Synthesis and Characterization of ZnO and Ag Nanoparticles

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Abstract

The objective of this work is to synthesize of ZnO nanoparticles by sol-gel method and silver nanoparticles by laser ablation. An aqueous zinc nitrates was dissolved in distilled water and then stirred for 30 mints. Starch was also dissolved in water and stirred at 60°C for 90 minutes. The two solutions were mixed and stirred for 12hr at 80°C in an oil bath. The gel obtained was calcined in an oven at 700°C for one hour to obtain ZnO nanoparticles. Square silver piece of 5g was placed in a glass with a current of nitrogen gas. The silver piece was then subjected to pulse laser beam of 532nm wavelength and 250 mW. Thereafter, the glass was washed with methanol and put under the sun, and then the silver powder was put in an oven at 60°C for 14 hrs until obtaining silver nanoparticles powder. UV, SEM, and XRD analyses were used to characterize the powders prepared, and it was found that the average size of ZnO particles was 13.8nm and for an Ag particle size was 12.4nm.
Key words: laser ablation, nanoparticles, preparation, silver, sol-gel, zinc oxide.

الخلاصة

يهدف العمل الى تكوين مادة نانوية من ثاني اوكسيد الزنك ZnO بطريقة المحلول–الغروي ومن مادة الفضة بطريقة تقشيط الليزر تم اذابة مادة نترات الزنك المائية في ماء مقطر ثم وضعت في هزاز امدة 30min. وتمت اذابة النشاء في الماء ووضعت في هزاز لمدة 90min بدرجة حرارة 60°C. خلط المحلولان ووضعا في هزاز لمدة 12hr تحت درجة حرارة 80°C في حمام زيتي، ثم وضع الحلول الغروي المنتج في فرن درجة حرارته 700°C لمدة ساعة واحدة للحصول على مادة اوكسيد الزنك النانوية. وضعت قطعة مربعة من مادة الفضة بوزن 5g في وعاء زجاجي مع امرار تيار من غاز النتروجين وتسليط شعاع الليزر النبضي على قطعة الفضة ذي الطول الموجي 532nm وبطاقة 250mw. بعد ذلك غسل القدح بالميثانول ووضع تحت الشمس, ثم وضع مسحوق الفضة في فرن بدرجة حرارة 60°C لمدة 14hr للحصول على المادة الفضة النانوية. تم تشخيص المادتين النانويتين بواسطة UV و SEM و XRD. وتم قياس الحجم الحبيبي بواسطة تقنية XRD ووجد ان قطر حبة ZnO هو 13.8nm وللفضة 12.4nm.
الكلمات المفتاحية: التقشيط بالليزر، مادة نانوية, اعداد المادة النانوية، فضة، المحلول–الغروي، اوكسيد الزنك.

Introduction

Nanotechnology is the studying of material in atomic and molecule scale (Bhushan, 2007). The aim of producing nanoparticles material is to generate a material with new properties (Nasir, 2010).

The nanotechnology science is not a modern but an ancient science (Drexler, 2010). The start of nanoscience was in 1950 by Richard Faynman, the owner of nano phenomenon (Drexler, 1992).

In 1974, professor Norio from Tokyo university defined the term "nanotechnology" as a separating or incorporating process for the material, atom by atom and molecule by molecule (Hall, 2005).

Since 2006, nanotechnology entered in many applications of the life, it occupied a wide area of many applied sciences like: medicine, biology, pharmacology, electronic materials, and chemistry, and it is used in several industrial sciences like drug delivery, semiconductors, and restorative dental materials as filler (Nasir, 2008).

Nanoparticles are small particles whose dimensions are from 1 to 100nm. Every nanoparticle of these particles behaves and reacts as a substance alone. Nanoparticles

can be classified into inorganic and organic compounds and according to its shape or size (Antonio *et al.*,2014).

For these properties of nanoparticles, the nanomaterials have been increasingly prepared in the last years because they have applications in industry, medicine, and industrial biomaterials as many of these materials have biocompatibility with human tissues (Darroudi *et al.*,2011).

Since the properties of Zinc Oxide Nanoparticles (ZnO-NPs) are useful, they could be used in medicine & industry, which are transparency, high isoelectric point, biocompatibility, and photo catalytic efficiency, so the synthesis of ZnO-NPs occupied a large area from the work of researchers (Qiang, 2001), and they were used in different fields like cosmetics, toothpaste, sunscreens, fillings in medical materials, textiles, and building materials.

Several methods for preparing ZnO-NPs are there, some of them are difficult and the others are easy. Recently, several techniques have been used to prepare ZnO-NPs using protein, biopolymer, and carbohydrates as a stabilizing or covering agent (Zamiri *et al.*,2013).

Starch is one of the biopolymer used to prepare ZnO-NPs because it prevents the nanoparticle from agglomerating and increaseing in size (Darroudi *et al.*, 2014).

Concerning the formation of silver Nano-sized particles, dolgaev *et al.*, in 2003, produced nanomaterial of silver by vapor laser, in laser ablation method, using several types of solutions (Dalgaev *et al.*, 2002).

In 2005, Kazakerich *et al.* formed nanoparticles of Ag, Au, and Ti by laser ablation using H_2O, C_2H_5OH, and $C_2H_4Cl_2$ using Nd: Yag laser of 1.06μm .

Barcik *et al.* (2006) Synthesized nano-sized of Ti, Ag, Au, and Co materials by ablation method using femtosecond laser in air.

Silver nanoparticles has a functional control agent against bacteria and micro-organisms(Zhang *et al.*,2013), and hence the researches, in generating silver nanoparticles, increased in this line as shown above.

The aim of this work

The aim of this work is to produce ZnO-NPs according to the preparation method mentioned in the paper of Darroudi *et al.* (2014) and silver nanoparticles.

Experimental procedures

Preparing of ZnO-NPs

1. **The material and apparatus used:**

   Chemical used

   $Zn(NO_3)_2.6H_2O$ (an aqueous zinc nitrates), starch, distilled water.

   Synthesis process of ZnO-NPs

   0.75g of an aqueous zinc nitrates was dissolved in distilled water (5ml) and stirred for thirty minutes. At the same time, 0.1g of starch was dissolved in distilled water and stirred at 60C° for 90 minutes. The two solutions were then mixed and stirred for 12 hours at 80C° in an oil path until obtaining a clear resin mixture. This mixture was then calcined in an oven at 700C° for one hour. At the end we got a powder of ZnO-NPs.

2. **Preparing nanoparticles of silver(Ag):**

   5g of $1cm^2$ of solid silver was placed in a glass. A current of nitrogen gas was passed on the silver piece, meanwhile the silver was subjected to the laser beam of (532nm wavelength, 6Hz (six pulses per second) frequency, and 250 mwatt the energy). Thereafter, the glass walls were washed by methanol to fall down all the silver nanoparticles that were glued on the walls. Thereat, the glass was put in the

sun for 10 minutes and then in an oven at 60° C for 14hrs. Finally Ag nanoparticles have been obtained.

Characterization

UV-VIS spectrophotometry for ZnO-NPs was made. Figure (1) shows UV spectrophotometry of ZnO-NPs prepared; the solvent used is water.

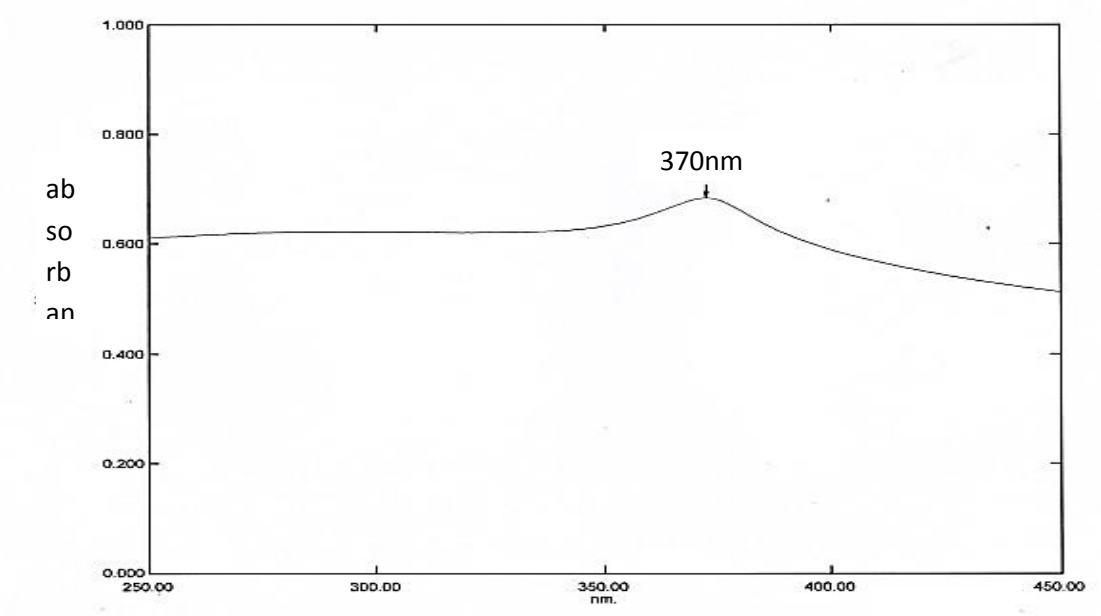

Fig.(1): the absorption spectrum of the ZnO-NPs powder in UV region. The x-axis represents the wavelength, and y-axis is for absorption.

Fig. (2) represents the particle size laser analyze for ZnO-NPs. Figure (3) is for XRD, and figure (4) is for (SEM).

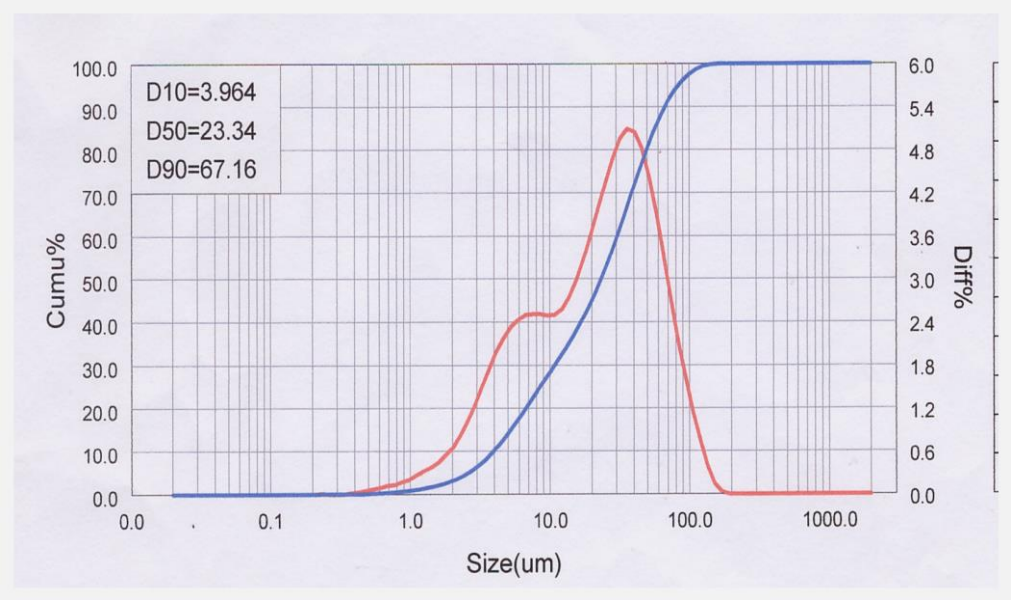

Fig.(2): the analyzing in PSLA where the x-axis represents the size of the particle, and the y-axis represents the percentage of the particle size distribution.

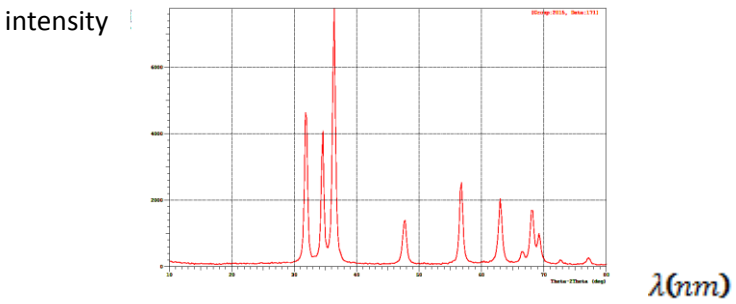

Fig.(3) XRD pattern for the ZnO-NPs prepared.

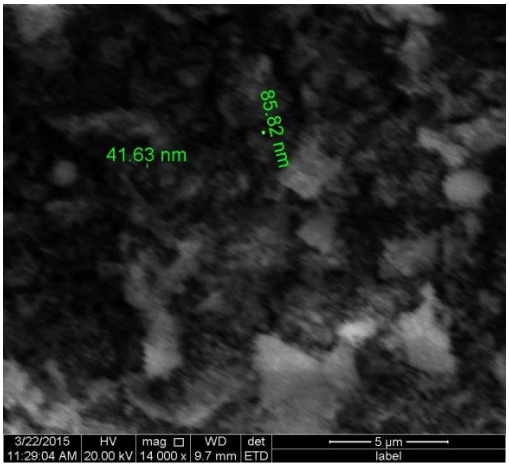

Fig. (4) SEM of the prepared ZnO-NPs

As to silver, UV-VIS graph is shown in figure (5), the XRD is shown in figure (6), and figure (7) represents SEM.

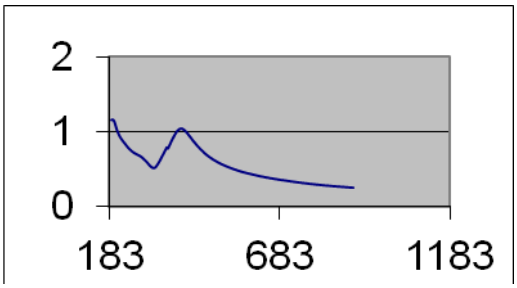

Fig. (5): UV spectrum of silver nanoparticles prepared where the x-axis is for wavelength in nanometer, and y-axis is for absorption.

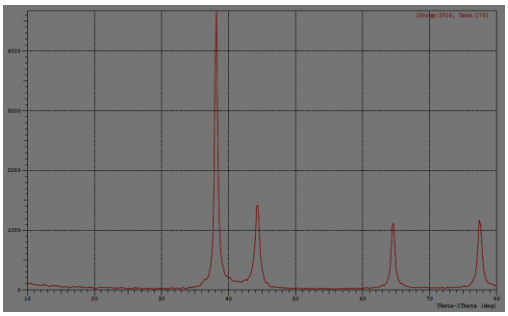

Fig. (6) XRD chart of the silver nanoparticles prepared.

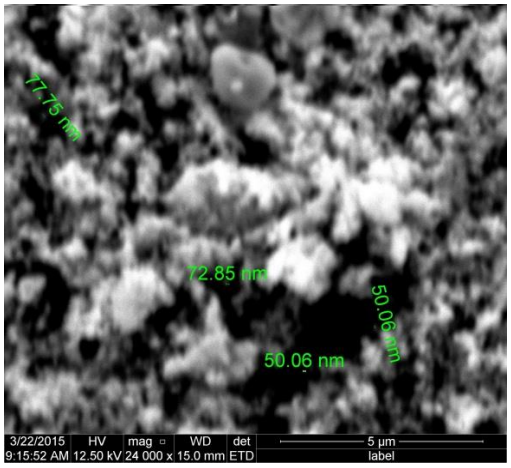

Fig. (7): SEM silver nanoparticles

Results and Discussion

For ZnO, the UV-VIS spectrum (figure 1) reveals that ZnO powder is in nanoscale because ZnO-NPs has a peak at 370 – 375 nm. This peak results from the electron transition from O_{2p} to Zn_3d across the band gap (Khorsand *et al*., 2013; khorsand *et al*., 2012).

From figure(2) we see the particle size distribution is from 1 to 100 µm. That because the nanoparticles agglomerate about themselves since they have an active surface, leading them to interact with each other (Hussein, 2014).

From Figure(3), the average particle size was measured by Debye-Sheerer equation (Darroudi *et al.*,2014)

$$D = \frac{0.89\ \lambda}{B\ COS\ \theta}$$

Where D is the grain size, λ is the wavelength at kα, it is intrinsic value for Cu, λ=0.154nm, B is the full wave half maximum of every peak, and θ is the diffraction angle, and it was found that the particle size is 13.8nm.

The standard XRD pattern, figure(8) (Aredi and Rezaei ,2012), was compared to that of our work, figure(3), the result is very good.

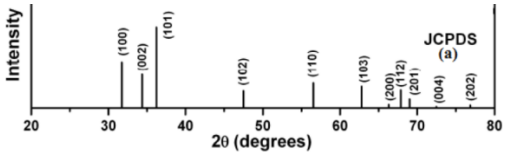

Fig.(8): the standard XRD pattern of ZnO-NPs.

Figure (4) shows the scanning electron microscope (SEM) of ZnO-NPs prepared. In the figure, it can be seen that the particles are agglomerated, but the grain sizes are apparent in nano range.

Regarding silver, UV-VIS spectrophotometry shown in figure(5) reveals that the silver powder prepared is in nano range and in spherical shape because nano silver particles have a peak at 390-420nm as shown in figure(9) (Debey *et al*., 2009).

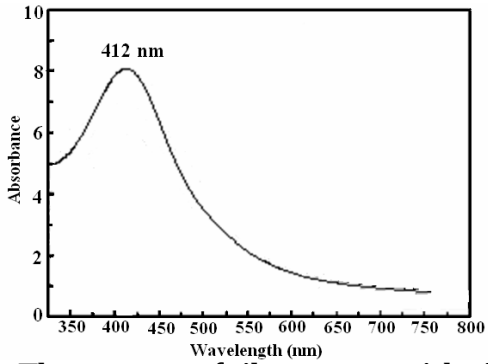

Fig. (9): The spectrum of silver nanoparticles in UV region.

XRD pattern of the silver powder prepared is shown in figure (6). Their peaks were compared with that of the standard ones figure(10) (http://s.) and found that they are correspondent. The grain size average was calculated using Deby-Sherer equation and found to be of 12.4nm, a good size, then the particles are in nano range.

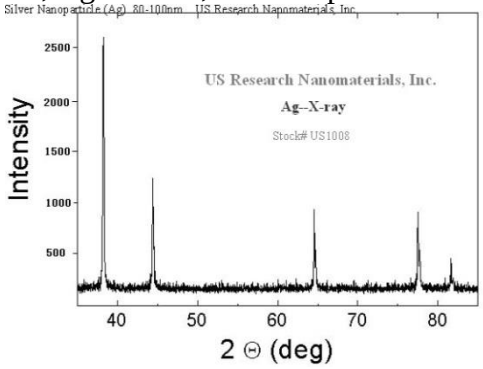

Fig. (10): The standard XRD flow chart of Nano silver.

Figure (7) shows us SEM of the nano silver and gives us the particle size of nearly 60nm that is because of the aggregation of the nanoparticles.

SEM in figures (4,7) reveale the agglomeration of nanoparticles about themselves. This agglomeration is because the nanoparticles have this feature (Scholz *et al*., 1998;Jiang *et al*., 2009;David and Maynard, 2007; Faure *et al*.,2013) mentioned that nanoparticles prepared from sol-powder suffer from hard agglomeration phenomenon, and Simord, J.M. mentioned that if ph during the operation is low, the nanoparticle size decreases, and agglomeration increases. So if we want to make nano grain, we must reduce the ph. Thereat, the agglomeration occurs (simard, 2007). To treat this phenomenon some of researchers used ultrasonic waves against agglomeration, at the last step of generating nanoparticles, to disperse the grains of an agglomerate (Sauter *et al*., 2008).

Conclusions

ZnO nanoparticles have been obtained by sol-gel method with a particle size of 13.8nm and silver nanoparticles by laser ablation with a particle size of 12.4nm even though the agglomeration appears in SEMs.

Acknowledgment

My acknowledgments are for my mother to her encouragement, my supervisor Dr. Nuhad abdul ameer for his treatment, Ph. D. student (in chemistry dept./college of science/ Babylon university) Firas Habeeb abdulrazzaq for his advice, head of my department Dr. Jabir hussein Alhamad, Dr. sayyed Adhnan Hammoodi Ala`araji for

his help, and Dr. Jassim (college of science for girls / Babylon university) for his help to me.

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