RESEARCH PAPER

Polymorphic Agglomerates of Nano-sized Zinc Oxide: Synthesis and Morphology Study

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ABSTRACT

This article explores the synthesis and characterization of zinc oxide nanoparticles produced via the sol-gel process, a method known for its ability to control particle size and morphology. The resulting nanoparticles exhibit a quartzite structure, as confirmed by X-ray phase analysis, with an average crystallite size of 17 nm. Hydrodynamic measurements, conducted using dynamic light scattering techniques, revealed an average particle radius of approximately 70 nm, indicating some degree of agglomeration in the colloidal solution. Scanning electron microscopy further supported these findings, showing that the zinc oxide nanoparticles tend to form polymorphic nano-sized agglomerates. The observed aggregation behavior is significant, as it may influence the material's properties and potential applications in fields such as catalysis, photovoltaics, and sensor technology. This study not only highlights the effectiveness of the sol-gel process in producing zinc oxide nanoparticles with controlled sizes but also provides insights into their aggregation tendencies, which are crucial for optimizing their functional performance in various applications.

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INTRODUCTION

The fabrication of nanostructured materials based on zinc oxide is the subject of an increasing number of investigations [1-3]. ZnO is used effectively in powder lasers and in various microelectronic devices, such as unipolar transistors and LEDs [4], due to its relatively inexpensive cost, short luminescence period, and radiation wavelength (550 nm). Regarding their polymorphism, nanostructures made of zinc oxide are very intriguing. Gas sensors [5], piezoelectric devices [6], photoelectric converters [7], solar batteries [8], and photoelectric converters can all be made with zinc oxide nanostructures of different forms. ZnO in the form of hollow microspheres and flower-like structures can be created in three dimensions using the techniques outlined in the study [9]. It's noteworthy to observe that ZnO[10] flower-like formations are made up of arrangements of Nano sheets and Nano rods. As gas sensors, 3D ZnO Nano flowers are employed [11] because of their high specific surface area and great sensitivity at ambient temperature. Furthermore, ZnO Nano flowers have been utilized

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in tissue engineering in the medical field [12]. This information outlines the experiments done as part of this endeavor to identify and examine the variety of polymorphic forms created by Nano sized zinc oxide.

MATERIALS AND MEHODS

Reagents of purity grade (chemically pure) were utilized to create zinc oxide nanoparticles. The synthesis process is shown in the section below. A zinc sulphate solution $ZnSO_4.7H_2O$ with a concentration of 0.015 M was made in the first step. A sodium bicarbonate solution with a 0.04 M concentration is added in the second stage. Then, while stirring continuously with a magnetic stirrer, a solution of sodium bicarbonate was added drop by drop to a solution of zinc sulphate. Zinc hydroxide precipitated once the solution turned hazy. The separated precipitate was then three times rinsed with distilled water in the following step. The precipitate that resulted from washing was then baked for three hours at 80°C. A muffle oven at 350°C for three hours was used to create the final sample of Nano scale zinc oxide. White powder was the outcome. An Empyrean X-ray diffract meter. series 2 was used to analyses the

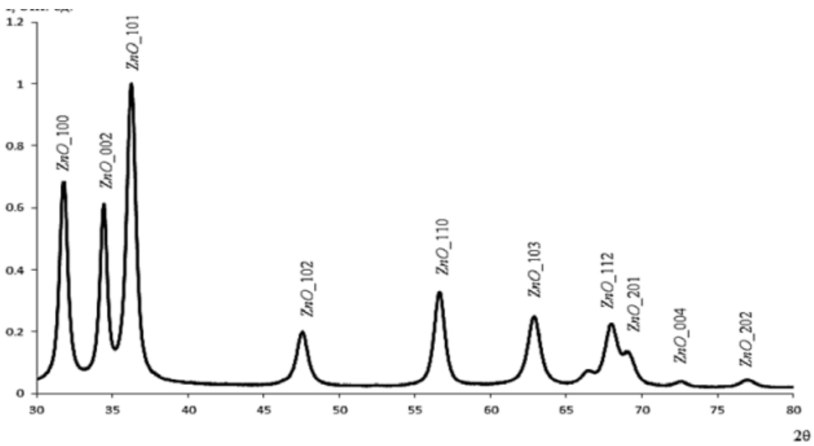


Fig. 1. X-ray Test of a zinc oxide.

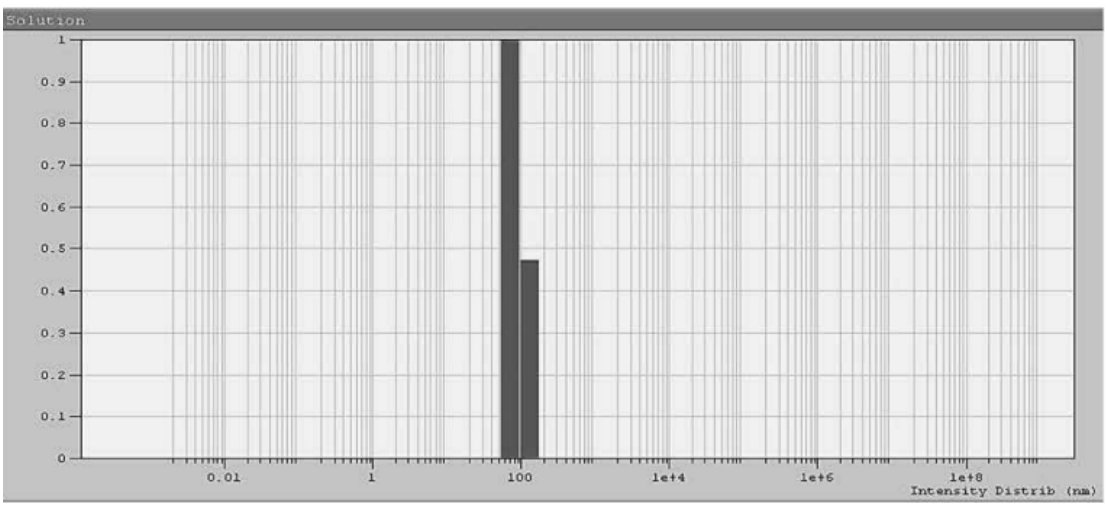


Fig. 2. Histogram of the hydrodynamic radius distribution of zinc oxide nanoparticles.

resultant sample in order to ascertain its phase composition and crystallographic properties. The produced ZnO nanoparticles were studied on a Photo or Complex spectroscope to ascertain the average hydrodynamic radius. The form of the acquired particles was specifically investigated using a Tuscan Mira 3 scanning electron microscope.

RESULTS AND DISCUSSION

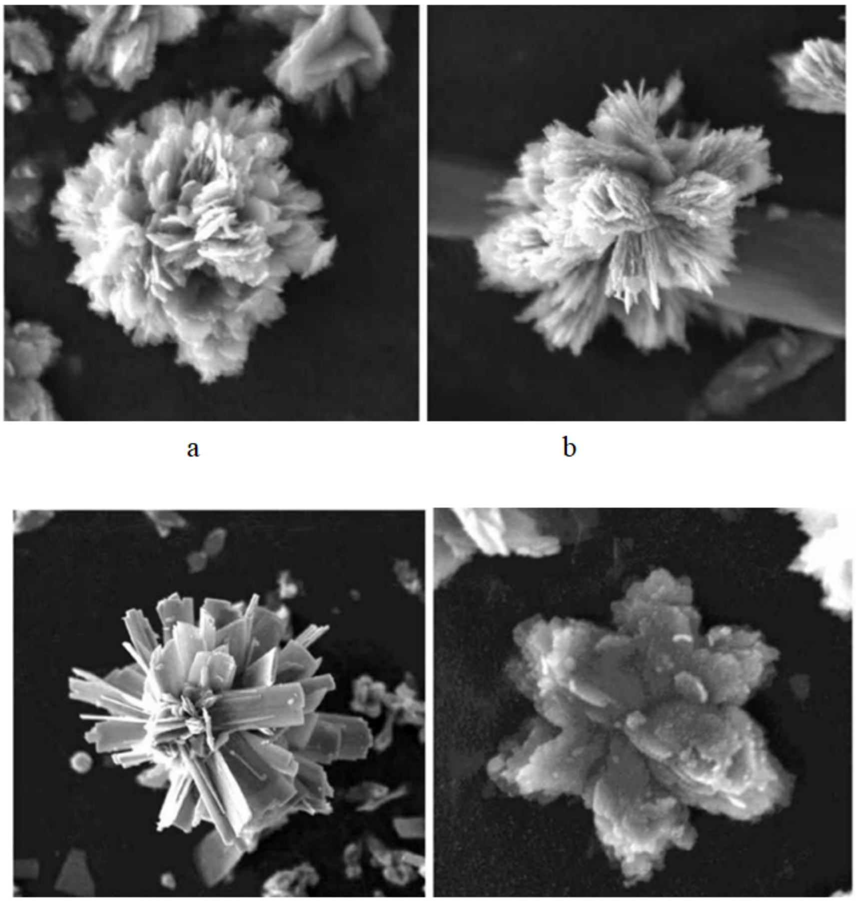
Utilizing X-ray phase analysis, it was possible to determine the structure and phase composition of the zinc oxide powder sample that was obtained. As a result, a diffraction pattern, represented in Fig. 1, was produced.

A database search was done to interpret the obtained diffraction pattern [13],[14]. It may be said as a consequence of the study done that the diffraction pattern comprises all the peaks typical of zinc oxide in a structure resembling quartzite. Furthermore, the strength of the principal peaks indicates that the resulting zinc oxide sample is a Nano scale creation with a “excellent” crystalline structure. collected systematic data on the analyzed material using the X-ray diffract meter Empery a 2 software, results shown in Table 1.

The findings in the Table 1 show that by-product-free synthesis of zinc oxide was feasible. The average crystallite size was 16.73 nm, as determined by X-ray phase analysis. The average hydrodynamic radius was then determined when a portion of the sample was placed back into the

Table 1. Crystallographic and Physical Parameters of ZnO Nanomaterial.

Relevant parameters of ZnO	
Structure and profile data:	
Formula sum:	$Zn_{1.00}O_{2.00}$
Formula mass / g / mol:	162.75588
Density (calculated) / g/cm3	5.6683
F(000):	76.0000
Weight fraction/ %:	100.000000
Space group (No.):	P 63 m c (186)
Lattice parameters:	
a / Å:	3.2507(2)
b / Å:	3.2507(2)
c / Å:	5.2096(3)
Alpha / °:	90
beta / °:	90
gamma / °:	120
V / 10^6 pm^3	47.67340
Crystallite (rms) Strain / %:	0.414
Crystallite Size / Å:	167.3



a b

c d

Fig. 3. microscopic images of zinc oxide.

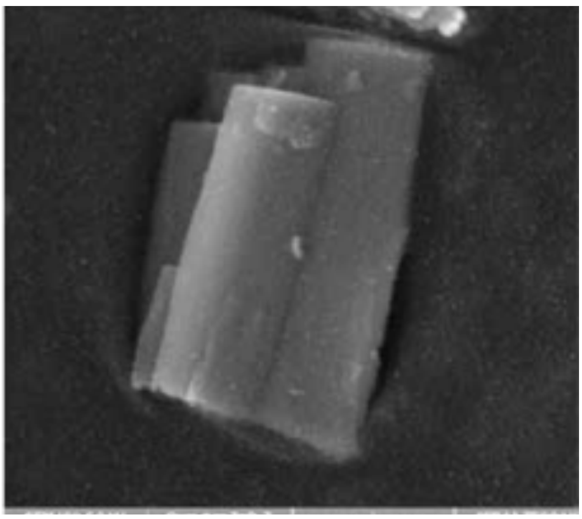


Fig. 4. A micrograph of a zinc oxide crystallite with lamellae.

water, and the findings are depicted in Fig. 2.

It can be stated after studying Fig. 2 that the majority of the produced nanoparticles had a hydrodynamic radius of roughly 70 nm. Using scanning electron microscopy, the generated zinc oxide sample was studied. Figs. 3-7 displays the research's findings.

Analysis of Fig. 3 revealed the presence in the zinc oxide sample of a number of irregularly shaped structures, which tend to be either spherical (a, b, c) or hexagonal star-shaped (d). These aggregates of different structures and levels are formed by lamellar "scaly" crystallites, which are depicted in Fig. 4.

Fig. 5 a, b, and c on the figure depicts the spherical zinc oxide forms that were produced by the above-mentioned approach in the sample. Fig. 5 analysis revealed that primary crystallites of diverse sizes and shapes are what combine to make zinc oxide spheres. The spheroid in Fig. 5a, for instance, is made up of lamellar crystallites, whereas those in Fig. 5b and 5c are cubic or tetragonal and have a diameter of around 30 to 50 nm, respectively (d).

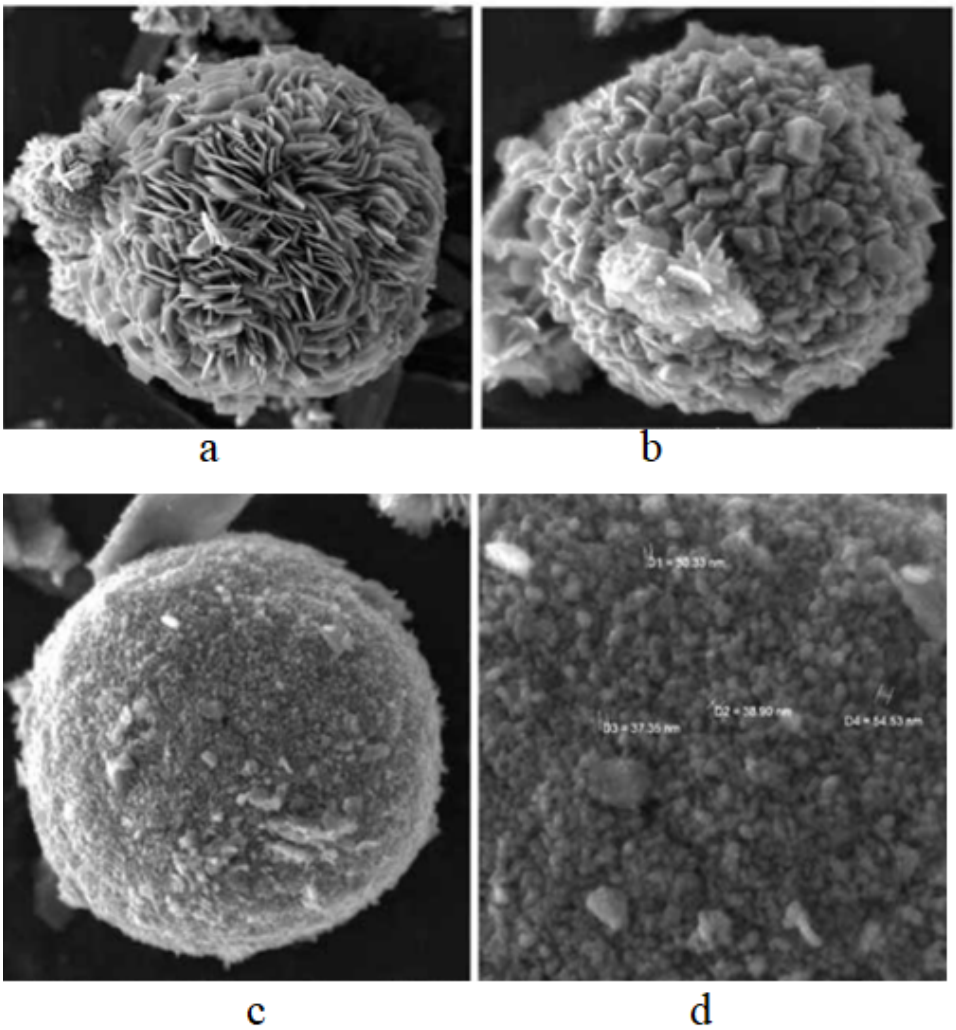


Fig. 5. Micrograph of a zinc oxide sample showing spherical aggregates of zinc oxide in panels a, b, and c, and primary spherical crystallites of zinc oxide in panel d.

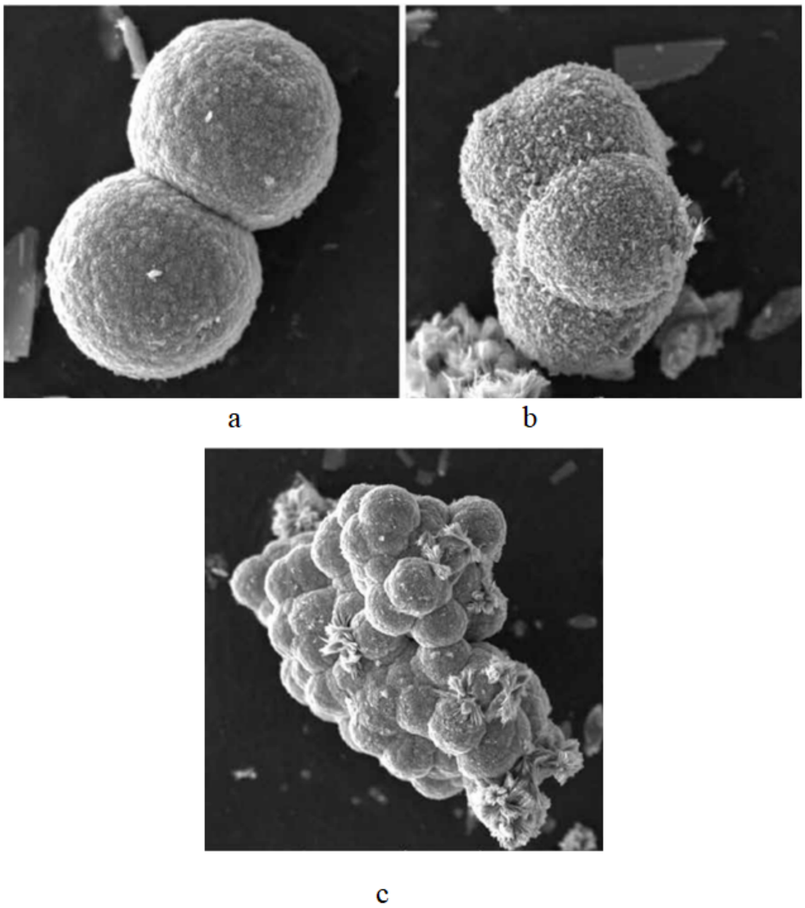


Fig. 6. shows a micrograph of zinc oxide spheres.

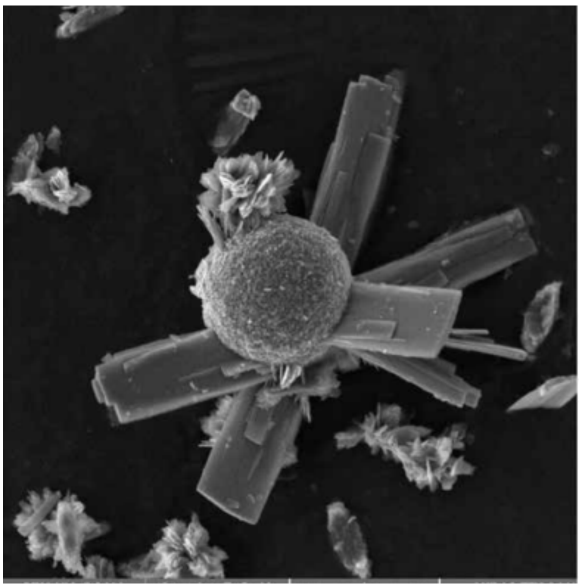


Fig. 7. shows a micrograph of an intricate zinc oxide aggregation.

Also, in the studied sample, aggregates of a higher order were found, presented in Fig. 6 a, b, and c.

It was discovered that cluster-shaped bi-, tri-, and polynuclear aggregates are formed by zinc oxide spheres. In addition to the aggregates previously described, aggregates with a higher degree of polycrystallinity were also discovered in the zinc oxide sample. One of these aggregates is depicted in Fig. 7.

CONCLUSION

As a result, it was clear from the analysis of the data that the approach described above produced a significant number of different agglomerates in varied sizes and shapes in the Nano sized zinc oxide. It can be said that all variations of ZnO can be synthesized under conditions that are thermodynamically advantageous. Furthermore, since no by-products were produced during the synthesis, the zinc oxide nanoparticles produced using this method can be employed to make electronic devices. In this context, process synthesis optimization is required to produce particles of a particular shape.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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