Solution Synthesis of Zinc Oxide Nanomaterials

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Abstract

Zinc oxide (ZnO) is a strong candidate for use in nanotechnology due to its enhanced properties at the nanoscale. A solution synthesis method was coupled with microscopic and spectroscopic characterization, allowing for an investigation of the morphology and prospective applications of ZnO nanostructures which include use in electronic and optical systems. Twenty experimental trials yielded ZnO nanostructures between approximately 40 nm and 1 μm, consistent with expectations based on quantum properties and prior literary work. The use of tin (Sn) as a structural dopant was also employed in exploring the properties of ZnO nanostructures. Characterization of the structures revealed potential electro-optical and nuclear applications. Based on comparisons with prior work and accepted standards.

1 Nanotechnology and Zinc Oxide

The fabrication of structures at the nanoscale is among the most rapidly evolving scientific pursuits today. Developments in nanotechnology have significant implications across a wide variety of fields, including information technology, energy, environmental science, medicine, homeland security, food safety, and transportation [1]. From chemical sensors to fission reactor coolant, and from specialized transistors to optical waveguides and cavities, nanotechnology's presence is growing, which necessitates the study of materials that can meet a diverse set of needs [2],[3].

The versatile properties of zinc oxide (ZnO) make it a strong candidate for nanoscale applications. Various ZnO nanostructures have been proposed for use in specialized transistors and lasers, variable electronics, field emission of various types of radiation, chemical and biological sensing, and nanoscale machines [4]. The structures have also proven invaluable in the development of advanced nano-research tools, including atomic force microscopy. In addition, ZnO nanostructures are already in use in a number of consumer applications, such as the enhancement of UV radiation protection in sunscreen and the addition of anti-bacterial properties to cotton fabrics [5],[6]. The experimental synthesis of nanoscale batteries and electrical generators has also enabled the use of nanotechnology across the energy industry, enabling significant optimization in the area of energy transport [7].

This wide variety of use cases makes zinc oxide a prime target for further research. One of the primary governing factors of the properties of ZnO nanomaterials is their structure [8]. Solution synthesis is used to conduct an accessible exploration of the effect of various factors on the structure of ZnO nanomaterials, as well as their potential applications.

2 Characteristics of Zinc Oxide

2.1 Bulk Properties and Crystal Structure

Zinc oxide is a II-VI semiconductor compound with a number of properties that make it viable in electronic and optical applications [8]. It has a wide bandgap (3.37 eV), which enables visible light emission, and a large exciton binding energy (60 meV), which allows
for room-temperature excitonic emission [9]. Excitons, which are the bound state of an excited electron attracted to the positive hole it leaves behind, enable ZnO to emit light at room temperature [10]. UV laser applications are made possible by a decrease in the lasing threshold, caused by excitonic recombination [11]. A large refractive index of 2.008 and optical cavities in the structure of ZnO allow it to be used as an optical waveguide for directional lasing [8] [9]. ZnO typically has a wurtzite crystal structure composed of bonded \([\text{Zn-O}]_4^{6-}\) tetrahedrons [12] (Fig. 1). This lacks an inversion center and is therefore non-centrosymmetric, leading to the induction of a dipole across the entire crystal (Fig. 2). The resulting piezoelectric and pyroelectric properties enable ZnO to generate a charge if mechanical stress or heat is applied [13].

2.2 Quantum Properties

When observed at the nanoscale, ZnO develops a set of specialized properties as a result of the increased impact of quantum interactions.

The primary interaction in question is quantum confinement, the alteration of electro-optical properties that occurs in samples of substances at the nanoscale. Quantum confinement is largely the result of the dual particle-wave nature of electrons. At the nanoscale, the dimensions of ZnO substructures begin to approach the de Broglie wavelength of electrons. The motion of the electrons is therefore confined to a radius within one, two, or three dimensions [14]. This effect produces fluctuations in the ZnO nanostructures' electrical and optical characteristics that yield several novel properties.

2.3 Structural Properties

One of the most defining aspects of ZnO nanomaterials is their wide range of morphologies. Among these are nanotubes, nanobelts, nanospirals nanohelices, nanocombs, nanostars, nanocages, nanotubes, nanofluids, and nanorods.

The fundamental ZnO nanostructure is the nanorod (also referred to as nanowires, in thinner variations). Generally taking on an elongated hexagonal shape, nanorods have a "I-D" structure, and are among the simplest nanostructures [15]. As such, they frequently serve as a component of other, more complicated structures. That said, the electro-optical properties of ZnO, combined with the versatile nature of the nanorod make it a prime subject of research and investigation, as it is easily adaptable to specific use cases [9]. As a result, nanorods are the primary morphological class investigated in this paper.

2.4 Solution Synthesis

The solution synthesis of ZnO nanomaterials offers the advantage of occurring at lower temperatures (< 100 °C) than conventional synthesis methods [12]. Unlike vapor transport processes, this method involves aqueous solutions of reactants that ultimately form a precipitate that is deposited on a substrate. Thermal treatment is then applied to obtain the desired nanostructures. Since solution synthesis is affected by many parameters such as species concentration, synthesis temperature and solution basicity, the morphology of the resulting ZnO nanostructures can vary greatly [16]. The work of Liu et al. has demonstrated the viability of solution synthesis using zinc nitrate and urea [12]. A proposed reaction mechanism is described as follows:

1. \( \text{CO}(\text{NH}_2)_2 + 3\text{H}_2\text{O} \rightarrow \text{CO}_2\uparrow + 2\text{NH}_3\text{H}_2\text{O} \)

2. \( 3\text{Zn}^{2+} + \text{CO}_3^{2-} + 4\text{OH}^- + \text{H}_2\text{O} \rightarrow \text{ZnCO}_3\cdot2\text{Zn(OH)}\cdot\text{H}_2\text{O} \cdot \uparrow \)

3. \( \text{ZnCO}_3\cdot2\text{Zn(OH)}\cdot\text{H}_2\text{O} \rightarrow 4\text{ZnO} + \text{H}_2\text{O} + \text{CO}_2\uparrow \)
The use of urea provides fine control of the basicity of the solution and the production of CO₂, which reacts with water to form the carbonate ion according to the following reaction:

\[ \text{CO}_2 + \text{H}_2\text{O} \rightarrow 2\text{H}^+ + \text{CO}_3^{2-} \]

Additional thermal treatment after the formation of the basic zinc carbonate precipitate allows for the formation of the desired ZnO nanostructures. The controlled release of OH⁻ ions enables the formation of nanoscale crystals rather than bulk material. A further degree of control can be obtained by changing the solvent during synthesis.

2.5 Doping

Doping of ZnO nanostructures involves the incorporation of certain ions into the synthesized materials and can be used to form various unique structures and enhance electrical properties. Spinels of ZnO nanomaterials are also of interest. The resulting structures are shown and compared with tin (IV) oxide in Figure 3.

Solution synthesis of zinc stannate is possible using tin chloride and offers a lower growth temperature than other vapor transport methods [17]. This synthesis process occurs through the following reaction mechanism:

\[ 8\text{OH}^- + 2\text{Zn}^{2+} + \text{Sn}^{2+} \rightarrow \text{Zn}_2\text{SnO}_4 + 4\text{H}_2\text{O} \]

![Zn₂SnO₄ and TiO₂](image)

The SnCl₂ must be the last reactant added before heating in order to minimize the reaction with water as shown below:

\[ \text{SnCl}_2 + \text{H}_2\text{O} \rightarrow \text{Sn(OH)}\text{Cl}_4 + \text{HCl} \]

3 Solution Synthesis of Zinc Oxide

3.1 Principles of Synthesis Method

Zinc oxide nanostructures were synthesized by heating solutions of zinc nitrate hexahydrate and urea on a hot plate. An initial test trial was conducted by heating 200 mL of 0.001 M urea and zinc nitrate solution at a relatively low temperature of 60 °C for three hours. Thereafter, the concentrations of reactants, reaction temperatures, and durations were varied for each trial according to Table 1. Subsequent trials used 40 mL solutions to enable the necessary heat transfer and facilitate the desired reaction. Two copper wires were placed in each solution to act as substrates for the deposition of the intermediate basic zinc carbonate (ZnCO₂Zn(OH)₂H₂O) precipitate. Aluminium foil was used to cover each solution to prevent evaporation of liquid during the heating process. Afterwards, the copper wires were extracted from the solutions and subjected to thermal treatment for 40 minutes in an oven to complete the formation of the nanostructures.

3.2 Implementation of Doping

Differing concentrations of stannous chloride dihydrate (SnCl₂·2H₂O) were introduced into urea and zinc nitrate solution according to Table 2. The method employed in these trials was consistent with the procedure of the previous trials, sans the addition of tin.

3.3 Analysis

The synthesized ZnO nanostructures and ZnO-based spinels were characterized by field emission scanning electron microscopy (FESEM) and energy-dispersive spectroscopy (EDS). Images were obtained across all regions of the wires using a Zeiss Sigma FESEM, to ensure accurate characterization of the structures. A wide range of magnifications were used to explore these formations individually and across a surface. An Oxford Instruments X-Max EDS was used in tandem with the FESEM to obtain composition profiles.
Table 1: Experimental Matrix for ZnO Trials

<table>
<thead>
<tr>
<th>#</th>
<th>Urea (mol/L)</th>
<th>Zinc Nitrate Hexahydrate (mol/L)</th>
<th>Temp. (°C)</th>
<th>Time (min)</th>
</tr>
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<tbody>
<tr>
<td>1</td>
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<td>240</td>
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<tr>
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<td>240</td>
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<tr>
<td>5</td>
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<td>0.01</td>
<td>90</td>
<td>240</td>
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<td>90</td>
<td>240</td>
</tr>
<tr>
<td>7</td>
<td>0.01</td>
<td>0.005</td>
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<td>240</td>
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<td>0.01</td>
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<tr>
<td>16</td>
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<td>0.01</td>
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</tr>
<tr>
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<td>360</td>
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</table>

Table 2: Experimental Matrix for Doped Trials

<table>
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<th>#</th>
<th>Urea (mol/L)</th>
<th>Zinc Nitrate (mol/L)</th>
<th>Stannous Chloride (mol/L)</th>
<th>Temp (°C)</th>
<th>Time (min)</th>
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<td>0.01</td>
<td>0.02</td>
<td>90</td>
<td>240</td>
</tr>
</tbody>
</table>

3.4 Equipment

After the use of a 250 mL Pyrex beaker in the first trial, 50 mL Pyrex beakers were used in all subsequent trials. Urea crystals of greater than 98% purity, zinc nitrate hexahydrate crystals of reagent grade purity, and tin chloride dihydrate crystals of 95% purity were supplied by Fisher Scientific.

4 Analysis of Synthesized Structures

4.1 Results of Experimental Trials

Twenty trials were conducted over the course of the experiment, each with its own unique configuration of the variable factors—reactant concentration, temperature, and synthesis duration—as depicted in Tables 1 and 2. A number of ZnO nanostructures—ranging in size from approximately 40 nm to 1 μm—were produced. Three of the trials were conducted with the addition of Sn as a dopant, yielding modified, rectangular-prismatic nanorods. With the exception of a small subset of the trials, the nanostructures synthesized tracked with both the expected theoretical outcomes and precedent from literature.

4.1.1 Initial Trial

Previous investigations into the formation of zinc oxide nanorods demonstrated the effectiveness of copper as a substrate [18]. As shown in Figure 4a, the trials were performed on an uncoated copper wire substrate in order to facilitate the reaction.

The first trial yielded partially formed nanoparticles, shown in Figure 4b, that lacked the hexagonal wurtzite structure generally characteristic of zinc oxide nanorods synthesized in solution. This was likely due to the relatively low concentration of the zinc nitrate hexahydrate and urea in solution and to the relatively low temperature of the system. Each nanoparticle had a highly irregular structure and a diameter of about 100 nm, and some appeared to have a roughly formed hexagonal basal plane, as shown in Figure 4c.

Two possible explanations exist for the presence of the white particles at the vertices of the nanoparticle pictured. The use of high-energy electrons in the FESEM process combined with the extreme conductivity of the samples at hand may have created an acute buildup of charge on the vertices of the structure. Due to the image production mechanism of the FESEM, overcharging of the sample registered as white areas on the image. Alternatively, zinc molecules
formed during the reaction may have attached to the vertices of the particles during the reaction. The data collected were inconclusive to definitively support either postulate.

### 4.1.2 Variation of Urea Concentration

The experimental conditions of subsequent trials were adjusted upward to produce more fully formed hexagonal nanorod structures. The next set of trials (2-5 in Table 1) were focused on variation of the concentration of urea in the solution, with time, temperature, and the concentration of zinc nitrate hexahydrate held constant.

Even the lowest experimental concentration of urea (0.001 M) yielded significantly more-developed nanorods due to the increase in the time and temperature of the system relative to those of the first trial. The nanorods had developed a rough hexagonal structure with indistinctly defined planar growth facets, but the basal plane of the nanorods remained blunt, as shown in Figure 5a.

Figure 5b depicts a linear formation of individual nanorods of varied lengths and diameters along a fissure in the copper wire substrate. The density of nanorods in this area may be due to the relatively high surface area of the fissure. The high concentration of urea in the solution of this trial resulted in distinct, well-formed hexagonal nanorods. As the concentration of urea was increased, the nanorods increased in both diameter and length.

and their hexagonal structure and planar facets became more easily discernible. This is possibly an effect of urea’s ability to regulate the rate of synthesis, and therefore the definition of the structures produced.

Two trials (2,3) produced relatively large splinter-like structures beneath the nanorods as shown in Figure 6a. Both trials occurred with relatively low concentrations of urea, at 0.001 mol/L and 0.005 mol/L. Each large structure was roughly 20–30 μm in length and appeared to be composed of porous shredded fragments layered over one another. The layered structures had no apparent orientation relative to the substrate and were highly irregular in appearance.

The composition of the structures was confirmed as zinc oxide by utilization of EDS equipment, and the EDS spectrum produced is shown in Figure 6b. The zinc and oxygen peaks are indicative of the elemental composition of the synthesized nanostructures. The additional peaks are likely due to the copper substrate and the carbon tape.
4.1.3 Variation of Zinc Nitrate Concentration

The next set of trials (6-9) focused on variation of the concentration of zinc nitrate hexahydrate in solution, with time, temperature, and the concentration of urea held constant. Both the low (0.001 M) and high (0.015 M) concentrations of zinc nitrate hexahydrate yielded distinctly shaped nanorods with well-defined hexagonal basal facets. However, specific aspects of the structural combinations of nanorods differed between low and high concentrations.

As shown in Figure 7a, the trial that occurred under the lowest concentration produced both nanorods and nanodisks that had hexagonal basal planes and relatively large diameters. The trials with higher concentrations of zinc nitrate hexahydrate did not yield nanodisks [19].

As the concentration of zinc nitrate hexahydrate increased though the trials, the orientation of the nanorods became more and more regularly perpendicular to the surface of the substrate. Figure 7b depicts a section of nanorods produced in high concentration conditions that were much more regularly oriented relative to those pictured in Figure 8a. The high concentration conditions also yielded irregular structures attached to the basal facets of the nanorods that appear to be composed of nanoparticles compressed into heterogeneous planar forms above the nanorods, as shown in Figure 7c. However, the mechanism of neither the planes’ nor the compressed shapes’ formation could not be definitively concluded.

4.1.4 Variation of Temperature

The next set of trials (10-12) specifically varied the temperature of the experimental system in order to confirm the effectiveness of the temperature utilized for the rest of the trials. As expected, nanorods formed at temperatures lower than 90°C were much less fully developed. Above 90°C, the temperature would have had a risk of reaching the boiling point of the solution due to the margin of error inherent in the experimental process. These findings confirmed the 90°C heating conditions employed for the other sets of trials performed.
4.1.5 Variation of Synthesis Duration

The next set of trials (13-17) were primarily concerned with the time variable of the experimental process. The nanorods grown for short periods of time resembled those of the first trial, whose nanoparticles were incompletely formed and without discernible wurtzite hexagonal structures. Greater periods of time yielded more substantial growth in the length and diameter of the nanorods.

Figure 8a depicts a collection of nanorods with widely varied structural morphology grown on a darkly colored area on the copper wire. It is postulated that the dark area contained ions that disrupted the growth process, producing the ridges visible in Figure 8a. The basal faces of the nanorods lacked the hexagonal structure of the nanorods produced previously. In addition, nanoscale ridge-like and plank-like structures were visible along the edges of the nanorods, as shown in Figure 8b.

4.1.6 Introduction of Dopant

The final three trials (18-20) employed tin oxide dihydrate as a dopant in order to alter the crystalline structure of the nanorods, altering the ratios of the reactants in order to morphological differences. As shown in Figure 9a, the zinc tin oxide nanorods formed had rectangular basal faces and were covered with small spherical zinc tin oxide nanoparticles. EDS analysis suggests the formation of the desired spinels. The EDS spectrum shown in Figure 9b has characteristic peaks for zinc, tin, and oxygen. The other peaks for chlorine, carbon, and nitrogen are likely caused by the remaining particles of the reactants and the carbon tape or the inherent background properties of the EDS.

4.2 Correlation to Theoretical Principles

The results of the experiment offered numerous experimental validations of the theoretical principles governing ZnO nanostructures and their solution synthesis.

In keeping with literature descriptions, the non-dopant trials produced ZnO nanostructures that demonstrated the expected hexagonal prismatic morphology, within acceptable variation. Similarly, the Sn-doped trials—while not yielding structures as defined as the non-dopant trials—produced viable samples of the expected, rectangular-prismatic structures.

Adding in Sn as a dopant had the expected impact, modifying the structure of the nanomaterials in line with the prior work of
Barth, et al. While the dopant structures lacked the morphological definition of the earlier trials, they were nevertheless easily identifiable as corresponding to the literature samples of doped ZnO nanostructures [20].

Increased concentrations of urea produce more structurally defined nanorods with significantly greater lengths and diameters, while increased concentrations of zinc nitrate hexahydrate produced more perpendicularly oriented nanorods. Also, there appears to be a positive correlation between the length and diameter of the nanorods and the time and temperature of the experimental apparatus. It was concluded that the temperature most suitable for zinc oxide nanorods solution synthesis methods was 90°C. In order to prevent unintended boiling of the solution due to experimental error. The effects of varied temperature, reactant concentration, and synthesis duration were all evidenced by the variation in the structures generated during the initial, non-dopant trials. As expected, altering each of these variable factors had a quantifiable impact on the dimensions of the nanorods produced, yielding cross-sectional diameters that spanned a range from approximately 100 nm, to just under 1 μm. Similarly, the lengths and face sizes of the also fluctuated based on the modulation of the aforementioned factors.

In several instances, areas of electronic charging were noted on the surface of the synthesized ZnO nanostructures, corresponding to morphological irregularities in the structures. This supports the observation of Hong, et al. that the electrical conductivity of the nanostructures can be influenced by the presence of deformities on their surfaces.

Also keeping in line with theoretical postulate was the observation of excess ZnO nanoparticles suspended in the solution at the conclusion of several trials. Structures with diameters of several hundred nanometers were generated in numerous trials, with certain trials yielding particles of approximately 43 nm in diameter. Based on the work of Buongiorno, et. al. these nanostructures would be viable candidates for use in critical heat flux (CHF) modulation of fission reactor cores, preventing overheating through suspension in nanofluid [2].

### 4.3 Completion of Experimental Objectives

The two primary experimental objectives – the exploration of the solution synthesis method and the investigation of the applications of ZnO nanostructures – were both successfully achieved.

Over the course of twenty experimental trials, the function, underlying chemistry, and methodology of the solution synthesis process were thoroughly investigated, as demonstrated by the data and observations. Variation of synthesis duration reactant concentration, and temperature aided in obtaining a fuller understanding of the process, as did the addition of dopants to certain trials, for comparative purposes.

Based on the structures obtained, a number of potential applications have been identified. As previously described, the synthesized ZnO nanostructures are candidates for use in fission reactors, where they can reduce overheating. The nanorod structures obtained largely matched the characteristics expected of ZnO nanorods, which lend themselves to applications in specialized nanoscale electronic and optical usage.
4.4 Experimental Problems & Shortcomings

There are several factors that can be improved upon to enhance the accuracy of the experimental procedure. Longer periods of time in which to operate the FESEM would remove the need for multiple calibrations, reducing inconsistencies by enhancing precision. In addition, increased access to the EDS would permit characterization of all trials, rather than just the three trials out of twenty that were actually characterized, permitting accurate verification of the elemental composition of the nanostructures of all trials.

5 A Viable Method of Exploration

The procedure set forth herein describes a simple yet effective method for using solution synthesis to produce zinc oxide nanomaterials, explore their morphologies, and employ characterization and analysis techniques to determine their potential applications. Through twenty experimental trials, ZnO nanorods ranging in size from approximately 40 nm to 1 μm were synthesized and characterized. Three of these trials were conducted with the addition of tin as a dopant, producing morphologically varied spinels. In certain instances, abnormal morphologies were observed. Overall, the synthesized ZnO nanomaterials followed the theoretical and literature-based expectations, demonstrating the general reliability of the solution synthesis method.

The characterization of the ZnO nanostructures suggests a number of potential applications, ranging from tunable nanoelectronics and optics to critical heat flux modulation in nuclear fission reactors. The experimental investigation of these potential applications is a prime subject for future work, as further investigation is required to determine the performance of the ZnO nanostructures in their various prospective roles. Testing of the conductive and switching capabilities of the synthesized ZnO nanorods using a nanoelectronic system will help ascertain their ability to serve as field emission transistors. Similarly, examining the ability of the structures to act as an optical waveguide will quantify their ability to serve as lasing chambers and UV emitters. Suspending the synthesized nanostructures in the aqueous coolant of a nuclear fission reactor will allow for measurement of the structures' ability to increase the CHF level and prevent overheating.

The successful synthesis, characterization, and analysis of potential applications represents a comprehensive investigation of the solution synthesis of zinc oxide nanomaterials. With this knowledge, future investigation can be directed towards exploring variations in the solution synthesis method, as well as the applications of ZnO nanostructures.

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References


