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Metal Oxide Nanoparticles: Synthesis Methods, Characterization And Applications-A Comprehensive Review

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Abstract:

Metal oxide nanoparticles (MONPs) are a class of nanomaterials known for their unique physical, chemical, and biological properties. Due to their high surface-to-volume ratio, stability, and functional versatility, MONPs have found wide spread applications in medicines, environmental remediation, food packaging, cosmetics, and catalysis. Commonly studied metal oxide include zinc oxide (ZnO), titanium dioxide (TiO_2), iron oxide (Fe_3O_4), and copper oxide (CuO), each offering distinct functionalities. These nanoparticles can be synthesized through various methods such as sol-gel, co-precipitation, hydrothermal, and green synthesis, which influence their size, morphology, and reactivity. Characterization techniques like XRD, SEM, TEM, and FTIR are essential to understand their structural and functional properties. The promising biomedical and industrial applications of MONPs are balanced with ongoing research into their toxicity, environmental impact, and safe use. This review aims to provide an overview of the synthesis, characterization, and emerging applications of metal oxide nanoparticles, highlighting their role in advancing nanotechnology-based solutions.

Keywords: Metal oxide nanoparticles, Metal oxide synthesis, Characterization, Inorganic nanoparticle, Biomedical applications.

Introduction:

Nanoparticles have merged as a key area of scientific research and technological innovation due to their unique properties and diverse applications. Measuring between 1-100 nanometers, these tiny particles exhibit distinct characteristics that differ from their bulk counterparts. Their small size enables enhanced surface area-to-volume ratios, leading to improved reactivity, catalytic activity, and optical properties. Precise control over nanoparticle size, shape, composition, and surface properties is crucial for harnessing their potential in various fields. Some potential applications of nanoparticles include research, energy storage and conversion, environmental remediation, and electronics. These inorganic materials are employed in medical application due to their effective anticancer and antimicrobial properties. However on of the primary concerns with inorganic nanoparticles (NPs) is their tendency to induce oxidative stress, a major factor contributing to their toxicity. To mitigate this issue and improve their biocompatibility, phytochemicals-known for their low toxicity and high biological activity-can be used as surface coatings to significantly reduce the cytotoxic effects of the nanoparticles. Despite their utility, most inorganic non-metallic substances tend to be brittle, lacking key mechanical properties such as toughness, plasticity, elasticity and ductility. In addition of biomedical uses, inorganic nanoparticles are gaining attention for their role in food preservation. They can inhibit bacterial growth when integrating into packaging materials. By incorporating inorganic nanoparticles in to film-forming solutions, nanocomposite films with improved physical and antimicrobial properties can be developed for food packaging applications. Metal oxide nanoparticles (MOx) are a diverse class of nanomaterials with applications in chemistry, physics, and materials science. Formed by metallic elements reacting with oxygen, they have unique properties at the nanoscale, making them suitable for various uses, including fluorescence and optical sensors, catalysts, photovoltaics, biomedicine, gas sensors, and anode materials for fuel cells, highlighting their importance in research and development.

APPROACHES OF INORGANIC NANOPARTICLES

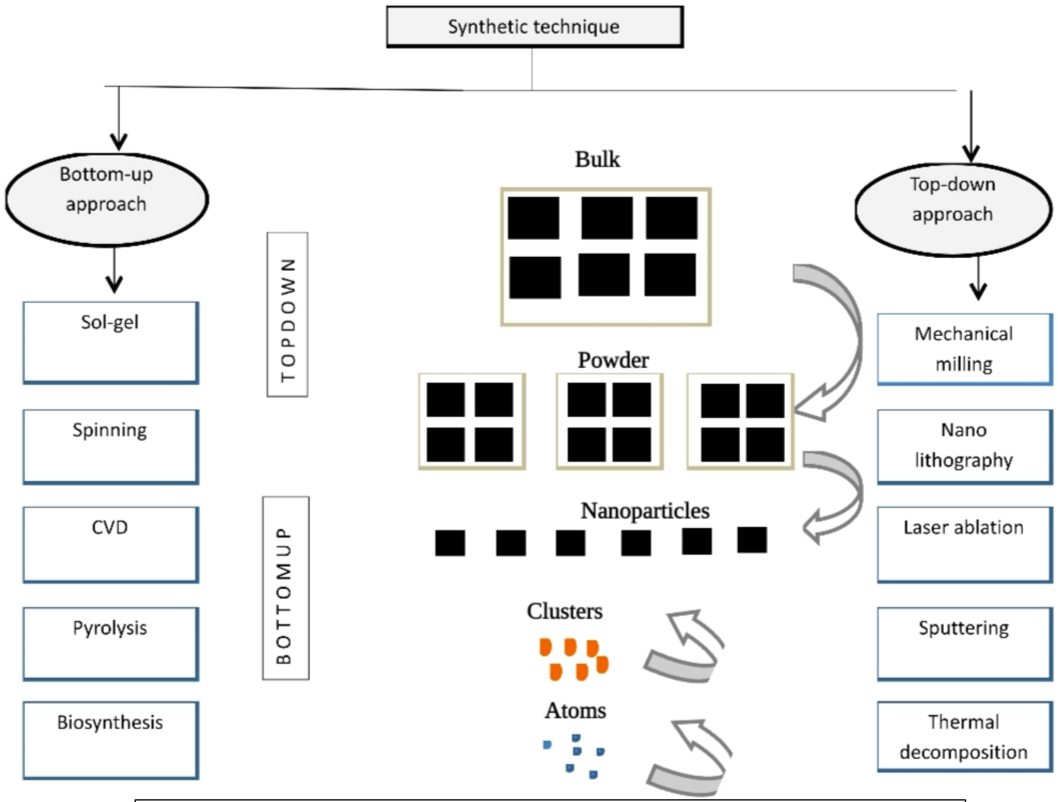

FIG. NO.1 APPROACHES OF INORGANIC NANOPARTICLES

Top-down approach

This approach involves breaking down bulk material into their atomic or nanoscale components. This method is especially suitable for materials with long-range order and structural connections at the macroscopic level. It allows for the reduction of large material segments into nanosized

particles. Although top down technique are relatively easy to implement, they are less effective in producing nanoparticles with precise shapes or uniform sizes. Common methods are mechanical milling, nanolithography, laser ablation, sputtering and thermal decomposition.

Bottom-up approach

This approach operates in the reverse manner, where nanoparticles are generated from basic or simpler substances. For this reason, it is known as bottom-up approach or building-up method. It typically offers better control over particle size, shape and structure. Common examples include sedimentation and reduction techniques. It encompasses methods such as sol-gel method, green synthesis, spinning and biochemical synthesis.

CLASSIFICATION OF NANOPARTICLES

1. ORGANIC NANOPARTICLE

Organic nanoparticles (NPs) are made of organic compounds like proteins, carbohydrate, lipids, and polymers. Examples include dendrimers, liposomes, micelles, and protein complexes. These nanoparticles are typically non-toxic, biodegradable, and sensitive to heat and light. They are often used in biomedical applications, such as targeted drug delivery and cancer therapy, due to their ability to carry payloads and interact with biological systems.

2. INORGANIC NANOPARTICLE

Inorganic nanoparticles are composed of metals or metal oxides, exhibiting unique properties due to their small size and high surface to volume ratio.

They can be categorized into:

I. Metal based nanoparticles
Created by reducing bulk metals to the nanoscale. Exhibit unique properties like UV-Vis sensitivity, conductivity, and catalytic activity. Applications including catalysis, biosensing, imaging.

II. Metal oxide-based nanoparticles
Formed through combination of metal ions and oxygen ions. Often exhibit superior properties compared to metallic equivalents. Examples including SiO_2, TiO_2, ZnO, Al_2O_3. Applications including advanced technological and biomedical applications.

3. CARBON BASED NANOPARTICLE

Carbon base nanoparticles mainly include fullerenes and carbon nanotubes (CNTs). Fullerenes are spherical, hollow carbon structures made from carbon allotropes, forming cage

like nanomaterials. They are widely used as fillers, efficient gas adsorbents for environmental cleanup, and as support materials for various inorganic and organic catalysts. (4)

IRON OXIDE NANOPARTICLES

Iron (III) oxide are reddish brown, paramagnetic inorganic compound and one of the primary forms of iron oxide, along with FeO and Fe_3O_4. Magnetic iron oxide nanoparticles (INPs), especially magnetite (Fe_3O_4) are widely synthesized using methods like sol-gel, hydrothermal, microemulsions, and especially chemical co-precipitation. Monocrystalline INPs (MIONs) can be stabilized using surfactants or polymers like polyethylene glycol and enhance biocompatibility. Stabilizers allow functionalization for targeted delivery. INPs serves as effective MRI contrast agents and can be conjugated with antibodies or ligands for tumor imaging. (7)

ZINC OXIDE NANOPARTICLES

Zinc plays a vital role in biological systems, functioning as a coenzyme, supporting the immune system, and acting as a signaling molecule in managing inflammatory responses. Zinc oxide nanoparticles are recognized for their strong antibacterial and viral properties. Due to their band gap similarity with titanium dioxide (TiO2), No nanoparticles can serve as photo catalysts under UV light or sunlight in the presence of water. This exposure led to the generation of reactive oxygen species (ROS) such as hydrogen peroxide, superoxide, and hydroxyl radicals on the nanoparticle surface, which can disrupt and destroy microbial membranes.

Synthesis:

Zinc acetate dehydrate (Impart, 99% purity) was used as the precursor, while triethanolamine (tea, Emparta, 99% purity) served as the surface-active agent. To begin the process, 40ml of TEA was mixed with 60ml of deionized water and stirred for 30minutes. Then, 5ml of ethanol was added gradually over 3-4minutes with continuous stirring. The mixture was then left undisturbed at room temperature at 30minutes.

Separately 11.4g of zinc acetate dehydrate was dissolved in 100ml of deionized water and stirred thoroughly for 10minutes. This solution was then combined with the TEA-ethanol mixture. To adjust the pH to 9, 25% ammonia solution (Emparta) was added. The resulting solution was heated gradually from 40°c to 70°c. within approximately 40minutes, the transparent mixture turned white and thick.

The white precipitate was separated using filter paper. The obtained residue was separated using filter paper. The obtained residue was washed with distilled water to eliminate impurities, then dried at 95°c for 4hours in an oven. Finally, the dried powder was subjected to calcination at different temperatures for 8hours to yield around 4grams of ZnO nanoparticles, depending on the calcination conditions. (8)

SILICA NANOPARTICLES

Silica nanoparticles (SiNPs) are inorganic materials with desirable properties, including uniform pore size, controllable particle size, large surface area and easily modified surfaces. These characteristics make them highly stable and biocompatible, suitable for various biomedical applications. Recent applications include their use as drug carriers and diagnostic reagents, targeting specific systems in the human body, such as the respiratory, nervous, digestive, and circulatory systems. Silica nanoparticles for biomedical are classified into two main types: mesoporous and nonporous(solid)NPs, both with an amorphous silica structure. Mesoporous silica NPs, characterised by pores (2-50nm in size), are particularly useful for delivering active payloads through physical and chemical absorption. (9)

Chemical methods:

Microemulsion technique: Using a water-in-oil (w/o) system, this method confines reactions to nanoscale "water cores" stabilized by surfactant and co-surfactants, allowing precise control over particle size and high monodispersity.

Sol-gel process: This method transforms a monomer into a colloidal solution (sol), which then evolves into a gel-like network. Typically involving metal alkoxides, the sol-gel method yields uniform, high-purity nanoparticles and is widely used due to its versatility.

Hydrothermal synthesis: Conducted under high-temperature and high-pressure aqueous conditions, this method offers advantages such as high purity, uniform dispersion and precise size control of the resulting nanoparticles. (10)

Experimental procedure:

The starting materials used in the synthesis included tetraethyl orthosilicate (Si $(OC_2H_5O_2)_4$), acetic acid (CH_3COOH), methyl acetate ($C_3H_6O_2$), and methanol (CH_3OH), all of which were procured from sigma-Aldrich. Silicon dioxide (SiO_2) nanoparticles were synthesized using the sol-gel technique.

To initiate the process, 20ml of methanol was mixed with 2.3ml of acetic acid and stirred continuously for 5min at room temperature. During this time, partial evaporation of water occurred, resulting in the formation of methyl acetate. Subsequently, 1.5ml of tetraethyl orthosilicate was added gradually, dropwise, at regular interval. After 90minutes of continuous stirring, a clear and homogeneous solution was formed.

Chemical reaction involved in the synthesis are as follows:

$$CH_3COOH + CH_3OH \longrightarrow C_3H_6O_2 + H_2O$$

$$C_3H_6O_2 + Si\ (OC_2H_5) \longrightarrow SiO_2 + C_{11}H_{26}O_4$$

After the reaction was complete, the resulting SiO_2 solution was left to dry at room temperature. The dried product was then ground into powder and subjected to calcination at 500ºc to yield fine silica nanoparticle. (11)

ALUMINIUM OXIDE NANOPARTICLES

Aluminum oxide, also known as alumina, is a compound composed of aluminium and oxygen with the chemical formula Al_2O_3. It functions as an electrical insulator while possessing high thermal conductivity. Due to its wide range of physical and chemical properties, alumina finds use in numerous applications. Aluminium-based materials are known for their high hardness, excellent stability, strong insulating properties, and transparency.

As a result, various synthesis methods for alumina nanoparticles have been explored, with particular attention given to cost-effective and environmentally friendly approaches. (12)

industrially, alumina is mainly extracted from bauxite and cryolite using the Bayer process, with its primary application being the production of aluminium metal. In addition to its biomedical applications, alumina's exceptional hardness makes it ideal for use in abrasive, and its high melting point makes it suitable for refractory application. (13)

Sol-gel method:

Aluminium oxide (Al_2O_3) nanoparticles were synthesized using a sol-gel method involving an ethanol solution of aluminium nitrate. In this process, Al $(NO_3)_3.9H_2O$ was fully dissolved in distilled water under continuous stirring at room temperature. Subsequently, ethanol was added dropwise to the solution, and the temperature was gradually raised to 80^{o}c. During the reaction, the solution's color changed from orange to dark brown. The pH was carefully maintained between 2and 3 throughout the synthesis. The resulting white product was subjected to evaporation for 3 hours, then cooled to room temperature, and finally calcined at 500^{o}c for 5 hours. (12)

MAGNETIC NANOPARTICLE (MNPs)

Magnetic nanoparticles (MNPs) have gained substantial attention due to their distinct magnetic properties, large surface area and nanoscale dimensions. Various structural forms of these nanoparticles have been developed, such as Nano-dots, Nano-rods, Nanowires, Nanotubes and core-shell nanoparticles. The chemical composition of MNPs can include pure metal, metal alloys, metal oxides and doped nanoparticles. Additionally, these particles can be coated and functionalized to suit specific application requirements. Within the wide spectrum of MNPs, magnetic nanoparticles (Fe_3O_4) are particularly noteworthy due to their favourable magnetic characteristics and versatility in various fields. (14)

Chemical synthesis:

Co-precipitation method

Magnetite nanoparticles (Fe_3O_4) were synthesized using the co-precipitation method by mixing ferric sulphate [Fe_2 $(SO_4)_3$] and ferrous sulphate [$FeSO_4$] In their stoichiometric ratio, in distilled water. Sodium hydroxide solution was added gradually, drop by drop, under constant stirring until the PH reached 11. The mixture was then continuously stirred and heated to 80^{o}C. After the reaction. The resulting nanoparticles were separated, washed with distilled water until the PH was neutralized to 7 and finally dried in an electric dryer at 105^{o}C. (15)

$FeSO_4 + Fe_2(SO_4)_3 + 8NaOH \rightarrow Fe_3O_4 + 4Na_2SO_4 + 4H_2O$

TITANIUM DIOXIDE NANOAPRTICLES

Titanium dioxide (TiO2) nanoparticles are a common type of nanoparticles used in various everyday products. They can be synthesized using physical, chemical, biological methods. Physical methods involve techniques like thermal decomposition, laser irradiation, and electrolysis, which require expensive equipment and vacuum conditions. Chemical methods use reducing agents like sodium borohydride or sodium citrate and are the most commonly used technique for nanoparticle synthesis. However, green synthesis, biological method, offers a more ecofriendly alternative, utilizing plant extract as stabilising agents, which reduces the use of toxic chemicals and environmental harm.

Their applications are diverse, ranging from nano fertilizers and heavy metal adsorption to antimicrobial activity and use in sensors and electronics. Additionally, titanium dioxide nanoparticles are used as pigments, food additives, and in cosmetics, leveraging their unique properties. They are found in many household items including personal care products and foods, but their widespread industrial use also leads to environmental discharge. (16) (17)

Chemical synthesis:

Titanium dioxide (TiO2) nanoparticles were synthesized using chemical precipitation method. The process involved mixing titanium trichloride (TiCl3) with double distilled water, followed by addition of polyethylene glycol (PEG) and sodium hydroxide (NaOH), resulting in the formation of white precipitates. After filtering, washing and drying at 50^{o}C for 4 hours, the precipitate was calcinated at 600^{Oc}C for 2hours to obtain a fine powder of titanium dioxide nanoparticles. This method produces high quality TiO2 nanoparticle powder. (18)

CHARACTERIZATION OF INORGANIC NANOPARTICLES

PARTICLE SIZE AND DISTRIBUTION

Particle size is a crucial parameter in the characterization of nanoparticles, influencing their classification as micro or nanoscale and affecting their distribution and overall behavior. Understanding the size and uniformity of nanoparticles is essential, as these properties affect reactivity, strength, and surface area.

Tools like dynamic light scattering (DLS) and Electron microscopy, including scanning electron microscopy (SEM) and transmission electron microscopy (TEM), is widely used to obtain high-resolution images that help in analyzing individual particles and clusters. For bulk solid samples, laser diffraction is commonly used to determine size distribution. (18)

In case of nanoparticles dispersed in liquid, techniques such as photon correlation spectroscopy (PCS) and centrifugation are employed for size measurement. However, imaging methods become less practical for particles in the gaseous phase. Therefore, the scanning mobility particle sizer (SMPS) is preferred due to its higher speed and accuracy in measuring airborne nanoparticles. (4)

SURFACE MORPHOLOGY

Nanoparticles exhibit a wide range of shapes and surface patterns, both of which significantly influence their functional properties. These shapes can include spherical, flat, cylindrical, tubular, conical, or irregular forms.

To analyze surface morphology, electron microscopy techniques such as scanning electron microscopy (SEM) and transmission electron microscopy (TEM) are commonly employed. (4)

SURFACE AREA AND POROSITY

The specific surface area is measured using techniques like BET (Brunauer-Emmett-Teller) analysis. This provides insights into the surface activity, which is crucial for catalysis and adsorption.

SURFACE CHEMISTRY AND FUNCTIONAL GROUPS

Understanding the surface chemistry is important for predicting the interaction of nanoparticles with their environment or biological systems.

Fourier transform infrared spectroscopy (FTIR) and x-ray photoelectron spectroscopy (XPS) are commonly used to identify surface functional groups and elements.

CRYSTALLINITY AND STRUCTURE

X-ray diffraction (XRD) is used to determine the crystal structure and phase purity of the nanoparticles.it reveals whether the sample is amorphous or crystalline and identifies the materials phase composition.

OPTICAL AND PHOTO LUMINESCENT PROPERTIES

Photoluminescence spectroscopy identified the lowest energy electronic transition at 1.72Ev, providing insights into the band gap and confirming their suitability as photo catalysts. (19)

THERMAL STABILITY

Thermogravimetric analysis (TGA) helps determine how nanoparticle respond to heat.it gives data on weight loss, decomposition temperature, and stability.

ELEMENTAL COMPOSITION

Energy-dispersive x-ray spectroscopy (EDX/EDS) and x-ray fluorescence (XRF) provide information about the elemental composition of nanoparticles, confirming the presence and proportion of specific elements.

ZETA POTENTIAL AND COLLOIDAL STABILITY

Zeta potential measurements help to evaluate the surface charge and predict the stability of nanoparticle suspension in different media. (18)

APPLICATIONS OF NANOPARTICLES

NANOTECHNOLOGY FOR CANCER:

Conventional cancer treatment such as surgery, chemotherapy, and radiotherapy, though commonly used, often come with drawbacks like damage to healthy tissues, loss of organ function, and severe side effects. In comparison, nanotechnology provides a more advanced, precise, and less harmful approach to cancer diagnosis and treatment. Nanoparticles (NPs) enhance drug delivery by increasing drug stability, circulation time, and targeting ability while minimizing toxicity. Their distinct physical, chemical, optical, and magnetic properties make them ideal for biomedical applications. Characterization techniques like X-ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM) help determine particle size, structure and morphology key factors in their performance.

In cancer treatment, NPs are used for drug delivery, controlled release, molecular imaging, biomarker detection, and tumor targeting mechanisms, allowing for more accurate diagnosis and effective therapy. Nanotechnology has significantly improved the personalization of cancer treatment and is playing an important role in developing solutions for other chronic disease as well. (20)

ENVIRONMENTAL APPLICATIONS OF NANOTECHNOLOGY:

The widespread use of engineered nanoparticles (NPs) in industrial and household products has led to their release into the environment, raising concerns about their mobility, toxicity and persistence. NPs in soil and groundwater pose key exposure risks. Their high surface area enables them to bind with pollutants, affecting contaminant behavior depending on their physical and chemical properties.

Nanotechnology in the environment is primarily applied in three area: sustainable product development, hazardous waste remediation, and environmental sensing. A major focus is the

removal of toxic heavy metals like mercury and lead using materials such as superparamagnetic iron oxide NPs. Challenges remain in detecting trace levels of NPs due to limited analytical tools. However, photo degradation using nanoscale materials has shown promise for pollutant breakdown, along with applications in fluorescence and sensing technologies. (5)

THERAPEUTIC APPLICATIONS OF POLYMERIC NANOPARTICLES:

Polymeric nanoparticles provide a highly effective platform for advanced drug delivery, particularly in the treatment of brain-related and neurodegenerative disorders. They protect therapeutic agents by encapsulating them, trapping them within their core, or enabling them to adhere to the particle surface. These nanoparticles can cross the blood brain barrier (BBB) through endocytosis and transcytosis mechanisms, facilitating targeted drug delivery to the brain. Additionally their polymeric coating reduces recognition by the immune system and limits clearance by the reticuloendothelial system. This lead to prolonged circulation time and increased drug accumulation in vital organs such as the brain, kidneys, and intestines. Beyond neurological applications, polymeric nanoparticles are also used in gene therapy, where they have demonstrated anti proliferative affects against breast cancer cells. (21)

NANOPARTICLES IN FOOD PACKAGING:

Nanomaterial-based antimicrobial emulsions and sensors offer effective solutions for food decontamination and contamination detection. Incorporating nanoparticles like gold, copper, and silver into packaging enhances antimicrobial activity, improves barrier properties, and extends food shelf life these innovations enable safer and more efficient food storage and transport. As technology advances, production and application methods are expected to improve, making commercialization easier. Continued research is needed to explore other metals and fully realize nanotechnology's potential in food engineering and packaging.

NANOPARTICLES FOR GENE DELIVERY:

Nanoparticles serve as efficient carriers for gene delivery in polynucleotide vaccines by introducing antigen-coding genes into host cells, where they stimulate immune responses. This intracellular protein synthesis activates both humoral and cell-mediated immunity. DNA, the key element in these vaccines, is simpler to manufacture and store compared to protein- based alternatives. Nanoparticles loaded with plasmid DNA sustained gene release by rapidly escaping degradation and reaching the cytoplasm. For instance, PLGA nanoparticles can transport therapeutic genes such as bone morphogenic proteins, supporting bone regeneration and repair. (21)

CONCLUSION

Inorganic nanoparticle (INPs) represents a significant advancement in nanotechnology, offering remarkable potential across biomedical, environmental, and industrial domains. Their unique physicochemical properties such as high surface area, tunable size, and distinctive optical, magnetic, and catalytic behaviors-enable targeted application in drug delivery, imaging, food preservation, and pollutant remediation. Despite their advantages, concerns about toxicity and oxidative stress remain, especially due to their interaction within biological system. However, the incorporation of surface modification using phytochemicals and biocompatible polymers has shown promise in mitigating adverse effects and improving biocompatibility. Ongoing research into the synthesis, characterization, and safe application of various INPs like gold, zinc oxide, silica, and iron oxide continuous to push the boundaries of science, offering tailored solutions for modern challenges. With careful evaluation and responsible design, inorganic nanoparticle holds the key to transforming diagnostic, therapeutics, and environmental protection in the future.

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