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Review Article

A Comprehensive Review on Nanoparticle Classification and Synthesis Methods

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ABSTRACT

Nanoparticles synthesis and the study of their size and properties are important in medicine as well as biological fields nanotechnology using nanoparticles such as metals, semiconductors and metal oxides are of great interest for a wide of variety of applications in the field of data, energy, environmental and medical tools due to their unique or improved properties. Current review focus on nanoparticle, types, characterization and most advanced application related to nanotechnology. It has the potential to revolutionize a series of medical and biotechnology tools and procedures so that they are portable, cheaper, safer, and easier to administer. They were synthesized by various methods for research and commercial uses which are classified into three types-chemical, physical and mechanical processes which had sawn a vast improvement. It plays a major role in the development of innovative methods to produce new products to suitable existing production equipment and to reformulate new material and chemicals with improved performance resulting in less consumption of energy and material and reduce harm to the environment as well as environmental remediation.

Keywords: -Nanoscience, Nanoparticle, Nanotechnology, Biotechnology, etc.

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INTRODUCTION

Nanoscience is a branch of science that comprises the study of properties of matter at the nanoscale, and particularly focuses on the unique, size-dependent properties of solid-state materials ^[1]. Nanotechnology is the branch that comprises the synthesis, engineering, and utilization of materials whose size ranges from 1 to 100 nm, known as nanomaterials ^[2]. The birth of nanoscience and nanotechnology concepts is usually linked to the famous lecture of Nobel laureate Richard Feynman at the 1959 meeting of the American Physical Society, “Tree’s Plenty of Room at the Bottom” ^[3]. However, the usage of nanotechnology and nanomaterials goes back in history long before that. Nanotechnology has gained huge attention over time. The fundamental component of nanotechnology is the nanoparticles.

The nanoparticles exhibit a unique physical, chemical and biological properties at nanoscale compared to their respective particles at higher scales. This phenomenon is due to a relatively larger surface area to the volume, increased reactivity or stability in a chemical process, enhanced mechanical strength, etc. ^[4]. These properties of nanoparticles have led to its use many applications. Today, due to their unique properties, nanomaterials are used in a wide range of applications, such as catalysis, water treatment, energy storage, medicine, agriculture, etc. ^[5-6]. Two main factors cause nanomaterials to behave significantly differently than the same materials at larger dimensions: surface effects and quantum effects ^[7]. These factors make nanomaterials exhibit enhanced or novel mechanical, thermal, magnetic, electronic, optical, and catalytic properties ^[7-8].

Classification of Nanomaterials: -

The key elements of nanotechnology are the nanomaterials. Nanomaterials are well-defined as materials where at least one of their dimensions is in the nanoscale, i.e. smaller than 100 nm^[9].

Based on their dimensionalities, nanomaterials are located into four different classes,

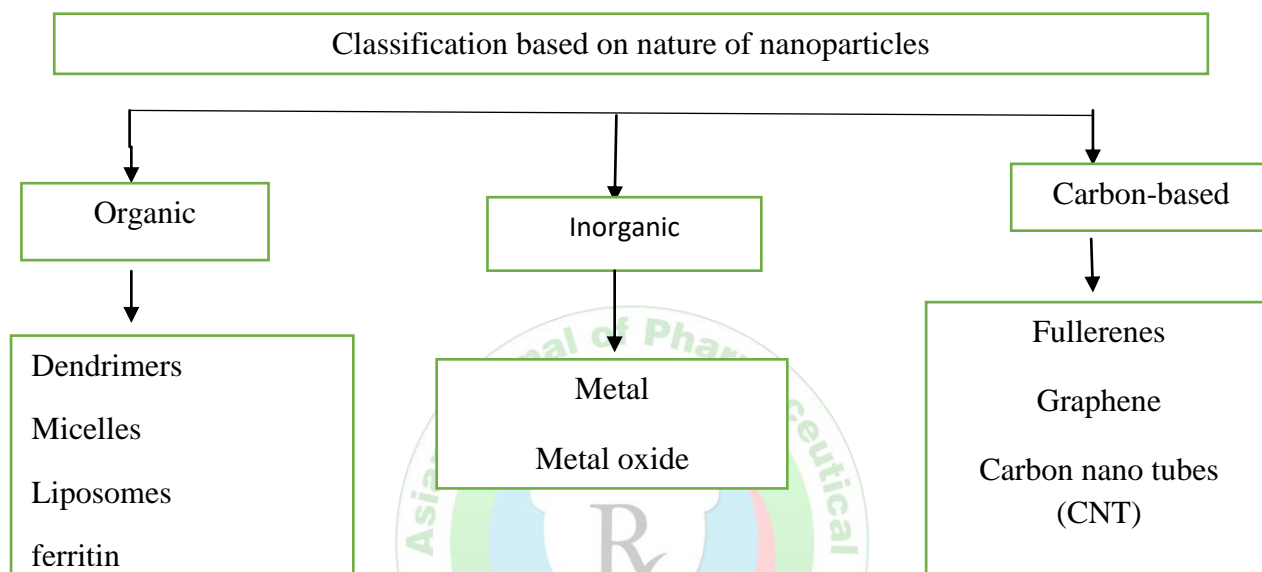
Zero-dimensional nanomaterials (0-D): the nanomaterials in this class have all their three dimensions in the nanoscale

range. Examples are quantum dots, fullerenes, and nanoparticles.

One-dimensional nanomaterials (1-D): the nanomaterials in this class have one dimension outside the nanoscale. Examples are nanotubes, nanofibers, Nano rods, nanowires, and Nano horns.

Two-dimensional nanomaterials (2-D): the nanomaterials in this class have two dimensions outside the nanoscale. Examples are nan sheets, nanofilms, and nanolayers.

Based on their Nature nanomaterials are located into three different classes



Tree-dimensional nanomaterials (3-D) or bulk nanomaterials

Organic Nanoparticles: -

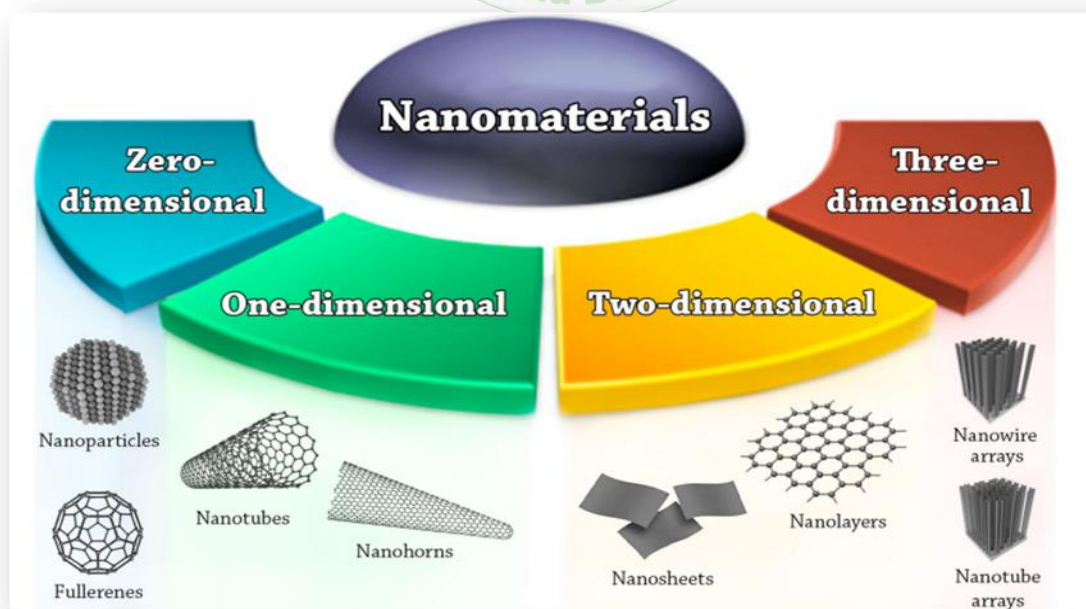


Figure 1: - Nanoparticle Classification based on dimensionality

The micelles, Dendrimers, ferritin and liposomes etc. are commonly known polymers or organic nanoparticles. These nanoparticles are non-toxic, biodegradable, and some atoms such as liposomes and micelles have a hollow core also known as Nano capsules and are sensitive to thermal and electromagnetic radiation such as heat and light.^[10] The organic nanoparticles are most widely used in the biomedical field for example drug delivery system as they are efficient and also can be injected on specific parts of the body which is also known as targeted drug delivery.

Examples: -Liposomes, Dendrimers, Micelles, Ferritin, etc.

Inorganic nanoparticles: -

Inorganic nanoparticles are particles which are not made up of carbon. Metal and metal oxide-based nanoparticles are generally categorized as inorganic nanoparticles.

I. Metal NPs:

Almost all the metals can be synthesised into their nanoparticles. [15] The commonly used metals for nanoparticle synthesis are aluminium (Al), cadmium (Cd), cobalt (Co), copper (Cu), gold (Au), iron (Fe), lead (Pb), silver (Ag) and zinc (Zn). These nanoparticles can be manufactured by chemical, electrochemical, or photochemical methods. In chemical methods, the metal nanoparticles are gained by reducing the metal-ion precursors in solution by chemical reducing agents. These have the ability to adsorb minor molecules and have high surface energy. These nanoparticles have applications in research areas, detection and imaging of biomolecules and in environmental and bioanalytical applications. For example, gold nanoparticles are used to coat the example before analysing in SEM. This was usually done to enhance the electronic stream, which helps us to get high quality SEM images. Due to their progressive optical properties, metal NPs find applications in many research areas.

II. Metal Oxides: -

a. Ceramic NPs: Ceramic nanoparticles are inorganic solids prepared of carbides, carbonates, oxides, carbides, carbonates and phosphates synthesized via heat and successive cooling. They can be found in polycrystalline, dense, amorphous, polycrystalline, dense, porous or hollow forms. Therefore, these NPs are getting great attention of researchers due to their use in applications such as catalysis, photo catalysis, photo degradation of dyes. By controlling some physical properties, these nanoparticles can be formulated in drug delivery system especially in targeting tumours, glaucoma, and some bacterial infections.

b. Semiconductor NPs: Semiconductor nanoparticles have properties like those of metals and non-metals. They are found in the periodic table in groups II-VI, IIIIV or IV-VI. These atoms have wide bandgaps, which on tuning shows different properties. They stand used in photo catalysis, electronics devices, photo-optics and water splitting applications. Semiconductor materials possess properties between metals and non-metals and therefore they found various applications in the literature due to this property. Some examples of semiconductor nanoparticles are GaN, GaP, InP, inas from group III-V; ZnO, ZnS, CdS, CdSe, s etc.

CdTe are II-VI semiconductors and silicon and germanium are from group IV.^[11]

c. Polymeric NPs: These are usually organic based NPs and in literature a special term polymer nanoparticle (PNP) is collectively used for it. Depending up on the preparation these are nan spheres or nano-capsular shaped. The former are matrix particles whose overall mass is usually solid and the other molecules are adsorbed at the outer boundary of the spherical surface. In the latter case the solid mass is summarized within the particle completely. The PNPs are readily functionalized and thus find bundles of applications in the literature. Some of the merits of polymeric nanoparticles are measured release, protection of drug molecules, ability to combine therapy and imaging, specific targeting and many more. They have applications in drug delivery and diagnostics. The drug transfers with polymeric nanoparticles are highly biodegradable and biocompatible.

d. Lipid-based NPs: Lipid nanoparticles are generally spherical in shape with a diameter ranging from 10 to 100 nm. It contains of a solid core made of lipid and a matrix containing soluble lipophilic molecules. The outside core of these nanoparticles is stabilized by surfactants and emulsifiers. These nanoparticles have application in the biomedical field as a drug carrier and delivery and RNA release in cancer therapy.

III. Carbon-based NPs: -

Carbon-based nanoparticles include two key materials, namely, carbon nanotubes (CNTs) and fullerenes. CNTs are nothing but graphene sheets rolled hooked on a tube. These materials are mainly used for the structural reinforcement as they are 100 times stronger than steel. CNTs can be classified into single walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs). CNTs are unique in a way as they are thermally conductive lengthways the length and non-conductive across the tube. Fullerenes are the allotropes of carbon consuming a structure of hollow cage of sixty or more carbon atoms. The structure of C-60 is called Buckminsterfullerene, and appearances like a hollow football. The carbon units in these structures consume a pentagonal and hexagonal arrangement.^[12] These have commercial applications due to their electrical conductivity, structure, high strength, and electron affinity. The rolled sheets can be single, double or many walls and therefore they are named as single-walled (SWNTs), double-walled (DWNTs) or multi-walled carbon nanotubes (MWNTs), respectively. They are extensively synthesized by deposition of carbon precursors especially the atomic carbons, vaporized from graphite by laser or by electric arc on to metal particles. Lately, they have been manufactured via chemical vapour deposition (CVD) technique. Due to their unique physical, chemical and mechanical characteristics, these materials are not only used in pristine form but also in Nano-composites for many commercial applications such as fillers, efficient gas adsorbents for environmental remediation and as support medium for different inorganic and organic catalysts.^[13]

Examples: - Fullerenes, Graphene, Carbon Nano tubes (CNT), Carbon nanofiber.

Synthesis of Nanoparticles: -

There are basically two approaches in synthesis of nanoparticle:

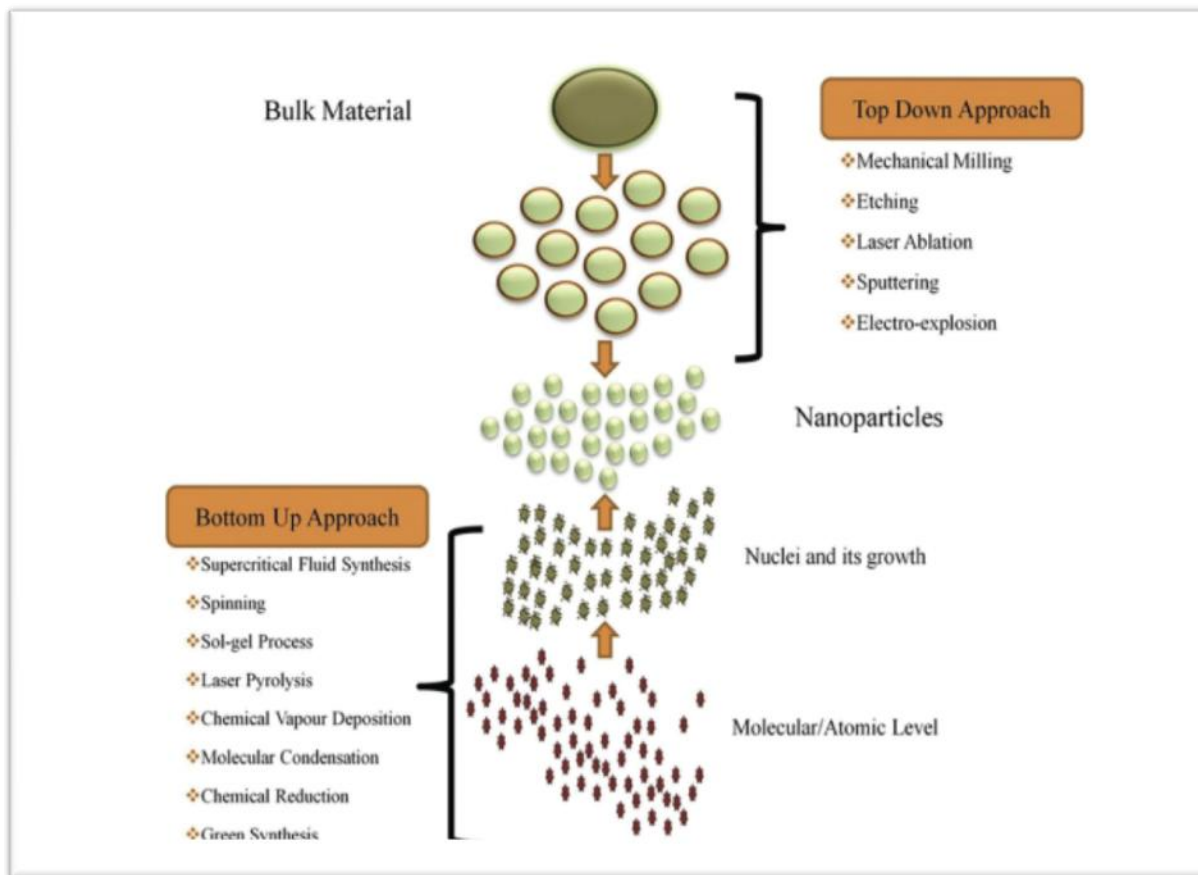


Figure 2: Basic approaches of preparation on Nanoparticles.

Top down approaches: -

It involves breaking down of large size bulk material into Nano size particles, could be done by milling, attrition process and electro explosion wire technique. It is a quick manufacturing process but requires more energy, so it is not suitable for large scale production. Another drawback of top down approach is imperfections of surface structure such defects have a significant impact on the physical and other properties of Nano particles^[38].

Cu Nano particles synthesized by the top down approach of electro exploding wire (EEW) technique^[39].

In this technique copper plate is kept inside suitable medium such as water, current approximately 1010 A/m² is applied to the medium through the copper wire which leads to melting and evaporation of copper metal plate occupied place. Evaporation of metal generates the plasma which readily dispersed in the media followed centrifugation to separate the particles. Similarly,^[40] synthesized the silver Nano particles and analysis the structural properties such as XRD, SEM, UV-Vis spectroscopy. 2-4 nm size of iron particle produced with agglomerate structure using high energy ball mill under room temperature followed by particle separation using sieve shaker and the structure morphology was established by high resolution electron micro scope (HREM). It is a popular method because of its applicability and suitability can be applied for all kind of constituents. Enhance the parameters such as milling time,

rotational velocity of agitator shaft, filling ratio of grinding media, flow velocity of circulation system for preparation of ZnO Nano particles consuming Taguchi method, Response Surface method and genetic algorithm method.

Bottom up approach

Bottom approach refers to structure of material from molecule by molecule, atom by atom and cluster by cluster. During the assembling procedure physical forces acting on the Nano structure used to association the particles in to a superior one. For synthesis of complex Nano structures, Nano technologist frequently select bottom up approach because the benefit of this approach is to specific control of particle size resulting good optical, electronic and other properties^[8]. The common methods elaborate in bottom up approach as follows,

Sol gel method The sol gel technique is well well-known technique for synthesis of colloidal Nano particles from liquid phase. The main benefits of sol gel technique are versatility, low temperature procedure and flexible rheology allowing easy decisive. Sol gel process is well fit for synthesis of oxide Nano particles and composite Nano powder. The most commonly used precursors are alkyl oxides due to their profitable convenience and high liability of MOR bond allowing eloquent tailoring in situ during processing. The process involving sol gel technique as follows^[9],

- Preparation of homogeneous solution by separation of metal organic precursor in the organic solvent or in organic salt solution.
- Conversion of precursor oxide in to a highly cross linked solid.
- Hydrolysis leads to sol, dispersion of colloidal particle in liquid done by appropriate reagents (mostly water).
- More condensation leads to gel, a rigid inter associated organic network.
- TiO₂ based Nano particles synthesis is one of the hot topic for the Nano technologist because of its wide range of applications in photo catalysis, shape memory alloy and solar cells. [10] elucidated the Ag₃ Nano particle produced by the sol gel technique had globular shape morphology. XRD verified that the molecules or atoms are arranged in the tetragonal lattice.
- Zinc oxide Nano particles created via zinc chloride, zinc nitrate, sodium hydroxide as a precursor [11]. NaOH was liquefied in distilled water and heated to 500°C and 900°C. Zinc Nitrate and Zinc Chloride were additional to a 1M concentrated NaOH solution for 26 minutes and 52 minutes. 23 full factorial designs were used to analyze the optimum size of crystal formation. Element was characterized by XRD, HRTEM and UV-Vis spectroscopy. The outcomes were determined that lesser size crystals are obtained by high temperature synthesis using zinc chloride precursors. The spectroscopy study showed that the strong absorbance was found at 300nm, showing that the sample was photosensitive in UV region.

a. Co Precipitation

Co precipitation is a classic wet chemical process, also called as solvent displacement method. It was commonly used method, due to ease, profitable and reproducibility. Key elements are needed to prepare the Nano particles are polymer part can be synthetic or natural, polymer solvent typically ethanol, acetone, hexane and non-solvent polymer. Nano particles are produced by a fast diffusion of polymer solvent into a non-solvent polymer phase by mixing the polymer solution finally. Interfacial tension between two phases creates a extreme surface area could leads to impulsive precipitation of Nano particles. Flow sheet for co precipitation technique are short below, Anti-microbial activity of CuO nano particles synthesized by precipitation technique using copper acetate and sodium hydroxide as a precursors and decreasing agent individually. Characterization of particles passed out by XRD, TEM and EDS analysis. XRD reveals that Nano particles are monoclinic crystal similarly EDS definite that lack of impurities in the prepared copper oxide Nano particles [42]. Nano fluids are new variety of fluids in which Nano size material such as Nanoparticles, Nano tube, Nano fibres, Nano wire, and Nano rods in a base [43]. The same author formed two types of zinc oxide (ZnO) nanoparticles, presence and absence of covering agents for analysing its behaviour in Nano fluids. The experimentation was showed by utilizing zinc nitrate, sodium hydroxide, starch and poly vinyl-2pyrrolidone which performance as precursors, reducing agents, stabilizers and capping agents respectively. The following conclusion was through by researchers, the

capped ZnO nanoparticles were agglomerate and had steady express structure than uncapped nanoparticles. Magnetite Nano particle show a vigorous role in many parts such as magnetic drug target, magnetic resonance imaging for clinical diagnosis, recording material and catalyst. Co precipitation of magnetic Fe₃O₄ Nano particle had the successful rate up to 99.7% [44].

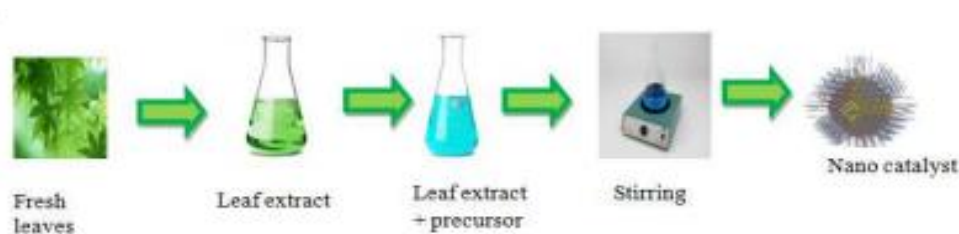
b. Inert Gas Condensation

The inert gas condensation (IGC) method, in which Nano particles are made via the evaporation of a metallic source in an inert gas, had been widely used to yield fine Nano particles. Possibly this method is well suitable for developed metal Nano particles, since metals are vaporized at reasonable rate at attainable temperature. Process for production of copper metal nanoparticles are shortened below,

- Metal is vaporized inside the chamber, into which an inert gas typically argon or helium or neon is periodically acknowledged.
- Once the atom is boil off instantly loss its energy, by colliding of vaporized atom with inert gas.
- The vapor cooled by liquid nitrogen, to form nanoparticles in the range of 2-100nm. Bimetallic Au/Pd Nano particles manufactured by highly size controlled inert gas condensation technique [45]. The subsequent Nano particles were analysed by TEM, mass spectroscopy, electron microscopy, atomic force microscopy, and XRD to verify the geometry and circulation of metals. [46] done a research on formation of Cu and Si Nano clusters by a sputtering or inert gas (Ar) aggregation type cluster source. The size of cluster was measured by the functioning factors such as sputtering pressure and gas-flow rate. The actual mean cluster size was associated with theoretical mean cluster size. The final result shown that the peak cluster size was decreased with increased Ar gas flow rate, due to the time spent by the cluster within the agglomeration region decreases resulting in lower size.

c. Green synthesis

Nano particles are formed by the physical and chemical method essential a longer time for synthesis to speechless this kind of problem, recent expansion of Nano technology is Green synthesis which use of biological system similar plants and microorganism [17]. More over green synthesis of Nano particle is an environmentally friendly method without any dangerous and expensive chemicals [18]. Synthesis of metal Nano particles using plant extracts are considered as a cost effective and hence used as an economically viable method for large scale production [19]. The mechanism overdue the formation of Nano particles clarified by several researchers, they postulated that bio reduction of Nano particles due to numerous bio molecules (vitamins, amino acids, proteins, phenolic acids, alkaloids, etc.) in the plant and microorganisms [17]. Phenolic acids are considered as a powerful anti-oxidant, possess hydroxyl and carboxyl groups that are able to bind metals. The active hydrogen may answerable for decrease of metal ions into formation of metal Nano particles [20].



The overview of green synthesis is easily understanding by the Fig 3, when precursor is added to the leaf extract it changes the colour which indicate the formation of Nano catalyst. Fig 3. Green synthesis of Nano catalyst Averroes bilimbi Fruit Extract broth synthesized gold and silver Nano particles using the salt chloroauric acid and silver nitrate respectively. Color change to yellow and violet indicated that formation of silver and gold Nano particles. The rate of development of Nano particles were slow at lower absorption of salt thus lowers the absorbance in UV spectrometer. They also recommended that alcohols, amines and phenols in the averroa bilimbi fruit might cause reduction and capping of Nano particles ^[21]. Another study proved for ZnO Nano particles from Calotropis Gigantea leaf extract by ^[22]. The synthesized particles were described by SEM from the spherical shape of Nano particles with a size of 10-15 nm and XRD study also revealed the size of the Nano particle. It was presented that rate of synthesis associated to reaction temperature, high temperature allows faster growth rate. More over increasing temperature leads to small size of Nano particles. Stabilization of nano particles using microwave heating is an emerging field of Nano particle synthesis. Gold Nano particles created from Hibiscus rosa-sinensis leaves and also optimizing the parameters like time and micro wave power have been executed by ^[23]. Finally, they decided that higher microwave power at lower time produce Nano particles at faster rate because of rapid constant heating. Anti-microbial activity against multiple drug resistant bacteria by silver Nano particles were formed from jamun leaf broth by utilizing silver nitrate as a precursor studied by ^[24].

1. Characterization of Nanoparticles

- a. **Zeta potential:** The zeta potential of a nanoparticle is commonly used to characterise the surface charge property of nanoparticles. It reproduces the electrical potential of particles and is influenced by the composition of the particle and the medium in which it is dispersed. Nanoparticles with a zeta potential between -10 and +10 mV are considered approximately neutral, while nanoparticles with zeta potentials of greater than +30 mV or less than -30 mV are considered strongly cationic and anionic, respectively. ^[29] The zeta potential can also be used to determine whether a charged active material is encapsulated within the centre of the Nano capsule or adsorbed onto the surface
- b. **UV-visible absorption spectroscopy:** Absorbance spectroscopy is used to determine the optical properties of a solution. A Light is sent through the sample solution

and the amount of absorbed light is measured. When the wavelength is diverse and the absorbance is measured at each wavelength. The absorbance can be used to measure the concentration of a solution by using Beer-Lamberts Law. ^[30] The optical measurement of UV-visible spectrophotometer has different absorbance peak like 410 nm.

- c. **X-ray diffraction (XRD) analysis:** X-ray diffraction is a conservative technique for determination of crystallographic structure and morphology. There is upsurge or decrease in intensity with the amount of constituent. This method is used to establish the metallic nature of particles, gives information on translational symmetry size and shape of the unit cell from peak positions and information on electron density inside the unit cell, namely, where the atoms are located from peak intensities. ^[31]
- d. **Fourier Transform Infrared [FTIR] spectroscopy:** It events infrared intensity vs. wavelength of light, it is used to determine the nature of associated functional groups and structural features of biological extracts with nanoparticles. The calculated ranges clearly reflect the well-known dependence of nanoparticle optical properties. The green synthesized silver nanoparticle by employing various leaf extract was analysed using Fourier Transform Infrared [FTIR] Spectroscopy showed characteristic peaks.
- e. **Microscopic techniques:** These methods namely SEM and TEM mainly used for morphological studies of nanoparticles. ^[32] Many researchers used these techniques to show that the synthesized nanoparticles were more or less uniform in size and shape.
- f. **Dynamic light scattering (DLS)** This technique is a common approach for the analysis of NP size and size distribution. This approach involves the measurement of light interference based on the Brownian motion of NPs in suspension, and on the correlation of NP velocity (diffusion coefficient) with their size using Stokes-Einstein equation [34]. The size distribution range of NPs is shown as the polydispersity index, which is the output of an autocorrelation function ^[33,35].
- g. **Nanoparticle tracking analysis (NTA)** This method is used for the analysis of NP size in suspensions based on their Brownian motion. Like in DLS, the rate of NP move- Joudeh and Linked Journal of Nano biotechnology (2022) 20:262 Page 13 of 29 meant is correlated with their size using Stokes-Einstein equation, allowing the measurement of scope

distribution profiles for NPs with 10–1000 nm diameter. Its advantage over DLS is that NP motion is analysed by video. Individual positional variations of NPs are tracked in two dimensions, which are used to determine NP diffusion rates, and by knowing the diffusion coefficient, the hydrodynamic width of the particles can be calculated. In DLS, individual NPs are not visualized, but instead, the time-dependent intensity fluctuations caused by Brownian motion are used to calculate the polydispersity index^[36]. NTA was found to be more precise for sizing monodisperse as well as polydisperse organic NPs compared to DLS^[37].

Applications

NPs Considering the unique properties discussed in Section 5, NPs can be used in variety of applications. Some significant of these are given below.

In drugs and medications

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

In manufacturing and materials

Nano crystalline materials provide very interesting substances for material science since their properties deviate

from respective bulk material in a size dependent manner. Manufacture NPs display physicochemical characteristics that induce unique electrical, mechanical, optical and imaging properties that are extremely looked-for in certain applications within the medical, commercial, and ecological sectors.

In energy harvesting

Recent studies warned us about the limitations and scarcity of fossil fuels in coming years due to their non-renewable nature. Therefore, scientists shifting their research strategies to make renewable energies from easily available resources at cheap cost. They found that NPs are the best candidate for this drive due to their, large surface area, optical behaviour and catalytic nature. Especially in photocatalytic applications, NPs are widely used to generate energy from photo electrochemical (PEC) and electrochemical water splitting.

CONCLUSION

Poorly soluble, poorly absorbed and labile biologically active substances are re-modified to promising deliverable drugs through the recent advancements of nanotechnology. The knack of nanotechnology to engineer matter at the smallest scale is re-developing areas such as information technology, cognitive science and biotechnology. Further research studies in nanotechnology, can be useful for every aspect of human life.

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