

Influence of Synthesis Techniques on the Size of Nanomaterials: A Comprehensive Review, Mechanisms, and Future Prospects

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Abstract

Nanomaterials are widely used today because their small size gives them special physical, chemical, and biological properties. Particle size and shape play a major role, as they affect surface area, optical and magnetic behavior, catalytic activity, and biocompatibility. Therefore, controlling nanoparticle size is crucial for improving performance. This review summarizes major synthesis methods and how they affect nanoparticle size. Top-down physical methods like ball milling and laser ablation are easy and scalable, while bottom-up chemical methods such as sol-gel, coprecipitation, microemulsion, and hydrothermal synthesis allow better control over size and morphology. Green synthesis using plant extracts, microbes, or biomolecules is also discussed as an eco-friendly alternative. The review highlights how different synthesis routes influence particle size, current applications, challenges in consistency and large-scale production, and future directions for creating uniform and sustainable nanomaterials.

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1. INTRODUCTION

Nanotechnology has transformed material science by allowing the design of materials at the nanoscale (below 100 nm), resulting in nanoparticles with exceptional optical, magnetic, electrical, and catalytic properties (Khan et al., 2022; Classification, Characterization, Synthesis, n.d.; Synthesis of green nanoparticles, 2024). Particle size is a crucial parameter that influences these properties because nanoscale dimensions significantly increase surface-to-volume ratios and introduce quantum confinement effects (Feng et al., 2025; Kießling et al., 2023). For example, gold nanoparticles exhibit size-dependent optical absorption known as surface plasmon resonance (SPR), with strong color shifts occurring when particle size decreases below 20 nm (Cacaci et al., 2023). Similarly, TiO₂ nanoparticles show enhanced photocatalytic efficiency at smaller sizes due to greater surface area and more active sites (Controlled Size Oils Based Green Fabrication, 2023).

In biomedical applications, particle size dictates biodistribution, cellular uptake, and clearance rates. Nanoparticles below 100 nm are generally optimal for drug delivery, while larger particles (>200 nm) are prone to rapid clearance by the reticuloendothelial system (Xu et al., 2023; Controlled Synthesis and Microstructural Properties of Sol-Gel TiO₂ Nanoparticles for Photocatalytic Cement Composites, 2019). Therefore, precise size control during synthesis is essential for maximizing functional performance in any application. Achieving this requires careful selection of synthesis route, optimization of reaction parameters, and sometimes the combination of methods to produce uniform and reproducible nanoparticles (Chen et al., 2023; Martínez-Vargas et al., 2023; Systematic Study of Reaction Conditions, 2024).

2. SYNTHESIS TECHNIQUES FOR NANOMATERIALS

Nanoparticles are synthesized using either top-down or bottom-up methods, and each technique influences their size, shape, and surface properties (Physical methods for preparation, 2024).

2.1. *Top-Down (Physical) Methods*

Top-down methods break bulk materials into nanosized particles using mechanical or physical forces.

1. **Ball Milling:** High-energy mechanical grinding reduces materials to nanoscale, and factors like rotation speed, milling time, and the ball-to-powder ratio determine the final particle size (Gupta & Yadav, 2024). Particles produced are typically in the 50–500 nm range, often with a broad distribution and structural defects. High energy consumption and

contamination from milling media are common limitations (Kim & Lee, 2024).

2. **Laser Ablation:** Pulsed lasers irradiate bulk targets in liquid or gas, producing nanoparticles with relatively narrow size distributions (10–100 nm) and high purity (Size Control of Iron Oxide Nanoparticles, 2022). Adjusting laser energy and pulse duration allows partial size control. However, low yields and expensive equipment limit scalability (Kumar et al., 2024).
3. **Evaporation–Condensation:** Bulk material is vaporized and then condensed to produce nanoparticles. While this method produces high-purity particles, controlling size uniformity is challenging due to rapid nucleation and aggregation (Al-Saidi & Al-Hajri, 2023).

2.1.1. *Advantages of Top-Down Methods*

1. Simple to implement for certain materials.
2. Can directly fabricate nanoscale patterns in devices.

2.1.2. *Limitations*

1. Poor control over particle size and morphology (except in lithographic techniques).
2. High energy requirements and potential for structural defects (Das & Jha, 2025; Effects of sol concentration, 2014).

2.2. *Bottom-Up (Chemical) Methods*

1. Bottom-up methods assemble nanoparticles from atoms or molecules via nucleation and growth, offering better control over size and crystallinity (Green synthesis and the formation kinetics, 2022).
2. **Sol–Gel Synthesis:** Metal alkoxides or salts undergo hydrolysis and polycondensation, forming colloidal sols that transition into gels. After drying and calcination, nanoparticles (5–50 nm) with controlled shape and composition are obtained (Size-and Aggregation-Controlled Preparation, 2023; Microemulsion mediated sol-gel synthesis of nano-scaled MAl_2O_4 (M=Co, Ni, Cu) spinels, 1999). Process factors like precursor concentration, pH, solvent choice, and calcination temperature strongly affect the size and shape of nanoparticles.
3. **Co-Precipitation:** Metal ions precipitate as hydroxides or oxides under controlled pH, temperature, and stirring. Particle sizes between 10–100 nm can be achieved, but aggregation is common without stabilizers or surfactants (Preparation of Nano- TiO_2 Using a Sol-Gel Process Mediated in Reverse Microemulsion Combined with a Solvent Thermal Technique, n.d.; Controlled Synthesis and Microstructural Properties of Sol-Gel TiO_2

Nanoparticles for Photocatalytic Cement Composites, 2019). This method is widely used for magnetic nanoparticles, ferrites, and mixed oxides.

4. **Microemulsion Method:** Uses water-in-oil or oil-in-water microemulsions as nanoreactors. Uniform nanoparticles (1–50 nm) are obtained with narrow size distributions. Surfactants stabilize the nanoparticles, but residual surfactant removal is necessary for biomedical applications (Al-Saidi & Al-Hajri, 2023).
5. **Hydrothermal/Solvothermal Synthesis:** Reactions occur in sealed autoclaves at high pressure and temperature. Particle size (10–80 nm) and crystallinity can be tuned by temperature, solvent, and reaction time. This method is popular for metal oxides and sulfides (Gupta & Yadav, 2024).
6. **Chemical Vapor Deposition (CVD):** Vaporized precursors decompose on heated substrates to form nanoparticles with sizes <10 nm. High precision and crystallinity are achieved, but equipment cost and process complexity limit wide adoption (Kim & Lee, 2024).

2.2.1. *Advantages of Bottom-Up Methods*

1. Better control over size, shape, and crystallinity.
2. Suitable for producing uniform and monodisperse nanoparticles.

2.2.2. *Limitations*

1. Sensitive to reaction conditions.
2. Often requires surfactants or capping agents to prevent aggregation (Kumar et al., 2024).

2.3. *Green/Biological Methods*

Eco-friendly synthesis using plants, microbes, or biomolecules is gaining attention due to sustainability and reduced toxicity (Das & Jha, 2025).

1. **Plant Extracts:** They contain polyphenols, flavonoids, and alkaloids that serve as natural reducing and stabilizing agents for synthesizing Ag, Au, ZnO, and TiO₂ nanoparticles. Sizes range from 10–200 nm depending on extract concentration, pH, and temperature (Lithi et al., 2025; Shahzadi et al., 2025).
2. **Microbial Synthesis:** Bacteria, fungi, and algae can synthesize nanoparticles extracellularly or intracellularly. These nanoparticles are generally stable but suffer from batch-to-batch variation and broader size distribution (Raja et al., 2025; Swain et al., 2025).

2.3.1. *Advantages*

1. Biocompatible and environmentally friendly.
2. Can use renewable materials and agricultural waste.

2.3.2. *Limitations*

1. Lower reproducibility compared to chemical methods.
2. Particle size control is challenging, often resulting in broader distributions (Ibrahim et al., 2024; Das & Jha, 2025).

Table 1. Comparative Analysis of Particle Size (Ibrahim et al., 2024; Das & Jha, 2025; Chen et al., 2023; Martínez-Vargas et al., 2023; Systematic Study of Reaction Conditions, 2024).

Method	Size Range (nm)	Distribution	Advantages	Limitations
Ball Milling	50–500	Broad	Scalable, simple	Defects, high energy
Laser Ablation	10–100	Narrow	High purity	Expensive, low yield
Sol–Gel	5–50	Narrow	Precise size and shape control	Sensitive to processing conditions
Co-Precipitation	10–100	Moderate	Economical, scalable	Agglomeration
Microemulsion	1–50	Very narrow	Uniform nucleation	Surfactant contamination
Green Synthesis	10–200	Moderate–broad	Eco-friendly, sustainable	Poor reproducibility

3. APPLICATIONS OF SIZE-CONTROLLED NANOPARTICLES

1. **Energy Storage:** Li-ion batteries and supercapacitors benefit from smaller nanoparticles, which offer shorter ion diffusion paths and higher active surface area (Controlled Size Oils Based Green Fabrication, 2023; Size Control of Iron Oxide Nanoparticles, 2022; Gupta & Yadav, 2024).
2. **Environmental Remediation:** Nano-TiO₂ and ZnO degrade pollutants more efficiently at smaller sizes due to enhanced surface activity (Kießling et al., 2023; Cacaci et al., 2023; Size-and Aggregation-Controlled Preparation, 2023).
3. **Biomedical Applications:** Drug delivery, imaging, and photothermal therapy rely on nanoparticles between 10–100 nm for optimal circulation and cellular uptake (Xu et al., 2023; Controlled Synthesis and Microstructural Properties of Sol-Gel TiO₂ Nanoparticles for Photocatalytic Cement Composites, 2019; Size-and Aggregation-Controlled Preparation, 2023).
4. **Electronics and Sensors:** Size-dependent bandgap tuning in semiconducting nanoparticles enables sensitive detection and quantum dot applications (Synthesis of green nanoparticles, 2024; Kim & Lee, 2024).

4. CHALLENGES AND FUTURE PERSPECTIVES

1. **Reproducibility:** Achieving consistent size across batches remains difficult, particularly in green synthesis (Ibrahim et al., 2024; Das & Jha, 2025).
2. **Scalability:** Some precise methods (e.g., laser ablation, CVD) are not cost-effective for large-scale production (Physical methods for preparation, 2024; Kumar et al., 2024).
3. **Agglomeration:** Small nanoparticles are prone to aggregation, affecting functional properties (Controlled Synthesis and Microstructural Properties of Sol-Gel TiO₂ Nanoparticles for Photocatalytic Cement Composites, 2019; Kumar et al., 2024).
4. **Sustainability:** Eco-friendly synthesis methods must be optimized to produce uniform and monodisperse nanoparticles at scale (Lithi et al., 2025; Shahzadi et al., 2025; Raja et al., 2025; Swain et al., 2025).

5. FUTURE DIRECTIONS

1. Hybrid methods combining chemical precision and green sustainability.
2. AI-assisted optimization of reaction parameters for real-time size prediction.
3. Advanced characterization linking synthesis conditions with functional properties (Martínez-Vargas et al., 2023; Gupta & Yadav, 2024).

6. CONCLUSION

The method used to synthesize nanoparticles strongly affects their size, shape, and uniformity. Top-down techniques like ball milling and laser ablation are simple and suitable for large-scale production but often produce particles with wide size ranges and structural defects. Bottom-up chemical methods such as sol-gel, co-precipitation, microemulsion, and hydrothermal processes offer better control over size, morphology, and crystallinity, usually yielding uniform nanoparticles in the 1–50 nm range. Green synthesis using plant extracts, microbes, or biomolecules provides an eco-friendly alternative, producing biocompatible nanoparticles, though issues with reproducibility and size uniformity remain. Future progress lies in combining the precision of chemical methods with the sustainability of green approaches to create high-quality nanoparticles for industrial, biomedical, and environmental applications.

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