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ZnO Nanostructures Synthesized by Chemical Solutions

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Abstract

Nanomaterials have been synthesized using several different techniques. Some of these techniques are sophisticated, expensive and need certain training before use. However, there are other highly efficient methods for preparing nanomaterials that are easy to work with and require no specialized equipment, making them relatively inexpensive routes for synthesis. The least expensive routes are those that are classified as solution-based techniques such as colloidal, sol-gel and microwave-assisted synthesis. The focus of this chapter is on a general description of each technique with recent advances in synthesis, doping processes and applications. Specifically, these processes are discussed in connection with the synthesis of ZnO compounds and its related nanomaterials.

Keywords: ZnO, synthesis, chemical solutions, nanostructures

1. Introduction

An important II–VI semiconductor is ZnO which has been well-studied and applied in a variety of applications. It has a band gap of 3.6 eV and large exciton binding energy of 60 meV. Nowadays this material is considered as one of the most important large band gap semiconductors due to its easy synthesis, stability at room temperature, eco-friendly properties, being a direct band gap material and fast mobility. This material exists in three different crystal phases such as zinc blende, cubic or rock salt and wurtzite or hexagonal. The first two phases are obtained only in certain well-controlled conditions such as certain pressures and

on specific substrates. However, the most common phase under ambient conditions is the wurtzite hexagonal crystal structure shown in **Figure 1**.

Another advantage of this compound is that it can be synthesized and deposited by employing different techniques. Slight variation in process conditions can result in different product morphologies and properties. Since the costs associated with research and industry is always an important consideration, it becomes necessary to use inexpensive and efficient methods to obtain the desired novel nanostructured materials with applications in different fields such as optoelectronics, solar cells, piezoelectric and sometimes in biological materials.

Sol-gel, colloidal solution and microwave-assisted synthesis are techniques that are still important in the synthesis of semiconductor nanomaterials. These techniques share some similar characteristics such as (i) they are relatively inexpensive; (ii) the efficiency of the synthesized materials is high; (iii) process parameters are easily controlled and (iv) these techniques are also well-studied. For these reasons, in this chapter we have focused on a review of these techniques, especially for the synthesis of ZnO, with emphasis on the recent advances in the synthesis of novel nanomaterials and its applications. A general overview of each process is also presented for ease of readability. The synthesized materials have been structurally characterized using X-ray diffraction (XRD) and scanning electron microscopy (SEM). **Figure 2** shows a representative XRD pattern of ZnO. XRD patterns of synthesized material can be compared to reference patterns to determine phase purity or if there is preferential crystal orientation. Most of the time, ZnO is obtained as a polycrystalline film or powder which can be identified by its numerous diffraction peaks at relative intensities. Depending on the processing conditions, single crystal or preferential growth can occur

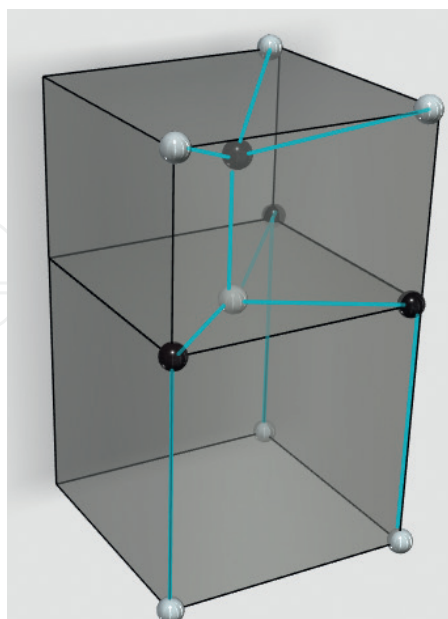


Figure 1. Representation of ZnO wurtzite crystal structure (black and grey balls corresponds to Zn and Oxygen atoms).

in thin films that result in different relative peak intensities or missing peaks compared to the reference pattern. The 2-theta values of the (100), (002) and (101) lines in **Figure 2** of the hexagonal crystal planes are located at 31.770, 34.422 and 36.253° for wurtzite ZnO (Ref. JCPDS card # 36-1451).

Different processing parameters may result in different microscopic product morphologies of ZnO. From SEM, we can observe that this material could be obtained as nanoparticles

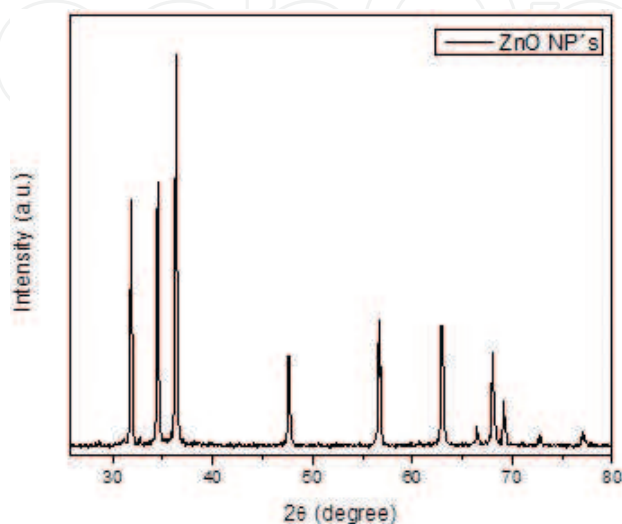


Figure 2. Typical XRD pattern of ZnO nanoparticles.

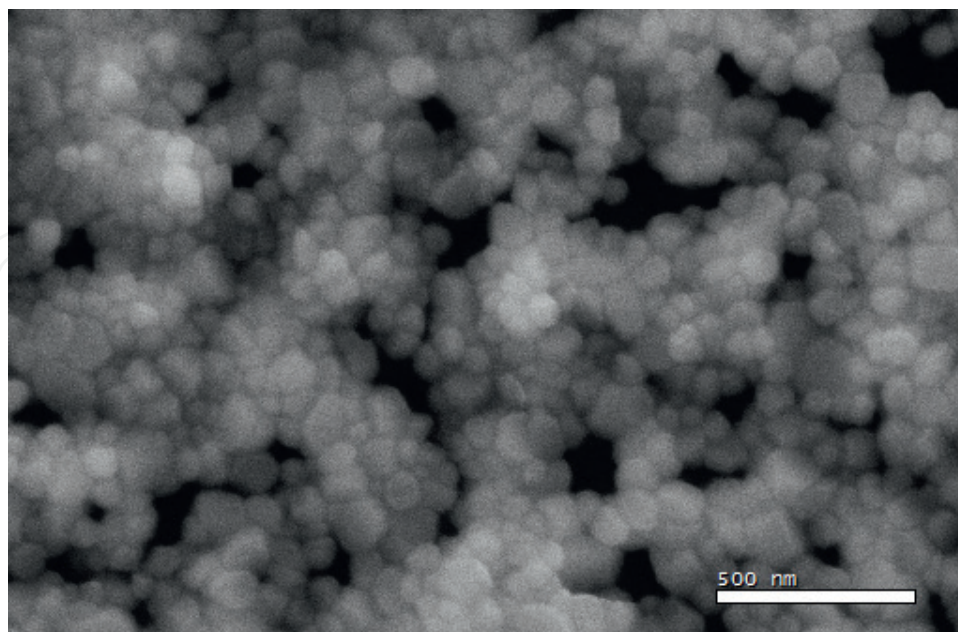


Figure 3. SEM image of ZnO nanoparticles obtained via colloidal synthesis. The scale bar is 500 nm.

(**Figure 3**), polycrystalline (**Figure 4**) and as a nanostructured thin film (**Figure 5**). All of these materials were synthesized under non-extreme conditions using colloidal synthesis to produce the source material. The crystal structure of these materials is the hexagonal wurtzite structure.

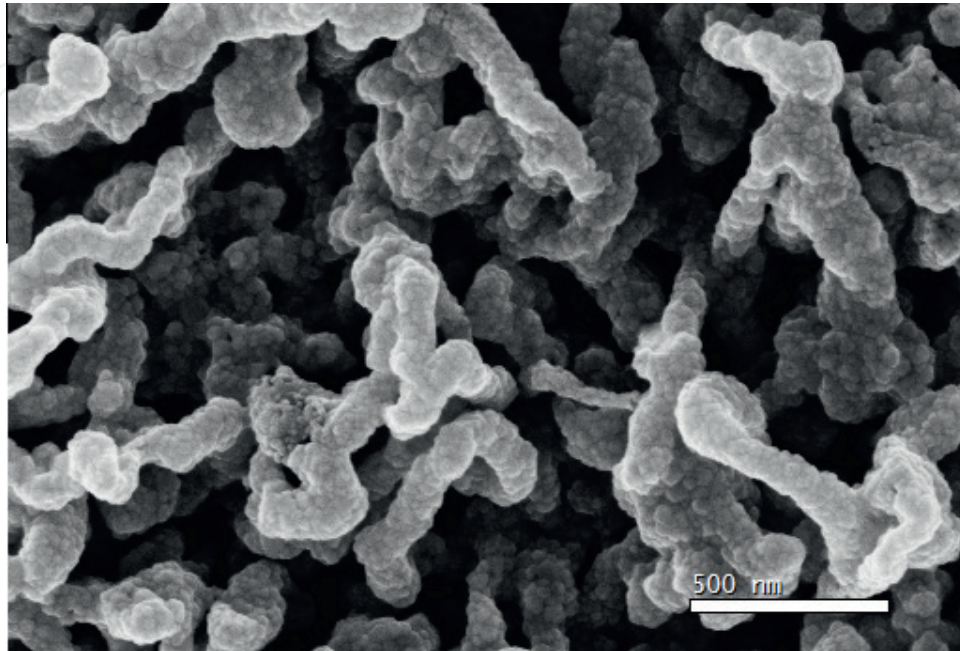


Figure 4. SEM image of polycrystalline ZnO thin film obtained through vacuum evaporation process, colloidal nanoparticles as source were used. The scale bar is 500 nm.

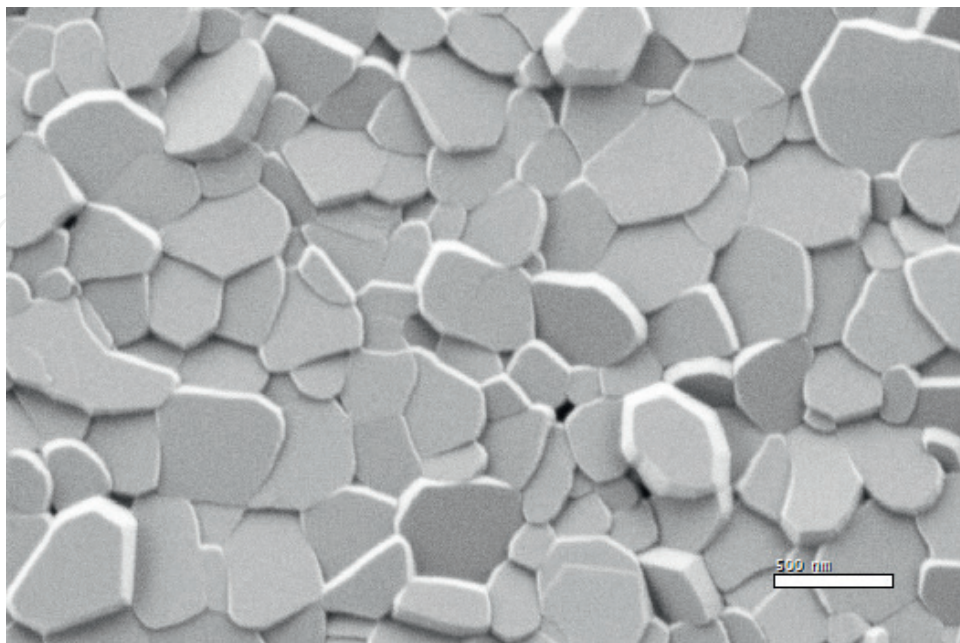


Figure 5. SEM ZnO nanostructures using colloidal nanoparticles as source. The scale bar is 500 nm.

2. Some techniques for synthesizing ZnO nanostructures and nanoparticles

2.1. Sol-gel

The sol-gel process encompasses a variety of precursors, solvents and additives. But in general, the basis of the sol-gel process includes some form of hydrolysis and condensation reactions. In the case of ZnO, usually a zinc salt such as zinc acetate is used with water or an alcohol as the solvent. An example of possible hydrolysis and condensation reactions for ZnO are shown in Eqs. (1) and (2), where Zn(OR)_2 is a soluble salt.



During the hydrolysis reaction, the soluble zinc precursor forms a zinc hydroxide intermediate that is able to condense with other intermediates to grow a zinc oxide inorganic polymer. The final product after drying has an amorphous structure and crystallization of ZnO particles require an annealing step. The morphology of the inorganic network can range from spherical nanoparticles to percolated gels and is highly dependent on the choice of precursors, water content, solute and solvent ratio, aging and additives. The sol-gel process has proven to be an inexpensive and relatively simple method of ZnO nanoparticle synthesis that is tailorable to produce unique nanostructures for different applications.

2.2. Colloidal solution

Colloidal synthesis is another well-known chemical solution method to obtain novel nanomaterials with different morphologies and sizes. All processing conditions involved in the system can be fixed to control nucleation and growth of the materials. The kind of interactions (physical and chemical) between particles include Vander Waals, electrostatic, Ostwald ripening and some other theoretical principles such as Derjaguin, Landau, Venvey and Overbeek theory (DLVO). These interactions can contribute to agglomeration and subsequently precipitation of the particles. Colloidal instability can be prevented through steric stabilization which usually requires a surfactant to maintain the colloidal suspension. Surfactants work in two ways: first, to prevent particulate interactions and second, to prevent the continuous nucleation and growth of particles.

2.3. Microwave-assisted synthesis

Microwave-assisted synthesis is a relatively recent technique that has been used for synthesis of nanomaterials. It has been considered as a promising approach to obtain novel nanomaterials in organic and inorganic fields. Additionally, microwave synthesis is considered as a green process and coheres perfectly to the principles formulated by Anastas et al. related to green chemistry [1].

Often a domestic microwave is used and the synthesis is carried out in solvent-free solutions. This technique allows for rapid and homogeneous heating of the system since energy is transmitted directly through molecular vibrations. The short heating ramp time of microwave synthesis allows for better control of particle size distribution compared to conventional

heating. On the contrary, the extremely high heating rate of microwave-assisted synthesis may cause the boiling point of the solution to increase by a few degree Celsius. Additionally, the microwave susceptibility will vary between different materials and temperatures.

The microwave energy is generated by a magnetron that transforms electrical energy into a strong magnetic field. The electromagnetic energy interacts with the solution, vibrating the molecules and giving sufficient activation energy to the system for chemical reactions to take place in seconds or minutes.

The reaction rate during microwave synthesis can be explained through the Arrhenius equation [Eq. (3)] as follows:

$$K = A e^{-\Delta G/RT} \quad (3)$$

where K is the rate constant, T is the absolute temperature (in Kelvin), A is the pre-exponential factor, a constant for each chemical reaction that defines the rate due to frequency of collisions in the correct orientation, ΔG is the activation energy for the reaction (in Joules) and R is the universal gas constant. Thus, the two parameters affecting the kinetics of a particular chemical reaction are temperature and activation energy.

Bilecka et al. reported that nanoparticle growth can be described using four thermodynamic parameters related to the Arrhenius equation through activation energy [2]. These variables are the activation energies for precursor solvation, monomer formation, nucleation and crystal growth. As with colloidal synthesis, nucleation and growth in microwave synthesis are governed by Ostwald ripening.

3. Synthesis of ZnO nanostructures and nanoparticles via chemical solutions: recent advances

Sol-gel, colloidal and microwave-assisted synthesis are effective techniques to efficiently obtain novel ZnO nanostructures. These techniques are relatively inexpensive and do not require sophisticated laboratory equipment. Additionally, slight variations in precursors or process parameters can produce different morphologies that can be applied in different technological fields.

3.1. Process, materials and precursors

The precursors used in these synthesis routes usually start with a basic salt of Zn, a solvent and a catalyser such as temperature. The Zn precursor must be soluble in the selected solvent such that it can provide the necessary Zn ions to produce ZnO particles. Other reagents may be added in order to substitutionally dope ZnO with metal cations such as Fe, Cu, Co and Ba. Additionally, surfactants may be added to maintain colloidal stability of the product or influence the morphology of the growing particles.

Different precursors used in sol-gel and colloidal techniques from recent publications have been summarized in **Tables 1** and **2**, respectively. The readers are asked to consult the relevant publications for details of these processes.

Precursor	Solvent	Stabilizing agent	Reference	Technique
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$	CH_3OH , $\text{C}_2\text{H}_5\text{OH}$, $\text{C}_3\text{H}_7\text{OH}$, $\text{C}_3\text{H}_7\text{OH}$, $\text{C}_4\text{H}_9\text{OH}$	$(\text{CH}_2\text{CH}_2\text{OH})_2\text{NH}$, $\text{N}(\text{CH}_2\text{CH}_2\text{OH})_3$	Pourshaban et al. [3]	Sol-gel
$\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}/\text{CuCl}$	2-methoxyethanol	$(\text{CH}_2(\text{OH})\cdot\text{CH}_2\cdot\text{NH}_2)$	Joshi et al. [4]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$, $\text{Ba}(\text{NO}_3)_2$	2-methoxyethanol	$(\text{CH}_2\text{CH}_2\text{OH})_2\text{NH}/\text{DEA}$	Kasar et al. [5]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$, $(\text{NH}_4)_2\text{CO}_3$, $\text{Fe}(\text{NO}_3)_3$	Distilled water/ethylene glycol	–	Bahari et al. [7]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$, $\text{Mn}(\text{CH}_3\text{CO}_2)_2 \cdot 4\text{H}_2\text{O}$	Isopropyl alcohol	Urea	Kumar et al. [6]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$, $\text{C}_2\text{H}_3\text{LiO}_2$	$\text{C}_2\text{H}_5\text{OH}$	$(\text{CH}_2(\text{OH})\cdot\text{CH}_2\cdot\text{NH}_2)$	Boudjouan et al. [8]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$, CaCl_2	CH_3OH , $\text{C}_2\text{H}_5\text{OH}$	–	Slama et al. [9]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$, $(\text{CH}_3\text{COO})_2\cdot\text{Co} \cdot 4\text{H}_2\text{O}$	CH_3OH	Mono ethanolamine $(\text{CH}_2(\text{OH})\cdot\text{CH}_2\cdot\text{NH}_2)$	Dhruvash et al. [10]	Sol-gel
$\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$	$\text{C}_2\text{H}_5\text{OH}$	–	Singh et al. [21]	Sol-gel
$\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}/\text{KOH}$	CH_3OH	–	Kim et al. [22]	Sol-gel
$\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$	2-methoxyethanol	$(\text{CH}_2(\text{OH})\cdot\text{CH}_2\cdot\text{NH}_2)$	Tabassum et al. [11]	Sol-gel
$\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}/\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}/\text{AgNO}_3$	$\text{C}_2\text{H}_5\text{OH}$	Diethanolamine (DEA)	Khan et al [12]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$, NaCl	$\text{CH}_3\text{OCH}_2\text{CH}_2\text{OH}$	$(\text{CH}_2(\text{OH})\cdot\text{CH}_2\cdot\text{NH}_2)$	Zhou et al. [30]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$	Isopropyl alcohol	$(\text{CH}_2(\text{OH})\cdot\text{CH}_2\cdot\text{NH}_2)$	Chebil et al. [23]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$, $\text{Cu}(\text{CH}_3\text{COO})_2$	Diethanolamine (DEA)	Agarwal et al. [14]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$	2-methoxyethanol	$(\text{CH}_2(\text{OH})\cdot\text{CH}_2\cdot\text{NH}_2)$	Haarindraprasad et al. [24]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$	Dimethyl formamide	Diethanolamine (DEA)	Bhunia et al. [25]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$, $\text{C}_2\text{H}_7\text{NO}_2$	Distilled water/glacial acetic acid	–	Para et al. [26]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$, $\text{Ga}(\text{NO}_3)_3 \cdot x\text{H}_2\text{O}$	2-methoxyethanol	$(\text{CH}_2(\text{OH})\cdot\text{CH}_2\cdot\text{NH}_2)$	Wang et al [27]	Sol-gel
$[\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}]$	2-methoxyethanol	$(\text{CH}_2(\text{OH})\cdot\text{CH}_2\cdot\text{NH}_2)$	Alfaro et al. [28]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$, LiOH , graphene	$\text{C}_2\text{H}_5\text{OH}/\text{EtOH}$	–	Li et al. [29]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$, $\text{Mg}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$, $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	Isopropyl alcohol	Diethanolamine (DEA)	Das et al. [13]	Sol-gel

Precursor	Solvent	Stabilizing agent	Reference	Technique
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$	1-butanol	$(\text{CH}_2(\text{OH})\cdot\text{CH}_2\cdot\text{NH}_2)$	Demes et al. [31]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$, $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$	Ethanol and chelating with glycerin	Acetic acid	Kose et al. [32]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$, $\text{Li}(\text{CH}_3\text{-COO})_2 \cdot 2\text{H}_2\text{O}$, $\text{Co}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$	$(\text{C}_2\text{H}_5\text{OH})$	$(\text{C}_2\text{H}_6\text{O}_2)$	Bashir et al. [15]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$	Ethanol ($\text{C}_2\text{H}_5\text{OH}$)	$(\text{CH}_2(\text{OH})\cdot\text{CH}_2\cdot\text{NH}_2)$	Ayana et al. [33]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$, $\text{Cu}(\text{CO}_2\text{CH}_3)_2 \cdot \text{H}_2\text{O}$	Ethanol ($\text{C}_2\text{H}_5\text{OH}$)	$(\text{CH}_2(\text{OH})\cdot\text{CH}_2\cdot\text{NH}_2)$	Wang et al. [16]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$, NaOH	2-Propanol	–	Zimmermann et al. [34]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$	Acetone	TEA	Efafi et al. [35]	Sol-gel
Zinc nitrate hexa hydrate/ Na-CMC	Deionized water		Muthukrishnan et al [36].	Sol-gel
$\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ / $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$, NaOH	Deionized water	PEG-6000	Liu et al. [37]	Sol-gel
$\text{Ti}(\text{OCH}(\text{CH}_3)_2)_4$, $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$	Isopropyl alcohol	–	Boro et al. [38]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$, NH_4VO_3	$\text{CH}_3\text{OH}/\text{MeOH}$	–	Slama et al. [17]	Sol-gel
ZnCl_2 , FeCl_3 , NH_4Ac , $\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$	$\text{C}_2\text{H}_6\text{O}_2$		Rabbani et al. [39]	Sol-gel
$(\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O})/\text{TiO}_2$	Isopropyl alcohol	$(\text{CH}_2(\text{OH})\cdot\text{CH}_2\cdot\text{NH}_2)$	Marimuthu et al. [40]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$, $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	Double distilled water	$[\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}]$	Birajdar et al. [18]	Sol-gel
$\text{Zn}(\text{NO}_3)_2$, citric acid and tetraethoxysilane	Ethanol ($\text{C}_2\text{H}_5\text{OH}$)	–	Sivakami et al. [41]	Sol-gel
Isopropyl orthotitanate (TTIP), zinc nitrate tetra hydrate	Ethanol ($\text{C}_2\text{H}_5\text{OH}$)	Diethanolamine (DEA)	Moradi et al. [42]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$	2-Methoxyethanol	$(\text{CH}_2(\text{OH})\cdot\text{CH}_2\cdot\text{NH}_2)$	Ocaya et al. [43]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$, CoCl_2		Polyvinyl alcohol	Verma et al. [19]	Sol-gel
$[\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}]/\text{Ga}(\text{NO}_3)_3$, gelatin	Distilled water	–	Khorsand Zak et al. [20]	Sol-gel
$\text{Zn}(\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}$	Distilled water/ethanol	$(\text{CH}_2(\text{OH})\cdot\text{CH}_2\cdot\text{NH}_2)$	Kiani et al. [44]	Sol-gel

Table 1. Precursors and solvents used in the synthesis of ZnO by the sol-gel process.

Precursor	Solvent	Stabilizing agent	Reference	Technique
Zn(CH ₃ OO) ₂ 2H ₂ O, sulfo propyl methacrylatepotassium	Ethylene glycol	–	Liua et al. [45]	Colloidal
Zn(CH ₃ OO) ₂ 2H ₂ O	Distilled water	Poly(vinyl alcohol) (PVA)	Nagvenkar et al. [46]	Colloidal
Zn(CH ₃ OO) ₂ 2H ₂ O, LiOH·H ₂ O	Ethanol (C ₂ H ₅ OH)	–	Yuan et al. [47]	Colloidal
Zn(CH ₃ OO) ₂ 2H ₂ O, tetraalkylammonium hydroxide	DMSO	NEt ₄ OH	Panasiuk et al. [48]	Colloidal
Zn(CH ₃ OO) ₂ 2H ₂ O	Ethanol	Triethylamine, diethylamine	Gupta et al. [49]	Colloidal
(Zn(NO ₃) ₂ 6H ₂ O), NaOH	Distilled water	1-Thioglycerol (TG) and 2 mercaptoethanol (ME)	Hodlur et al. [50]	Colloidal
Zn(CH ₃ OO) ₂ 2H ₂ O	Deionized water	Hexamethyl netetramine	Guo et al. [56]	Colloidal
Zn(CH ₃ OO) ₂ 2H ₂ O, KOH	Methanol	–	Rahman [51]	Colloidal
Zn(CH ₃ OO) ₂ 2H ₂ O, KOH	Methanol	PVP	Gutul et al. [52]	Colloidal
Zn(CH ₃ OO) ₂ 2H ₂ O, KOH	Ethanol	3-aminopropyltriethoxysilane	Moghaddam et al. [53]	Colloidal
Zn(CH ₃ OO) ₂ 2H ₂ O, NaOH	Ethyl alcohol	–	Liu et al. [54]	Colloidal
Zn(CH ₃ OO) ₂ 2H ₂ O	Diethylene glycol.	–	Xie et al. [60]	Colloidal
Zn(CH ₃ OO) ₂ 2H ₂ O	Ethanol	LiOH	Verma et al. [61]	Colloidal
Zn(CH ₃ OO) ₂ 2H ₂ O, NaOH	2-propanol	–	Moghaddam et al. [64]	Microwave
GO, Zn(NO ₃) ₂ NaOH	Deionized water	–	Tian et al. [65]	Microwave
Zn(CH ₃ OO) ₂ 2H ₂ O, NaOH	Distilled water	Guanidinium carbonate, acetyl acetone,	Hamedani et al. [66]	Microwave
Zinc hydroxide	Distilled water	Cetyltrimethylammonium bromide	Rai et al. [67]	Microwave
Zn(CH ₃ OO) ₂ 2H ₂ O, NaOH, NH ₄ OH	Dieonized water	–	Yanga et al. [69]	Microwave
(Zn(NO ₃) ₂ ·6H ₂ O), hydrazine hydrate	Distilled water	–	Krishnakumar et al. [70]	Microwave
ZnSO ₄ ·7H ₂ O, GO, NaOH	Distilled water	–	Lua et al. [71]	Microwave
Zn(CH ₃ OO) ₂ 2H ₂ O	Deionized water	–	Zhu et al. [72]	Microwave

Precursor	Solvent	Stabilizing agent	Reference	Technique
ZnSO ₄ , NaOH	Deionized water	–	Liu et al. [73]	Microwave
Zn(NO ₃) ₂	Deionized water	–	Rassaeia et al. [74]	Microwave
Zinc oxide, ammonium hydroxide	Deionized water	–	Lu et al. [75]	Microwave
ZnSO ₄ , NaOH	Deionized water	–	Limaye et al. [76]	Microwave
Zinc acetylacetonate monohydrate	Water	Ethoxyethanol, ethoxyethanol, and n-butoxyethanol	Schneider et al. [77]	Microwave

Table 2. Precursors and solvents used in the synthesis of ZnO by colloidal/microwave synthesis.

3.2. Recent studies and applications

Various morphologies of ZnO can be obtained from the sol-gel process including nanorods [3], inhomogeneous films [4, 5], inhomogeneous nanoparticles [6] and nanocomposites [7].

The structural effects of cation doping on ZnO nanoparticles was investigated in several studies. When doped with lithium, it was found that the concentration of Li^+ ion substitution for Zn^{2+} directly affected the XRD intensity of the (002) plane, but did not affect the grain size or crystallinity of the nanoparticles [8]. When ZnO was doped with Ca^{2+} ions, the average particle size was increased to 40–90 nm which could be attributed to the larger ionic radius of Ca^{2+} that substituted for Zn^{2+} ion sites [9]. Likewise, the average grain size reduced when a small radius ion is substituted for Zn^{2+} (0.74 Å) in the hexagonal wurtzite structure such as Co^{2+} (0.58 Å) [10]. Doping with Al^{3+} ions also showed the same tendency in reducing particle size, however, impurity phases such as Al_2O_3 and ZnAl_2O_4 were also observed [11]. Additionally, co-doping of ZnO with Ag^+ and Al^{3+} ions showed the formation of crystal defects due to the difference in ionic radius between Ag^+ , Al^{3+} and Zn^{2+} . Crystallinity improved proportionally with increased Ag^+ doping concentration, however, lattice defects and dislocations increased with Al^{3+} substitution [12]. Further dopant studies also demonstrated that limited dopant precursor solubility provoked a random distribution of dopant throughout the product [13]. Most research about doping ZnO has resulted in improved optical and electrical properties due to improved morphology or intrinsic material properties [14–20].

Synthesis of ZnO of different morphologies without doping is also important to consider since product morphology alone can affect device properties. Without any dopant ZnO can be obtained under normal laboratory conditions with well-aligned nanorods, agglomerated nanoparticles and inhomogeneous thin films composed of nanoparticles, quantum dots, nano-wires, spheres or nano-cubes [21–44].

Colloidal synthesis technique can be utilized to obtain nanocomposites of ZnO and other materials. Nano-sheets of poly (styrene-methyl methacrylate-sulfopropyl methacrylate potassium)/ZnO nanocomposites were obtained by Liua et al. [45]. Dissolving ZnO in other materials can result in a great combination and co-application of materials such as ZnO/PVA (Polyvinyl alcohol) [46]. The same process was done to produce ZnO/ TiO_2 multilayer thin films [47]. This technique allows obtaining well size-controlled nanoparticles such as those reported with use of dimethyl sulfoxide, but the author reports that the solvent and post-annealing treatment are also important factors in the crystallization process and average particle size [48].

Several authors have reported that the product morphology can be altered between flakes, hexagons, particles and flower-like morphologies by adding different surfactant material [49]. Agglomeration of ZnO nanoparticles was reduced by adding capping agents to different thiol molecules during synthesis [50]. It was demonstrated that the colloidal stability of nanoparticles can be maintained after dispersion in monoethanolamine (MEA). Also, hybrid structures can be obtained through this method like ZnO-Au reported recently [51]. Dispersion of nanomaterials could also be maintained through an additive such as poly (N-vinylpyrrolidone) which has been shown to maintain colloidal stability for more than a couple of months [52]. In the same way agglomeration of ZnO quantum dots can be prevented through a capping

agent such as 3-aminopropyltriethoxysilane in order to maintain their quantum properties [53]. Stabilization of the colloidal particles ensures that particle size and shape does not change with time allowing for more repetitive results for each batch of material. Stable colloidal solutions have also been used to grow novel nanostructures on several kinds of unique substrates such as wood that can allow for new ecological applications in future [54–63].

Colloidal and sol-gel processing are both chemical techniques that can be used to easily obtain different nanomaterials; similarly, microwave-assisted synthesis can obtain similar products but has been explored very little. In microwave-assisted synthesis, most reactions take place in a short amount of time and have resulted in the synthesis of good ZnO nanostructures. The technique has obtained spherical nanoparticles that are stable in solution for up to 50 days, and can be deposited several times on a substrate without any change in its morphology. Similarly, it is possible to obtain composites such as ZnO-nanoparticles on reduced graphene oxide. Also, the morphology is highly dependent on the complexing agent where the reaction takes place or if a dopant is added, such as that reported for obtaining ZnO nanoflowers, nanorods and nanoparticles. Additionally, a research group has confirmed the formation of flower-like to rod-like nanostructures by changing the system temperature. Other works have also reported about dumbbell-shaped nanoparticles, nano-flowers, graphene-ZnO nanocomposites, straw-bundle, chrysanthemum and nanorod-based microspheres obtained under certain temperature conditions. [2, 64–78].

4. Conclusions and future directions

The techniques listed in the above paragraphs remain as the most important chemical solution-based routes to synthesize ZnO. Within the same processing method, a variety of material morphologies and properties can be obtained by subtle changes in temperature, additives, dopants or other parameters. There has been a wide range of organic and inorganic particles that have been synthesized and applied in different fields through these techniques. Investigating the effects of processing conditions on ZnO nanoparticles is still a hot topic in current research for their applications in optoelectronic and solar cell devices.

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