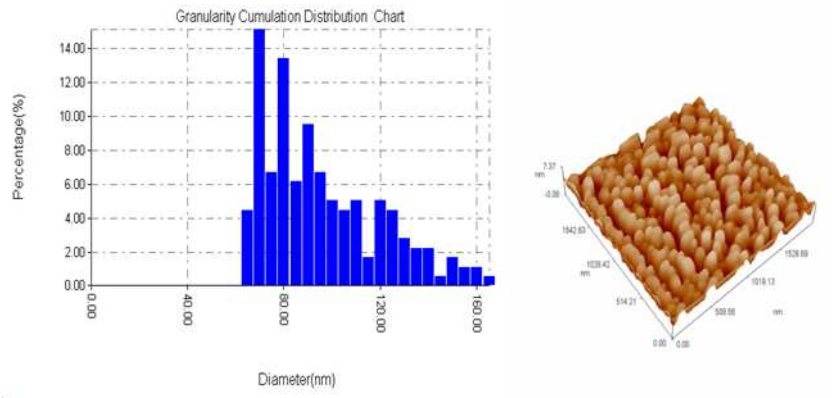


Fig. 3.b: AFM of MgO NPs prepared by chemical method

4. UV-visible and Fluorescence spectra:

Fig.4 shows the study of the absorption (U.V.-Vis.) and fluorescence spectra of MgO NPs prepared by PLAL method at number of pulse 200 pulse and concentration of solution SDS 0.01M.Where the absorption peak at (199)nm and the fluorescence peak at the wavelength (397)nm.

While the absorption peak at (226)nm and the fluorescence peak at the wavelength (396)nm when MgO NPs prepared by chemical method. Shown fig.(5).

Absorbance change as a function of wavelength in figures 4,5, through these figures a shift in wavelength red shift type is noticed. The reason for this shift is due to the change in energy levels result in effect of vibrational energy levels on molecules MgO, and also because of the increased grain size of MgO, an increase of grain size increases the shift in wavelength red shift type.

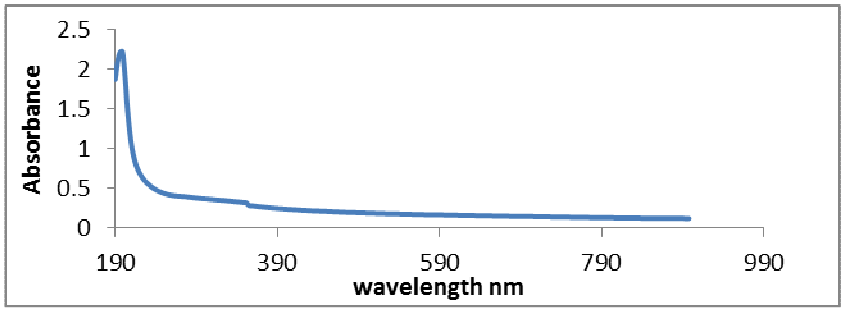
The area under the curve fluorescence spectrum is calculated by using GEUP program. The lifetime (τf) and quantum yield (φf) of fluorescence are calculated by using equations (2), (3), table (1).

Table 1: Linear optical properties of samples.

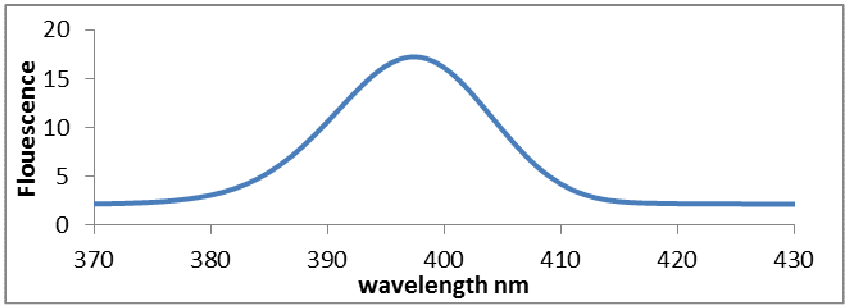
Method	Wavelength (nm)	Absorption coefficient (αo) cm^{-1}	Refractive index (n_o)	Quantum yield (φ_f)	Lifetime of fluorescence (τ_f) sec
Chemical	226	4.306061	2.15	1.238714	$1.874x10^{-5}$
PLAL	199	9.107849	1.35	1.499528	$2.063x10^{-5}$

The absorption coefficient is valued at when preparing MgO nanoparticles by pulse laser ablation method higher than valued at when preparing chemical method, but the refractive index is lower. Because of shift that happen in wavelength.

The lifetime and quantum yield of fluorescence of MgO NPs is different in both methods. When the MgO NPs prepared by PLAL the lifetime longer than if the MgO NPs prepared by chemical, As well as the quantum yield is the largest when MgO NPs prepared by PLAL. The reason for this result is due to shift the wavelength red shift, this leads to reduced space to spread molecules MgO which restricts her freedom of movement which increases the energy transfer processes radioactive rate and thus long lifetime and increases quantum yield of fluorescence.

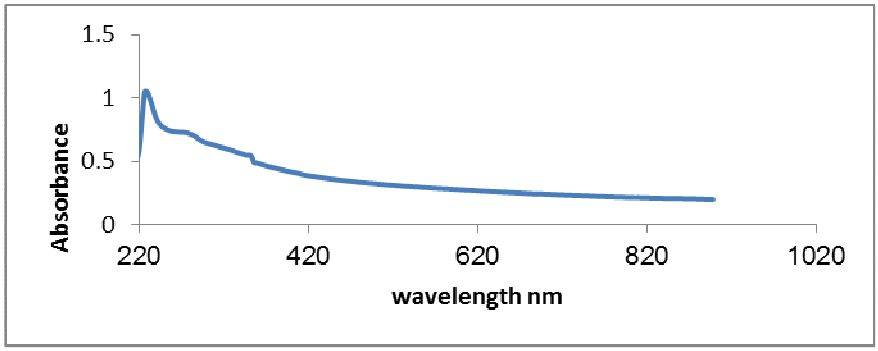


(a)

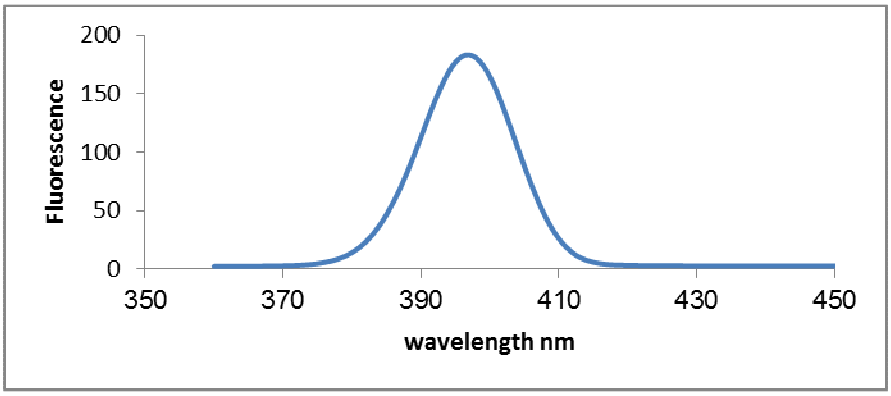


(b)

Fig. 4: Absorbance (a) and Fluorescence (b) spectra of MgO NPs prepared by PLAL method



(a)



(b)

Fig. 5: Absorbance (a) and Fluorescence (b) spectra of MgO NPs prepared chemical method

Conclusion:

1- X-Ray Diffraction (XRD) shows that the material obtained is MgO. It shows that MgO nanoparticles have cubic crystallites.

2- SEM images indicate shape of the MgO NPs chemically prepared is a large spherical unlike nanoparticles prepared by pulse laser ablation where a tube shapes.

3- From the absorption spectrum, the absorbance can be found at maximum wavelength λ_{max}. It is found that the highest value when it is prepared by pulse laser ablation.

4- From transmission spectrum, linear absorption coefficient and refractive index are found. It is noticed that the highest value of the absorption coefficient when the pulse laser ablation method was used, but the refractive index was found that the highest value when the chemical method was used.

5- The chemical method produce large grain size nanoparticles which led to a red shift in absorption peak, while the pulse laser ablation method produced smaller grain size which shows a blue shift in the peak absorption.

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