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Methods for the Synthesis of Metal Oxide Nanoparticles (MONPs): A review

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Abstract: The nano-science and nanotechnology play a vital role and attracted a lot of attention among researchers due its uses and application. Nanoparticles have dimension between 1 to 100 nm in size. Several methods have developed for the synthesis of NPs due to its applications. Synthesis of NPs were classified as bottom-up and top-down approach. There are three broad methods i) physical method ii) chemical method and iii) Green synthesis. Out of all methods chemical and green methods are widely used and favorable among researchers. The size of NPs is very small and can't measure by scale. The size and shape of NPs measured by different characterization such as Scanning Electron Microscope (SEM), X-ray Diffraction (XRD), Tenulling Electron Microprobe (TEM), Inductive Coupled Plasma (ICP), Atomic force microscope (AFM) etc. After confirmation of the size the synthesized NPs were investigate for its applications in different field.

Keywords: Nanoparticles, Methods of synthesis of NPs, Physical methods, Chemical method, Green synthesis. Bottom-up approach, Top-down approach.

Introduction:

Science & technology at the nanometric scale have attracted a lot of attention from researchers due to its use & its varied applications, nanometric materials and nanoparticles because of its multifunctional properties and efficiency such as optical,magnetic,electronic& catalytic properties its distinct structure, composition & morphology characteristics have drawn attention to different areas such as chemistry, physics, materials, science & engineering, etc. [1] Green nanoscience & nanotechnology are growing very fast today. Nanoparticles are those particles which has dimension between 1 & 100 nanometers in size [2]. Nanoparticles have wide range of applications in areas such as health care, cosmetics, food & feed, environmental health, mechanics, optics, biomedical sciences, chemical industries, electronics, space industries, drug-gene delivery, energy science, optoelectronics, catalysis, single electron transistors, light emitters, nonlinear optical devices & photoelectrochemical application[3]. In recent medical application, the use of metal nanoparticles has made its standard. It has been observed that the use of silver nanoparticles is extensively used for medical purposes due to its better chemical stability & good antimicrobial activity[4]. The synthesis of nanoparticles using modern techniques has emerged as an important application in the biomedical & human health care field for a broad range of products [5]. Advantages of Nanoparticles: - Nanoparticles offer different advantages over other drug delivery systems [6].

- Enhancement of Solubility & bioavailability.
- Enhancement of Pharmacological activity.
- Sustained drug delivery.
- Protection from degradation.
- Enhancement of Permeability.
- Decreased side effects compared to conventional drug delivery.
- Improved therapeutic effect.

Plants considered the main factory for the green synthesis of metal nanoparticles (MNPs) & metal oxide nanoparticles (MONPs), and until now, different plant species have been used especially plant leaf extracts [7]. Synthesis of Nanoparticles through biological method is a good, environment friendly& economically alternative method. Synthesis of green nanomaterials & their characterization is an emerging field of nanotechnology from the past few decades, because of their applications in the fields of physics, chemistry, biological & medicine [8]. There are basically three broad ways of synthesizing metal nanoparticles: chemical, physical, and green synthesis. Chemical and physical methods are quite expensive and potentially hazardous to the environment.

Thus, it is necessary to use safer (environmental and biological) and cost-effective methods to synthesize metallic nanoparticles. This has given birth to the synthesis of nanoparticles using "Green Chemistry" [9].

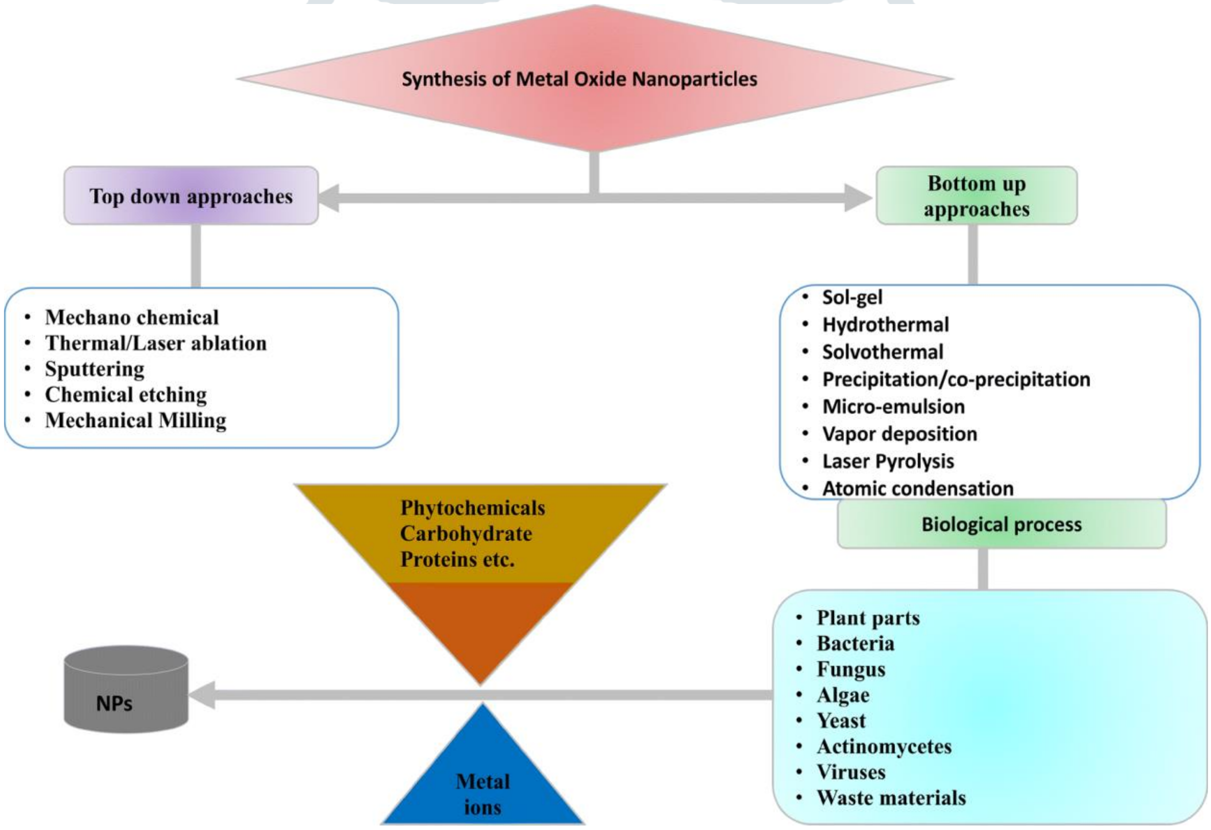
Method of synthesis of NPs:

Nanoparticles can be synthesized by different methods like chemical, physical and biological, but due to increasing popularity of green methods, different works has been done to synthesize metal and metal oxides nanoparticles using different sources. A list of tables had been put to summarize the valuable work done in chemical, physical and biological fields.

A. Chemical Approach: -

In the chemical approach, the main components are the metallic precursors, stabilizing agents & reducing agents (inorganic & organic both) [10]. Wet chemical techniques using chemical approaches such as precipitation, sol-gel & solvothermal processes are usually simpler & proved to be very effective for large scale production [11]. Moreover, employing hazardous chemicals & reagents during the synthesis process & generation of byproducts is lethal to humans & the environment also [12], Therefore, specifically such kind of nanoparticles is limited for biological applications.

Chemically nanoparticles can be classified as "top-down" & bottom-up" by the direction of nanoparticles approach fig.1



The bottom-up approach begins from the atomic level, through forming molecules. In the top-up approach, the scale of the resultant nanoparticles is larger, so that a mechanical process or the addition of acids is required to reduce the particle size. The top-down technique requires the use of complex & complicated instrumentation [13].

B. Physical Approach: - Physical approach for synthesizing nanoparticles is mainly "top-down" approach [14]. Nanoparticles are formed by size reduction method that means suitable bulk materials reduces to small units with the use of appropriate lithographic methods, for ex: -crushing, spitting, milling.

1. Ball Milling: - High ball milling, first developed by John Benjamin in 1970 to synthesize oxide dispersion strengthened alloys capable of withstanding high temperature & pressure [15]. Milling is generally represented as the public face of top-down processes, as it involves the direct breaking of bulk materials into micro/nanostructures. In mechanical milling,

the kinetic energy of the rollers/ balls is transferred to the bulk material, which results in the reduction in grain size [16]. This is one of the simplest ways of making nanoparticles of some metals and alloys in the powder form. [17]

2. Sputtering: - Sputtering is one of the most commonly used synthesis protocols that includes the deposition of nanoparticles as a thin layer generated by the collision of ions over the substrate & followed by annealing. This method is also referred as the physical vapor deposition (PVD) method. [18] The efficiency of this method mainly depends on factors such as layer thickness, substrate type, annealing duration & temperature which directly influence the size & shape of nanoparticles [19].
3. Laser Abalation :- Laser abalation is one of the methods that is considered to be a suitable replacement for conventional chemical methods due to its fast conventional chemical methods due to its fast processing times, providing better control over the size & shape of the particles & high yields with better long term stability [20]. In laser abalation process, a solid surface (generally a plate of pure metal) is irradiated with laser beam, leading to a low-flux plasma plums, which is finally evaporated or sublimated to for nanoparticles [20]. A wide range of nanoparticles can be produced through this technique, such as metal nanoparticles, carbon-nanomaterials, oxide composites & ceramics. Pulsed laser ablative deposition (PLD) is an attractive synthetic method owing to its ability to produce nanoparticles with a narrow size distribution & a low level of impurities [21]

C. Chemical Approach: -

1. Sol-gel method:- In the sol-gel methods there are two types of component's viz 'sol' which is a colloidal suspension of solid particles in a liquid & 'gel' which are polymers containing liquid. Thus, this process includes the creation of 'sols' in the liquid that lead to the formation of a network of discrete particles or network polymers by the connection of sol particles [22]. It comprises the condensation, hydrolysis & thermal decomposition of metal alkoxides or metal precursors in solution. A stable solution is formed, known as the sol. Upon hydrolysis or condensation, the gel is formed with increased viscosity. The particle size can be monitored by changing precursor concentration, temperature & pH values. A mature step is mandatory to empower the development of solid mass it may take few days for the removal of solvent, Ostwald ripening & phase alteration could happen. The unstable reagents are detached to produce nanoparticles [23]
2. Co-precipitation method:- It is a wet chemical process, also called as solvent dispersion method. Polymer phase can be synthetic or natural; polymer solvents are ethanol, acetone, hexane & non solvent polymer. Nanoparticles are produced by rapid diffusion of polymer phase by mixing the polymer solution at last. Nanoparticles are produced by interfacial tension at two phases [24]
3. Hydrothermal Synthesis:- It is one of the most usually used methods for the preparation of nanoparticles. It is principally a chemical reaction-based approach. Hydrothermal synthesis involves a broad temperature for the synthesis of nanoparticles. This method has a wide range of advantages over physical & biological methods. The nanomaterials generated through hydrothermal synthesis may be unstable at higher temperature ranges [25].
4. Laser Pyrolysis: - The process of synthesis of nanoparticles by using a laser is known as laser pyrolysis. An intense laser beam is focused to decompose the mixture of reactant gases in the existence of some inactive gas like helium or argon. The gas pressure shows a significant part in determining the particles sizes & their distribution [26].

D. Biological method: -

Green nanotechnology is an emerging field to design novel nanoparticles using a green chemistry approach. Biological methods of nanoparticles synthesis provide a new possibility of synthesizing nanoparticles using natural reducing & stabilizing agents. It is an economical & environmentally friendly alternative to chemical & physical approaches with no usage of energy & toxic chemical. Biological synthesis of nanoparticles is a bottom-up approach that involves the use of simple unicellular to complex multicellular biological entities like bacteria, fungi, actinomycetes & yeast, algae & plant materials [27]. Biological entities is easy (one-pot synthesis), fast, cost-effective & ecofriendly process for the production of nanoparticles. So biological approaches using microorganisms & plants or plants extracts for synthesis of metal nanoparticles have been suggested as safe alternatives to chemical methods [28].

But synthesis of nanoparticles by using microorganisms is somewhat difficult because it involves elaborate process of maintaining cell cultures, intracellular synthesis & multiple purification steps.

In present times "green" method in the synthesis of nanoparticles has greatly become a topic of interest because the conventional chemical methods are expensive & require the use of chemical compound/organic solvents as reducing agents which are toxic as well [29].

1. Bacterial based synthesis of metal oxide nanoparticles: -

   The synthesis of metals & oxides can occur via an extra or intracellular mechanism when green synthesis through using bacteria [30]. Bacteria species have been widely utilized for commercial biotechnological applications such as bioremediation, genetic engineering &bioleaching [31].

Synthesis of metal oxide using bacteria: -

Bacteria	Oxide obtained	Particle Size (nm)	Morphology	Reference
Lactobacillus Plantarum	MgO	30	Cubic	[32]
Lactobacillus sporogeneses	MgO	30	Cubic	[33]
Desulforibrio, cepa LS4	Fe_2O_3	19	Rounded	[34]
Streptomyces Sp.	ZnO	16-25	Spherical	[35]
Morganela morganii	CuO	13	Spherical	[36]
Proteus mirabilis	CuO	1.44-14.9	Spherical & monodispersed	[37]
Lactobacillus casei	CuO	200	Spherical	[38]

2. Fungi based synthesis of nanoparticles: -

Fungi can be very promising in synthesizing nanoparticles on a large scale, as they can be easily cultivated in both the laboratory & on an industrial scale. Like bacteria fungi also use two pathways for synthesis: intracellular & extracellular [39]

Green synthesis of metal oxide using fungi as source: -

Fungus	Oxide Obtained	Particle Size (nm)	Morphology	Reference
Lactobacillus plantarum	MgO	30	Cubic	[40]
Aspergillus niger	MgO	43-91	Spherical	[41]
Fusarium keratoplasticum	MgO	3-38	Hexagonal	[42]
Trichoderma asperellum	CuO	110	Spherical	[43]
Penicilluimchrysogenum mediated	CuO	9.70	Spherical	[44]
Botrytis	CuO	60-80	Spherical	[45]
Aspergillus terrus	CuO	15-75	Spherical	[46]

3. Algae based synthesis of nanoparticles: -

Algae are eukaryotic aquatic oxygenic photosynthetic organism which ranges from unicellular form to multicellular ones [47]. The algae mediated synthesis of nanoparticles utilizes four different methods 1. Whole algal cells harvested from their culture media at a given phase of growth using centrifugation & then dispersed directly into an aqueous solution of metallic salt. 2. Cell-free aqueous extract made from freshly harvested or lyophilized cells. 3. An aqueous extract filtrate or supernatant of ground, fresh or dried algae. 4. An aqueous filtrate of an algal broth [48].

Algae	Metal Oxides nanoparticles	Size	Shape	Reference
Sargassum muticum	ZnO	30-57	Hexagonal	49
Bifurcariabifurcate	CuO	20.66	Spherical	50
Chlamydomonas reinhardtii	ZnO	21	Hexagonal wurtzite	51
Sargassum wightii	ZnO	20-62	Spherical	52
Ulva lactuca	ZnO	10-50	Sponge like	53

4. Plants based synthesis of nanoparticles: - The use of plant system has been considered as a green route and a reliable method for the biosynthesis of metal oxide nanoparticles to its environmentally friendly nature [54]. The major advantage of using plant extract for silver nanoparticle synthesis is that they are easily available, safe and nontoxic in most cases, have a broad variety of metabolites that can aid in the reduction of metalions and are quicker than microbes in synthesis [55]. Despite the fact that "green synthesis" of nanoparticles using plant material is of considerable interest, it is worth studying the equivalence of these nanoparticles with nanoparticles produced through physical and chemical methods, especially with regard to their potential applications and production scalability. For ex:- it is well known that traditional physiochemically synthesized metal nanoparticles have been used in cancer therapy, the targeted delivery of drugs, molecular imaging, wastewater treatment, catalysis, biosensor development, fuel elements, coatings, cosmetics and as

antiseptic. Nanoparticles produced in plants/ plants extracts have been tested so far only in a small number of practical applications. Plant extract mediated synthesis used a plant component (leaves, stems, roots, shoots, flowers, barks & seeds) extract for the synthesis of nanoparticles, the major advantage of this method is the ability of the extract to serve as both the reducing & stabilizing agent [56]. In an exploration of different antioxidant constituents of the extracts of blackberry, blueberry, turmeric& pomegranate, the pomegranate was found to have the ability to produce more uniform size & shape nanoparticles in the range of 20-500nm. These nanoparticles could be used for the management of cancer & the antioxidant therapy [57].

Plants have biomolecules (like carbohydrates, proteins & coenzymes) with exemplary potential to reduce metal salt into nanoparticles. Like other biosynthesis processes, gold & silver metal nanoparticles were first investigated in plant extract assisted synthesis. Various plants including aloe vera ,oat(avena sativa), alfalfa (Medicago sativa), Tulsi (Osmium sanctum), lemon grass have been utilized to synthesize silver nanoparticles & gold nanoparticles as listed in the table

Green Synthesis of Metallic Nanoparticles from various Plant Extract: -

Sr. no.	Plant Origin	Nanoparticles	Size(nm)	Morphology	Applications	Reference
1.	Aloe barbadensis Miller (Aloe Vera)	Gold & Silver	10-30	Spherical, triangular	Cancer hyperthermia, optical coatings	[58]
2.	Apiin extracted from henna leaves	Silver & Gold	39	Spherical, triangular & quasi-spherical	Hyperthermia of cancer cells & IR-absorbing optical coatings	[59]
3.	Azadirachta indica(neem)	Gold, silver & silver gold alloys	5-35 & 50-100	Spherical, triangular, hexagonal	Remediation of toxic metals	[60]
4.	Camellia sinensis (black tea leaf extracts)	Gold & silver	20	Spherical, prism	Catalyst's sensors	[61]
5.	Brassica juncea (mustard)	Silver	2-35	Spherical	Catalyst's sensors	[62]
6.	Cinnamomum camphora (camphor tree)	Gold & silver	55-80	Triangular, spherical (Au), and quasi-spherical (Ag)	Catalyst's sensors	[63]
7.	Canca papaya (papaya)	Silver	60-80	Spherical	Catalyst's sensors	[64]
8.	Citrus limon (lemon)	Silver	<50	Spherical, spheroidal	Catalyst's sensors	[65]
9.	Coriandrum sativum (coriander)	Gold	6.75-57.91	Spherical, triangular, truncated triangular, decahedral	Drug delivery, tissue/ tumor imaging, photothermal therapy	[66]
10.	Cymbopogon flexuous (lemongrass)	Gold	200-500	Spherical, triangular	Infrared-absorbing optical coatings	[67]
11.	Eucalyptus citriodora (neelgiri)	Silver	20	Spherical	Antibacterial	[68]
12.	Eucalyptus hybrida (safeda)	Silver	50-150	Crystalline, spherical	Antibacterial	[69]
13.	Garcinia mangostana (mangosteen)	Silver	35	Spherical	Antimicrobial activity against E coli and S aureus	[70]
14.	Gardenia jasminoides Ellis (gardenia)	Palladium	3-5	Spherical	Nano catalysts for	[71]

					p-nitrotoulene hydrogenation	
15.	Syzygiumaromaticum (clove buds)	Gold	5-100	Irregular	Detection and destruction of cancer cells	[72]
16.	Jatropha curcas (seed extract)	Silver	15-50	Spherical	Detection and destruction of cancer cells	[73]
17.	Medicago sativa (alfalfa)	Gold	2-40	Irregular, tetrahedral, hexagonal platelet, decahedral, icosahedral	Labeling in structural biology, paints	[74]
18.	Morus (mulberry)	Silver	15-20	Spherical	Antimicrobial activity against E.coli, B. subtilis	[75]
19.	Nelumbo nucifera (lotus)	Silver	25-80	Spherical, triangular, truncated triangular, decahedral	Larvicidal activity against malaria and filariasis vectors	[76]
20.	Pear fruit extract	Gold	200-500	Triangular, hexagonal, crystalline	Catalysis biosensing	[77]

Oxides Synthesized by the Green synthesis method using Plant Extract:

Sr.no.	Plants	Plant Parts	NPs	Size (nm)	Morphology	Reference
1.	Eichhornia crassipes	Leaf	ZnO	32	Spherical	[78]
2.	Trifolium pratense	Flower	ZnO	60-70	Spherical	[79]
3.	Aloe vera	Leaf	ZnO	25-40	Spherical	[80]
4.	Boswellia ovalifoliolata	stem bark	ZnO	20	Spherical	[81]
5.	Garlic (Allinum sativum)	leaves	ZnO	14	Spherical	[82]
6.	Potato		ZnO	20	Hexagonal(wurtzite)	[83]
7.	Potato		Fe_3O_4	40	Spherical	[84]
8.	Kalopanax pictus	leaves	MnO_2	19.2	Spherical	[85]
9.	Tradescantia pallida	leaves	ZnO	23-27	Rod	[86]
10.	Punica granatum	seeds	Fe_2O_3	-	Spherical	[88]
11.	Avicennia marina	flowers	FeO	37.5	Spherical	[89]
12.	Cocos nucifera	pulp	CuO	24	Cubic momodispersed	[90]
13.	Azadirachta indica	leaves	CuO	12	Spherical	[91]
14.	Anacyclus pyrethrum	root	RuO_2	13.9	Almost spherical	[92]
15.	Carica papaya	leaves	ZnO	50	Spherical	[93]
16.	Banana	Peel	CuO	23	Spherical	[94]
17	Camellia japonica	leaves	CuO	15-25	Spherical	[95]

Applications of nanoparticles: -

Nano-sized inorganic particles of either simple or complex nature, display unique, physical and chemical properties and represent an increasingly important material in the development of novel nanodevices which can be used in numerous physical, biological, biomedical and pharmaceutical applications [96]

NPs have drawn increasing interest from every branch of medicine for their ability to deliver drugs in the optimum dosage range often resulting in increased therapeutic efficiency of the drugs, weakened side effects and improved patient compliance [97]Iron oxide particles such as magnetite (Fe_3O_4) or its oxidized form maghemite (Fe_2O_3) are the most commonly employed for biomedical applications [98] Liposomes have been used as a potential drug carrier instead of conventional dosage forms because of their unique advantages which include ability to protect drugs from degradation, target to the site of action and reduce the noxiousness and other side effects. However developmental work on liposome drugs has been restricted due to inherent health issues such as squat encapsulation efficiency, rapid water leakage in the commodity of blood components and very poor storage, and stability. On the other hand, polymeric NPs promise some critical advantages over these materials i.e. liposomes. For instance, NPs help to increase the rateability of drugs or problems and possess convenient controlled drug release properties [99]

Most of the semiconductor and metallic NPs have immense potential for cancer diagnosis and therapy on account of their surface plasmon resonance (SPR) enhanced light scattering and absorption. Au NPs efficiently convert the strong absorbed light into localized heat which can be exploited for the selective laser photo thermal therapy of cancer [100] The increasing area of engineered NPs in industrial and household applications leads to the release of such materials into the environment. Assessing the risk of these NPs in the environment requires on understanding of their mobility, reactivity, Eco toxicity and persistency Most of environmental applications of nanotechnology fall into three categories:

1.Environmentally benign sustainable products (e.g., green chemistry or pollution prevention).
2.Remediation of materials contaminated with hazardous substances and
3.Sensors for environmental stages [101]

Nanoparticles are produced by engineering methods or through combustion techniques. Healthcare, cosmetics, environmental preservation, and air purification are processes that involve nanoparticle technology. These particles transport chemotherapeutic drugs across the human body for the treatment of cancer. They can transfer even to the regions where the arteries are damaged. Aerospace engineers use carbon nanotubes for the morphing of aircraft wings. Zinc oxide nanowires applied in the solar cells help in environmental preservation. The nanoparticles hence have several other applications [102].

Conclusion:

Biosynthesis of nanoparticles using eco-friendly approach has been the area of focused research in the last decade. Green Sources act as both stabilizing and reducing agent for the synthesis of shape and size-controlled nanoparticles. Future prospect of plant-mediated nanoparticle synthesis includes an extension of laboratory-based work to industrial scale, elucidation of photochemicals involved in the synthesis of nanoparticles using bioinformatics tools and deriving the exact mechanism involved in inhibition of pathogenic bacteria. The plant based nanoparticle can have huge application in the field of food, pharmaceutical and cosmetic industries and thus become a major area of research.

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