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COMPARISON OF DIFFERENT NANOPARTICLE PREPARATION METHOD - A REVIEW

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ABSTRACT: Nanoparticles have gained significant attention in various scientific and industrial field due to their unique physical, chemical and biological properties. The method of nanoparticle synthesis plays a crucial role in determining their size, shape, stability and functional efficiency. This review provides a detailed comparative analysis of various nanoparticle preparation method, primarily focusing on top-down and bottom-up approaches. Top-down methods such as laser ablation, ball milling offers past production and high purity; however, they often lack precise control over particle size. Bottom-up approaches, including sol-gel, spinning, biological synthesis employing plant extracts, bacteria and fungi has emerged as an eco-friendly and cost-effective alternative, although achieving consistent yield and particle size remain as challenge. This review highlights the advantages, limitations of each synthesis. The study also emphasizes the importance of selecting an appropriate synthesis technique based on the intended application and desired nanoparticle properties. Additionally, recent advancements in nanoparticle preparation and future directions of sustainable and large-scale productions are discussed. This comparative overview aims to assist researchers in selecting the most effective preparation method to enhance nanoparticle performance for targeted applications.

INTRODUCTION: Nanotechnology has been a prominent field of research since previous century. The term nanotechnology was introduced by Richard P. Feyman in his famous 1959 lecture, "There is plenty of room at the bottom [1]." Nanoparticles (NPs) have gained significant attention in recent years due to their unique physiochemical properties, which make them valuable across various fields, including electronics, energy storage, and environmental applications.

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Their small size, typically ranging from 1 to 100 nm and large surface area to volume ratio allows them to exhibit enhanced surface area, reactivity and tunable optical and mechanical properties compared to bulk materials [2]. Nanoparticle can be classified as 0D, 1D, 2D, 3D based on their shape [3]. The importance became evident when researchers discovered that size influence the physiochemical properties of material including their optical characteristic.

Several approaches exist for nanoparticle synthesis, broadly categorized into top down and bottom-up methods. Top-down approaches such as mechanical milling and laser ablation, involve breaking down materials into nanosized particles, whereas bottom up approaches, including chemical reduction, sol-gel synthesis and biological synthesis, build nanoparticles atom by atom or molecule by molecule. Each method has its advantages and limitations concerning particle size control, stability, cost-effectiveness, environmental impact, and application suitability [4, 5].

Classification of Nanoparticles: Nanoparticle vary in dimensions, shapes, sizes depending on their composition [6]. They can be classified based in their dimensionality: zero dimensional nanoparticles have all three spatial dimensions confined to a single dot such as nanodots. One dimensional structure extends only in one direction, as seen in material like graphene. Two-dimensional nanoparticle have both length and bredth but are extremely thin, for example carbon nanotubes. Three dimensional nanoparticles possess length, breadth and height, such as gold nanoparticles. Nanoparticles also exhibit diverse shapes, size and structures, they may appear as spherical, cylindrical, tublar, conical, hollow core, spiral, flat or irregular forms, with sizes typically ranging from 1 nm to 100nm. Their surfaces can be smooth or rough, uniform or varied. Additionally, nanoparticles can exist in crystalline or amorphous state and may be composed of single or multiple crystals, either as discrete entities or agglomerated structures [7].

Classification: Nanoparticles are broadly categorized into inorganic, organic and carbon based.

Organic Nanoparticles: This category of nanoparticles, known as organic nanoparticles (ONPs) is composed of organic molecules and has a size of 100 nm or smaller [8]. This category includes widely recognized nanoparticles or polymers such as ferritine, micelles, dendrimers, and liposomes. Micelles and liposomes, which feature a hollow interior known as nanocapsule, exhibit sensitivity to heat and light [9]. These nanoparticles are biodegradable and non-toxic, making them ideal candidates for drug delivery applications, their effectiveness and areas of use are influenced by factors such as drug carrying capacity, stability and delivery mechanism – wheather through drug entrapment or adsorption. Additionally, their size, composition and surface characteristic play a crucial role in their overall performances [10].

Inorganic Nanoparticles: Inorganic nanoparticles are those that do not contain carbon in their composition. They are primarily classified into metal and metal oxide nanoparticles.

Metal Based Nanoparticles: Metal based nanoparticle are synthesized from metals at nanometric scale s using either top-down (destructive) or bottom-up (constructive) approaches. Nearly all metals can be converted into nanoparticles, with commonly used including aluminum (Al), [11] cadmium (Cd), [12] cobalt (Co), [13] copper (Cu), [14] gold (Au) [15], iron (Fe), [16] lead (Pb), [17] silver (Ag) [17], zinc (Zn) [18]. These nanoparticles exhibit unique characteristic such as sizes ranging from 10 to 100 nm, high surface area to volume ratio, specific pore sizes, surface charge properties, and crystalline or amorphous structures. Their shapes can vary including spherical and cylindrical forms, while their properties such as color, magnetic and sensitivity can be influenced by environmental factors like air moisture, heat and sunlight [19].

Metal-oxide Based Nanoparticles: Metal-oxide compounds are formed through the combination of positively charged metal ions and negatively charged oxygen ions, resulting in strong and stable ionic interactions due to electrostatic forces [20]. For example, iron (Fe) nanoparticles readily oxidized to form iron oxide (Fe_2O_3) nanoparticles upon exposure to oxygen at room temperature. This transformation significantly enhances their reactivity compared to their pure metal counterparts. Metal oxide nanoparticles are synthesized to modify and improve the properties of their metal-based forms, leveraging their increased reactivity and efficiency [21]. Commonly synthesized metal-oxide includes silicon dioxide (SiO_2), titanium dioxide (TiO_2), and aluminum oxide (Al_2O_3), zinc oxide (ZnO) [22-26]. These nanoparticles exhibit exceptional properties when compared to their metallic equivalents, making them valuable for various applications.

Carbon Based Nanoparticles: Nanoparticles made primarily of carbon are referred to as carbon-based nanoparticles. These nanoparticles can take various shapes including tubular, horn shaped, spherical and, or ellipsoidal forms.

They are broadly classified into two main categories fullerenes and carbon nanotubes (CNTs). Additionally, other types of carbon-based nanoparticles include graphene, carbon nanofibers, and carbon black [27].

Fullerenes: Fullerene is the general term for C_n cage molecules composed of hexagonal and pentagonal faces [28]. Kroto and Smally named the family of molecules observed in their gas-phase experiments [29] "fullerenes" due to their resemblance to the geodesic domes designed by R. Buckminster Fuller [30, 31]. The term "Buckminster fullerene" or simply "buckyball" specifically refers to the C_{60} molecule [28].

Graphene: Graphene, an allotrope of carbon, is crafted from sp2-hybridized carbon atoms meticulously arranged in a two-dimensional honeycomb lattice structure. It serves as the foundational unit for graphite materials across various dimensions, including zero-dimensional fullerenes, one-dimensional nanotubes, and three-dimensional graphite structures [32-34]. In a graphene sheet, every atom forms σ bonds with its three closest neighbours, along with a delocalized π-bond. This collective bonding arrangement contributes to a valence band that spans the entire sheet. This bonding pattern mirrors that found in carbon nanotubes and polycyclic aromatic hydrocarbons, as well as partially in fullerenes and glassy carbon structures [35]. In 2004, the material was rediscovered, isolated and investigated at the University of Manchester [36], by Andre Geim and Konstantin Novoselov. In 2010, Geim and Novoselov were awarded the Nobel Prize in Physics for their "groundbreaking experiments regarding the two-dimensional material grapheme [37]."

Carbon Nanotubes: Carbon nanotubes (CNTs) are carbon allotropes first discovered by Japanese scientist S. Lijima in 1991 [38]. They exhibit remarkable properties including high rigidity, strength and elasticity. Additionally, CNTs possess excellent thermal and electrical conductivity. Structurally they are cylindrical nanomaterials composed of rolled graphene sheets, with diameters in the nanometerrange [39]. Based on their structural characteristics CNTs are categorized into two main types: 1. Single walled carbon nanotubes (SWCNTs) [40] which have dimeters ranging from 1 to 3 nanometer and extends a few micrometers in length, and 2. Multi walled carbon nanotubes (MWCNTs), [39] which have diameters between 5 and 40 nanometer and lengths of approximately 10 micrometers.

Synthesis of Nanoparticles: Nanoparticles are synthesized *via* different methods. A schematic diagram is shown below.

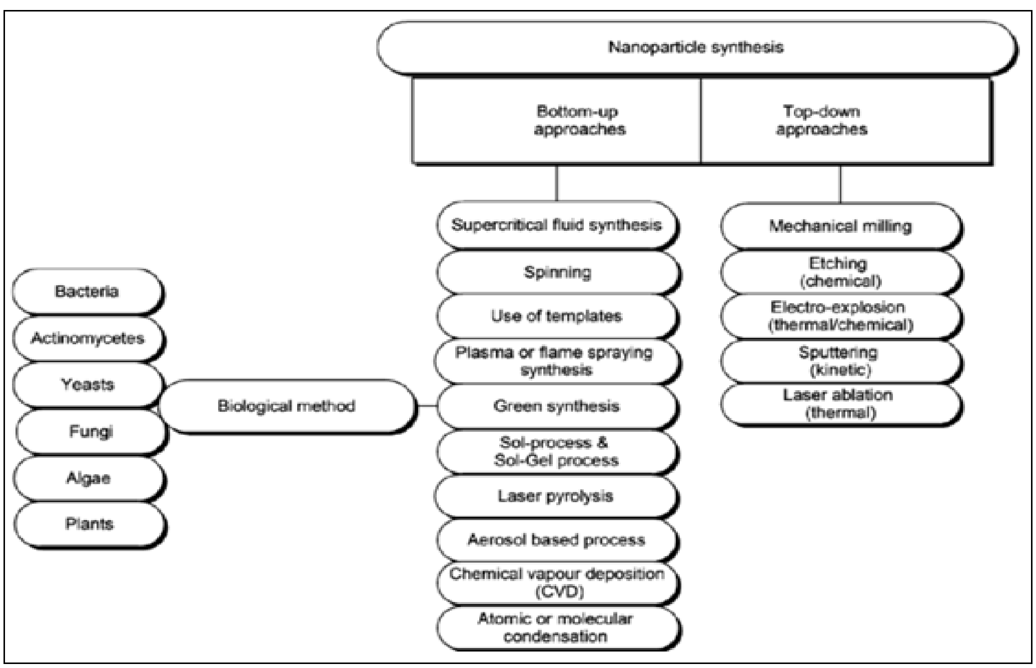

FIG. 1: COMPARISON OF NANOPARTICLES SYNTHESIS METHODS: ADVANTAGES, DISADVANTAGES, AND APPLICATIONS

From the above diagram we have the idea that the synthesis of nanoparticles are divided into two main classes:

a. Top down synthesis

b. Bottom up synthesis

Top-Down Synthesis: The top-down approach also known as the destructive method, involves breaking down larger materials into their smallest building blocks, such as atom. This method works best for materials with well-organized structures at larger scale. It allows larger pieces of material to be reduced into tiny, nano-sized particles. While top-down methods are easier to use, they are not ideal for creating nanoparticles with precise shape and sizes. One major challenge of this approach is controlling the final particle size and form. Some common techniques used in this method include mechanical milling, nanolithography, laser ablation, sputtering and thermal decomposition.

Mechanical Milling: mechanical methods are among the most cost-effective techniques for large scale production of nanoparticles [41]. One of the simplest approaches is ball milling, which involves grinding material in a rotating chamber filled with small balls made of iron, hardened steel, silicon carbide and tungsten carbide [42].

In ball milling a powdered material is placed inside the chamber. As the mill rotates, the kinetic energy from the balls is transferred to material, breaking it down to nanoscaledimensions through mechanical attrition [43]. This widely used top-down technique is employed for synthesizing various nanoparticles and metal alloys. During the milling process, friction and collision between the milling balls generate heat and pressure [44]. At high temperatures, these forces can cause significant changes in the particle structure, leading to phase transformations. Ball milling is commonly used to: reduce particle size, modify surface properties, induce structural changes in materials, trigger certain reactions that do not occur typically at room temperature.

Several key factor affect the Mechanical Milling Process [45]: plastic deformation influences particle shape, fracture leads to a decrease in a particle size, cold welding causes an increase in particle size. Mechanical milling is often performed in an inert atmosphere to prevent oxidation and enhance the properties of the synthesized nanoparticles [46].

Despite its Advantages, Ball Milling has Some Limitations: the final nanoparticle structure structures are highly sensitive to the milling environment and may become contaminated by the grinding media, producing extremely small particles requires prolonged milling time, noise pollution and environmental disturbances are major drawback associated with this process [47].

Nanolithography: nanolithography is a part of nanotechnology that focuses on creating and designing extremely small structures, usually at nanometer scale. It involves forming patterns with at least one dimension ranging from the size of a single atom up to about 100 nm [48]. Lithography techniques are categorized into two types based on the use of maskes: masked lithography and maskless lithography.

Masked lithography [48] uses masks or templates to transfer patterns over a large area at once allowing fast and high-volume production, processing multiple wafers per hour. Examples include photolithography, soft lithography, and Nanoimprint lithography [49-51].

Maskless lithography [48] such as electron beam lithography, focused ion beam lithogtaphy, and scanning beam lithography creates pattern directly without masks by writing them one at a time [51, 52].

These methods allow for extremely fine details, with feature sizes as small as a few nanometers. However, because it works in a step-by-step manner, it is much slower and not suitable for large scale manufacturing [51].

Laser Ablation Method: Laser ablation is a technique used to create different types of nanoparticles, such as semiconductors, quantum dots, carbon nanotubes, nanowires and core-shell nanoparticles. In this process a laser beam hits a material, turning it into vapor. The nanoparticles then form as the vapor cools and condenses in a gas. Because the cooling happens very quickly, this method helps produce very pure nanoparticles, often smaller than 10 nanometers [53]. In recent years, laser ablation synthesis in solution (LSAiS) has become a dependable alternative to traditional chemical methods for making noble metal nanoparticles (NMNp). LSAiS is an ecofriendly method that produces stable NMNp in water or organic solvents without needing extra chemicals or stabilizers. The resulting nanoparticles are easy to modify for different applications or can be used directly when pure nanoparticles are needed [54].

However, this method has some drawbacks, when laser ablation is used for a long time, too many nanoparticles build up in the solution. This blocks the laser beam and causes the nanoparticles to absorb the laser energy instead of the targeted material. As a result, the ablation process slows down [55].

Sputtering: Sputtering is a process where fast moving particles hit a material (called the target) and knock off its atoms. These atoms then move through the air and settle on another surface (the substrate) to form a thin layer [56]. There are many types of sputtering like DC sputtering- uses an electric field to create a charged gas(plasma) which shoots ion at the target, removing atom that deposite on the substrate. Reactive sputtering, similar to DC sputtering but includes a reactive gas (like oxygen or nitrogen) to form chemical compounds (e.g. oxides, nitrides). RF sputtering is used when the target is an insulator. It switches the electric charge back and forth to prevent buildup that would stop the process. Magnetorn sputtering uses magnets to improve efficiency by increasing the number of collisions, creating a denser plasma and a faster process [56, 58].

Sputtering works with metals, semiconductors and compounds like oxides, nitrides, sulfides and carbides [56].

The drawbacks of the sputtering method include the effect of the sputtering gas on the surface structure, composition, texture and optical properties of the nanocrystalline metal oxide coating. Additionally sputtering is slower than thermal evaporation, and most of the energy turns into heat when hitting the target which needs to be removed [59, 60, 61].

Thermal Decomposition Method: Thermal decomposition is a widely used method to create inorganic nanoparticles. This technique produces stable and uniform nanoparticles that can self-assembles into structures. Nucleation (initial formation of nanoparticles) happens when a metal precursor is added to a heated solution containing a surfactant (a stabilizing agent that prevents clumping). Growth of the nanoparticles occurs at a high temperature, allowing them to develop into the desired size and shape. Hence, it is an endothermic process [62]. The decomposition temperature is the specific temperature at which substance breaks down chemically. Nanoparticles formed as a result of the metal decomposing at this particular temperature [63].

The thermal decomposition method has some limitations. Many metal compositions and combinations are difficult to deposite. Additionally, there are only a few processing options to controls the properties of the film and the available source materials may not always be sufficient [64].

Bottom-up Synthesis: The bottom-up approach is a method of making nanoparticles assembling materials from their smallest building blocks, such as atoms or molecules [65]. This technique helps create materials with unique properties that differ from larger, bulk materials [66]. Compared to other methods, bottom-up synthesis is more cost effective and produces less waste.

This approach ensures precise control over nanoparticle size, shape and distribution [67]. It is commonly used in chemical synthesis, where precursor solutions are mixed to form uniform nanoparticles, such as zinc oxide, magnetite and brushite [68]. One example is the sol-gel method, which is used to create titanium dioxide (TiO_2) nanoparticles, assessed using environmental tools [69].

A key challenge of the bottom-up method is ensuring nanoparticles properly attach to surfaces. However, it is widely used for making luminescent nanoparticles [70] and has great potential in creating functional nanomaterials. This method enables extreme miniaturization and could be a cost-effective alternative to top-down nanofabrication [65]. Some bottom-up techniques are sol-gel, spinning, chemical vapor deposition (CVD), pyrolysis and biosynthesis.

Sol-gel Method: The sol-gel technique is a wet-chemical process used to synthesize metal oxide nanoparticles from a liquid precursor solution. The solution undergoes hydrolysis (breaking down in water or alcohol) and condensation (forming a gel like structure) to create nanoparticles [71].

The process involves typically metal alkoxide precursors. The sol-gel method of nanoparticles synthesis follows several steps. First, metal oxide undergoes hydrolysis in water or alcohol, forming a sol. Next, condensation increases the solution viscosity, creating porous structures that are left to age. During polycondensation, hydroxo (M-OH-M) and oxo (M-O-M) leading to metal-polymer networks in solution [72]. As aging continues, structural and porosity changes occur, with decreasing porosity and increasing particle spacing. After aging, the materials undergo drying to remove water and solvents. Finally, calcination at high temperature completes the nanoparticle formation [73]. The key factors affecting the final product are precursor type, hydrolysis speed, aging time,ph and water to precursor ratio [74].

The advantages of sol-gel methods are it is low cost, ecofriendly and works at lower temperatures. It produces high quality, uniform nanoparticles for films, powders or coating and enables complex nanostructure formation [73]. This method is widely used for iron oxide (Fe_3O_4) nanoparticles, often resulting in spherical shapes [75, 76].

The main challenge of using the metal alkoxide-based sol-gel method includes its sensitivity to moisture and the limited availability of suitable precursor, especially for mixed-metal oxides. Since different metals react with water at varying rates, it can be difficult to control the hydrolysis process, leading to inconsistent mixing. As a result, instead of forming a uniform material, the components may separate, creating unwanted mixed phase in the final product [77].

Spinning: A spinning disc reactor (SDR) is a machine used to synthesize nanoparticles by rapidly rotating a disc inside a controlled chamber. The reactor's temperature and gas environment (often nitrogen or other inert gas) can be adjusted to prevent unwanted reactins [78]. The process is that liquid reagents are fed onto the center of spinning disc(rotating at 300 to 3000 rpm). As the disc spins, the liquids spreads into a thin film (1 to 200 nm thick), allowing fast mixing and heat transfer. The strong drag forces between the liquid and the disc surface ensure efficient micromixing, which helps control the formation of nanoparticles and prevent clumping. Micromixing happens extremely fast (within a millisecond), ensuring even chemical reactions.

The disc can be smooth or grooved to enhance mixing by creating waves in the liquid layer. Temperature control is achieved through a heating coil or a thermostatic fluid flow beneath the disc. Some SDRs have a vertical setup with a horizontally rotating shapt for easy slurry removed.

The advantages of SDR are continuous operation allows large-scale production. It is easier to scale up compared to traditional stirred tank reactors. The safer operation is due to controlled conditions. It can process upto 150 kg/h of low- viscosity materials (like water) using a 500 mm disc [50, 79].

Chemical Vapor Deposition: Chemical vapor deposition is a process where gases, called precursors, react in the air to form a solid material that sticks to surface [80]. In CVD reaction can occur in two ways: 1. The gases can react in the air first, then attach to the surface, 2. The gases can attach to the surface first, then react and form the solid material on the surface. The second method generally gives better adhesion to the surface [81].

One of the main problems with CVD is the potential hazards from the gases used, which can be explosive, poisonous or corrosive. Another challenge is the difficulty in depositing materials that have more than one component [82].

There are different types of CVD based on the energy used to start the reaction, including thermally activated CVD, plasma-enhanced CVD, photo-initiated CVD [80].

Prolysis: One promising technique for the synthesis of nanoparticles is CO_2 laser pyrolysis, which involves using a laser to heat gas and vapor phase reactants to create nanoparticles. This method allows for controlling the size and the shape of the particles and can produce nanoparticles in quantities large enough for testing in particle applications.

It has been used to create a variety of oxide and non-oxide nanopowders especially silicon-based nanoparticles (e.g. SiC, Si3N4) which are used in high temperature structural materials. The laser pyrolysis technique has also been used to produce other nanoparticles like TiO_2 for UV protection and links for printing. This demonstrates the versatility of laser pyrolysis in creating nanoparticles for various applications, with ongoing development in both the synthesis process and the use of the materials [83]. The method of laser pyrolysis is highly flexible and shows promise for producing nanoparticles for a wide range of applications.

However, it is difficult to get the right size of nanoparticles with this method because once they leave the hot chamber, they tend to stick together and form chains. This is the main problem using pyrolysis to make nanoparticles [84].

Biosynthesis: Biosynthesis is an ecofriendly and sustainable method for making nanoparticles that are non-toxic and biodegradable [85]. It involves using bacteria, plant extracts, fungi and other natural sources along with precursors to create nanoparticles, instead of using traditional chemicals for reduction of capping. The nanoparticles produced through biosynthesis have unique and improved properties, making them ideal for biomedical applications [86].

The biological method for synthesizing nanoparticles is ecofriendly and non-toxic. However, a challenge with this approach is that plant extracts contain many other compounds, and it takes considerable time to remove impurities. Despite efforts to purify, there is still a risk of unwanted particles remaining in the final product [87].

TABLE 1: FLOW DIAGRAM OF VARIOUS TYPE OF NANO PARTICLES

Aspect	Top-down	Bottom-up
General approach	Starts with bulk material and reduces them to nanoscale particles.	Starts with atoms of molecules that assemble to form nanoparticles.
Process	Bulk material is broken down using mechanical and physical methods.	Atoms, molecules or ions assemble or react to form nanoparticles.
Control over size and shape	Challenging to control size and shape; often irregular particles.	Better control over size, shape and composition, particles are more uniform.
Material usage	High material wastage as bulk material is broken down.	More efficient with lower wastage since nanoparticles are built from the atomic level.
Cost and scalability	Can be expensive and challenging to scale; energy intensive.	More cost effective and easier to scale up, especially for large scale production.
Material types	Primarily used for metallic and ceramic nanoparticles.	Can be used for a wide range of materials (metals, polymers, semiconductors, biomolecules).
Example of synthesis methods	Ball milling, laser ablation, photolithography etc.	Sol-gel process, spinning, CVD.
Applications	Large scale production, catalysis, sensors, magnetic materials.	Advanced applications requiring uniform nanoparticles(e.g. drug delivery, electronics).
Advantages	Large scale production, simple for bulk materials, suitable for basic nanoparticle synthesis.	Better control over properties, less material waste, versatile in material types and applications.
disadvantages	Less precise shape and size control, high energy consumption, expensive equipment.	Complex synthesis, difficult to scale up in some cases, require precise conditions.

Top-down is generally more suitable for large scale industrial production of nanoparticles where precise control over size and shape is not critical, especially for simple materials like metals and ceramics. Bottom-up is preferably when high precision, and control over nanoparticle characteristics (such as size, shape and composition) are required. This method is particularly beneficial for specialized applications, including advanced materials and biomedical applications.

TABLE 2: COMPARISON BETWEEN TOP-DOWN AND BOTTOM-UP APPROACHES FOR NANOPARTICLE SYNTHESIS

Method	Advantages	Disadvantages	Common application
Ball milling	Scalable, simple, cost effective.	Broad size distribution, contamination.	Metal and ceramic nanoparticles.

Laser ablation	High purity, no chemical contamination.	Expensive, low yield.	Biomedical, catalysis.
Sputtering	Controlled composition, high purity.	Expensive, requires vacuum.	Coating, electronics.
Nanolithography	High precision, nanoscale control.	Expensive, complex, limited scalability.	Nanoelectronics, biosensors.
Sol-gel	High purity, controlled shape/size.	Time consuming, costly precursor.	Optical coating, catalysis.
Spinning	Scalable, uniform particles.	Limited to polymer based nanoparticles.	Drug delivery, tissue engineering.
Chemical vapor deposition(CVD)	High purity, uniform particles.	Expensive, high temperature, toxic chemicals.	Semiconductor industry, nanocoatings.
Green synthesis	Eco-friendly, cost effective.	Poor size control, slow reaction.	Biomedical, environmental.

Top-down approaches focuses on mechanical, laser based or vacuum assisted techniques to synthesize nanoparticles. They offer high purity and precise control, but methods like ball milling lack uniformity, while nanolithography and sputtering require expensive equipment. Bottom-up methods are widely used for their versatility, cost-effectiveness and scalability. Sol-gel, CVD *etc*. allow controlled synthesis but may aloe toxic precursors, high temperatures and long processing times. Biosynthesis use natural resources to synthesize nanoparticles, making them eco-friendly and biocompatible. However, they have limited scalability and size control, making them less reliable for industrial applications.

CONCLUSION: The synthesis of nanoparticles involve various top-down and bottom-up approaches, each offering unique advantages and challenges. Top-down method such as ball milling, laser ablation, sputtering, and nanolithography enable high purity and precise nanoparticle fabrication, though they often require specialized equipment and hence scalability limitations. Bottom-up methods including sol-gel, CVD etc. provide good control over particle size and shape, making them suitable for diverse applications in catalysis and drug delivery. However, some methods require high temperatures, toxic precursors and long processing times. Nanolithography a high precise fabrication method, enables the patterning of nanoparticles at the nanoscale, making it essential for electronics and biosensor applications. Despite its advantages in precision, it remains an expensive and complex technique. Biosynthesis, using plant extracts or microbes, offer an ecofriendly alternative but struggle with reproducibility and scalability. The choice of synthesis method depends on application requirements, including cost, scalability and environmental considerations. As nanotechnology advances, hybrid approach combining multiple techniques and green synthesis methods are likely to gain importance, balance efficiency, sustainability and precision.

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