

Review of Production Routes of Nanomaterials

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Abstract Manufacturing and commercialization of nanomaterials are often hampered due to lack of appropriate large scale production of these nanomaterials. In this review article, we discuss different types of commercial scale production, including vapour, liquid and solid phase synthesis. The choice of production methods will influence the nanomaterials' properties and functionalities, scalability and production costs. This review emphasized on the fundamental process of each production method, including merits and drawbacks.

1 Introduction

Nanomaterials can be classified as materials with the size less than ~ 100 nm diameter and exhibit unique properties due to their nanoscale dimensions. With the development of new techniques and tools to synthesis and manipulate the size, shape and structure of nanomaterials, nanomaterials have been utilised in numerous technological applications, including catalytic systems, hydrogen storage, chemical sensors, biomedical applications, electronic, etc.

Although there is an extraordinary growth of using nanomaterials in the consumer end-products, many unprecedented materials innovations are still remain on the laboratory bench. One of the main challenges faced in commercialising the application of these nanomaterials is lack of appropriate large scale production of these nanoparticles. This chapter will discuss different types of commercial scale

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production, with the focus on the inorganic nanoparticles and carbon-related nanoparticles.

There are two main approaches to fabricate nanomaterials, namely top down and bottom up approaches. Figure 1 showed the schematic illustration of top down and bottom up approaches. The top down approach involved decreasing the size of the large uniform piece of material in order to create the required nanostructures from it. This method is very much used in the electronic industry, whereby lithography and etching techniques are applied to create the unique electronic circuitry. Mechanical milling is another common top down approach to generate nanoparticles. However, it poses some difficulties to generate uniform nanoparticles and is more likely to introduce internal stress, structural defects and contaminations. The bottom up approach, on the other hand, arranges the basic building blocks such as atoms or molecules into large nanostructures using chemical or physical forces.

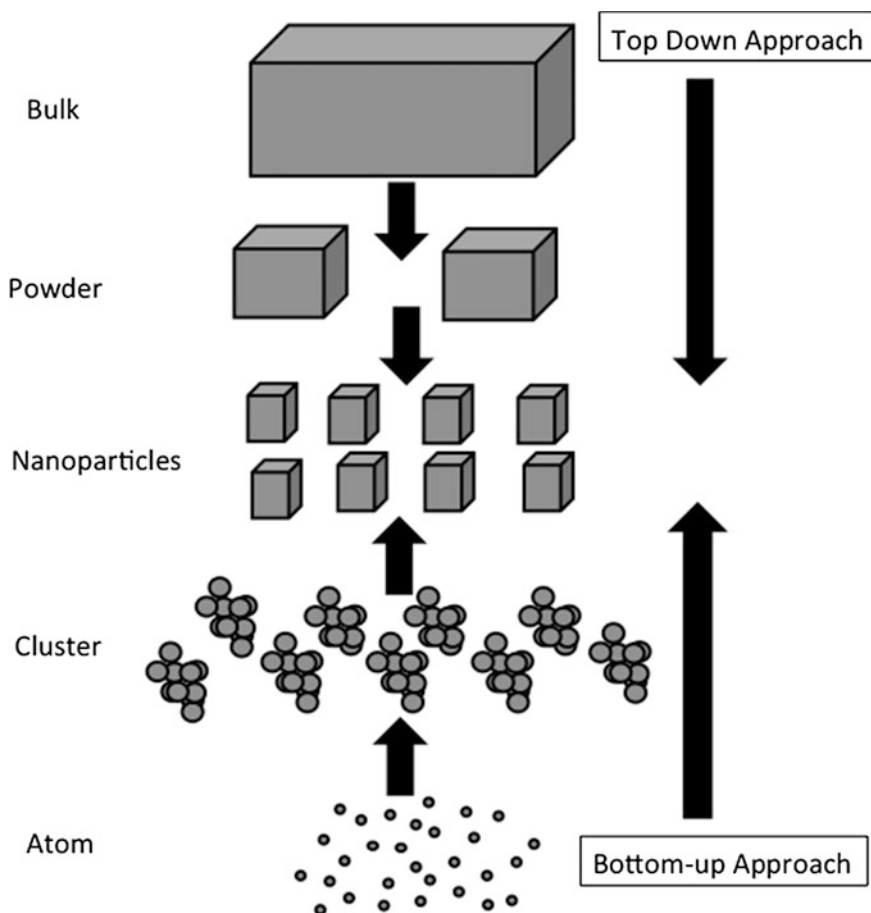


Fig. 1 Top down and bottom up approaches of nanoparticles production

The growth and self-assembly of the building blocks allows designing and controlling nanoparticles with more precise structures, shape, size and chemical composition.

This chapter will discuss the pros and cons of different production methods of creating nanomaterials. Scientists and engineers have to weigh between the needs for quantity or quality of the nanoparticles before they make their decision on choosing the right production methods to produce the nanoparticle with the desired properties.

2 Vapour Phase Synthesis

The works on vapour phase synthesis have evolved considerably since the early 1960s. Since then, there has been numerous of reports and references detailing the works on direct gas to particles conversion synthesis methods. Formation of nanoparticles through the vapour phase synthesis generally undergoes three main processes. It first started with nucleation of clusters from the supersaturated vapours, either homogeneously or heterogeneously. Homogeneous nucleation occurs when the vapour molecules condense to form nuclei without the presence of foreign particles or ions. Heterogeneous nucleation, on the other hand, occurs on the foreign nuclei, ions or surfaces. The nanocrystal nuclei condense from the constituent vapour later on undergoes growth due to collision and coalescence. Finally, the primary particles coagulate and form aggregates as the temperature decreases.

A number of different vapour phase methods of synthesis have been developed to increase the production of nanoparticles. This section will discuss the vapour synthesis methods including flame synthesis, chemical vapour condensation, arc discharge and laser ablation in more details.

2.1 Flame Synthesis

Flame synthesis is an inexpensive and widely used method for commercial production of nanoparticles. The most common examples of nanoparticles produced using the flame synthesis are titania, fumed silica and carbon blacks (Swihart 2003; Stamatakis et al. 1991), with the annual production rate reaching several million metrics tons. These nanoparticles are used extensively as pigments, catalysts, flowing aids and in telecommunication.

In flame synthesis, synthesis of particles is carried out within the flame, produced by the combustion reactions. Flame heat is employed to initiate the chemical reactions and to form flames of dust clouds comprising the corresponding metals. The volatile precursors are oxidized within the flame to form metal-oxide monomers, which later nucleate and coalescence to form aggregated nanoparticles. Unlike the wet chemistry synthesis route, the desired crystal phases are formed directly without the need for a calcination step due to the high temperature of the

flame. Figure 2 shows a typical flame synthesis unit, which consists of a precursor unit, a burner incorporated with the gas delivery systems, and a filter unit for particle collection.

The main fuels used in flame synthesis are methane, hydrogen or ethylene and air or oxygen as oxidants. These gases sometimes are diluted with inert gases such as argon, helium or nitrogen. An example of the flame synthesis is synthesis of iron oxide nanoparticles from iron pentacarbonyl precursor using $\text{CH}_4/\text{O}_2/\text{Ar}$ flame, as reported by Buyukhatipoglu and Morss Clyne (2010). It was found that change of flame configuration from diffusion flame configuration to inverse diffusion flame configuration reduces the size range of the nanoparticles from 50–60 to 6–12 nm.

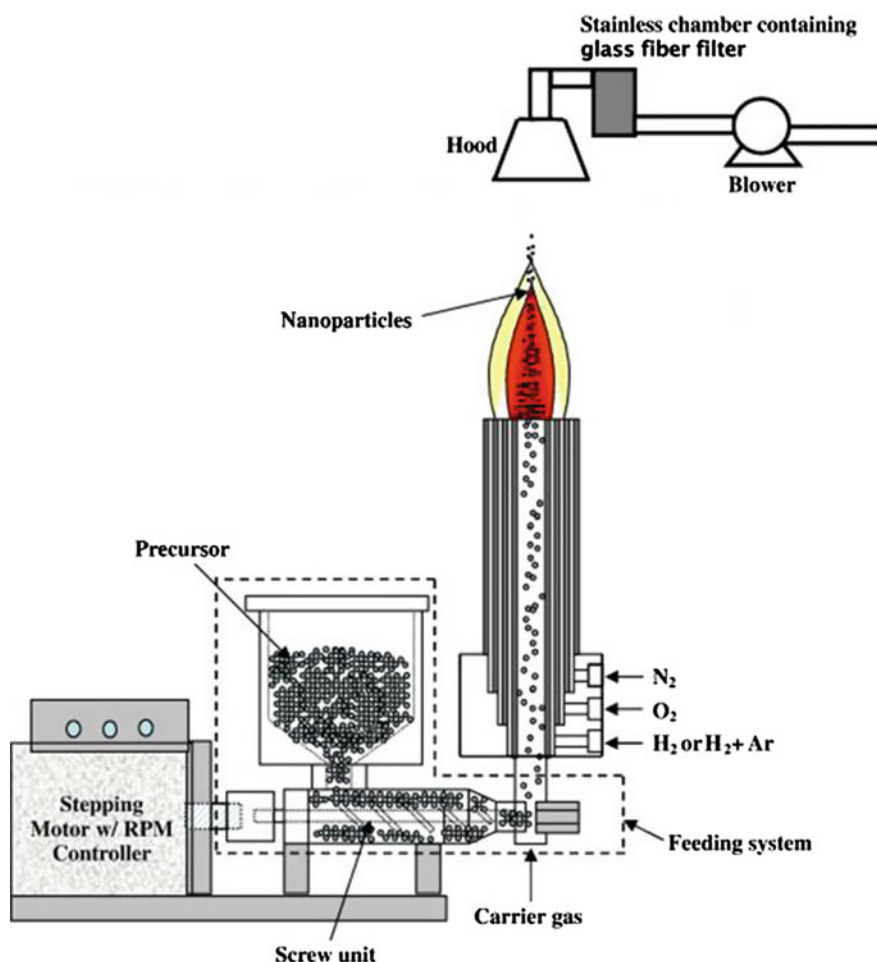


Fig. 2 Typical set up for flame synthesis (Yang et al. 2010). Reprinted from Powder Technology, 2010, 197(3), Yang, S. et al., A flame metal combustion method for production of nanoparticles, pp. 170–176. Copyright 2010, with permission from Elsevier

The addition of N_2 gas cooling further lowers the flame temperature and leads to the formation of magnetite Fe_3O_4 instead of hematite $\alpha-Fe_2O_3$ (Buyukhatipoglu and Morss Clyne 2010). The main parameters influencing the flame synthesis of iron oxide are the flame temperature and particle residence time. The characteristics of the flame-made nanoparticles are also controlled by the composition of the precursors, burner configuration, turbulence, precursor injection and particle collection location, supplementary laser irradiation and external electric fields (Buyukhatipoglu and Morss Clyne 2010; Yang et al. 2010).

Apart from the simplicity of experimental set up, flame synthesis involves only a 'one-step' process. A wide array of precursors can be employed using the flame synthesis. Thus, a broad range of the nanosized powders can be synthesized using this method. Johannessen et al. also reported other complex products including composite metal oxide (e.g. $ZnAl_2O_4$, $MgAl_2O_4$ spinel) and supported noble catalysts are synthesized using the flame synthesis (Johannessen et al. 2004). The synthesized products require no subsequent post-processing and less process waste is obtained as flame synthesis is a solvent free process. In spite of simplicity and efficiency of flame synthesis of nanoparticles, agglomeration of nanoparticles remains a significant challenge for this synthesis method.

2.2 *Laser Ablation Method*

Since the discovery of laser decades ago, laser has been used extensively in various applications including nanomaterials synthesis, surface cleaning and surface nanopatterning (Zhong et al. 2012). Synthesis of nanoparticles using laser ablation involves vaporization of source materials or precursor through laser irradiation. A wide variety of materials including high melting point elements or compounds can be produced at low temperature, including metals such as silver (Tsuji et al. 2008), silicon (Khang and Lee 2010), ceramics, oxide composites and nitrides (Kononenko et al. 1997). Figure 3 displays an illustration of laser ablation systems connected with particles analyser.

The most common source laser source for ablation is neodymium-doped yttrium aluminium garnet (Nd:YAG). During laser ablation, the laser beam is irradiated onto the surface of the target materials at specific incident angle, either 90 or 45 degree. The energy of the laser beam can be transferred to the target materials through different pathways depending on the laser power, wavelength and pulse duration. Photothermal ablation will take place if low laser power (100–500 MW/cm²) and longer pulse length (>10 ns) are used (Wang et al. 2002). During the photothermal ablation, the free or bound electrons on the target materials will absorb the energy. The excited electrons transfer the energy through the collision interaction with other electrons and lattice phonons, resulting in heat transfer and increase of the temperature of the material, which melts the target material (Zhong et al. 2012). The melted material continues to absorb the energy from the laser beam and subsequently forms the plasma plumes on the surface. The atoms on

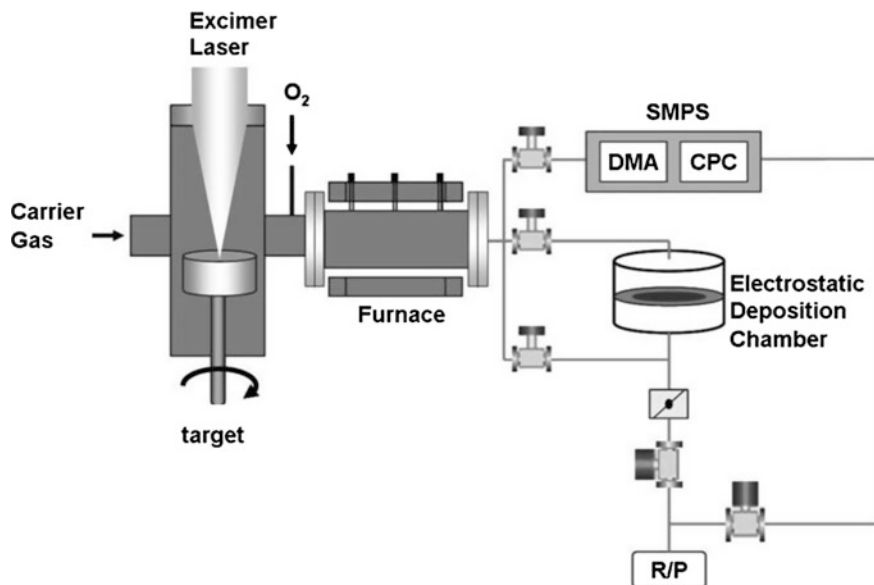


Fig. 3 Schematic illustration of laser ablation systems connected with particles analyser (Khang and Lee 2010). Reprinted from Journal of Nanoparticle Research, 2010. 12(4), Khang, Y. and Lee, J., Synthesis of Si nanoparticles with narrow size distribution by pulsed laser ablation, pp. 1349–1354. Copyright 2009, with permission from Springer

the heated surface are finally ejected and vaporized due to the rapid volume expansion.

Plasma assisted ablation is the second pathway, in which temporary plasma is produced above the target material when high peak power in the laser pulse is used. Generally, high laser power ($>500 \text{ MW/cm}^2$) is applied to provide high-energy ions, which bombard the surface of the target materials. The atoms of the target materials are then heated and ejected from the surface. Due to the high energy of the laser ablation, both photothermal and plasma assisted ablation will leave craters on the surface of the target material. At the same time, the liquid droplets of the melted materials solidified and create a rim. Photochemical or photolytic ablation, on the other hand, occurs when laser photon energy similar to the bonding energy of the molecules is absorbed and lead to dissociation of the bond. This later causes the ejection of the material. Unlike photothermal and plasma assisted ablation, only the target material irradiated by the laser beam is ablated and the remaining area remains unaffected for the photochemical ablation Wang et al. (2002).

Laser ablation technique is applicable to synthesis a wide range of materials with the complex chemical structure and stoichiometry composition similar to the target. The properties of nanoparticles synthesized using this method can be easily controlled by altering the laser pulse energy, pulse fluence, repetition rate, pulse duration and laser spot size. Due to the monochromatic characteristics of laser,

selective excitation of molecules will occur by applying the appropriate laser wavelength. Apart from having the ablation carried out in vacuum and gas, there are also numerous studies reported to have laser ablation in the liquid medium (Tsuji et al. 2008; Neddersen et al. 1993).

In the last decade, Pulsed Laser Ablation in Liquid (PLAL) has emerged as an alternative synthesis technique to generate, excite, fragment, and conjugate a large variety of ultrapure nanostructures for large scale production (Intartaglia et al. 2014). This technique has overcome most of the shortcomings of traditional wet chemical synthesis, presenting a simple, straightforward and versatile technique to produce ligand-free nanoparticles which are a prerequisite for specific biological or electrical applications (Barcikowski and Compagnini 2013). In addition, the liquid phase can be tailored in order to produce viscosities suitable for ink jet printing or other conventional controlled fluid surface coating techniques (Bagga et al. 2015). Ablation in liquid medium offers a number of advantages including higher cooling rate which tends to form smaller size nanoparticles, allows surface modification, and increase stability of nanoparticles. While the liquid can be as benign as deionised water, other solvent species used could affect the chemical composition of the nanoparticles synthesized in a positive or negative manner depending on application.

2.3 Arc Discharge Method

Fabrication of nanoparticles using arc discharge method has gained much interests due to its simplicity in apparatus set up and ability to scale up the production rate of nanoparticles (Ashkarran et al. 2009). Different types of nanoparticles have been reported to synthesize using this method, ranging from metal to refractory metals. One of the most widely reported nanoparticles synthesized using this method is carbon nanotubes (CNTs) using the direct current (DC) arc discharge. For the production of single walled carbon nanotubes (SWNTs), catalyst metals for instance Ni, Y, Fe, Co or Mo are required, whereas it is not required for the multi-walled carbon nanotubes (MWNTs).

Arc discharge involves very simple setups with two electrodes acting as cathode and anode respectively. These electrodes are subjected to a potential and subsequently cause an electrical breakdown. Arc discharge generally requires low voltage (few to several tens of V) and high current density (10^5 – 10^{11} A/m²) (Xu 2007). Synthesis of nanoparticles using arc discharge method can be either in pulsed (Su et al. 2014) or continuous mode. To produce MWNTs and SWNTs, high purity graphite are used as electrodes and arc discharge can be carried out in helium (Waldorff et al. 2004) or hydrogen gas (Zhao et al. 1997). Figure 4 showed a schematic illustration of the experimental set up for the arc discharge.

During the arc discharge, electrons are emitted either through heating or field emission due to large electric field generated. Plasma is thus formed due to the ionization and this exerts large amount of heat, which subsequently evaporate the

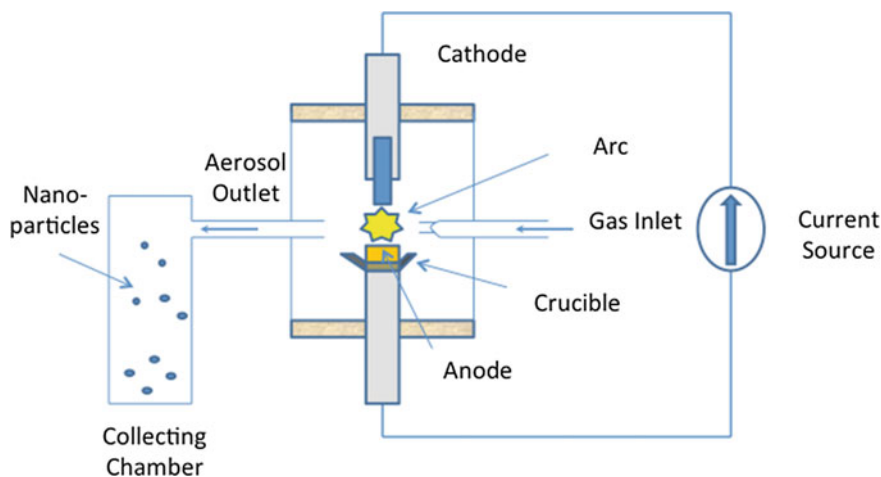


Fig. 4 Schematic illustration of the experimental set up for arc discharge in gas chamber

metal of interest into metal vapor. The plasma also enhances the kinetics of the chemical reactions, dissociating atoms and radicals. The metal vapor later nucleates and forms nanoparticles in the surrounding cooling medium (Ashkarran et al. 2008). The metal vapour can either be quenched by mixing with cold gas or by the surrounding liquid medium.

Su et al. has reported synthesis of ZnO nanoparticles with the size varies from 10 to 25 nm using the pulsed arc discharge in deionized water (Su et al. 2014). Compare with the arc discharge in vacuum, arc discharge in deionized water is more economical, does not involve the use of organic solvent and chemicals and so has potential for the environmentally friendly and biocompatible production of nanoparticles. At the same time, it also avoids the use of toxic, explosive and corrosive gases and reduces the use of costly vacuum equipment. In most cases, nanoparticles synthesized using the arc discharge method are subjected to high cooling rates. Due to the non-uniform cooling, the uniformity of the nanoparticles produced using this method often deteriorates. Thus, control of particle nucleation and growth continuously require much attention.

3 Liquid Phase Synthesis

Liquid phase synthesis or wet chemical method is another important nanoparticles synthesis approaches widely used by the industries. A typical liquid phase synthesis involves mixing solution of different ions with well-defined quantities and subjected it to controlled heat, temperature and pressure to promote the formation of insoluble nanoparticles, which precipitates out of the solution. Unlike the vapour phase synthesis, liquid phase synthesis is more energy efficient and generally

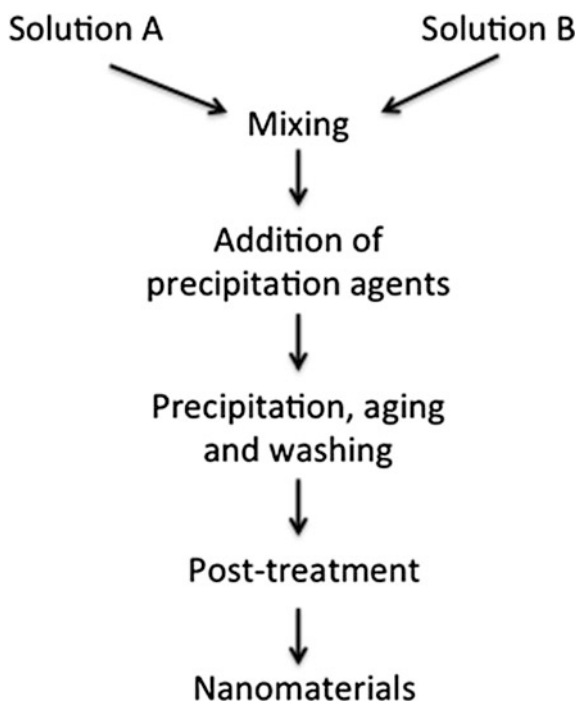
carried out at low temperatures and below the atmospheric pressure. It does not require high temperature, expensive energy source or high pressure. Besides, liquid phase synthesis also enable more precise control of the stoichiometry composition of the end products and the nanoparticles synthesized can be stabilized or functionalized during the synthesis process.

Although liquid phase synthesis offers many advantages, it still faces some challenges for their industrial applicability and scalability. Very often, the chemicals used in the liquid phase synthesis are toxic, explosive and corrosion, which pose a significant risk to occupational health and environments. Furthermore, nanoparticles synthesized require further processing and purification. The purity of the nanoparticles synthesis using liquid phase synthesis is usually less than those synthesized using the vapour phase synthesis.

3.1 Chemical Precipitation

Chemical precipitation is one of the most widely used liquid phase synthesis method to synthesize complex oxides. The main advantages of this method are due to its simplicity and ability to yield final products of near perfect stoichiometry without high temperature treatment. Figure 5 summarized an overall process involves in the chemical precipitation.

Fig. 5 A summary of the procedures for chemical precipitation of nanoparticles (Zhong et al. 2012)



It first starts with dissolving the starting materials, usually metal oxides, metal salts or their hydrates to form aqueous solution. The solution later on mixed with another solution with dissolved precipitation agent either directly or by drop-by-drop method. Meanwhile, co-precipitation will take place when the reactant products dissolve in the solvent and precipitation agent is added to solution to precipitate the reaction products. The precipitates later undergo aging process to obtain larger particles and these particles are collected through filtration or centrifugation. Further washing with distilled water, ethanol or other solvents is required to clean the impurities and to obtain high purity nanoparticles. Post treatment, including annealing, sintering or calcination is carried out to obtain nanoparticles with desired crystal structures and morphology.

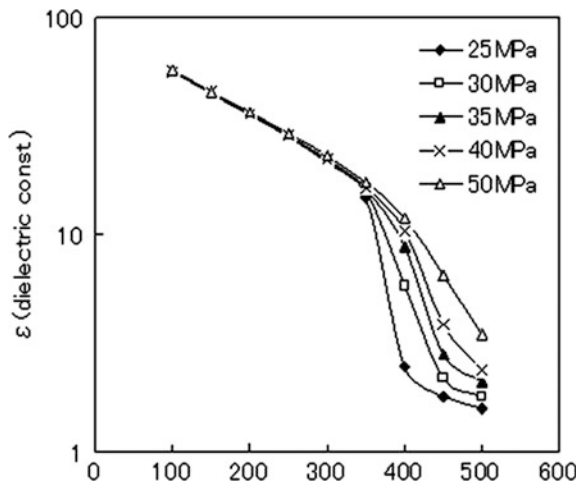
Chemical precipitation involves simultaneous occurrence of nucleation, growth, coarsening and/or agglomeration. However, the fundamental mechanism of chemical precipitation is not fully understood due to the difficulties in isolating each process for independent studies (Cushing et al. 2004). The critical step to precipitation is nucleation, which is governed by the degree of supersaturation. A large number of small crystallites are initially formed and subsequently grow through secondary processes, for instance Ostwald ripening and aggregation to form more stable particles. Control of the nucleation and growth processes is necessary, as it will affect the characteristic of the nanoparticles synthesized. The main parameters influencing the as-synthesized nanoparticles are surface charge or zeta potential, reactant concentration, reaction temperature, pH, degree of mixing and recrystallization of the precipitates. By controlling these parameters, the particles size, particle size distribution and stoichiometry of the nanoparticles can be manipulated. The drawbacks of this method are it requires the use of a lot of chemicals and may generate potentially hazardous waste products. Besides, most of the nanoparticles synthesized using chemical precipitation requires stabilizers and a number of post-processing treatments are required to improve the purity of the end products.

3.2 *Hydrothermal Method*

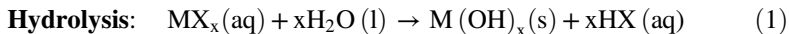
Hydrothermal method is a simple and versatile method for the synthesis of inorganic nanomaterials under high temperature and high-pressure condition. It is possible to alter the characteristic of the nanoparticles by adjusting processing conditions including temperature, pressure or precursor concentration. The density of water and dielectric constant are highly dependent on the temperature and pressure, as shown in Fig. 6 (Hayashi and Hakuta 2010). A drop in the dielectric constant of the water is closely related with the increase of the temperature and decrease in pressure. As the dielectric constant of the water decreases, the reaction rate enhances significantly and ionic species starts to precipitate to form solid phase.

Hydrothermal synthesis is usually carried out in a closed vessel and the precursors solutions, for instance metal nitrate, metal chloride or acetates undergo two steps formation mechanism to form the nanoparticles, as shown in Eqs. (1) and (2).

Fig. 6 Dielectric constant of water at various temperatures and pressures (Hayashi and Hakuta 2010). Reprinted from Materials, 2010. 3(7), Hayashi, H. and Hakuta, Y., Hydrothermal Synthesis of Metal Oxide Nanoparticles in Supercritical Water, pp. 3794–3817. CC BY-NC-SA 3.0 licence



M denotes the metal and X the anions. The hydrated metal ions are initially hydrolysed to form metal hydroxide. The metal hydroxides later undergo dehydration to form metal oxide (Adschiri et al. 1992).



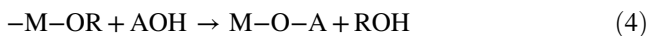
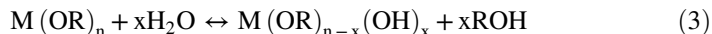
The hydrothermal synthesis method has been used extensively to fabricate TiO_2 , which is an important photocatalyst for the decomposition of toxic chemicals. Mi et al. has reported a fast and one step synthesis of rutile TiO_2 nanoparticles using isopropanol-water as solvent at temperature of 300 °C and pressure of 25 MPa (Mi et al. 2012). TiO_2 with the average size of 22 nm was synthesized at the supercritical condition, which allow precipitation of nanoparticles instantaneously as the temperature of the metal salt brought very rapidly above the critical point. Hydrothermal method is easily scalable with the development of the continuous flow hydrothermal reactors. Furthermore, it can be used to synthesize complex inorganic nanoparticles by altering the synthesis parameter. However, the disadvantage of this method is there is a need to find proper chemical reactions and processing conditions for different nanoparticles.

3.3 Sol-Gel Method

Similar with the chemical precipitation and hydrothermal method, sol gel synthesis takes place in an inexpensive reactors, allow precise stoichiometry control and the

nanoparticles synthesized are generally having smaller particles size and narrow size distribution. As the name implies, it involves the formation of a sol-gel containing nanomaterials which later dries, sinters and calcinates at specific temperature in order to obtain desired nanomaterial.

Typically, the starting materials can be metal alkoxides or metal chlorides used to form solvated metal precursor (sol). The precursors are hydrolysed with water or alcohol to produce hydroxide, following the Eq. (3) below. Apart from water and alcohol, an acid or base can also help to hydrolyse the precursor. Equation (4) shows the reaction takes place between alkoxide and the acid (Tavakoli et al. 2007)



The hydroxide molecules later form oxide- or alcohol bridged network (the gel) through polycondensation or polyesterification reactions. At this stage, gelation occurs and a dense gel consisting of three-dimensional skeleton with interconnected pores is obtained. The properties of the final products are largely influenced by the rates of hydrolysis and condensation. Slower and more controlled hydrolysis will yield smaller particles. The rate of hydrolysis is dependent on the electronegativity of metal atoms and its coordination number. More electronegative metal tends to have faster hydrolysis rate. Meanwhile, the hydrolysis rate reduces as the coordination number of the metal increase due to the steric hindrance (Srivastava 2013).

After the solution condensation to a gel, it will follow by aging of the gel, during which polycondensation continues until the gel form a solid mass. At the same time, the solvent is removed from the gel pores and accompanied by contraction of gel network. The gel is subsequently calcined at high temperature. This results in densification and decomposition of the gel, whereby the pores of the gel network collapsed and remaining organic species are volatilized (Cushing et al. 2004). The thermal treatment is crucial to obtain nanoparticles with desired crystal structure, crystallinity and size.

Sol gel method has been used to synthesize numerous of metal oxide nanoparticles and nanocomposites. Niznansky has reported synthesis of Fe_2O_3/SiO_2 nanocomposites by one-step sol-gel method. Commercial precursors (TEOS and iron (III) nitrate) were dissolved in the methanol solution. After gelation and drying, the transparent nanocomposites with the size 2–10 nm was obtained, depending on the Fe concentration and heating temperature (Niznansky et al. 1997). Despite the advantages of nanoparticles synthesis using sol-gel method, there are also some drawbacks on using this method including high cost of raw materials, presence of residual carbon, health hazard of organic compounds and possible cation segregation during thermal decomposition (Wang et al. 2002).

4 Solid Phase Synthesis

Compare with vapour phase and liquid phase synthesis, solid phase synthesis is the oldest techniques to synthesize nanomaterials. Solid phase synthesis involves physically pounding coarse powders into finer powders. Most of the solid phase synthesis is carried out using planetary or rotating ball mills. The advantages of these techniques are they are simple and require low cost equipment. However, large particle size, broad size distribution and contaminations often pose a major challenge for this type of synthesis.

4.1 Mechanical Milling

Mechanical milling refers to mechanical disintegration of the solid metals into smaller particles after subjected to the high-energy collision from the balls. This is a common process used for the production of hard metals, oxide powder, inter-metallics, ceramics and composites. Mechanical milling is largely applied for particle size reduction, solid state alloying, mixing or blending and particle shape changes.

Grinding process is carried out, by rotating the jar filled with hard balls and powdered metals to be milled at high speed. Collision between the balls and the metals later crushed it into powders with finer size. Apart from causing fractures, milling operation also deforms and cold-welds the impacted particles. As a result of cold-working during the mechanical milling, the average grain size can be reduced from the range of 50–100 μm to the range of 2–20 nm. Milling can be performed in either wet or dry conditions. To reduce oxidation on the freshly created particle surfaces, mechanical milling is preferably conducted in protective atmosphere.

Mechanical milling is relatively simple operation with the ease of scalability and ability to form nanostructured powders with different structures (nanocrystalline, amorphous, quasicrystalline) and atomic bonding (metal/metal, metal/semiconductor, metal/ceramic, etc.) (Wang et al. 2002). However, the major drawback of mechanical milling is non-uniformity of particles sizes. Although there has been significant improvement in the milling instrument and quality of grinding media, nanoparticles produced using mechanical milling generally has large particles size distribution. Furthermore, mechanical milling also have high tendency to introduce contamination from the milling media and atmosphere. Milling products are very sensitive to the grinding conditions. Very often, surface-active grinding aids are used to reduce surface energy of particles and enable shorter grinding time. To further reduce the particle size, longer milling time is required (Schatt and Wieters 1997). This subsequently introduced more impurities into the powder particles. It is always problematic to remove these impurities and grinding aids.

5 Summary

Vapour phase synthesis generally is a clean process and able to yield nanoparticles with high purity. However, the nanoparticles synthesized using this method are mainly polycrystalline in nature and it also suffers from the agglomeration problem which can annihilates the desired nanoparticles properties. Liquid phase synthesis or wet chemical method, on the other hand, allows precise control of the stoichiometry composition and it is relatively easy to scale-up as it does not require high temperature and high-pressure condition. However, purity of the nanoparticles synthesized remains a major issue as stabilizers or capping agents needs to be added to prevent agglomeration. Similar with the liquid phase synthesis, solid phase synthesis also faces the problem on contaminations. However, it can be used to form alloy compounds at low temperature and is a relative simple and efficient method for scale up production.

All the vapour, liquid and solid synthesis methods have its own merits and drawbacks. Fabrication and processing of nanoparticles often faces the challenges to ensure nanoparticles have desired size, uniform size distribution, morphology, crystallinity, chemical composition and microstructures. Furthermore, the issues of Ostwald ripening and agglomeration of nanoparticles need to be overcome. Although there has been continuous research and development to meet these challenges, scalability and production costs of the production methods need to take into consideration. Hence, the choice of the production methods is very much dependent on the need and application of the nanoparticles.

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