

Nanomaterials: Preparation and Characterization—A Review

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Abstract: Nanomaterials have become an important new class of advanced materials because their physicochemical properties can have significant differences from the properties of the corresponding bulk materials. These differences may arise due to their unique features namely nanoscale dimensions, high specific surface area, quantum-size effects, surface energy and size-dependent structural effects. Nanomaterials are being used in catalysis, electronics, energy conversion and storage, environmental control, sensing, medicine and biotechnology. Materials in the nano regime can exhibit significant enhancement in certain properties and it is a fundamental requirement that these properties can be accurately predicted and controlled which in turn requires fine control of the physical and chemical properties of the materials concerned. As the performance of a nanomaterial is critically influenced by its composition, size, morphology, crystallinity, surface properties and degree of aggregation, the controlled preparation and thorough characterization of nanomaterials are vital. This review considers the main methods available for the preparation of nanomaterials with particular emphasis on top-down and bottom-up approaches. Physical techniques (including ball milling, laser ablation, sputtering and laser ablation, lithography) and chemical techniques (including sol-gel, precipitation, hydrothermal/solvothermal syntheses, chemical vapour deposition and microemulsion methods), along with biological or environmentally friendly preparation routes are summarized. A broad discussion of the main methods used to characterize nanomaterials including X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), atomic force microscopy (AFM), dynamic light scattering (DLS), Fourier-transform infrared (FTIR) spectroscopy, ultraviolet-visible (UV-Vis) spectroscopy, X-ray photoelectron spectroscopy (XPS), energy dispersive X-ray analysis (EDS) and Brunauer-Emmett-Teller (BET) analysis as well as the relationship between preparation conditions and measurable structural and surface properties are presented. The question of reproducibility, agglomeration, scale-up, environmental impact and the complementary nature of different techniques are then considered.

Keywords: Nanomaterials; nanoparticles; nanostructures; synthesis; top-down; bottom-up; characterization; XRD; TEM; SEM; DLS; green synthesis.

INTRODUCTION

Nanotechnology is the science and technology of designing and making matter with features at dimensions generally in the nanometre range. Nanomaterials are typically described as materials with one or more exterior dimensions in roughly the 1-100 nm range [Cao, G., & Wang, Y. 2011; Poole, C. P., & Owens, F. J. 2003], although there are differences in the definition according to application and discipline. These small size can lead to novel behaviour and function originating from the increased surface/volume ratio, the changes in the electronic structure, the surface effects and the quantum confinement in very small structures.

Depending on their dimensions we can distinguish four categories: zero dimensional (0D) nanoparticles and quantum dots, one dimensional (1D) nanowires, nanorods and nanotubes, two dimensional (2D) nanosheets and thin film structures and three dimensional (3D) nanostructured assemblies. They can be classified according to chemical composition as metallic, metal-oxide, semiconductors, carbon based, polymeric, ceramic, magnetic, and composite

nanomaterials [Cao, G., & Wang, Y. 2011; Poole, C. P., & Owens, F. J. 2003; Gleiter, H. 1995].

Beyond the chemical composition, other parameters (particle size, shape, crystallinity, surface charge, defects, porosity, surface functionalization, and aggregation state) strongly influence optical, electrical, magnetic, catalytic and biological properties; preparation and characterization should be thus considered as interconnected processes rather than separate stages: the synthesis method determines the nucleation and growth conditions and it significantly influences the final properties such as size, morphology, crystal phase, surface chemistry and dispersibility [Poole, C. P., & Owens, F. J. 2003; Sajid, M., & Płotka-Wasyłka, J. 2020].

Nanoparticles are currently widely investigated for analytical chemistry, environmental, agriculture, pharmaceutical and biomedical applications [Sajid, M., & Płotka-Wasyłka, J. 2020]. The successful development of these nanomaterials with well-controlled sizes and reproducible properties is thus essential for translating these experimental findings from the laboratory to the real world.

PREPARATION OF NANOMATERIALS

Nanomaterial preparation methods are broadly divided into top-down and bottom-up approaches. In top-down processes, bulk materials are physically or chemically reduced to nanoscale dimensions. In bottom-up processes, nanoparticles or nanostructures are assembled from atoms, ions or molecules through nucleation, growth and self-assembly mechanisms [Cao, G., & Wang, Y. 2011; Poole, C. P., & Owens, F. J. 2003].

Top-Down Approaches

Top-down methods begin with a bulk solid and reduce its dimensions through physical or physicochemical processes. Important methods include mechanical milling, lithography, laser ablation, sputtering, arc discharge and etching.

Mechanical Milling

Mechanical or ball milling is one of the simplest approaches for producing nanostructured powders. Bulk material is placed together with milling media and subjected to repeated impact, fracture and cold welding. Continued milling reduces crystallite size and can produce nanocrystalline alloys, oxides, carbides and composite materials.

The principal advantages are relatively simple equipment, scalability and the ability to process large quantities. However, contamination from milling media, broad particle-size distributions, structural defects and agglomeration can occur. Consequently, subsequent characterization is necessary to distinguish particle size from crystallite size.

Lithography and Etching

Lithographic techniques are particularly important for fabrication of nanoscale patterns and devices. Photolithography, electron-beam lithography and related techniques allow controlled patterning of surfaces. Chemical or plasma etching can subsequently remove selected regions.

The major advantage is excellent dimensional and positional control. The disadvantages include high equipment cost, complex processing and relatively low suitability for high-volume production of free nano powders.

Laser Ablation

Laser ablation involves irradiation of a bulk target with a high-energy laser pulse. Material is vaporized or ejected from the target and subsequently nucleates to form nanoparticles. Depending on the surrounding medium,

nanoparticles may be generated in vacuum, gases or liquids.

Laser ablation can produce relatively pure materials because chemical reducing agents and stabilizers may be avoided. However, energy consumption, equipment cost and comparatively low production rates can limit its large-scale application.

Bottom-Up Approaches

Bottom-up synthesis constructs nanomaterials from atoms, ions or molecules. It generally provides greater control over nucleation and growth and is therefore widely used for producing nanoparticles with controlled morphology and composition.

Chemical Precipitation and Reduction

Chemical precipitation is widely employed for metal oxides and other inorganic nanoparticles. It commonly involves controlled addition of precipitating agents to a precursor solution followed by nucleation, growth, washing and drying. For metallic nanoparticles, reduction of metal ions can generate nanoscale nuclei that subsequently grow into particles.

The classical synthesis of colloidal gold using citrate reduction established an important basis for controlled nanoparticle formation [Turkevich, J. *et al.*, 1951]. In such systems, precursor concentration, reducing-agent concentration, pH, temperature and stabilizer concentration affect nucleation and particle growth.

Sol-Gel Method

The sol-gel method is particularly useful for oxide nanomaterials such as SiO₂, TiO₂, ZnO and related mixed oxides. Metal alkoxides or inorganic precursors undergo hydrolysis and condensation to produce a colloidal sol, followed by gelation, drying and, when required, calcination.

The method offers good compositional homogeneity and allows substantial control over particle size, porosity and morphology. The hydrolysis ratio, pH, precursor concentration, solvent and aging conditions are important variables. The classical Stöber process demonstrated controlled formation of relatively uniform silica particles through hydrolysis and condensation of alkyl silicates in alcoholic media using ammonia as catalyst [Stöber, W. *et al.*, 1968].

Hydrothermal and Solvothermal Synthesis

Hydrothermal synthesis uses aqueous solutions at elevated temperatures and pressures, whereas solvothermal synthesis uses non-aqueous solvents. Precursors are sealed in an autoclave and transformed under controlled temperature and pressure.

These methods are useful for preparing crystalline metal oxides, sulphides, phosphates and other inorganic nanostructures. Crystal growth can occur under relatively controlled conditions, frequently producing well-defined morphologies. Reaction temperature, duration, solvent, precursor concentration and mineralizer concentration influence the final structure.

Chemical Vapour Deposition

Chemical vapour deposition (CVD) involves decomposition or reaction of volatile precursors at a heated substrate. It is widely employed for carbon nanotubes, graphene, semiconductor nanostructures and thin films. Parameters including substrate temperature, precursor concentration, carrier-gas flow and pressure determine deposition rate and morphology.

CVD can produce high-quality nanostructures and is compatible with device fabrication, although relatively high temperatures and specialized equipment may be required.

Microemulsion and Reverse-Micelle Synthesis

Microemulsions provide nanoscale reaction environments through surfactant-stabilized droplets. These droplets can act as nanoreactors, restricting nucleation and growth and thereby assisting particle-size control. Water-in-oil reverse micelles are particularly useful for synthesizing metallic, oxide and semiconductor nanoparticles.

The method provides good control of particle dimensions but can involve significant amounts of surfactants and organic solvents, requiring additional purification.

GREEN AND BIOLOGICAL SYNTHESIS

Increasing environmental concerns have stimulated interest in biological and green synthesis. Plants, microorganisms, enzymes and biomolecules can act as reducing, stabilizing and/or capping agents during nanoparticle formation.

Plant-mediated synthesis is particularly attractive because plant extracts contain phytochemicals such as polyphenols, flavonoids, terpenoids,

proteins and sugars that can participate in metal-ion reduction and nanoparticle stabilization [Yadi, M. *et al.*, 2018; Marslin, G. *et al.*, 2018; Saratale, R. G. *et al.*, 2018]. Such methods can reduce reliance on some conventional reducing and stabilizing chemicals.

However, green synthesis should not automatically be considered environmentally benign. Extract composition can vary with plant species, geographical origin, season, extraction procedure and storage. Such variability may affect particle size, morphology, surface chemistry and reproducibility. Therefore, standardized extraction and rigorous physicochemical characterization are essential [Yadi, M. *et al.*, 2018; Marslin, G. *et al.*, 2018]. Reviews published by 2020 reported extensive use of plant extracts for synthesis of silver, gold and other metallic nanoparticles and highlighted both their potential and reproducibility challenges [Yadi, M. *et al.*, 2018; Marslin, G. *et al.*, 2018; Saratale, R. G. *et al.*, 2018].

FACTORS CONTROLLING NANOPARTICLE FORMATION

Regardless of synthesis route, several parameters determine the final properties of nanomaterials.

Precursor concentration: Increasing precursor concentration can increase nucleation and growth rates, but excessive concentrations may promote aggregation.

pH: pH influences precursor hydrolysis, reduction potential, surface charge and particle stability. It is therefore particularly important in precipitation and sol-gel processes.

Temperature: Temperature affects reaction kinetics, nucleation rate, diffusion and crystallization. Higher temperatures can increase crystal growth and improve crystallinity but may also increase particle size.

Reaction time: Longer reaction periods may promote crystal growth, Ostwald ripening and aggregation.

Reducing and capping agents: Reducing agents control the conversion of ions into atoms, whereas surfactants or polymers can restrict growth and prevent aggregation.

Solvent: Solvent polarity, viscosity and coordinating ability can modify precursor chemistry and growth kinetics.

Post-treatment: Washing, drying, calcination and annealing can substantially modify phase

composition, crystallinity, surface chemistry and particle aggregation.

Thus, optimization should focus not merely on obtaining a nanoscale particle but on achieving the required combination of size, morphology, crystallinity, surface chemistry and stability.

CHARACTERIZATION OF NANOMATERIALS

Characterization is essential for confirming successful synthesis and establishing structure–property relationships. No single technique can provide complete information about a nanomaterial; consequently, complementary techniques are normally required.

X-Ray Diffraction

X-ray diffraction (XRD) is primarily used to determine crystal structure, phase composition, lattice parameters and crystallinity. Diffraction peaks provide fingerprints of crystalline phases. The mean crystallite size can frequently be estimated using the Scherrer equation:

$$D = \frac{K\lambda}{\beta \cos \theta}$$

Where D is the mean crystallite size, K is the shape factor, λ is the X-ray wavelength, β is the peak broadening and θ is the Bragg angle.

Importantly, the crystallite size obtained from XRD is not necessarily equivalent to the physical particle size. A particle may contain several crystallites, and peak broadening may also arise from micro strain and instrumental effects. Therefore, XRD results should be correlated with electron microscopy.

Scanning Electron Microscopy

SEM provides information about surface morphology, particle shape, aggregation and approximate particle dimensions. Modern SEM systems can achieve nanoscale spatial resolution, although the exact resolution depends on the instrument and sample.

SEM is particularly useful for comparing morphology before and after processing. Coupling SEM with energy-dispersive X-ray spectroscopy (EDS) provides qualitative or semi-quantitative elemental information and helps identify compositional heterogeneity.

Transmission Electron Microscopy

TEM is one of the most important tools for nanoparticle characterization because electrons transmitted through a thin sample can provide direct images of individual particles. Particle size,

shape, aggregation and morphology can therefore be assessed.

High-resolution TEM (HRTEM) can reveal lattice fringes, allowing direct observation of crystalline planes. Selected-area electron diffraction (SAED) can provide information about crystallinity and phase structure. TEM is especially valuable when nanoparticles are below the resolution or reliable measurement range of conventional optical techniques.

Atomic Force Microscopy

AFM produces three-dimensional surface topography by scanning a sharp probe over the sample surface. It can operate under ambient conditions and can provide information about surface roughness, particle height and morphology.

An advantage of AFM is that conductive samples are not necessarily required. However, tip–sample interactions and tip geometry can affect apparent particle dimensions.

Dynamic Light Scattering

DLS is widely used to determine the hydrodynamic diameter and size distribution of nanoparticles dispersed in liquids. The technique measures fluctuations in scattered light caused by Brownian motion.

DLS is rapid and requires relatively little sample, but the measured hydrodynamic diameter includes the particle and its associated solvation/capping layer. Moreover, scattering intensity strongly depends on particle size, so a small fraction of aggregates can disproportionately influence the measured distribution. Consequently, DLS should not be treated as interchangeable with TEM particle-size measurements.

Fourier-Transform Infrared Spectroscopy

FTIR spectroscopy is useful for identifying chemical bonds and surface functional groups. It can reveal hydroxyl, carbonyl, amine, carboxylate and other groups associated with capping agents, polymers or biological extracts.

For green-synthesized nanoparticles, comparison of FTIR spectra before and after nanoparticle formation can provide evidence that phytochemicals participate in reduction or surface stabilization. However, FTIR alone generally cannot establish a definitive reaction mechanism.

UV-Visible Spectroscopy

UV-Vis spectroscopy is particularly useful for metallic nanoparticles and semiconductor

nanomaterials. Metallic nanoparticles can display localized surface plasmon resonance (LSPR), producing characteristic absorption bands whose position and intensity depend on particle size, shape, composition and surrounding medium.

UV–Vis spectroscopy is therefore frequently used for monitoring nanoparticle formation and stability. Changes in absorption peak position or broadening may indicate aggregation or changes in particle morphology.

X-Ray Photoelectron Spectroscopy

XPS provides information about surface elemental composition and chemical states. It is particularly valuable for determining oxidation states, surface functionalization and elemental composition within the sampling depth of the technique.

Because many nanomaterial properties are controlled by surface chemistry, XPS can provide information not readily obtained from bulk techniques.

BET Surface-Area Analysis

Gas adsorption using the Brunauer–Emmett–Teller (BET) method is widely employed to determine specific surface area and, when appropriate, pore characteristics. The original BET model was introduced by Brunauer, Emmett and Teller in 1938 [Brunauer, S. *et al.*, 1938].

High specific surface area is frequently associated with nanoparticles and porous nanomaterials, although surface area depends on particle size, aggregation and porosity. BET therefore provides useful complementary information rather than a direct measurement of nanoparticle diameter.

CORRELATION OF PREPARATION AND CHARACTERIZATION

An important principle in nanomaterials research is that synthesis and characterization should be interpreted together. For example, a reduction in XRD crystallite size accompanied by a decrease in TEM particle size may support nanoscale crystallization, whereas disagreement between the two measurements may indicate polycrystalline particles.

Similarly, a narrow TEM size distribution but substantially larger DLS hydrodynamic diameter may indicate aggregation or a thick surface coating. FTIR and XPS can then help determine whether surface ligands or functional groups are responsible for colloidal stability.

For porous materials, SEM/TEM can establish morphology, XRD can determine crystalline phase, and BET can quantify surface area. For metallic nanoparticles, UV–Vis spectroscopy can monitor plasmonic behaviour, TEM can determine morphology and XPS can determine surface chemical states. Thus, reliable characterization generally requires a multimodal analytical strategy rather than reliance on a single technique.

CHALLENGES AND FUTURE PERSPECTIVES

Despite substantial progress, several challenges remain. First, reproducibility is a major issue because small changes in synthesis parameters can substantially alter nanoparticle properties. This problem is particularly significant for biological synthesis, where the chemical composition of biological extracts is inherently variable [Yadi, M. *et al.*, 2018; Marslin, G. *et al.*, 2018; Saratale, R. G. *et al.*, 2018].

Second, agglomeration can occur because nanoparticles possess high surface energy. Agglomeration changes effective particle size and may modify optical, catalytic and biological performance.

Third, laboratory-scale synthesis does not necessarily translate directly to industrial production. Scale-up can change mixing, heat transfer, mass transfer and nucleation conditions. Consequently, maintaining the same size distribution and morphology at larger scales can be difficult.

Fourth, characterization data require careful interpretation. For example, XRD measures crystalline domains rather than necessarily individual particles, whereas DLS measures hydrodynamic size and TEM measures projected physical dimensions. Reporting only one measurement can therefore produce an incomplete description of the material.

Finally, environmental and biological safety should be considered alongside synthesis efficiency. Surface coatings, residual reagents, particle size, dissolution behaviour and aggregation can influence toxicity and environmental fate. Green synthesis offers an important route toward reducing hazardous reagents, but claims of sustainability should be supported by systematic evaluation of material inputs, energy consumption, waste generation and product toxicity.

CONCLUSIONS

Nanomaterials can be prepared through a diverse range of physical, chemical and biological approaches. Top-down methods such as milling, lithography and laser ablation provide direct routes from bulk materials, whereas bottom-up approaches such as precipitation, sol-gel, hydrothermal synthesis, CVD and microemulsion techniques construct nanostructures through nucleation and growth. Biological and plant-mediated methods provide additional possibilities for reducing the use of conventional reagents, although reproducibility and process standardization remain important considerations.

The final properties of nanomaterials depend strongly on synthesis conditions, including precursor concentration, pH, temperature, reaction time, solvent and stabilizer concentration. Consequently, characterization must be integrated into the synthesis workflow. XRD provides structural and phase information; SEM and TEM reveal morphology and particle dimensions; DLS determines hydrodynamic size in dispersion; FTIR and XPS provide information about surface chemistry; UV-Vis spectroscopy is useful for optical properties; and BET analysis provides specific surface area and porosity information.

A combination of complementary techniques is therefore essential for establishing reliable structure-property relationships. Future progress in nanomaterials will depend not only on developing new synthesis routes but also on improving reproducibility, standardization, scale-up, environmental compatibility and comprehensive characterization.

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