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An Overview on synthesis of metal oxide nanoparticles

K.Murugesan[1],P.Sivakumar[2], P.N.Palanisamy[3*]

[1]Department of Chemistry, Sri Shanmugha College of Engineering and Technology, Sankari, Salem, Tamil Nadu 637 304, India.
[2]Department of Chemistry, Aringer Anna Govt. Arts College, Namakkal, Tamil Nadu 637 002, India.
[3]Department of Chemistry, Kongu Engineering College, Perundurai, Erode, Tamil Nadu 638 052, India.

*Corresponding Author : K.Murugesan
E-mail: drpnp@gmail.com

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Abstract

Presently there is a growing interest in the preparation and application of nanometal oxides. They can exhibit excellent results when applied in various kinds of industries like pharmaceutical, fertilizers, engineering etc,. Several techniques have been proposed for the synthesis nanometal oxides such as CVD, Sol-Gel Synthesis, Microwave synthesis etc. Preparing the nanometal oxides by these techniques has the better performances potentially. This review analyses the various technique for the synthesis of nanometal oxides, which is applied to the mass production of nanometal oxides like nanofibres, nanowires, nanotubes etc. It is highlighted from the review that the most relevant characteristics of each techniques and the type of new material that can be produced from novel sources.

Keywords: CVD, Sol- Gel, Nanofibres, Microwave, Nanowires, Nanotubes.

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1.Introduction

Now-a-days, research and development is mainly concentrated on new types of materials especially nanomaterials because of their numerous realistic applications. Mainly nanomaterials are attractive because of their improved physical and chemical properties. The technology which produces the above said extraordinary materials is nothing but nanotechnology which is highly described by a physical Nobel Laureate, Richard Feynman in 1959. Richard Feynman is commonly considered to be the father of nanotechnology due to his speech in 1959 entitled “There’s plenty of room at the bottom”, but the term “nanotechnology” was first used in 1974 by Norio Taniguchi.And another well-liked defintion about this technology is: “Nanotechnology mainly consists of the processing of separation, consolidation, and deformation of materials by one atom or one molecule.”

According to this definition nanotechnology only describes the manipulation of materials on the molecular level and it refers to structures that are typically between 1 and 100 nm (1 nm = 10^{-9} m) in size. This technique not only focuses on reducing the size but also on increasing the efficiency of materials. The materials or particles produced by nanotechnology are between 1 and 100 nm, in each spatial dimension. The general aim of the technology development is miniaturization. Nano technology proved that by producing smaller, faster, lighter and cheaper devices with greater functionality while using less raw materials and consuming less energy. Research on the materials that was synthesised by this technology is the initiation towards miniaturization that will play a vital role towards a sustainable usage of raw materials and energy [1]. Nanotechnology applications and products make use of characteristics which occur in the transition area between the atomic and the mesoscopic scale. This means nanoscale particles can have different physico-chemical properties with respect to microscale or macroscale particles of the same material. The review is focused on rapid synthesis of nanometal oxides by a novel method especially microwave method and its special features. Therefore, nanomaterials are being designed and delivered into a broad and ever increasing range of applications.

2. Synthesis Methods

For the synthesis of nanomaterials, there are large numbers of techniques available by which we can produce the nanomaterials in the form of colloids, clusters, powders, tubes, rods, wires, thin films etc. All the techniques are generally based on two approaches. One approach is summarized under the so-called “top-down” technology and refers to the production of very small structures out of material building blocks by grinding, etching or other mechanical processing. The millionfold electronic microchips which are produced fall under this category. The desired conductor paths are predetermined through lithography. The distances and widths of the conductor paths currently are at less than 100 nm. On the other hand, nanomaterials can also be manufactured according to so-called “bottom-up” technology. In this case structures are built atom by atom or molecule by molecule. Generally metal nanoparticles can be prepared by various methods such as solution-phase chemical reduction [2], UV Photolysis [3], metal vapor deposition [4] thermal decomposition [5], sonochemical decomposition [6], Electrochemical techniques [7], Laser ablation, sputtering and ball milling/ mechanical milling [8-10]. However, these methods require specific apparatus, vacuum conditions and chemical costs. Microwave irradiation as a heating method has found a number of applications in chemistry. The utilization of microwave irradiation in the preparation of nano particles has been reported in recent years [11]. Compared to the conventional methods, the microwave synthesis has the advantages of producing small particle size metal oxides with high purity owing to short reaction time [12-16]. Moreover, microwave assisted synthesis of nanomaterials have attracted great attention among the researchers because the time required to prepare metal oxides by this technique is too short and the material is high purity and exhibits good stability.

Microwave-assisted synthesis is a novel method to produce materials, since microwave heating is an in-situ mode of energy conversion and the microwave heating process is fundamentally different from conventional heating processes. Heat will be generated internally within the material, instead of originating from external sources. By means of this method, many functional materials and compounds with novel structures and properties have been obtained [17-19].

3. Synthesis of metal/metal oxide nanoparticles by Microwave method

Deepali Sharma et al [20], obtained ZnO nanoparticles by a facile route which involves the reaction of zinc sulphate heptahydrate and sodium hydroxide through drop by drop mixing synthesis and instant mixing synthesis and also under the influence of microwave radiation. Moreover, the synthesis under the influence of microwave radiation resulted with the product of smaller particle size. According to TEM studies, the size of ZnO nanoparticles is in the range of 10-15 nm. The as-prepared ZnO nanoparticles also showed good photocatalytic activity for the photodegradation of methylene blue dye.

Kushal D. Bhatte et al [21] employed microwave-assisted additive free synthesis of nanocrystalline ZnO from zinc acetate and 1,4 butanediol. It was found that crystalline ZnO nanoparticles exhibiting high catalytic activity for the synthesis of propargylamines. Moreover, this is the first report on additive free microwave-assisted synthesis.

E.Savary et al [22] prepared the nano-sized ZnO using zinc acetate, ethanol and dehydrate oxalic acid by microwave sintering method. In order to get the above said nano particle, a liquid route was employed. The size of the nanoparticle obtained through this method is 20nm, which was achieved after thermal treatment.

Sousan Rasouli et al [23] synthesised ZnO-Co nano-pigments by microwave-assisted gel combustion method using zinc nitrate, cobalt nitrate as an oxidant and citric acid as a fuel and complexing agent. The results reveals that the microwave-assisted combustion reaction was very simple and rapid to prepare the as-prepared nanocrystalline pigments with the size of 27-37 nm which depends on the amount of fuel content.

Chun-Chieh Tseng et al [24] reported the synthesis of zinc oxide nanoparticles starting from chloride precursor by microwave-assisted hydrothermal method using urea as a fuel under 800W irradiation for 5 min. And the optical properties of the as-prepared ZnO nanoparticles were analyzed which revealed that the intensity of 393 nm emission enhanced with the calcination temperature as the defects were reduced and the crystallinity was increased. Initially, the morphology of ZnO is sponge-like appearance and that could change to a net-like structure after thermal treatment and finally transformed to compact ZnO nanoparticles. After the calcination of sponge-like ZnO nanoparticles at 500^0C, pure ZnO nanoparticles were obtained.

De Moura et al [25] synthesized ZnO nanoparticles by an efficient microwave-assisted hydrothermal (MAH) method using a cationic surfactant, cetyltrimethylammonium bromide (CTAB) as the structure-directing template.

Wen-hui Li [26] studied the synthesis of single crystalline Co_3O_4 nanorods through thermal decomposition of the precursor cobalt chloride ($CoCl_2.6H_2O$) and urea ($CO(NH_2)_2$) by microwave-assisted hydrothermal route. The resulting oxide was pure, single-crystalline Co_3O_4 nanorods which was confirmed by SEM and TEM images. Also the optical properties of as-prepared nanorods were studied which indicates that the absorption peak of the nanorods shifted towards short wavelength. And the blue shift phenomenon might be ascribed to the quantum effect.

Renugadevi et al [27] prepared silver nanoparticles by microwave-assisted irradiation using Azadirachta indica leaf extract as a reducing agent. The main advantage of using microwave irradiation is it takes less time to reduce the silver ions. Apart from that the antibacterial and anticancer activity of the as prepared silver nanoparticle was tested against Vibrio chlorae and Bacillus subtilis. The report revealed that silver nanoparticle were more toxic to cancer cell.

Pires et al [28] followed the microwave-assisted hydrothermal method to prepare the nanocrystalline SnO powders using $SnCl_2.2H_2O$ as a precursor and three different mineralizing agents such as NaOH, KOH and NH_4OH. By changing the processing time, temperature, mineralizing agent and its concentration, SnO crystals having different sizes and morphologies could be achieved. Generally, for the preparation of SnO_2 nanocrystals by chemical method, the precursor most commonly used was $SnCl_4.5H_2O$ with the oxidation state of tin is IV. Moreover, this is the first report, in which the precursor used with the oxidation state II of tin in solution. The synthesised SnO nanoparticle have very good potential for applications in gas sensors, photo-catalysis and photo-electrochemical cells.

Ming-Guo Ma et al [29] studied the preparation of hierarchical Bi_2O_3 spheres assembled from nanosheets with pore structure by microwave method using bismuth nitrate, polyvinylpyrrolidone and ethanol under the influence of microwave irradiation for 10min at 150^0 C.

Newaz Mohammed Bahadur et al [30] prepared the TiO_2 coated ZnO nanocomposite particles by a novel and rapid microwave method. The photocatalytic activity of uncoated ZnO nanoparticles and ZnO-TiO_2 nanocomposite particles were determined by the photodegradation of Orange G in aqueous solution under UV radiation. Finally the report concluded that the photocatalytic activity of ZnO -TiO_2 nanocomposite particles was smaller than that of uncoated ZnO. Moreover, the report revealed that the prepared composite is promising candidate for use as sunscreen material as well as UV lasing materials.

Hui Wang et al. [31] employed microwave method for the synthesis of CuO nanoparticles under the influence of microwave irradiation using copper (II) acetate and sodium hydroxide as the starting materials and ethanol as the solvent. The as-prepared CuO nanoparticles are having the monoclinic structure it has regular shape, small size, narrow size distribution and high purity.

K.J.Rao et al [32] synthesized many nanometal oxides using a microwave-assisted solvothermal method. Some of the nanometal oxides are MgO, NiO, ZnO, Al_2O_3, ZrO_2 etc. The report revealed that the primary requirement is that the oxide precursor must have a high dielectric constant than the solvent. Moreover, the formation of nanometal oxides is confirmed by the presence of a suitable capping agent. During the preparation of ZrO_2 and Al_2O_3, capping agent is not necessary. In addition the heat treatment also provides control on the particle sizes of the resulting nanomaterials.

Sambandam Anandan et al [33] synthesized Bi_2O_3 short nanorods by microwave method using bismuth (III) nitrate in the presence of polyvinylpyrrolidone (PVP), as a stabilizing polymer under the influence of microwave irradiation in aqueous solutions for 6 min. This report also pointed out an information that a microwave assisted route to prepare Bi_2O_3 short nanorods has not been reported yet. Apart from that this methodology could be used for synthesising a number of various semiconductor systems involving other metal oxides with 1-D morphology.

Nadi Shojaee et al [34] were successfully synthesized the ZnO nanorods using zinc nitrate and methenamine aqueous solutions in a microwave- hydrothermal process. They analysed the effect of concentration and heating conditions on above said synthesis process of ZnO nanorods. It was found that the concentration of precursors and microwave irradiation power plays a significant role in the dimensions of the grown nanorods. While increasing the concentration of precursor, automatically there is a decrease in the mean diameter of ZnO nanorods. The remarkable change was observed while using the alumina slab and zinc foil were used as the substrate.

Zhiqin Chen et al [35] applied the microwave hydrothermal method to synthesize nanocrystalline rutile (TiO_2) using aqueous $TiCl_4$-HCl solution. In this procedure, the mixture solution is processed in microwave hydrothermal autoclave first at 120^0 C for 1.5h and then at 180^0C for 1h to precipitate rutile powders with the particle size of average diameter of 10nm.

Lun-Hong Ai et al [36] synthesized nanocrystalline Co_3O_4 by a microwave-assisted combustion method using urea as a fuel. The study suggested that the application of microwave heating, which makes the nanocrystalline Co_3O_4 particles porous and it also reveals that their microwave assisted method for the preparation of as-prepared nanomaterial is an economical.

L.C.Nehru et al [37] followed the microwave-assisted combustion method for the rapid sythesis of nanocrystalline ZnO using dissolution of zinc nitrate (as oxidant) and urea (as fuel) as the starting materials and water as solvent The as-synthesized nanomaterial has hexagonal structure and this route is not only an eco-friendly but also economical with respect to energy, time and simplicity.

Luc Huy Hoang et al [38] successfully synthesised the $Zn_{1-x}Co_xO$ nanopowders by a simple microwave method using sodium hydroxide (NaOH), Zinc acetate dihydrate [$Zn(CH_3COO)_2.2H_2O$], cobalt acetate tetrahydrate [$Co(CH_3COO)_2.4H_2O$] and Triethanolamine (TEA) as a surfactant. Moreover, the surfactant Triethanolamine (TEA) has negligible influence on the phase of the final product. The as-prepared $Zn_{1-x}Co_xO$ nanopowders has good crystalline quality and good surface morphology.

Naghmeh Faal Hamedani et al [39] prepared the ZnO nanocrystals with various morphologies via a fast and facile microwave assisted method using zinc acetate as starting material, guanidinium and acetyl acetone as structure directing agents and water as solvent. By adjusting the reaction conditions, templates and pH of the reaction medium, the various morphologies like nanorod, nanoparticle and nanoflowers of as-prepared ZnO nanocrystals could be obtained. The samples which was prepared by this novel technique used as sensing materials for the fabrication of selective sensing of CO, CH_4 and ethanol.Finally the report revealed that the performance of sensors fabricated by the ZnO nanostructures. The report concluded that the performance was totally depends upon the morphology of ZnO. The performance varies with various morphologies.

Elin Hammarberg et al [40] synthesized aluminium doped ZnO (AZO) and indium-doped ZnO (IZO) nanocrystals via microwave assisted heating in diethylene glycol (DEG) as a high-boiling multidentate alcohol i.e. a polyol medium. The as-prepared doped nanocrystalline ZnO via polyol mediated synthesis using the solutions of $Zn(CH_3COO)_2.2H_2O$, $InCl_3.4H_2O$ and $AlCl_3.6H_2O$ in DEG under the influene of microwave irradiation at 200^0 C for 30min. Moreover, the microwave-assisted polyol synthesis is a superficial one and also economical.

Yongjun He [41] reported the synthesis of ZnO nanoparticles with very narrow size distribution by coupling homogeneous precipitation with microemulsion under pulsed microwave heating using CTAB (Cetyltrimethyl Ammonium Bromide), 1-hexyl alcohol, n-hexane, zinc nitrate and methyl oxalate. The ultraviolet absorptivity of ZnO nanoparticles is much better than that of normal ZnO powders. Moreover, there is an increase in average size as well as in yield of the ZnO nanoparticles with increasing microwave power and duration of microwave radiation.

Chris Y. Fang et al [42] synthesised the nano-sized barium titanate (BT) powders using barium titanyl oxalate (BTO) as a precursor by microwave processing at 2.45 GHz at a temperature range between 600^0C and 750^0C with different heating rates from 10^0C/min to 20^0C/min without isothermal holding. This work mainly focused on the feasibility study of synthesizing nano-BT by microwave heating which has a great potential for accelerated kinetics in BT synthesis. Finally the pure nano-sized cubic BT has been synthesised at a temperature of 680^0C by microwave heating without holding. The average particle size of the as-prepared nanoparticle was found to be 70nm by BET which was confirmed by SEM examination. This report also reveals that the microwave processing accelerated the formation kinetics of cubic BT from barium titanyl oxalate which is not possible by normal conventional method at same temperature and heating rate.

Lin Ma et al [43] reported the synthesis of high –purity $Pr(OH)_3$ nanorods with uniform diameter in large scale by microwave-assisted heating method, and Pr_6O_{11} nanorods by subsequent thermal decomposition of the as-prepared $Pr(OH)_3$ nanorods at 500^0C for 6h. After annealing the $Pr(OH)_3$ nanorods with hexagonal phase were converted to Pr_6O_{11} nanorods with face centered cubic phase. They explained about the effect of concentration of alkali and microwave heating time during the synthesis.

Miroslaw Zawadzki [44] followed the microwave-assisted hydrothermal method (MAH) for the synthesis of nanosized and microporous zinc aluminate ($ZnAl_2O_4$) with a spinel structure of interesting properties including high specific surface area (220 m^2/g), microporosity and narrow pore size distribution. The precursors used for this synthesis were aluminium hydroxyl nitrate having the empirical formula $Al_2(OH)_{6-x}(NO_3)_x$, wherein x was equal or close to 1 and hydrated zinc acetate compare to other methods, this MAH method is very rapid and does not require a high temperature heating to attain the spinel structure. The above said interesting properties of the nanosized and microporous zinc aluminate spinel was confirmed by the liquid nitrogen adsorption-desorption measurements.Apart from that, the as-prepared $ZnAl_2O_4$ with spinel structure make them an advanced material suitable for use in catalytic or high tech ceramic applications.

Joseph Lik Hang Chau [45] synthesised the Ni and bimetallic FeNi nanopowders by microwave plasma (MP) method. Initially, nickel nanopowders was synthesised by MP synthesis method using nickel (II) chloride powder ($NiCl_2$) as the precursor raw material. Although nickel carbonyl ($Ni(CO)_4$) can also be used as the precursor raw material in the MP chemical synthesis. Due to safety and environmental issues, $NiCl_2$ was used. By changing the feeding rate of the precursor material, the average size of the Ni powder can be easily controlled.Next, the bimetallic FeNi nanopowder can be prepared by premixing the precursor materials nickel (II) chloride and iron (III) chloride before feeding into the reaction region during the MP synthesis process.

Ming-Guo Ma et al [46] have synthesised the ZnO micro and nanostructures with various morphologies using $Zn(NO_3)_2.6H_2O$ and pyridine as an additive by a microwave-assisted aqueous solution method at 90^0C for 10min. Here, pyridine plays a major role in controlling the morphology of ZnO. By adjusting, the concentration of pyridine, various morphologies such as hexagonal columns, linked hexagonal needles, hollow structures and hexagonal nanorings etc., were obtained. Not only with the pyridine, but with the other alkaline additive such as aniline and triethanolamine, the effect on the morphology was investigated. The report concludes that this method is simple, fast, surfactant-free and low cost for the preparation of ZnO with various morphologies.

Anil Kumar Kodge et al [47] used the microwave irradiation for the complete conversion of cobalt oxalate precursor into a cobalt oxide nanomaterial using polyvinyl alcohol as an efficient fuel. Moreover, this technique is very simple and energy efficient to obtain materials at nanolevel. Apart from this, the above said technique can adopt for the synthesis of other metal oxides at nano dimensions not only for laboratory preparations, it can also be extended for large scale synthesis of metal oxide nanoparticles.

Qingjun Sun et al [48] reported the rapid synthesis of monodisperse crystalline $WO_3.2H_2O$ ($H_2WO_4.H_2O$) nanospheres with $Na_2WO_4.H_2O$ and L (+) tartaric acid as a protective agent by microwave hydrothermal synthesis. The main advantage of this method is it requires only short time and also the reaction process was very simple. The morphology of the nanostructures were controlled by the protective L (+) tartaric acid. The as-prepared $WO_3.2H_2O$ nanostructures can be used in many fields such as sensor devices, catalysts and also electrochromic materials, etc.

Xuejun Zhang et al [49] have successfully synthesised the one-dimensional hematite (α-Fe_2O_3) nanorods through a facile microwave-assisted hydrothermal method using $FeCl_3.6H_2O$ and polyvinylpyrrolidone (PVP) as precursor raw material. The as-prepared nanorods are single crystalline with average diameter of about 60nm and length upto several micrometers. During the whole process, the capping agent or stabilizer (PVP) and microwave heating played important roles in the formation of the 1-D hematite nanorods. The magnetic investigation indicates that the weakly magnetic property of the nanorods may be ascribed to the shape anisotropy.

Hao Wang et al [50] reported the synthesis of $CeVO_4$ nanoparticles with controlled particle size through a surfactant free microwave irradiation method for only 10min. The particle size of the as-prepared $CeVO_4$ nanopowders was in the range of 6-18nm and is very sensitive to the pH value.At pH=7, the size of the nanoparticles is very smallest, while both the increase and decrease of pH results in forming larger particles. The microwave technique will ensure higher purity in the products and greatly reduce the production cost. In addition, this is the simple and rapid route for synthesising other functional inorganic materials.

4.Conclusion

In recent years, the application of microwave heating in synthetic chemistry is a fast growing area of research. In this review, the microwave assisted synthesis of various metals and metal oxides were discussed. Along with the oxides the synthesis of 1D nanoscale materials were also reported. Many one dimensional nano materials such as nanotubes, nanobelts, nanorods and nanowires have been prepared by different approaches. The main advantage of microwave heating are rapid volumetric, fast reaction rate, short reaction time, high reaction selectivity and energy saving. Due to inherent difference in the heating mechanism, microwave processing of the materials has many advantages over the conventional technique, if the material system is properly chosen. The reaction kinetics can be significantly enhanced by microwave heating of a material system. Other conventional route generally needs higher reaction temperature and longer reaction time. But microwave heating is fundamentally different from conventional heating processes. Microwave heating differs from other heating processes such as the use of heating fluid, gas, steam or electrical heating. In microwave heating, heat is generated within the material instead of originating from external heating sources .The distinctive advantage of the method is that the process no heat treatment or calcinations at a high temperature.The microwave-assisted synthesis approach may be extended to synthesize nanostructures of many metal oxides.

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