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Different Methods of Fabrication of Zinc oxide Nanoparticles: Review

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***Abstract*— *Nanoparticles gained considerable attention because of their versatile applications in various fields. Especially, zinc oxide nanoparticles are highly preferred among the metal oxide nanoparticles, having a vast list of fascinating properties. Due to their unique characteristics, they have a lot of biological applications such as antibacterial, antifungal, anticancer, antioxidant, anti-diabetic and anti-inflammatory, etc. The synthesis protocol plays a major role in the properties of synthesized zinc oxide nanoparticles, small changes led to drastic variation in size and morphology. Therefore, careful selection of synthesis method is most essential for the product with desired properties and applications. This review reveals the different techniques used for the fabrication of zinc oxide nanoparticles.*

***Keywords*— *Synthesis, ZnO nanoparticles.*

I. INTRODUCTION

Nanotechnology is an emerging interdisciplinary science for the synthesis of nanoparticles at the nanoscale. [1] Among the various metal and metal oxide nanoparticles, zinc oxide is one of the most widely used n-type semiconducting metal oxides with a wide band gap (3.37 eV) and large excitonic binding energy (60 meV), which have made zinc oxide nanoparticles important for both scientific and industrial applications. [2][3] It appears as a white powder and is almost insoluble in water. Also an amphoteric oxide exhibits both basic and acidic properties. [4] Nowadays, the unique properties of nanomaterials have motivated researchers to develop many simpler and inexpensive techniques to produce nanostructures of technologically important materials. [3] Different methods result in different particle sizes of ZnO depending on the type of precursor, solvent, pH and temperature of the reducing solution. [5] The morphology, shape and size of these materials play an important role in determining their properties such as optical, electrical and magnetic properties. [6]

Synthesis of nanoparticles

Various methods can be used for the synthesis of nanoparticles (NPs). These methods can be broadly classified into two main classes, namely 1) bottom-up approach and 2) top-down approach. These approaches are further divided into different subclasses based on the mode of operation, methods, reaction conditions and protocols used. The various properties such as size, shape and surface chemistry of the ZnO NPs can be fine-tuned with different reaction parameters such as temperature, solvent, time, concentration etc. Since these properties affect the application and toxicity of ZnO NPs, it is important to select the synthesis mode properly. The synthesis protocol must be selected based on the expected application. [7]

Top-down synthesis

In this method, destructive approach is employed. The larger molecules, which are decomposed into smaller units and then these units are converted into suitable nanoparticles. Examples of this methods are grinding/ milling, Chemical Vapour Deposition (CVD), Physical Vapour Deposition (PVD) and other decomposition techniques.

Chemical vapour deposition

In 1993, Endo first reported chemical vapour deposition (CVD) technique. CVD is one of the most trusted method. By using a carbon source that is in the gas phase and an energy source, such as a plasma or a resistively heated coil, to transfer energy to a gaseous carbon molecule, substances that are in the vapour phase can be condensed to produce material in the solid phase. [29] Methane, carbon monoxide, and acetylene are used as carbon sources in the CVD process. While heated to a high temperature in an oven, the hydrocarbons flow through the quartz tube. At high temperature the bond between hydrogen and carbon breaks producing pure carbon molecules. Then, the carbon diffuse towards the substrate, which is heated and coated with a catalyst where it bonds. CVD is classified by three parameters. 1. The operating conditions. 2. Physical characteristics of vapour. 3. Heating of the substrate. [31]

Physical vapour deposition

Thermal evaporation process is basically a Physical Vapour Deposition method, and it is one of the simplest and most popular method, and has proved to be very successful in the production of nanowires and nanobelts with various characteristics. The basic process of this method is sublimation of the starting materials in powder form at high temperature, and the subsequent deposition of the vapour in a certain temperature range to form the desired nanostructures. The most common PVD processes are sputtering and evaporation. [32] The process for PVD is as follows: (1) the material to be deposited is converted to vapour by a high temperature vacuum or gaseous plasma, (2) the vaporized material is transported to the substrate, and (3) the vapour condenses on the substrate to form a thin film. [33]

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Bottom-up synthesis

This approach is employed in the opposite way to top-down synthesis, because here the nanoparticles are formed from relatively simple substances. Therefore, this approach is also referred to as the building-up approach. It is similar to the "seed" model, where the beginnings are small but eventually grow in complexity and completeness. These include sol-gel, hydrothermal, solvothermal, wet chemical, green synthesis, etc. Some of the methods are explained in detail.

Precipitation method

The simplest method for the synthesis of ZnO nanoparticles is chemical precipitation, which usually involves a reaction between a zinc precursor such as $Zn(CH_3COO)_2$. $2H_2O$, [8] $Zn(NO_3)_2$. $6H_2O$, [9] [10] $Zn(SO_4)_2$. $7H_2O$, [11] [12] $ZnCl_2$ [13] and a precipitating agent such as sodium hydroxide (NaOH), [9] [6] ammonium hydroxide (NH_4OH), [13] potassium hydroxide (KOH), [14] urea etc. Mixing of these solutions results in the formation of an intermediate product zinc hydroxide ($Zn(OH)_2$) at pH 10, which ultimately converts to ZnO after calcination at high temperature. [15] The formation of ZnO NPs by the chemical precipitation method is not only simple and easily controlled but also easily industrialized. However, due to the surface effects of nanoparticles, the nano-oxide prepared by the chemical precipitation method could form agglomerates easily. [16] And this method does not work well if the reactants have very different precipitation rates. [17]

Wet chemical synthesis

The wet chemical synthesis of ZnO NPs is a modification of the precipitation method. It is very simple and cost effective procedure to synthesize a wide range of nanomaterials without involving complicated equipment. [34] In this approach, an additive is used to stabilize the NPs formed. The mechanism of stabilization involves either increasing the viscosity of the solution or forming a complex with metal ions via hydroxyl groups. [18]

Hydrothermal synthesis

Hydrothermal method is another standard preparation route, especially for powdery nanostructure. In this method, distilled water is used as a primary solvent. [35] An aqueous solution of precursor is heated in a stainless steel autoclave above the boiling point of water and consequently, the pressure within the reaction autoclave is dramatically increased above atmospheric pressure. This synergistic effect of high temperature and pressure provides a one-step process to produce highly crystalline materials without the requirement of further annealing treatments. [36] The temperature determines the internal pressure and the total amount of solution added to the autoclave. [35] The parameter such as pressure, temperature, concentration of the precursors, pH and ageing time affects the composition, crystallinity and morphology of the end product. [19]

Solvothermal synthesis

Solvothermal synthesis is a generally adopted method to synthesize metal oxide nanostructures. [37] In solvothermal method, water is replaced by an organic solvent. The properties of solvent render strong effect on the morphology of the end product. [34] This reaction typically occurs at elevated temperatures close or above the boiling point of the reaction medium in a closed vessel called an autoclave. [38] The autoclaves are made up of a strong alloy such as steel, to withstand the high pressure developed during the reaction. Generally, the autoclave contains a teflon liner to protect it from corrosion and to provide chemically inert vessel for the reaction. [39]

Sol-gel synthesis

Sol-gel method is an outstanding route for the synthesis of NPs and nanocomposites. [40] This method involves the preparation of a colloidal solution called as 'Sol' that gradually transforms to a 'gel' like biphasic system composed of both liquid and solid phase. The method has the following steps; In the first step, the sol is prepared by suspending precursor in appropriate liquid such as water or organic solvent (solvation). The second step involves the conversion of colloid sol into gel by polymerization (either hydrolysis or condensation reaction). [41] [18] The intermediate requires further heat treatment in order to obtain high crystalline nanoparticles. [42] The products obtained from sol-gel method are highly modifiable, crystalline and pure. However, the use of toxic chemicals and long reaction time are some of the drawbacks of sol-gel approach. [29]

Solid-state pyrolytic method

The solid-state pyrolytic method is a simple, rapid, cost-effective method and reliable for large scale production. Typical solid-state pyrolytic synthesis involves mixing of zinc acetate and sodium bicarbonate. The mixture is then pyrolyzed. This reaction occurs only at elevated temperatures. Zinc acetate changes to ZnO NPs, while sodium bicarbonate changes to sodium acetate, which can be washed away by deionized water. The size of the NPs can be controlled by adjusting the pyrolytic temperature. The byproducts also have an important role in controlling particle growth and agglomeration. Sodium acetate can be distributed on the surface of the NPs, preventing them from agglomeration. [18]

Solution-free mechanochemical method

Solution-free mechanochemical preparation of ZnO NPs is a two-step synthesis method. In the first step $Zn(CH_3COO)_2$ and $H_2C_2O_4.2H_2O$ powder mixtures are ground for a certain time to form $ZnC_2O_4.2H_2O$ nanoparticles. [20]

$Zn(CH_3COO)_2$ (solid, large particles) + $H_2C_2O_4.2H_2O$ (solid, large particles) → $ZnC_2O_4.2H_2O$ (solid particles) + $2CH_3COOH$ (liquid and gas).

The second step is the thermal decomposition of $ZnC_2O_4.2H_2O$ at very high temperature to get ZnO NPs

$$ZnC_2O_4.2H_2O \rightarrow ZnC_2O_4 + 2H_2O \uparrow$$
$$ZnC_2O_4.2H_2O \rightarrow ZnO + CO_2 \uparrow + CO \uparrow + 4H_2O \uparrow$$
$$CO + O_2 \rightarrow CO_2 \uparrow$$
$$H_2C_2O_4.2H_2O + O_2 \rightarrow 4CO_2 \uparrow + 4H_2O \uparrow$$
$$CH_3COOH \text{ (liquid)} \rightarrow CH_3COOH \text{ (gas)} \uparrow$$

The advantages of this method are the low production costs and high homogeneity of the crystalline structure and morphology. But the morphology of the ZnO NPs strongly

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depends on the milling time of the reactant mixture, a longer time of milling led to a smaller particle size. [16]

Vapour transport synthesis

The vapour transport process is a very prevalent method for the synthesis of ZnO nanostructures. Oxygen plays an important role in vapour transport process. In this process, zinc and oxygen or oxygen mixture vapour are transported and the mixture reacts with each other resulting in the formation of ZnO nanostructures. Another direct method includes heating zinc powder while oxygen is being pumped through it. In order to produce the necessary nanostructures, careful monitoring of the zinc to oxygen ratio is required. The change in this ratio results into a large variation in size and geometry of nanostructures. [3] The optimum temperature and flow rate for introducing oxygen was found to be 900°C and 25 m^3/s respectively. [21]

Green synthesis

Green chemistry is an innovative research field that reduce or eliminate the use and generation of hazardous substances. [1] The principle mainly focuses on the choice of reagents which are eco-friendly. [3] The biological method of synthesizing metal oxide nanoparticles may be of three ways, which includes the use of microorganisms, plants or with enzymes. [22] In recent years, the green synthesis approach has been gaining attention, which eliminates the use of toxic chemicals and applies environmentally friendly routes. Bio synthesis also becomes very important from the economical perspective. [23] Almost all types of organisms have been tested to synthesize biologically mediated nanoparticles which are non-hazardous in nature. [24] The literature survey shows that using plants offer important advantages over other biological systems. The plants are easily available and safe to handle, and the nanoparticles synthesized by plant extracts are more stable. Plant extracts contain different concentrations and combinations of bio compounds, and can act both as reducing and capping agents in the synthesis process of nanoparticles. [25] The plant phytochemicals with antioxidant properties are accountable for the preparation of metal and metal oxide nanoparticles. [9] Green synthesis of nanoparticles can overcome the problems related to chemical and physical methods. [8] Synthesis of nanoparticles using microorganisms involves elaborate process of maintaining cell cultures, intracellular synthesis and multiple purification steps. [26] Also, the microorganism based synthesis is not of industrial viability. [27]

II. Conclusion

The chemical method consumes less power and can be carried out in a robust atmosphere during the synthesis. [9] But, the chemicals used for the synthesis of nanoparticles and their stabilization are toxic, which leads to non-eco-friendly by-products. [3] Chemical method also requires special type of vessel like polypropylene vessel for nanoparticles synthesis. [28] Although physical and chemical methods are quick and easier for nanoparticles synthesis, the biogenic technique like green is better and eco-friendly. [3]

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