Hydrothermal Synthesis of ZnO

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Abstract

This paper outlines explicit highlights of hydrothermal synthesis of zinc oxide nanoparticles. Hydrothermal synthesis permits control of the ZnO nanostuctural morphology and nanoparticle size since the synthesis factors can be fluctuated to influence these features. In this survey, the hydrothermal synthesis factors and their effects on the particulate properties are unraveled.

Keywords': Hydrothermal, synthesis, ZnO, Sintering temperature.

1. Introduction

The investigation of materials on nano-scale has turned out to be central in science and innovation as decrease in a material's size brings forth novel electrical, mechanical, chemical, optical properties coming about because of surface and quantum confinement [1, 2].

ZnO is a compound semiconductor (transparent to visible light) with a wide band-gap of 3.37eV and high exciton restricting vitality of 60 meV [2, 3]. Synthesis of ZnO thin films had begun during the 1960s when ZnO materials were connected in sensors, transducers and as photocatalysts [1]. ZnO nanaostructures have various innovative applications in optoelectronics, surface acoustic wave channels or filters, photonic crystals, photodetectors, light radiating diodes, photodiodes, gas sensors, optical modulator waveguides, solar cells [4, 5] and varistors [1, 3, 6].

Nanomaterials are typically classified into three groups: 0-dimensional, 1-dimensional, and 2-dimensional. 0-dimensional nanostructures, referred to as quantum dots or nanoparticles with an aspect ratio near unity, have been extensively used in biological applications [7, 8]. 2- dimensional nanomaterials, such as thin films, have also been widely used as optical coatings, corrosion

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protection, and semiconductor thin film devices. Zinc oxide crystals can be prepared in the form of nanopowders, films, ceramics, and single crystals [9]. One-dimensional (1D) semiconductor nanostructures such as nanowires, nanorods (short nanowires), nanofibres, nanobelts, and nanotubes have been of intense interest in both academic research and industrial applications because of their potential as building blocks for other structures [10]. 1-D nanostructures are useful materials for investigating the dependence of electrical and thermal transport or mechanical properties on dimensionality and size reduction (or quantum confinement) [11]. They also play an important role as both interconnects and functional units in the fabrication of electronic, optoelectronic, electrochemical, and electromechanical nanodevices [12]. Among the one-dimensional (1D) nanostructures, zinc oxide (ZnO) nanowire is one of the most important nanomaterials for nanotechnology in today's research [13].

2. Basic synthetic approaches and growth mechanisms

The synthetic techniques used for the synthesis of ZnO are broadly divided into three types: chemical, biological, and physical methods [14].

In the physical (also called gaseous phase [6]) method, physical forces are involved in the attraction of nanoscale particles and formation of large, stable, well-defined nanostructures[15]. This method of synthesis uses gaseous environment in closed chambers with high temperatures from 500°C to 1500°C to carry out the synthesis. Its examples includecolloidal dispersion method, vapor condensation, amorphous crystallization, physical fragmentation, vapour phase transport (which includes vapour solid (VS) and vapour liquid solid (VLS) growths), physical vapour deposition (PVD), chemical vapour deposition (CVD), metal organic chemical vapour deposition (MOCVD), thermal oxidation of pure zinc and condensation, microwave assisted thermal decomposition and many others are expensive and complicated [1].

Biosynthesis of nanoparticles (NPs) is an approach of synthesiswhich uses microorganisms and plants for the formation of ZnO NPs having biomedical applications. This approach is an environment-friendly, cost- effective, biocompatible, safe, green approach [16]. Green synthesis can be carried out through plants, bacteria, fungi, algae etc. They allow large scale production of ZnO NPs free of additional impurities [17].

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The chemical (also called solution phase)method is done in water with growth temperatures less than the boiling point of water which makes it simple and has favorable growth conditions[7]. Chemical reactions in aqueous systems are usually considered to be in a reversible equilibrium, and the driving force is the minimization of the free energy of the entire reaction system, which is the intrinsic nature of wet chemical methods [2, 6]. ZnO solution phase synthesis methods include: Sol-gel method (a chemical solution processused to make ceramic and glass materials in the form of thin film, fibers or powder), Co-precipitation method, Hydrothermal method (method of synthesis of single crystal that depends on the solubility of minerals in hot water under high pressure [2, 18, 19]), Pulsed laser deposition method (a physical vapour deposition technique where a high-power pulsed laser beam is focused inside a vacuum chamber to strike a target of the material that is to be deposited)[1, 2, 20].

Hydrothermal method unlike other methods is attributed with the following favorable circumstances which makes it more appropriate and suitable than different other methods for ZnO synthesis: utilization of simple equipment, minimal effort and low cost, catalyst-free growth, enormous area uniform circulation of as-grown particles [21], less perilous and natural agreeableness, does not require the utilization of natural solvents or extra handling of the item (crushing and calcinations) which makes it basic, the likelihood of completing the synthesis at low temperatures nonattendance of complex vacuum setup [22],assorted shapes and dimensions of the subsequent crystals relying upon the piece of the beginning mixture and the procedure temperature and pressure, high level of crystallinity of the as-orchestrated product, high purity of the material acquired, it very well may be utilized on large areaand/or flexible substrates, can be utilized for creation of unsupported nanostructures, accomplishment of exact control of the nanostructure size and direction [21]. Therefore, it has been a subject of exceptional research lately.

2.1 Zinc oxide crystal structure

The ZnO crystal is hexagonal wurtzite and exhibits partial polar characteristics [48, 49] with lattice parameters a = 0.3296 and c = 0.52065nm [4, 49]. The structure of ZnO can be described as a number of alternating planes composed of tetrahedrally coordinated O^{2-} and Zn^{2+} stacked alternately along the c-axis. The piezoelectric and pyroelectric properties are ascribed to the absence of inversion symmetry in ZnO which gives rise to tetrahedral coordination [49].

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suggested that Na^+ is attracted by the OH^- around the nanocrystal and forms a virtual capping layer; thus, inhibiting the nanocrystal growth.

$$Zn^{2+} + 2OH^- \longleftrightarrow Zn(OH)_2 (1)$$

$$Zn(OH)_2 + 2OH^- \longleftrightarrow [Zn(OH)_4]^{2-} (2)$$

$$[Zn(OH)_4]^{2-} \longleftrightarrow ZnO_2^{2-} + 2H_2O \quad (3)$$

$$ZnO_2^{2-} + H_2O \longleftrightarrow ZnO + 2OH^- (4)$$

$$ZnO + OH^- \longleftrightarrow ZnOOH^- \quad (5)$$

The main reactions involved in the growth are illustrated in the above equations [6, 9].

3.2 Growth mediated by hexamethylenetetramine (HMTA) aqueous solution

The most commonly used chemical agents in the existing literature for the hydrothermal synthesis of ZnO nanoparticles are $Zn(NO_3)_2$ and HMTA. In this case, $Zn(NO_3)_2$ provides Zn^{2+} ions required for building up ZnO nanowires. When HTMA (a nonionic cyclic tertiary amine [9]) and $Zn(NO_3)_2$ are chosen as precursor, the chemical reactions can be summarized in the following equations [24].

$$(CH_2)6N_4 + 6H_2O^- \rightarrow 6HCHO + 4NH_3 (1)$$

$$NH_3 + H_2O \longleftrightarrow NH_4+ + OH^- (2)$$

$$2OH^- + Zn^{2+} \rightarrow Zn(OH)_2 (3)$$

$$Zn(OH)^{2-} \rightarrow ZnO + H_2O (4)$$

H_2O molecules in the solution, not at all like for the instance of alkali-mediated growth, provides and gives O^{2-} ions. It has been recommended that HMTA amid the ZnO nanowire growth goes about as a bidentate Lewis base that directs and extends two Zn^{2+} ions and furthermore goes about as a frail base and pH buffer. Contingent upon the given pH and temperature, Zn^{2+} can exist in a progression of intermediates, and ZnO can be framed by the lack of hydration of these intermediates [6]. On the off chance that a ton of OH^- is created in a brief period, the Zn^{2+} ions in the arrangement will

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precipitate out rapidly because of the high pH condition, and, along these lines, Zn^{2+} would contribute little to the ZnO nanowire development and in the long run outcome in the quick utilization of the supplement and forbid further development of the ZnO nanowires [25]. In this manner, the convergence of OH^- ought to be controlled in the solution to keep up low supersaturation levels amid the entire nanowire development process [24].

Table 1.0: **Summary of hydrothermal method of obtaining Zinc Oxide with different morphologies and sizes**

S /N	Precursors	Synthesis conditions	Properties and applications	Reference
1	Nitrate.ethylene glycol, NaOH	reaction temperature:180 °C	flower 5µm and rods	[26]
2	Acetate dihydrate. ethylene glycol	reaction temperature:160 °C	sphere 5µm and nanorods	[27]
3	Acetate dihydrate. ethylene glycol	reaction temperature: 180 °C	sphere 5µm and nanocrystallites 10 nm	[28]
4	Acetate dihydrate. ethylene glycol	reaction temperature: 160 °C	sphere0. 12 µm and ovoids 10 nm	[29]
5	$ZnCl_2$, NaOH	reaction: 5–10 h, 100–220 °C in teflon-lined autoclave	particle morphology: bullet-like (100–200 nm), rod-like (100–200 nm), sheet (50–200 nm), polyhedron (200–400 nm), crushed stone-like (50–200 nm)	[30]

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6	tetramethylammonium hydroxide (TMAH). Ethanolic solution of zinc acetate dihydrate	heating temperature: room temperature	particle morphology snowflakes sized from 10 to 20nm	[31]
7	$Zn(CH_3COO)_2$, NaOH, HMTA (hexamethylenetetramine)	reaction: 5–10 h, 100–200 °C, HMTA concentration: 0–200 ppm	particlesize and morphology: diameter 55–110 nm, spherical shape	[32]
8	$Zn(CH_3COO)_2$, $Zn(NO_3)_2$, LiOH, KOH, NH_4OH	reaction: 10–48 h, 120–250 °C	particlesize and morphology:hexagonal (wurtize) structure, microcrystallites: 100 nm–20 μm	[33]
9	$Zn(CH_3COO)_2$, NH_3, zinc 2-ethylhexanoate, TMAH, ethanol, 2-propanol	reaction time and pH of solution: 15 min, 2–72 h, final pH: 7–10	particles with irregular ends and holes; aggregates consist particles of 20–60 nm, *BET* 0.49–6.02 m^2/g	[34]
10	Tetramethylammonium chloride, 4-picoline N-oxide, HCl, toluene, ethylenediamine	reaction time and temperature: 24–100 h, 180 °C	particlesize and morphology hexagonal structure, nanorods (40–185 nm), nanoparticles (24–60 nm)	[35]

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	(EDA), tetramethylethylene diamine (TMEDA)			
11	$Zn(NO_3)_2 \cdot 2H_2O$, NaOH, CTAB	reaction time and temperature: 120 °C for 24 h	particlesize and morphology-nanorods with diameters ~0.5–1 μm and lengths ~5–6 μm (without additive). nanoflowers with diameter ~4–5 μm (with additive)	[36]
12	$Zn(CH_3COO)_2$, $Zn(NO_3)_2$, ethanol, imidazoliumtetrafluoroborate ionic liquid	reaction temperature: 150–180 °C; drying temperature: 80 °C in vacuum oven; calcination temperature: 500 °C	particlesize and morphology:hexagonal (wurtize) structure, hollow microspheres (2–5 μm) consisted nano-sized particles and contained channels (10 nm); hollow microspheres consisted of nanorods (~20 nm), flower-like microspheres (2.5 μm)	[37]
13	zinc acetylacetonate, methoxy-ethoxy- and n-butoxyethanol, zinc oxinate	precursor concentration: 2.5–10 wt%; heatingtime and temperature: in 800 W microwave, 4 min; drying temperature: 75 °C in air	particlesize and morphology: zincite structure; average crystallite size: 9–31 nm; diameter: 40–200 nm; BET: 10–70 m^2/g	[38]

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reaction temperature, mechanical stirring duration, heatingrate, additives or template and substrate type.

The concentration of precursors affects ZnO nanoparticle by advancing its formation. In this manner formation of the nanoparticles (nucleation rate) increases with increase in alkaline concentration of the precursors [51]. Varying the time of reaction influences the diameter of the as-synthesized ZnO particle such that the diameter increases with increase in reaction time and vice-versa. The rate at which heating is ramped up directly influence the kinetics of nucleation and precipitation severely. The reaction temperature guarantees that a ZnO phase is formed changing from 1D to 2D and then to 3D as reaction temperature is increased from 90-100 °C, and from 120-160 °C, and then from 180-200 °C [50]. A high temperature and heating cause increase in the average diameter of the as-synthesized ZnO nanoparticle [50, 51].

Table 2.0: **Modification Methods of Zinc Oxide**

Classification of modifiers	**Type of modifying agents**	**Modification effect**
Inorganic compound	Si_2O, Al_2O, $LiCo_2O$, metal ions	Change of surface area and particle size, reduced photocatalytic action of the oxide, improvement of dispersion degree of ZnO particles.
Organic compound	Carboxylic acids, silanes	Introduced characteristic groups on the surface of ZnO and altered its physicochemical properties, increased the compatibility of ZnO with an organic matrix, reduced aggregation of particles and enhanced the long-term stability in an organic matrix, improved ZnO dispersion in rubber matrices.

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Polymer matrices	Poly(ethylene glycol), polystyrene, poly(methyl methacrylate), poly(methacrylic acid), chitin	Improved electrical, thermal and optical properties of ZnO polymer composite

4. Characterization of ZnO

Under general conditions, ZnO is single crystalline and exhibits a hexagonal wurtzite structure. The optical properties could be obtainable from UV-Visible absorption spectroscopic analysis. The structure of ZnO nanowires could be revealed by Light Microscopy, X-ray diffraction (XRD) and scanning electron microscopy (SEM). Further structural characterizations can be carried out by, transmission electron microscopy (TEM), Raman spectroscopy, Scanning Force Microscopy (SFM), high resolution transmission electron microscopy (HRTEM) and Rietveld Refinement [1, 2, 6, 16, and 40].

The composition analysis could be carried out through X-ray absorption spectroscopy (XAS), energy dispersive spectroscopy (EDS), energy dispersive X-ray (TEDX), X-ray fluorescence spectroscopy (XRF), Auger electron spectroscopy (AES), proton induced X-ray emission spectroscopy (PIXE), atomic absorption spectroscopy (AAS), neutron absorption spectroscopy (NAS), Fourier transform infrared spectroscopy (FTIR), X-ray photoelectron spectroscopy (XPS), wave length dispersive spectroscopy (WDS).

5. Conclusion

This survey paper portrays ZnO hydrothermal synthesis for ZnO nanoparticle development and formation utilizing diverse zinc salts and alkali solutions, its focal points over different other methods and its characterization techniques. The hydrothermal synthesis technique is cordial, costless, straightforward and effective and it has gotten expanded consideration because of the extreme utilization of ZnO in research and industry holding to its remarkable highlights. A blend of zinc nitrate and hexamine is the most well known decision of antecedent for hydrothermal synthesis. In

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light of this paper, the morphology and dimension of nanostructures incorporated by hydrothermal method are enormously impacted by precursor concentration, pH of solution, reaction time, reaction temperature, mechanical mixing or stirring duration, heating rate, additive or template and substrate type. Thus the need to comprehend the ZnO hydrothermal synthesis to empower designing and refined the as-combined ZnO nanoparticles to wanted prerequisite.

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