Synthesis and Characterisation of Zinc Oxide Nanopowders Prepared by Precipitation Method

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Received: 28 September 2017 Accepted: 3 January 2018

Abstract

In this study, the basic understanding of precipitation method to synthesis of Zinc Oxide nanopowders has been demonstrated. The synthesised nanopowders were formed by mixing Zn $(O_2CCH_3)_2$ $(H_2O)_2$, diethylene glycol (DEG) and deionized water, then the mixed solution was heated at two different temperatures 140 ^{0}C and 180 ^{0}C for 2 hours. The synthesised nanopowders have been studied by different characterisation techniques as X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), Dynamic Light Scattering (DLS), and Energy-Dispersive X-ray spectroscopy (EDX). Results demonstrated that the pure Zinc oxide nanopowders with low trace elements, with uniform morphology, controllable size and narrow-size distribution in diameter were achieved. The synthesized nanopowders can be used as active filler for rubber and plastic, catalyst and gas sensor.

Keywords*:* Precipitation method, Zinc oxide nanopowders, XRD, SEM, TEM.

DIYALA JOURNAL FOR PURE SCIENCES

Synthesis and Characterisation of Zinc Oxide Nanopowders Prepared by Precipitation Method

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تخليق وتوصيف مسحوق اوكسيد الزنك النانوي المحضر بطريقة الترسيب

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الخلاصة

تم في هذه الدراسة تقديم المفاهيم الاساسية لطريقة الترسيب في تحضير مسحوق اوكسيد الزنك النانوي. تم تشكيل المسحوق النانوي المحضر بواسطة خلط $Zn(O_2CCH_3)_2(H_2O)$، الكلايكول ثنائي الإثلين (DEG) و ماء لا ايوني، ثم تم غلي المحلول المخلوط عند درجتين حراريتين مختلفتين ^0C (140 و 180) لمدة 2 ساعة. تم دراسة المسحوق النانوي باستخدام تقنيات مختلفة مثل حيود الأشعة السينية (XRD)، المجهر الإلكتروني الماسح (SEM)، المجهر الإلكتروني النافذ (TEM)، تشتيت الضوء الديناميكى (DLS) ومطيافية تشتت الطاقة بالأشعة السينية (EDX). أظهرت النتائج أن مسحوق اوكسيد الزنك النانوي مع نسب نزرة من عناصر اخرى، مع مورفولوجيا موحدة و توزيع ضيق فى الحجم و مسيطر عليه. ان المسحوق النانوي المحضر يمكن ان يستخدم كحشو نشط للمطاط والبلاستيك و كمحفز و متحسس غازي.

كلمات مفتاحية: طريقة الترسيب، مسحوق اوكسيد الزنك النانوي، المجهر الإلكتروني الماسح، المجهر الإلكتروني النافذ، حيود الأشعة السينية.

Introduction

The nanosized ZnO has various properties in different fields and that due to the large surface area to volume ratio. It can be used as a UV absorber in cosmetics and antivirus agent in coating since it has high ultraviolet (UV) absorption properties. Beside this, ZnO can be used for applications such as gas sensor, active filler for rubber and plastic and catalyst [1-3]. More recent interest on its optoelectronic and semiconducting properties has been focused due to its application on solar cells [2]. Additionally, for skin treatment, ZnO is highly recommended in products, antidandruff shampoo, in calamine cream, baby powder and barrier creams to prevent a range of skin problems. Moreover, it can be used as antiseptic ointments [3].

There are different methods for synthesis of ZnO nanoparticles (NPs), which are organometallic method, sol- gel technique, mechanochemical method, homogeneous precipitation, mechanical milling, thermal evaporation, spray pyrolysis and microwave method. Nevertheless, because of

the property of the large surface area and high surface energy, ZnO nanoparticles are prone to aggregate. In case of having a perfect dispersion, it is important to modify the surface of ZnO (NPs) [4-6].

In this work, the precipitation method was used followed by controlled and drying processes. The materials achieved were thermally treated. The structural, textural, and morphological characteristics of the substances were analysed by using powder X-ray diffraction, scanning electron microscopy (SEM), transmission electron microscopy (TEM), dynamic light scattering (DLS), and energy dispersive X-ray spectroscopy (EDX).

Materials and Methods

After cleaning the equipment, the oil bath is placed on a magnetic stirrer with hot plate, and 100 ml round bottom flask is fixed with clamp stand above the oil bath. (0.5 g) of Zinc acetate was used as a precursor, in which the purity was about 97% (Combined Chemical Services (UK) Ltd). Then a weigh boat is used to transfer it carefully into the round bottom flask. After that, 50 ml of diethylene glycol (DEG) was measured and transferred into the round bottom flask. By using oval stirrer, which is attached inside the round bottom flask, the solution was stirred to get a homogeneous solution. Furthermore, 1 ml of deionised water was measured by using 1 ml syringe, and transferred to the round bottom flask. Also, the flask is put in the oil bath while heating, and the temperature of the solution is kept at 140 ^{0}C and the magnetic stirrer is adjusted to 700 rpm. The heating process continued at the temperature 140 ^{0}C for an hour and the flask was covered with foil. After1 hour, the temperature was increased to nearly 180 ^{0}C and then the flask was left for more than 2 hours, before removing it from the oil bath and allowing it to cool down in room temperature.

Results and discussion

XRD Analysis

X-ray diffraction, (Philips PW1050, scanning range of 3-105°), studies reveal the properties through the X-ray diffraction pattern, which is shown in figure (1). In this figure, 11 peaks showed of Zinc Oxide phase. The diffraction peaks located at 31.75, 34.44, 36.25, 47.54, 56.55, 62.87, 66.38, 67.91, 69.05,72.61 and 76.95 have been keenly indexed as hexagonal phase

(wurtzite) of ZnO, and these values correspond to the file (JCPDS Card No.00-005-0664). It could be noticed that no peaks due to impurity were observed. Moreover, when the peaks broaden, the particle size would be small [2]. Then, the produced ZnO nanoparticles crystallite size was determined by using Scherrer's equation, $D= 0.89 \lambda/\beta \cos \theta$, where 0.89 is Scherrer's constant, λ is the wavelength of X-rays, θ is the Bragg angle, and β is the full width at half maximum (FWHM) of the diffraction peak for the highest peak which is 36.25, as a result the average particle size of the sample was found to be 16.21 nm according to Scherrer's formula [5].

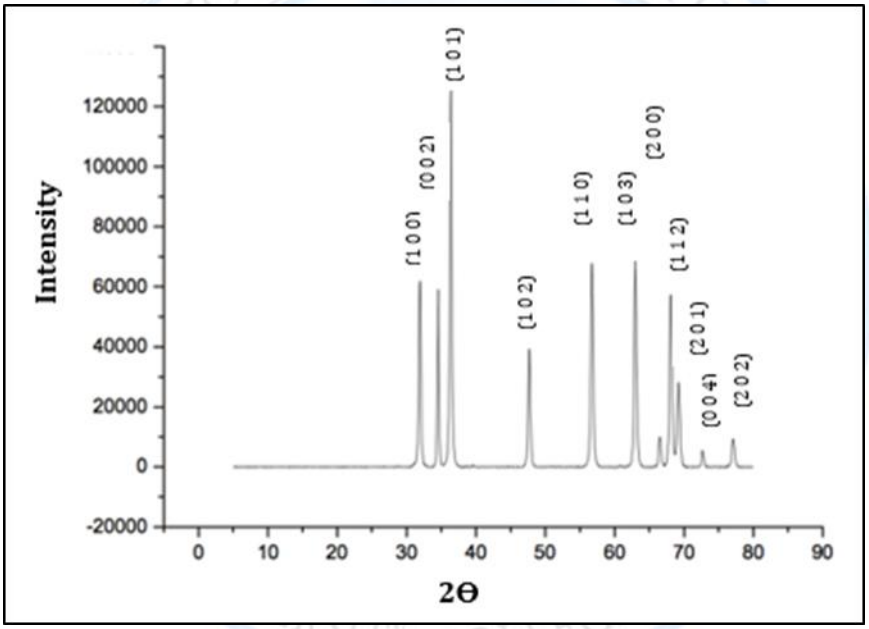

Figure 1: The XRD pattern of zinc oxide hexagonal phase

SEM Analysis

In this study, SEM (Hitachi SU8230: high performance cold field emission) has been used. In figure (2. a), the SEM image demonstrates a uniform structure and size for ZnO nanoparticles. However, in some places, the size of particles is bigger, and they are agglomerated by different groups. In addition, it demonstrates the ZnO NPs are well dispersed in the powder shape.

TEM Analysis

According to the TEM (FEI Titan3 Themis 300: X-FEG 300 kV S/TEM with S-TWIN objective lens, monochromator (energy spread approx. 0.25 eV)) image of the ZnO NPs in figure (2. b), structures have grown in the shape of hexagonal, which demonstrates the good feature of the ZnO nanoparticles. Within hexagonal shape, the sphere like particles in some places can be observed, and different dimension sizes would be noticed with various groups that they are agglomerated and the average size about 20-120 nm.

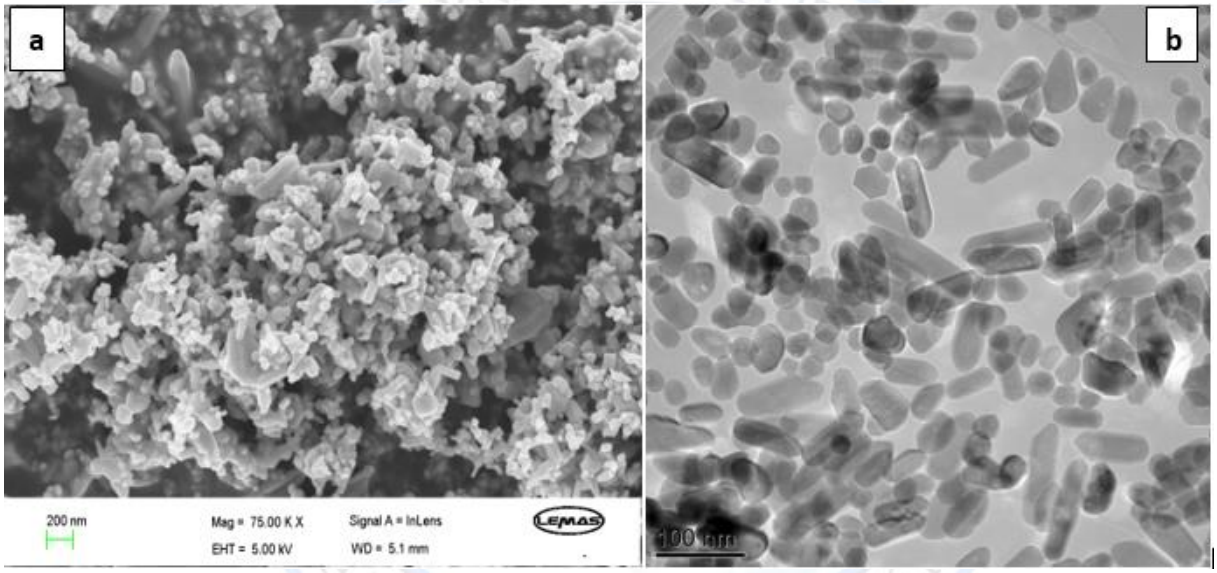

Figure 2: a (SEM image of ZnO nanoparticles and b) TEM image of ZnO nanoparticles.

DLS Analysis

Dynamic light scattering (DLS), (Malvern Instruments Zeta Sizer HPPS), (sometimes referred to as photon correlation spectroscopy or Quasi-elastic light scattering) had been used, which is a method for calculating the size of particles typically in the sub-micron region. In this technique, the interaction occurs between particles, light and changing with intensity, the small particles make the small peaks; while, the large peaks indicate the big-sized particles. In Figure (3), the distribution of particle size according to intensity, number and volume have been shown. In the case of intensity (Red line), the particle sizes are in the range of 50 nm to about 900 nm, while approximately 9% of particles in the synthesised powder would be 160 nm.

Moreover, according to the particle size distribution number (black line); it is between 30 and 200 nm, where 16% of the particles were about 60 nm. Then, if the particle size distribution is converted from number to volume (blue line), the peaks would be different due to volume of sphere, that is when the particles volume was in the range of 30-500 nm, and also 10% of ZnO would be 80 nm.

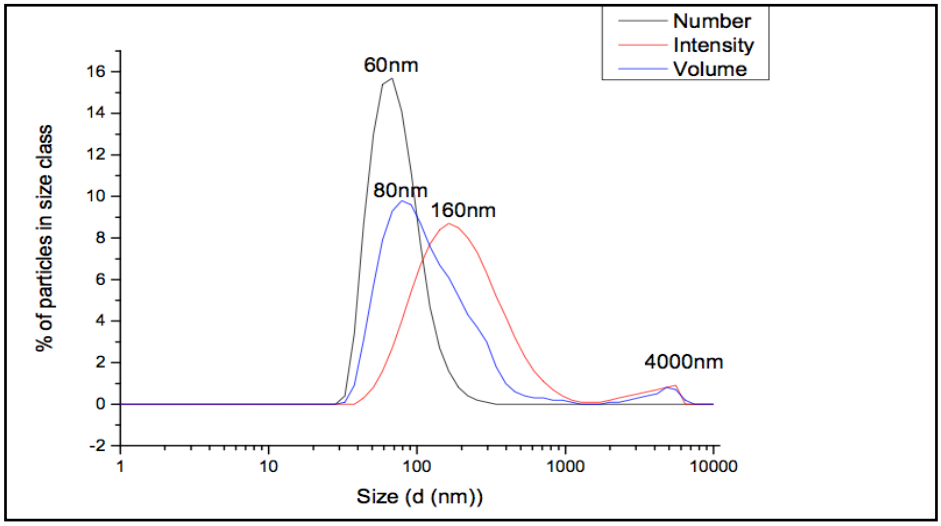

Figure 3: The distribution of particle sizes of ZnO NPs, with intensity, number and volume

EDX Analysis

According to energy dispersive X-ray spectroscopy (EDX), (SHIMADZU, EDX-7000) pattern showed that zinc (Zn) and oxygen (O) are not the only two elements in the sample which means that trace elements (other elements) have also been detected in synthesised nanopowder [7]. However, the elements of ZnO with respect to atomic percentage and weigh percentage showed 100% of elements O and Zn. This result indicated that the quantities of sharing substances taking in a reaction or forming a compound of ZnO was resulted as shown in figure (4) which affirmed an atomic ratio of Zn: O=1:1.

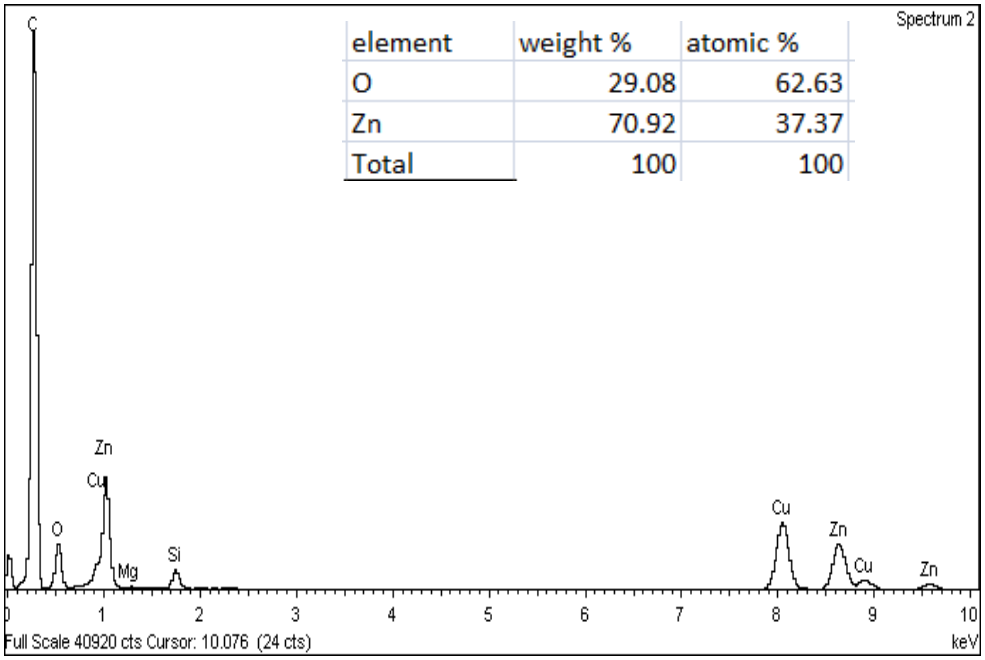

Figure 4: EDX pattern of ZnO NPs

Conclusion

ZnO NPs were successfully synthesised by wet chemical synthesis technique which was precipitate method, by using 0.5g of Zn $(O_2CCH_3)_2$ $(H_2O)_2$ and 50 ml of diethylene glycol (DEG) with 1 ml of deionized water. Synthesised powders were characterised by different techniques. XRD showed the purity of ZnO with the form of hexagonal phase with lattice constants a=b= 0.324 nm and c= 0.521 nm. SEM and TEM studies confirmed that the nanostructures for ZnO NPs have perfectly formed. DLS technique showed that the particle size distribution, according to intensity, number, and volume appoints sequentially the following range: 160 nm, 60 nm and 80 nm respectively. EDX technique showed that (O) and (Z) are not the only two elements in the sample, however ZnO was achieved.

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