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CHEMICAL SYNTHESIS BY PRECIPITATION OF ZINC OXIDE FOR BOIMEDICAL APPLICATION

ABSTRACT

The objective of the study is the chemical synthesis of ZnO powders, from $ZnCl_2$ and NaOH solutions according to an appropriate procedure. The powders (a) and (b) obtained underwent various characterizations such as: optical microscopy, SEM, UV, BET, IR, XRD and antimicrobial activity. The results showed the inhomogeneous distribution, the nanometric size, the absorbance at 353 and 346 nm and the specific surface of 25.701 and 30.534 cm^2/g of the particles, the presence of all the characteristic bands of ZnO which was confirmed by XRD and very good bacterial sensitivity of the two ZnO powders.

Keywords: *Chemical synthesis; Optical properties; Antimicrobial activity*

INTRODUCTION

Currently, intense research has been relaunched on the study of zinc oxide, due to its availability and its non-toxicity. This metal oxide has very interesting properties and is used in such diverse and varied fields as the pharmaceutical, electronic, cosmetic and medical industries [1].

Zinc oxide is an inorganic substance with the chemical formula ZnO. It comes in the form of a water-insoluble white powder [2, 3]. It is found in Earth crust in the form of zincite mineral, although most ZnO utilized for commercial applications is synthesized [4]. This oxide exists in three different types of crystallographic structures depending on the production conditions; Under very high pressures appears the first cubic structure (Rock-Salt), under high pressures appears the second cubic structure (Blende) which is unstable and under normal conditions appears the hexagonal structure (Wurtzite), this one is thermodynamically the more stable [5].

Zinc oxide, is a multifunctional material, with its unique physical and chemical properties, such as high chemical stability, high electrochemical coupling coefficient, wide range of radiation absorption and high photostability [6-8]. In materials science, zinc oxide is classified as a semiconductor in group II-VI, whose covalence is on the boundary between ionic and covalent semiconductors. A broad energy band (3.37 eV), high bond energy (60 meV) and high thermal and mechanical stability at room temperature make it attractive for potential use in electronics, optoelectronics and laser technology [9,10, 8].

Zinc oxide has very interesting physical and chemical properties that make it a suitable candidate for a large number of applications, ranging from optics to environmental sciences, including biomedicine. Moreover, the versatility of ZnO is also due to the existence of many morphologies with very high specific surfaces [11]. This metal oxide is a potential semiconductor material for many applications such as catalysts [12], solar cells [13], gas sensors [14], light emitting diodes [15], l rubber additive [16], and pigments [17]. Due to the advantage of anti-UV absorption properties of ZnO, they are increasingly used in personal care products, such as cosmetics and sunscreens [18]. In addition, for its new physical properties are characterized by their photocatalytic and photo-oxidative ability to resist chemical and biological species [19]. Zinc oxide is used potentially in clinical purposes it is more competent for biosynthesis of nanoparticles than that of other metals [20-22].

The normal ZnO and its nanoparticles are commonly added to plastic, glass, ceramics, cement, and rubber materials, as well as pigments, paints, food supplements, batteries, and non-flammable materials. The reason for this is their wide range of suitable properties, which is also linked with the easy availability and low price of the chemical. These properties include relatively high electrical and thermal conductivity and stability in high temperatures with a neutral pH and mild antimicrobial effects [23, 3].

Recently, researchers have been attracted to ZnO nanoparticles because of their wide biomedical applications [24]. ZnO nanoparticles are versatile materials with distinct chemical, optoelectronic and wettability properties. They are easy to manufacture and widely used in a variety of industries including wastewater treatment [25, 26].

Zinc oxide nanoparticles show promising and far-reaching prospects for the biomedical field, especially for antibacterials, anti-cancer drug/gene delivery, cell imaging, biosensing, etc. [27, 28]. Zinc oxide nanoparticles are prepared by the precipitation method. Their ability to reflect and scatter UV-A and UV-B radiation makes them excellent UV inorganic filters [29, 30]. These nanoparticles are commonly used in various fields due to their particular physical and chemical properties [31, 32]. ZnO nanoparticles are first applied in rubber industry because they can provide wear resistance of rubber composite, improve the performance of high polymer in their toughness and intensity and anti-aging, and other functions [33, 34]. In addition, ZnO NPs have superior antibacterial, antimicrobial and anti-UV properties. Therefore, in the textile industry, fabrics finished by adding ZnO NPs exhibited the attractive functions of ultraviolet and visible light resistance, antibacterial and deodorant [35].

Thanks to this study, zinc oxide powders were synthesized by the chemical method based on precipitation. This is easily done in the laboratory. Different characterization techniques, such as SEM, UV, IR, BET, XRD and antimicrobial activity were used. The results obtained showed that these powders, composed of nanoparticles and due to their optical properties, their antibacterial activity and their non-toxicity can be used in the biomedical field such as the preparation of a dermal ointment.

MATERIALS AND METHODS

Used materials

The materials used for the synthesis of ZnO powders were Zinc chlorides, Sodium hydroxide, Ethanol 95%, Distilled water and ice cubes.

Preparation of ZnO powder

The zinc oxide powders were prepared chemically, by the precipitation method, according to this process: By instantaneous mixtures of two solutions of the same volumes (250ml) and equimolar (0.5M) of zinc chloride and hydroxide of sodium, under magnetic stirring for two hours and under pH control (5 – 7), until white clouds are obtained. The latter were filtered and washed several times with distilled water and then with ethanol to obtain white precipitates. The preparation steps are summarized in Figure 1.

Fig. 1. Experimental protocol

Finally, the precipitates thus obtained were dried differently in an oven and under vacuum to obtain white powders called samples (a) and (b) respectively.

ZnO powder characterization techniques

Different characterization methods were carried out on our synthesized and dried powder samples by two different methods, such as observations under an optical microscope and scanning electron microscope, infrared spectroscopy, optical properties, BET measurements, X-ray diffraction and antibacterial activity.

Antibacterial activity

The biological tests were carried out at the level of the microbiology laboratory of the University Hospital of Tizi Ouzou in Algeria.

The antibacterial activity of the synthesized ZnO powders was determined by the well agar diffusion method. This method is equivalent to an antibiogram and provides preliminary results on the sensitivity of the strains and the antibacterial activities, thanks to the diameters of the zones of inhibition appearing around the wells measured in millimeters.

Two bacterial strains (Staphylococcus aureus (gram positive) and Escherichia coli (gram negative) were tested in a culture medium. The two bacterial strains were subcultured by the streak method on Mueller-Hinton agar and then incubated at 37° C. for 24 hours.

From these young cultures, pure colonies were isolated to prepare the bacterial inoculum. Each colony was placed in a cell suspension prepared in sterile physiological water containing approximately 108 bacteria per ml, with an optical density of 0.08 to 0.10 read at 625 nm and calibrated at 0.5 MF [36].

The seeding of the inoculum was carried out by swabbing, by making tight streaks on the agar while turning the dish 3 times by 60° in order to ensure a good distribution of the inoculum.

Wells of about 10 mm were drilled using a Pasteur pipette and the zinc oxide powder was directly deposited, according to Figure 2.

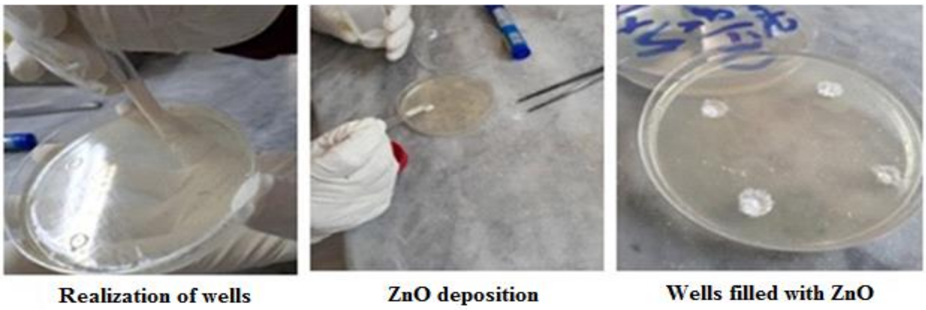


Fig. 2. Creation of wells on agar and deposition of ZnO powder

After incubation at 37°C for 24 hours, the clear inhibition diameters around the wells were measured.
The sensitivity of ZnO powders was classified by the diameter of the inhibition halos [37].

- Not sensitive for diameters less than or equal to 8 mm.
- Sensitive for diameters from 9 to 14 mm.
- Very sensitive for diameters of 15 to 19 mm.
- Extremely sensitive for diameters greater than 20 mm.

RESULTS AND DISCUSSION

Observation under an optical microscope

Observation under an optical microscope of the two samples gave the results illustrated in Figure 3.

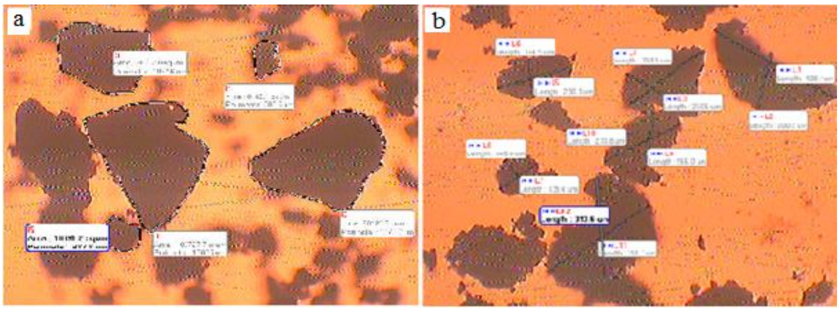


Fig. 3. Microscopic observation of samples (a) and (b) (objective magnification × 10)

Analysis of Figure 3 shows that the distribution of the particles of the two powder samples (a) and (b) is non-homogeneous. Indeed, a difference in size and shape of the particles is observed in the two samples.

Analysis by scanning electron microscopy (SEM)

In order to know the morphology of the two samples (a) and (b), SEM analyzes were carried out and the results obtained are displayed in Figure 4.

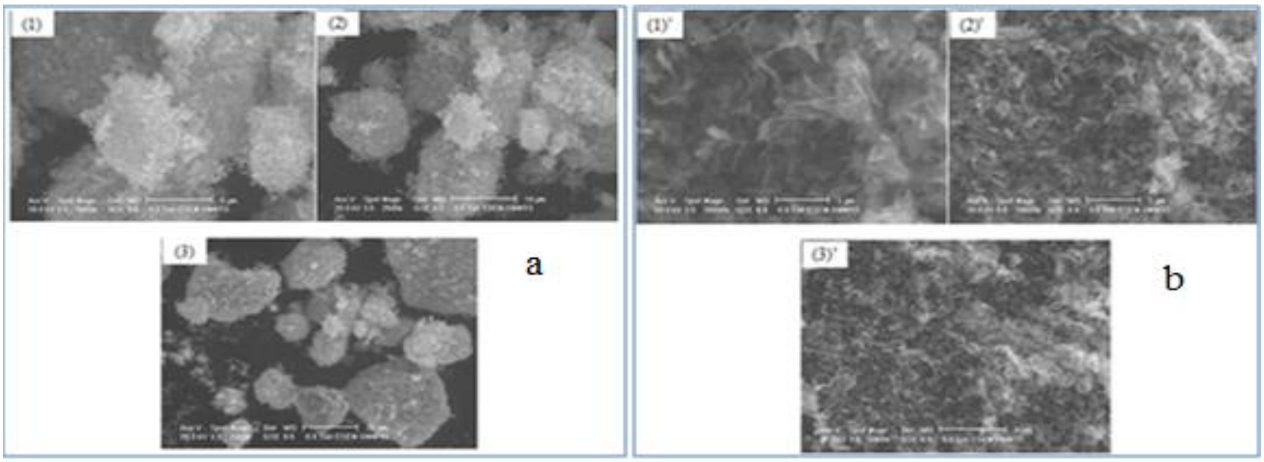


Fig. 4. SEM images of ZnO powders

The SEM micrographs reveal that the particles of the two powder samples are of nanometric sizes. At equal magnification, it is observed that the grains of the vacuum-dried sample (b) are significantly smaller than those of the oven-dried sample (a).

Optical properties

The evaluation of the optical properties of our samples was carried out by UV-Vis analysis and the results obtained are presented in Figure 5.

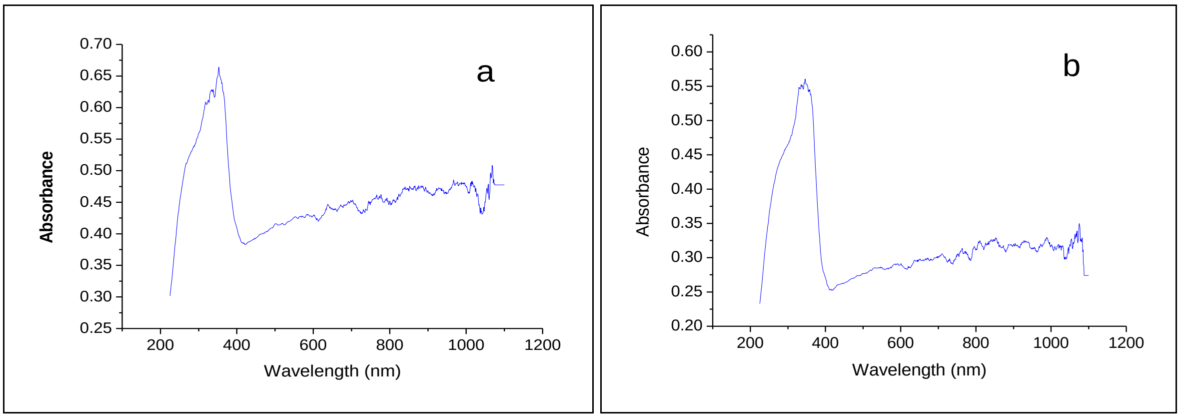


Fig. 5. Absorbance spectra of ZnO powders

Figure 5 shows the two UV-Vis absorption spectra. The two samples absorb in the same wavelength range and the width of the absorption band is of the same order of magnitude with maximums located around 353nm and 346nm for the respective samples (a) and (b).

Analysis by infrared spectroscopy

Infrared spectroscopy tells us about the main chemical groups that may exist in the two samples of zinc oxide. The analysis results are shown in Figure 6.

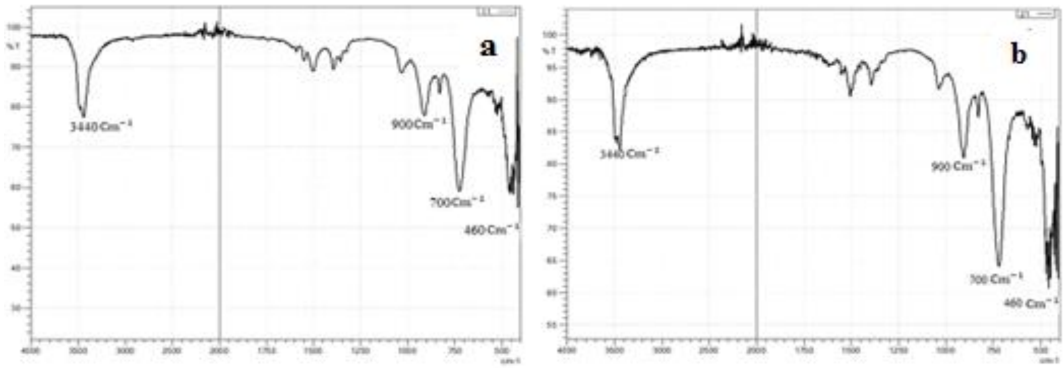


Fig. 6. Infrared spectra of ZnO powder samples

The two spectra reveal the formation of ZnO by the chemical precipitation method:

- The peaks at 460 cm^{-1} and 700 cm^{-1} clearly represent the Zn-O bond that forms in the range of [400 - 700] cm^{-1}[38].
- The band at 900 cm^{-1} is attributed to the O-H bond strain vibration.

The deepest bond is located at the wave number equal to 3440 cm^{-1}, this is attributed to the O-H bond relating to the presence of free water (humidity), this band is the result of the experimental conditions.

So, drying has no effect on the properties of the O-H and Zn-O bonds, but the intensity of the peaks is important in the case of vacuum drying.

Analysis by the Brunauer-Emmett–Teller (BET) method

The nitrogen adsorption/desorption isotherms on the two samples are shown in Figure7.

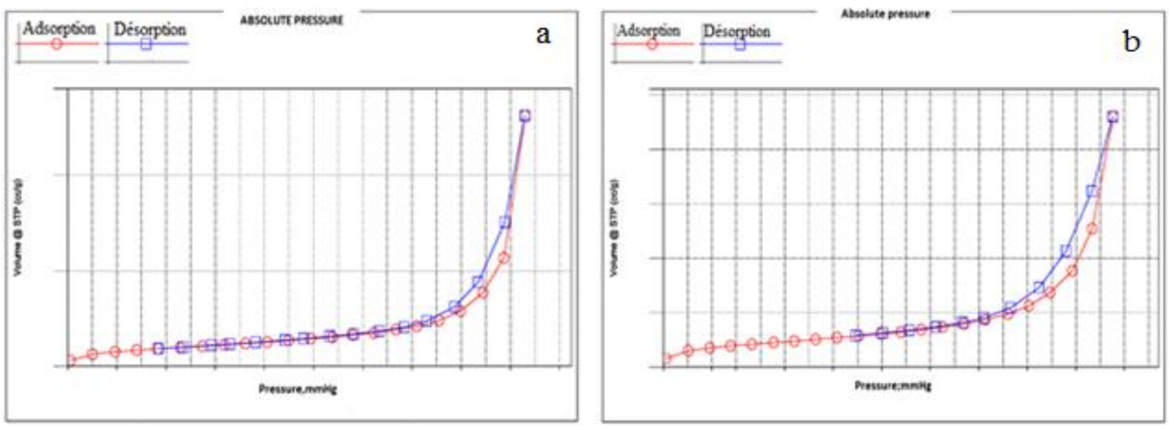


Fig. 7. Nitrogen adsorption isotherms on the two ZnO samples

The isotherms of the two samples studied are of type IV. The type IV isotherms are typical of mesoporous materials. For these materials, single-layer-multilayer formation is followed by pore condensation, which means that the gas condenses to a liquid-like state in a pore at a pressure below the saturation pressure p0 of the bulk liquid [39-41].

The hysteresis is of H3 type and loops of this type are given by non-rigid aggregates of plate-like particles, but also if the pore network consists of macropores which are not completely filled with pore condensate [42].

The properties deduced from these isotherms are shown in Table 1:

Table 1. *Results of analysis by nitrogen adsorption on zinc oxide powders*

Dried sample	Area (m^2/g)	Pore volume (cc/g)	Pore sizes (Å)
In the oven	25.701	0.160	75.532
Under a vacuum	30.534	0.141	70.007

The analysis of table 1 gives these main results:

- The vacuum-dried sample has a higher adsorption capacity (30.534 m^2/g) compared to the oven-dried one (25.701 m^2/g) since it has the largest specific surface area.
- The pore volume of the oven-dried sample (0.160 cc/g) is greater than that of the vacuum-dried sample (0.141 cc/g).
- The pore size of the oven-dried sample (75.532 Å) is larger than that of the vacuum-dried sample (70.007 Å).

Therefore, vacuum drying increases the specific surface of ZnO by about 16% compared to oven drying.

X-ray diffraction

The two synthesized ZnO powders were analyzed by X-ray diffractometer and the results obtained are shown in Figure 8.

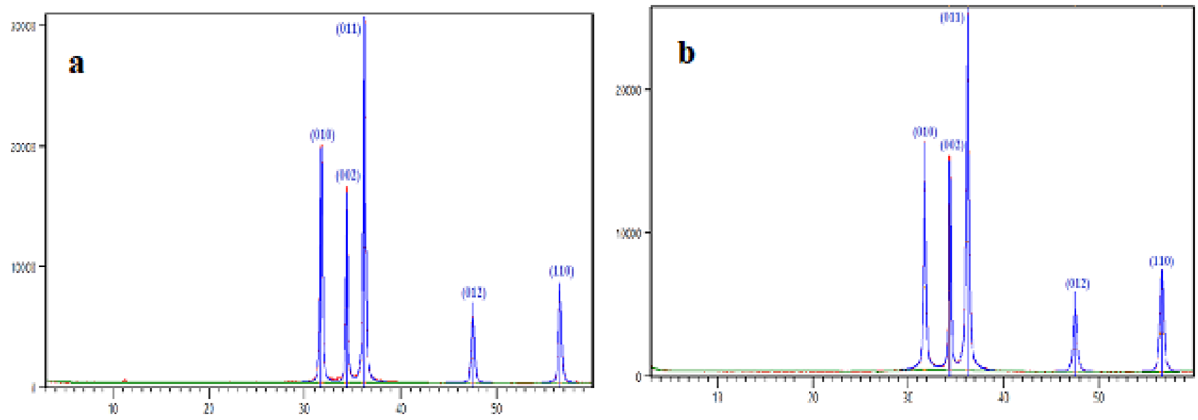


Fig. 8. Diffractograms of ZnO powders

The main peaks of the two diffractograms (a and b) of Figure 8 correspond to Bragg reflections with 2θ values of 31.73°, 34.37°, 36.21°, 47.49° and 56.54°, the reflections were indexed to the crystal planes (010), (002), (011), (012) and (110) respectively.

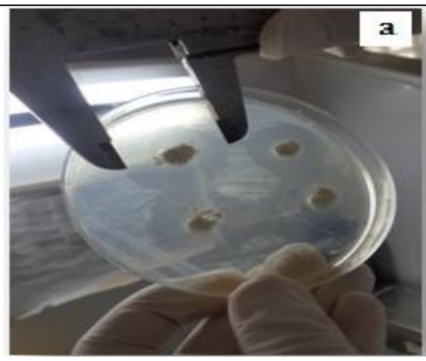
The results confirm that all the diffraction peaks of the two samples correspond to the hexagonal wurtzite structure (a = 3.2530 Å and c = 5.2070 Å) according to the reference code 96-900-4182.

Antimicrobial activity

The antimicrobial activity was carried out on the two ZnO powder samples by adopting the well diffusion method. This method provides preliminary results on the susceptibility of the strains and the antibacterial activities, thanks to the diameters of the zones of inhibition appearing around the wells measured in millimeters.

The results of the antibiograms obtained are grouped in Table 2.

Table 2. *Results of antibiograms of samples (a) and (b) of ZnO*

Bacterial strain	Inhibition diameter	
Staphylococcus aureus	 28 mm	 23 mm
Escherichia Coli	 24 mm	 23 mm

Staphylococcus aureus is extremely sensitive to ZnO powder with an inhibition zone of 28 mm for sample (a) and 23 mm for sample (b).

Escherichia coli showed good sensitivity to zinc oxide powder with an inhibition zone of 24 mm for sample (a) and 23 mm for sample (b).

CONCLUSIONS

The objective of this study is the development and characterization of ZnO powders. According to the chemical method by precipitation; a simple, inexpensive and easy-to-implement method. The samples thus obtained were dried differently in an oven and under vacuum, and then characterized by different techniques such as optical microscopy, scanning electron microscopy, spectrophotometry, the Braunuer-Emmett – Teller (BET) method, infrared spectroscopy, X-ray diffraction and the antimicrobial activity. The main results obtained are as follows:

- The optical microscope shows the inhomogeneous distribution of ZnO particles.
- SEM images reveal the nanometer size of the ZnO particles obtained by vacuum drying.

- The UV-Vis absorption spectra confirm the transparency of our samples (a) and (b) which absorb in the same wavelength range and whose maximums are respectively at 353 nm and 346 nm.
- The specific surface of sample (b) (S=30.53 m^2/g) is greater than that of sample (a) (S=25.70 m^2/g).
- The infrared spectra show the presence of broad peaks between 460-700 cm^{-1}, more intense in the case of the vacuum-dried sample, which correspond to the Zn-O metallic bond and medium bands around 3440 cm^{-1} which correspond to the OH bond due to the presence of humidity.
- X-ray diffraction reveals characteristic peaks of the ZnO powder and confirms the previous results (IR and UV-visible).
- Both bacteria (Staphylococus aureus and Escherichia Coli) showed good sensitivity to both ZnO samples.

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