Synthesis of Nanomaterials by Chemical Route

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Shilpa Taneja, Pinki Punia, Preeti Thakur, and Atul Thakur

Abstract

Nanoferrites are the most frequently used ceramic nanomaterials due to their excellent physical and chemical properties. The properties of nanomaterials are more significant than their bulk counterparts. Selection of specific synthesis route plays a vital role in the preparation of nanoparticles. The reaction conditions and other physical parameters can affect the properties of particles to be developed. Several wet chemical methods have been introduced in the past few decades. Sol-gel, polyol, electrochemical, citrate precursor, sonochemical, solvothermal, co-precipitation, hydrothermal, etc. are some trendy methods to synthesize nanoparticles. Various synthesis routes are explained briefly in this chapter. Various factors such as reaction conditions, energy utilization, reagent compositions, and costs of production are discussed as well.

Keywords

Nanomaterials · Synthesis methods · Sol-gel method · Chemical properties

S. Taneja · P. Thakur (✉)
Department of Physics, Amity School of Applied Sciences, Amity University Haryana, Gurugram, India

P. Punia
Department of Physics, Guru Jambheshwar University, Hisar, India

A. Thakur
Centre of Nanotechnology, Amity University Haryana, Gurugram, India

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4.1 Introduction

Science and engineering have made rapid advances in the synthesis of nanomaterials to attain unique properties that differ from those of bulk materials. The particle has fascinating features below 100 nm, mostly due to two physical factors. The quantization of electronic states appears, resulting in very sensitive size-dependent effects such as optical and magnetic properties. The high surface-to-volume ratio modifies the thermal, mechanical, and chemical properties of materials. Because of their unique physical and chemical properties, nanoparticles are ideal for a variety of specialized applications. The top-down and bottom-up approaches to synthesize metal nanoparticles have been identified. Milling, lithography, and repeated quenching are examples of top-down methods. The bottom-up method, which involves building a material, atom by atom, molecule by molecule, and cluster by cluster (Hazra and Ghosh 2014; Shaikh et al. 2020), is the method most commonly used by researchers in the synthesis of nanoparticles. Using chemical reductants in solvents, numerous chemical processes have been recognized to synthesize colloidal metal nanoparticles from various precursors (aqueous and nonaqueous). Electrochemical method (Wang et al. 2008), sonochemical method (Gogate and Pandit 2004), radiolytic method (Uttayarat et al. 2015), and photochemical method (Parra et al. 2002) are some of the chemical processes which are used for industrial applications.

4.2 Synthesis Methods

4.2.1 Chemical Methods

4.2.1.1 Polyol Method

In the polyol method, nonaqueous liquid (polyol) is used as a reducing agent and solvent for the benefit of reducing surface oxidation and agglomeration. This method regulates the size, texture, and shape of nanomaterials. It can also be used in the mass production of nanomaterials (Zhao et al. 2010). If the synthesis is carried out at an elevated temperature with precise particle growth, this process can be used as a sol-gel method for synthesis of oxide (Fi et al. 2018). Due to its strong reducing ability, high dielectric constant and boiling point, ethylene glycol is used as solvent in this method. This solvent is also used as a cross-linking substance to form metal glycolate, which leads to oligomerization (Woei and Ying 2012). When as-synthesized glycolate precursors are calcined in air, they can be converted to metal oxide derivatives (Quievryn et al. 2014). Metallic alloys and core-shell size nanoparticles have also been synthesized using the polyol synthesis process (Kyun et al. 2007; Hoon and Kim 2012; Chang and Chen 2005; Dong et al. 2015). Yang et al. used this method to form icosahedral and cubic gold particles in the range of 100–300 nm (Kim et al. 2004). To control the molar ratio between silver nitrate and PVP, Xia et al. studied the management of morphologies like nanocubes and nanowires (Xia et al. 2009).

4.2.1.2 Microemulsions

An emulsion is a fluid that is dispersed in another fluid. As long as the polymer solution is liquid, it can produce emulsions. Emulsions are classified as macro, mini, or microemulsions based on the size of the droplet (Drmota et al. 2012). Using the microemulsion synthesis, the nanoparticles are formed. Immiscible liquids are separated into two phases when mixed (Foroughi et al. 2016; Mathew and Juang 2007). To create water-oil, energy is required to mix the two phases. In order to associate both phases, energy is required to create a water-oil connection; afterward it replaces the water-water and oil-oil contacts. Surfactants can reduce the interfacial tension between two liquids. Hydrophilic and lipophilic groups are found in surfactants (Puliová et al. 2013). If there are enough surfactant molecules, the interface between oil and water can be aligned and established by lowering the interfacial tension. The most common method for preparing nanomaterials in both phases is shown in Fig. 4.1. After mixing two microemulsions, due to the collision between micelles, Brownian motion occurs. Good collisions result in the reactants coalescing, fusing, and mixing well. The reaction between solubilizates produces metal nuclei. From the nucleation stage, Bönnemann et al. studied zerovalent metal atoms (Bönnemann and Richards 2001).

The nucleation point is a collision between a reverse micelle moving a nucleus and moving product monomer due to intermicellar conversation throughout the growth stage. The size and shape of nanodroplets, as well as the type of surfactant, determine the size and morphology of nanomaterials. Surfactants are commonly used to stabilize particles and prevent them from growing (Malik et al. 2012).

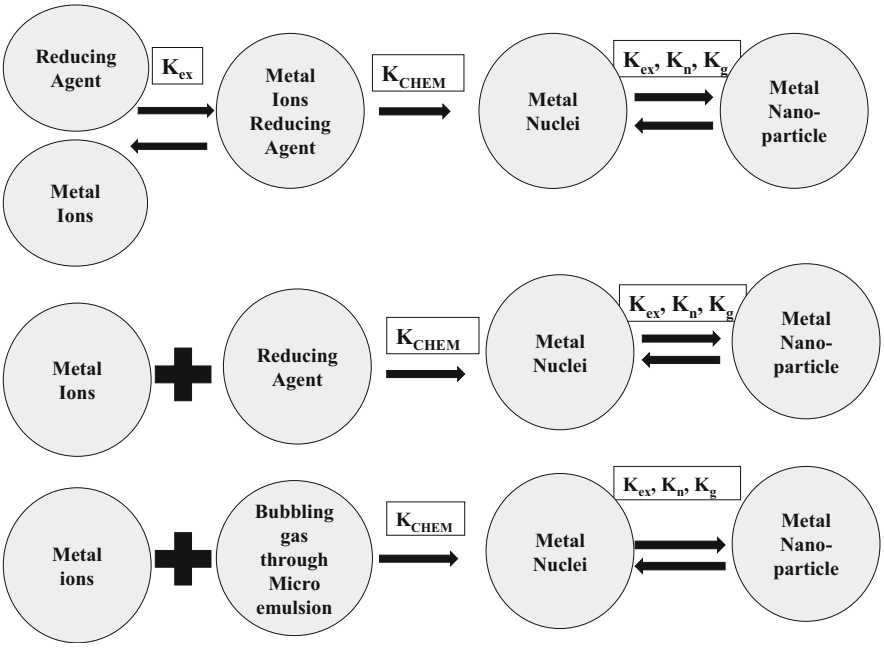

Fig. 4.1 Schematic diagram of nanoparticle preparation (Zhi et al. 2006)

Wongwailikhit et al. investigated the creation of Fe_2O_3 by adding water in oil microemulsion. Spherical nanoparticles were obtained with a diameter of approx. 50 nm. The size of the particles was determined by the amount of water in the microemulsion system. In an oil microemulsion, increasing the water fraction resulted in a larger particle size (Wongwailikhit 2011).

Sarkar et al. used water-in-oil microemulsion method to synthesize pure zinc oxide nanomaterials in a variety of shapes (Sarkar et al. 2011). Maitra was the first to use the microemulsion technique to create chitosan nanoparticles. Chitosan nanoparticles cross-linked with glutaraldehyde in the aqueous core of reverse micellar droplets (Mitra et al. 2001).

4.2.1.3 Thermal Decomposition

Thermal decomposition is a chemical method in which heat is needed to break chemical bonds in the compound being decomposed in this method, and the reaction is endothermic. A positive feedback loop is created when decomposition is sufficiently exothermic (Parra et al. 2002). Arshad et al. used TG-DTA-DTG techniques to study the thermal decomposition of metal complexes in an air atmosphere. To investigate the thermal behavior and mode of decomposition, thermo-analytical techniques were used in a static air atmosphere. When heated to 740°, the complexes and ligands disintegrated in two steps. The residue corresponded to metal oxide at temperatures above 740°; the thermal stability improves in the order Co(II) < Cu (II) < Zn(II) < Cd(II) (Arshad and Qureshi 2008).

Patil et al. investigated metal acetates and dicarboxylates to study TG-DTA-DTG to regulate the metal acetate bonding (Patil et al. 1968). George et al. studied the formation of thermal decomposition of n-butyl copper. This investigation (Whitesides et al. 1970) reported an example of reaction between metal hydride and metal alkyl to create the products of a thermal decomposition.

Logvinenko et al. (Logvinenko et al. 2007) investigated the thermal decomposition of bismuth and silver carboxylates using different characterization techniques. Kinetic studies were conducted using non-isothermal thermogravimetric data. All of the decomposition methods were multistep. Ewell et al. studied nearly pure talc at different temperatures, both unheated and after heating. The heat effects and weight losses occurred when heating talc was calculated. The crystal structure of talc did not change when heated to 800 °C. Enstatite gradually changed to clinoenstatite at temperatures around 1200 °C, whereas silica gradually directs to cristobalite at temperatures around 1300 °C (Punia et al. 2021; Liu and Hu 2014).

4.2.1.4 Electrochemical Synthesis

The combination of chemical compounds in an electrochemical cell is known as electrochemical synthesis. The ability to accept the potential is the main benefit of electrochemical synthesis (Tourillon et al. 2000). The electrochemical synthesis of Ag nanoparticles has received a lot of attention in recent years. In one such investigation, the electrochemical method employed consisted of dissolving a metallic anode in a solvent. The formation of Ag nanoparticles in the size range 2 to 7 nm was observed using this method. By varying the current density, the particle size was determined. The impact of various parameters on the size of nanoparticles was investigated. Two different silver clusters were detected in the UV-Vis spectra (Balakumaran et al. 2016).

Dobre et al. described the synthesis of colloidal silver solutions by the "sacrificial anode" method, which was carried out with a stirrer and a current pulse generator. The researchers created Ag particles of spherical geometry with a diameter of 10–55 nm. The absorption band at 420 nm was visible in the UV-Vis spectra, indicating the presence of Ag nanoparticles (Hajos et al 2011). The synthesis of Ag nanoparticles in aqueous PVA solution has also been studied which is a low-cost synthetic polymer with numerous mechanical properties. The average diameter of Ag nanoparticles was found to be 15 ± 9 nm (Starowicz et al. 2006).

The synthesis of silicon (Si) nanoparticles under UV excitation is useful for optics and other applications (Choi et al. 2014). More research was completed on the electrochemical method for silver nanoparticles in aqueous solutions. The size distribution of the produced silver nanoparticles ranged from 2 to 20 nm. On the surface of the cathode, Ag crystals are obtained with size less than 40 nm (El-sherbiny et al. 2012).

The study focused on synthesizing highly pure silver nanoparticles using an electrochemical method which was chosen because it is simple to control at room temperature and does not require the use of hazardous chemicals. The anode oxidation and cathode reduction were brought up by the experimental setup. The geometry of silver nanoparticles was found to be with a size of less than 50 nanometers (Khodashenas and Ghorbani 2015). Islam et al. investigated the electrochemical deposition method for the synthesis of platinum nanoparticles. Variations in electrolysis parameters were used to control particle size, and the composition of electrolytic solutions was used to improve platinum particle homogeneity. The particle sizes of platinum nanoparticles were larger than 10 nm (Islam and Islam 2013).

4.2.1.5 Precursor Method

Decomposition of precursor compounds is used to make complex oxides (Assar and Abosheiasha 2012). The hydrate of Li [Cr (C2O4)2 can be used to make LiCrO2. Metal oxides are typically synthesized using alkoxides and carboxylates as precursors. A variety of oxide metals have been prepared using hydrazine precursors. The thermal decomposition of ammonium oxalate precursors yielded ceramic composites. Semiconducting compounds such as GaAs and InP have been synthesized using organometallic precursors.

4.2.1.6 Combustion Synthesis

Combustion synthesis is a well-known procedure for producing a wide range of solids (Ehi-eromosele et al. 2015). This method has been used to make borates, carbides, oxides, etc. The mixture of reactants must be highly dispersed for combustion to occur. The combustion method can be used to produce the product by combining a fuel and an oxidizer. The powder mixture of reactants (0.1–100 m particle size) is commonly used in combustion synthesis. Then, select a gas medium that promotes the ignition of an exothermic reaction (Cheruku et al. 2012). Depending on the reaction, the combustion temperature ranges from 1500 to 3000 K because the desired products are obtained quickly after combustion. The

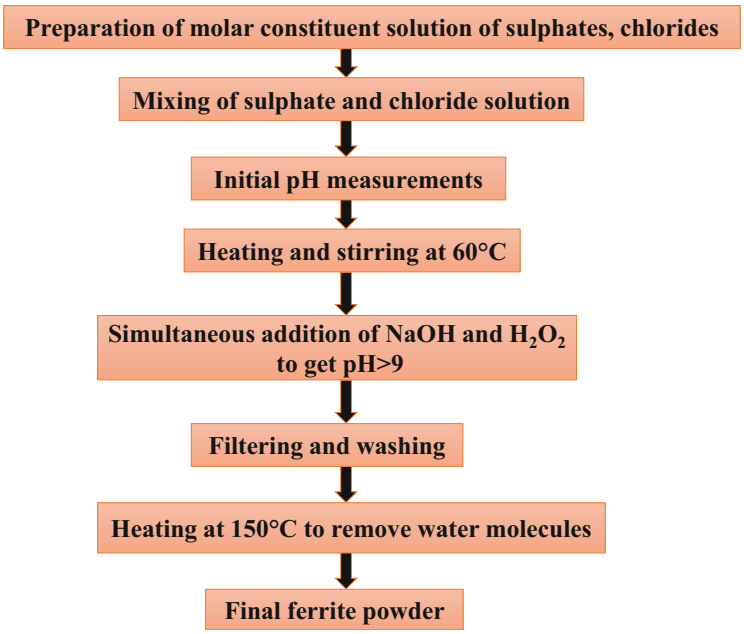

Fig. 4.2 Flowchart of wet chemical coprecipitation method

synthesis of silicates and carbides does not require the use of a gas medium. This method can be used to make superconducting cuprates, ferrites, and various oxides.

4.2.1.7 Wet Chemical Coprecipitation Method

Chemical coprecipitation can be used to make wet ferrites at different properties at low temperatures (55 °C). The oxidation method for making ferrite powders comprises ferrous ion and ferrite powders (Thakur et al. 2011). Many researchers have studied wet chemically prepared ferrites in depth. (Kumar and Kumar 2015; Sheikh and Jain 2016; Chahar et al. 2022; Rana et al. 2018). Ferrites are made by air oxidizing an aqueous suspension containing stoichiometric proportions of constituent cations. The starting solutions are made by stoichiometrically mixing 50 ml of aqueous solution with the appropriate sulfate. As a precipitant, a two-molar (2 M) solution of NaOH is prepared. Continuous stirring of all the precipitates has converted into brownish oxides of soft ferrites, and finally all the samples have been filtered, washed, and dried. The flowchart for the wet chemical method (Ataie et al. 1995) is shown in Fig. 4.2.

4.2.1.8 Sol-Gel Method

Sol-gel method is a wet chemical method used for preparing inorganic oxides by combining both chemical and physical processes (Bel-hadj-tahar and Mohamed 2014). The rise in viscosity is the conventional characteristic of sol-gel formation. The important characteristics of the sol-gel method are good size, homogeneity,

morphology, high purity, lower cost, and temperature. The six steps in sol-gel method are as follows:

1. **Hydrolysis** – In this process, a mixture of metal alkoxide and water in a solvent is stirred at the elevated temperature.
2. **Polymerization –** In this step, condensation of adjacent molecules was eliminated, and metal oxide linkages are designed to form liquid (sol) state.
3. **Gelation:** Then, the polymer networks join up to arrange a 3-D network throughout the liquid. The system becomes rigid due to removing the solvent from the sol.
4. **Drying:** H_2O and alcohol are removed at modest temperatures by reducing a hydroxylated metal oxide with some residual content.
5. **Dehydration:** In this step organic residues and chemically bound water exit out.
6. **Densification:** For compression temperature in the range of 1200–1400 K is used to form the dense oxide product.

The sol-gel procedure has been used to prepare metal oxide powders with a less particle size distribution and different particle shapes. Metal-ceramic composites and organic-inorganic composites have been ready by the sol-gel process.

Sol-gel route comprises three different techniques, namely, (a) autoignition, (b) auto-combustion, and (c) Pechini synthesis.

4.2.1.8.1 Sol-Gel Autoignition Method

In this method, the starting compounds were reserved in nitrates to form homogeneous powders. The flowchart is given in Fig. 4.3. The figure shows the detailed

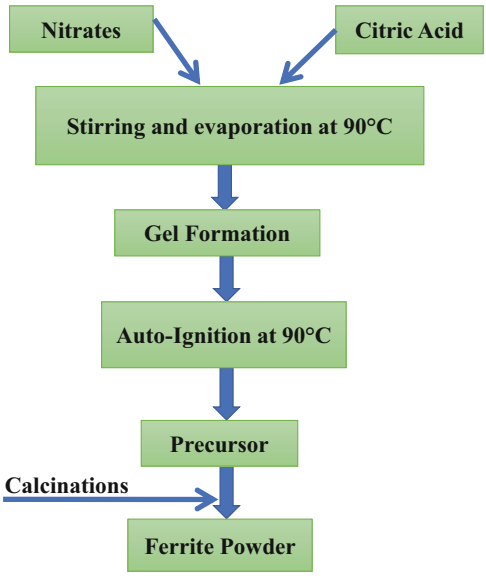

Fig. 4.3 Flowchart of sol-gel autoignition method

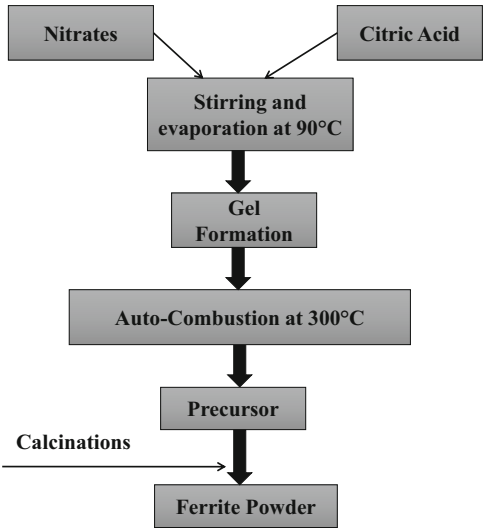

Fig. 4.4 Flowchart of sol-gel auto-combustion method

process of obtaining the required ferrite powders by sol-gel autoignition method (Raghavender et al. 2011).

The nitrates were used as starting materials and citric acid as chelating material. The metal nitrates were dissolved together in a deionized water to get a pure solution. To adjust the pH at 7, an aqueous solution of citric acid was mixed with metal nitrate solution. After full mixing of the chemical solutions, the mixed solution was placed onto a hot plate with nonstop stirring at 90 °C. After evaporation, the solution became viscous and finally is shaped to viscous brown gel. After several minutes the gel mechanically ignited and burnt with shining flints. The autoignition was completed finally with the brown colored.

4.2.1.8.2 Sol-Gel Auto-Combustion Method

Sol-gel auto-combustion method is alike to the method as described above till the gel formation. Once the gel is formed, the beaker with gel is moved onto the mantle, and the temperature is increased to 300 °C (Fig. 4.4).

As the temperature of the beaker reaches high, the entire gel is transformed into glowing flints, and the entire process would not stop till the citric acid is not consumed. The obtained precursor powders will also show some interesting properties, but the structural changes, which are taking place at low temperature, i.e., the initial phase of the compound formation, cannot be investigated. This is because the obtained powders by this method are presintered at 300 °C.

4.2.1.8.3 Pechini Method

This is also one of the sol-gel techniques employed by Pechini (Parvin et al. 2019; Massoudi et al. 2020; Jebeli Moeen et al. 2010). The metal nitrate mixture was heated to 90 °C, at which point ethylene glycol was added at a mass ratio of 4060 with respect to citric acid (Fig. 4.5). The temperature was maintained constant up to gel formation, which polymerized at 300 °C.

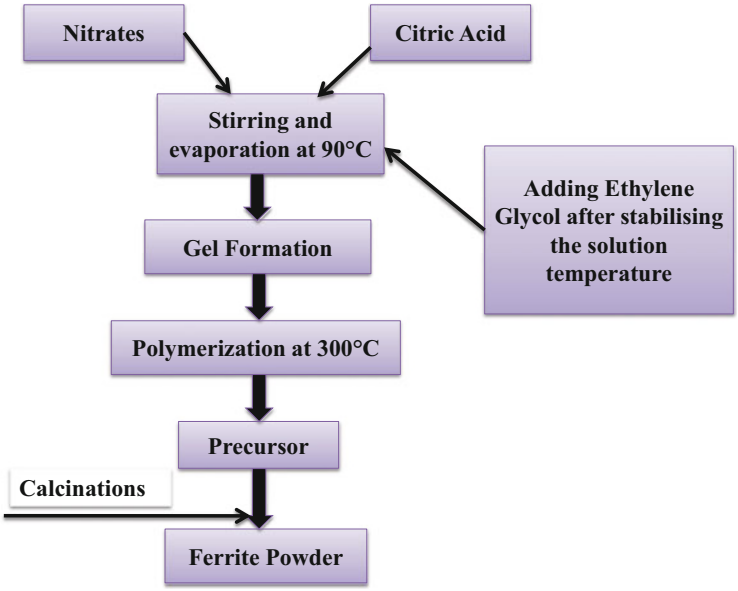

Fig. 4.5 Flowchart of Pechini method

4.2.1.9 Hydrothermal Synthesis

Among all the synthesis routes, hydrothermal synthesis is one of the most frequently used solution reaction-based procedure for synthesizing nanomaterials. This process requires very high temperature for the formation of nanoparticles. The process could be employed at a very low or very high pressure for the controlled morphology, and the process is nondestructive at high vapor pressure (Gan et al. 2020). The size of nanomaterials depend on hydrolysis rate and solubility of the metal compounds. First of all, the precursor solutions are poured into a Teflon container and then loaded in an autoclave reactor. Then, the solution is heated at specific temperatures up to 300 °C for several hours. Quenching with cold water is used to end up the reaction, and the prepared samples are washed with acetone or water many times to remove impurities (Dunne et al. 2015). The residue material is then dried at low temperature (60–80 °C) before calcination. Several metal nanoparticles like metal oxides, metal sulfides, metal phosphates, metal ferrites, etc. can be fabricated using this route. For nanoparticle formation the hydrated metal compounds undergo hydrolysis process, and then precipitates of metal nanoparticles are formed using dehydration (Hayashi and Hakuta 2010). Hydrolysis and dehydration processes during metal oxide nanoparticle formation take place according to the following reactions:

$$MA_x + xH_2O \rightarrow \quad M(OH)_x + xHA$$

$$M(OH)_x \rightarrow MO_{x/2} + x/2H_2O$$

Hydrolysis is also the combination of an electrostatic reaction between metal and hydroxyl ions. Though the conventional hydrothermal process is much effective for nanomaterial fabrication, hydrothermal synthesis with supercritical water is superior due to the 1000 times higher reaction and high crystallinity. This process is eco-friendly as it can degrade alkaline concentrations and other toxic by-products. Other integrated hydrothermal synthesis methods such as microwave-assisted hydrothermal method and template-free self-assembling catalytic synthesis are also introduced with better results for the formation of nanomaterials (Machmudah et al. 2014). Controlled synthesis of nanoparticles is also possible through liquid phase or multiphase chemical reactions. Nanomaterials generated via hydrothermal process have low stability at high temperatures. Beyond these great advantages, the process holds some drawbacks also. As the hydrothermal process needs high temperature and high pressure, it is quite difficult to attain the required values of temperature and pressure. Second and the most serious issue is that the process takes much more time than some other conventional synthesis routes like precursor and sol-gel method.

4.2.1.10 Solvothermal Method

Solvothermal synthesis is basically a process having chemical reactions within any solvent at specific physical parameters like pressure and temperature. Usually, temperature above the boiling point of the solvent and pressure more than 1 bar is significant for the process to fabricate metal nanoparticles, but the parameters may vary according to the required properties of nanomaterials to be formed. Solvothermal synthesis is a different process from hydrothermal synthesis as it consists of chemical reactions within nonaqueous solutions at comparatively high temperatures (Peh and Zhao 2020). Numerous organic and inorganic solvents or alcohol, etc. can be used as medium for solvothermal process. In this process temperature and pressure are the key parameters like in hydrothermal synthesis for crystallization of the precursor solution. Further addition of solvents gives rise to the mobility of dissolved ions, and a fine mixture of all the reagents can be formed. The technique is useful for complex materials also as it gives a single-step reaction route for complex elements. Different reagents such as chlorides, acetates, nitrates, etc. undergo solvothermal process; form precipitates in the form of hydroxide or any other compound depending upon the specific nanomaterial to be prepared. Sodium hydroxide, potassium hydroxide, or any other base could be mixed with the reagent solution for precipitation. The properties of the solvent like dielectric constant, thermal conductivity, density, etc. change with temperature varying toward higher values (Shaikh et al. 2020). Thus, desirable characteristics of the resultant product can be attained by controlling the reaction parameters. Autoclave reactors are used for the heat treatment of the prepared solutions. These reactors are usually made up of strong materials or alloys like steel so that the reactor can uphold the pressure

created inside the autoclave. To get a fine chemically inert vessel and prevent it from corrosion, a layer of Teflon is used inside the walls of reactor (Nunes et al. 2019).

Batch reactors are also very strong candidates for uninterrupted nanomaterial formation via solvothermal route. In one such reaction, Graphene oxide (GO) was prepared by mixing concentrated H_2SO_4 and $NaNO_3$ to the flake graphite powder while $KMnO_4$ was added to control the temperature of the solution. Graphene oxide nanoparticles were formed after the solution was kept in an ice bath for a few hours prior to the heat treatment, and residue was washed away with HCl solution and water many times (Yang et al. 2007). On the other hand, silver nanoparticles were successfully synthesized by one-step seedless solvothermal reduction route by using silver nitrate as a reagent and dimethylformamide (DMF) as solvent with polyvinylpyrrolidone (PVP) in a Teflon autoclave reactor at different temperatures (Liu et al. 2014). These solvents are mild reductant used to stop agglomeration of particles and growth mechanism of crystalline faces. Other than the conventional solvothermal technique, some integrated synthesis routes like microwave-assisted solvothermal synthesis have also shown excellent results in the formation of nanoparticles (Chella et al. 2015; Zhang et al. 2014).

4.2.1.11 Sonochemical Method

Nanoparticles could be synthesized by countless procedures, but controlled chemical reaction is the key factor to prepare nanomaterials with desired properties. The chemical reaction during the synthesis process strongly depends on many physical parameters like temperature, feed inlet, pressure, time, etc. Among all these factors, the feed inlet or energy supply is one of the most important parameter to control the reaction (Hankare et al. 2011; Patil and Bhanage 2016). Though each and every energy type has specific properties along with their reaction conditions, ultrasonic radiations give unique and most effective reaction conditions than other conventional energy source techniques (Xu et al. 2012). The effect of ultrasonic waves in liquids was first observed by Robert Williams Wood. The interaction between acoustic wave and matter was nil at atomic level that shows the zero interaction between chemical species and ultrasound at molecular level (Ali Dheyab et al. 2021). Further the acoustic cavitation process involves formation of bubbles and their disintegration that gives rise to intense heat emission. This intense heat is generated with high pressure abruptly and leads to the high-energy chemical reactions. This technique of sonochemistry is effectively applicable to the formation of nanostructured materials. Numerous nanomaterials including metal nanoparticles, metal oxide nanoparticles, nanostructured carbides, etc. have been synthesized using sonochemical synthesis route till now. Kumar et al. prepared silver nanoparticles by sonochemical route using starch and silver nitrate in specific amounts. Further for complete dissolution the mixture was stirred and agitated under sonication, and ultrasonic processor (DAIGGER GE 505, 500 W, 20 kHz) was used for radiation feed under effective operating conditions. The reduction of silver ions was observed at different time intervals for 22 days using a UV-visible spectrophotometer (Kumar et al. 2014). Different kind of solvents are used to dissolve the reagents before sonication in sonochemical synthesis, but some traditional solvents include

dichloromethane, hexane, hexadecane, isopropyl ether, diethyl ether, pentane, etc. D. Mahajan and R. R. Adzic used hexadecane and hexane in equal amounts as solvent and polyvinylpyrrolidone (PVP) as stabilizer in formation of molybdenum and palladium nanoparticles. In addition sodium borohydride ($NaBH_4$) was mixed with the solution as a reducing agent, and monitoring of sonolysis was completed through CO gas extraction for 3 h (Okoli et al. 2018). Sonochemical synthesis method is proven to be very effective in formation of nanoparticles of Ag, copper, and cobalt oxide using distilled water as solvent. The prepared mixture was sonicated for 10 min using high-intensity ultrasonic horn (Kis-csitari et al. 2008). The sonochemical process is a simple, easy, and rapid path to form nanostructured materials, but the main problematic step is to control the reactions among the precursors due to the ultrasonic radiations. The process basically involves two-step reactions, first is the reaction between reagents, and second is the actual formation of nanoparticles. The properties of final product can be specified if these two reactions are controlled.

4.3 Conclusion

The synthesis of uniformly sized nanoparticles is increasing day by day due to their unique and modified physical properties. These properties of nanomaterials are much more efficient than the properties of the bulk materials. The electrical, structural, optical, and magnetic characteristics of the nanoparticles are dependent on the synthesis procedure. Several synthesis methods have been evolved according to the required properties of materials and ease of operation. Among all the synthesis methods, sol-gel, polyol, electrochemical, citrate precursor, sonochemical, coprecipitation, hydrothermal, etc. are some most frequently used methods to synthesize nanoparticles. A number of synthesis routes are discussed in this chapter including the abovementioned methods. Selection of a particular synthesis process for nanoparticles depend on the required properties of final product and availability of resources. Energy consumption, reaction conditions, reagent compositions, and cost-effectiveness are some factors which should be taken care of during the selection of synthesis route. Some integrated techniques have shown better results than the conventional methods and proven to be of great advantage.

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