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ACOUSTICAL TECHNIQUES FOR CHARACTERIZING METAL OXIDE NANOPARTICLES IN ENERGY STORAGE SYSTEMS

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ABSTRACT

The authors present a comprehensive review of research on metal oxide nanoparticles, which includes their synthesis strategies, techniques, nanoscale physicochemical properties, and specific industrial applications across a variety of domains of applied nanotechnology. This review acknowledges the significance of metal oxide nanoparticles as technological materials. In this work, a complete analysis of the recent developments in semiconducting metal oxide gas sensors for ambient gases such as carbon dioxide, oxygen, oxygen, and nitrogen, as well as highly dangerous gases such as carbon monoxide, hydrogen sulfide, and nitrogen dioxide, flammable gases such as carbon monoxide, hydrogen sulfide, and liquefied petroleum gas, and volatile organic compounds, is presented. Different metal oxide nanoparticles have been subjected to independent analysis in order to investigate their gas sensing properties for a variety of different target gases. It has been discovered that metal oxide nanoparticles have the potential to be used for the sensitive and selective detection of particular gases. In this work, metal oxide sensors are classified according to gas analysis, and the primary techniques and production procedures that are utilized in nanotechnology are described. In addition, a complete investigation into the methodologies, sensing processes, and applications related with semiconducting metal oxide materials is carried out. Among the many applications that are associated with ultra trace-level gas sensors, batteries, magnetic storage media, various types of solar cells, metal oxide nanoparticles in catalysis, energy conversion, antennas (including micro strip and patch-type optically transparent antennas), rectifiers, optoelectronics, and electronics are a few examples.

KEYWORDS: Metal oxide · MONPs · Gas sensors · Batteries · Antennas · Solar cells · Optoelectronics

INTRODUCTION

Nanotechnology has made tremendous advancements in a variety of sectors over the course of the last decade, particularly in the domains of materials science and engineering. A scientific field that focuses on the creation of technologies that make use of nanomaterials that range in size from one nanometer to one hundred nanometers and exhibit a variety of shapes and morphologies is known as nanotechnology.Nanostructured materials with miniscule grain sizes have been proven to exhibit excellent strength and toughness, increased diffusivity, superior sintering capabilities, and a variety of other qualities, according to research. The dimensions and configurations of the materials are suited to a variety of scenarios in the fields of biomedicine, energy generation, storage systems, coatings, fertilizers, and agriculture. It should not come as a surprise that nanostructures can be modified and included into biomedical devices. This is because nanostructures are

naturally present in the majority of biological systems, such as viruses, membranes, and protein complexes. Currently, some of the most exciting and demanding uses of nanostructures are found in the fields of medicine and biomedical technology. There are a number of biomedical applications that have been developed as a result of the continual development and progress in nanostructures. These applications include scaffolds for tissue engineering, implant engineering, cell culture, and drug delivery systems. There are numerous stages that are included in the field of nanotechnology. These stages include the design, manufacture, characterization, and application of functional devices at the nanoscale. These devices are designed to take advantage of their distinct chemical, physical, and biological capabilities in comparison to those of bulk materials. Researchers have examined nanomaterials in medical domains such as bio-imaging and biosensors due to developments in characterization techniques and their specific features. This has led to the development of various diagnostic instruments that are crucial to the field. This behavior is greatly amplified on nanostructures with a high surface-to-volume ratio, which may present more active areas for subsequent interactions with biological entities. Proteins bind to material surfaces in a manner that is not specific to the material. Proteins that are adhered to a surface are known as poisonings because they alter the chemical composition of the surface as well as its state through their presence. The characterization of nanostructures through the application of contemporary approaches is a vital step that must be taken before studying the interfacial features and applications. Because of the sensitivity of the materials and the different ways in which they are handled, virtually all methods of characterization are not appropriate for all types of nanostructured materials (metals, carbon-based materials, polymers, biomolecules, and so on). In the first place, the nature of the materials and the surface qualities of the materials are the primary factors that determine which approach is used. During the process of classifying the biological sample, it is essential to use extreme caution in order to maintain its vitality. Because of the possibility of morphological alterations, imaging biological materials is a task that is both critically vital and extremely challenging. For the purpose of biomedical applications, a great number of nanostructured materials have been investigated. In rod, tube, cylinder, and spherical forms, the materials that have been investigated the most thoroughly are various types of carbon, silica, and metals. The most important aspects of toxicity and biocompatibility are determined by a number of different factors, including as size, surface area, carboxyl groups, quantity, and dosage.Techniques such as Field Emission Scanning Electron Microscopy (FESEM), Dynamic Light Scattering (DLS), Scanning Probe Microscopy (SPM), Near-field Scanning Optical Microscopy (NSOM), and Confocal Microscopy can be utilized in order to carry out topological analysis of nanostructures. This analysis involves the examination of spatial relations and geometric properties. Techniques such as X-ray diffraction (XRD), Transmission Electron Microscopy (TEM), and Magnetic Resonance Force Microscopy (MRFM) are utilized in order to carry out the investigation of the internal structure. Furthermore, X-ray Photoelectron Spectroscopy (XPS), Energy Dispersive X-ray Spectroscopy (EDS), Auger Electron Spectroscopy (AES), Secondary Ion Mass Spectrometry (SIMS), and X-ray Photoelectron Spectroscopy (XPS) are all techniques that can be utilized in order to carry out composition analysis. The primary focus of this study was to conduct an exhaustive investigation of the approaches listed above, which included case studies. Additionally, this research included a wide range of applications of nanostructured materials in the field of biomedicine**.**

METAL OXIDE NANOPARTICLES: SYNTHESIS, CHARACTERISTICS, SURFACE MODIFICATION AND CHARACTERIZATION

Synthesis Methods and Key Characteristics of Metal Oxide Nanoparticles

The production of nanoparticles can be performed through the use of either a "top-down" or "bottom-up" methodology. Methods of size reduction, such as lithographic techniques, milling, grinding, laser ablation,

and sputtering, are utilized in the top-down strategy, which involves the reduction of bulk materials into nanoparticles (NPs). The bottom-up strategy entails the synthesis of nanoparticles (NPs) by the utilization of chemical, physical, and biological means. These methods include the utilization of plant materials, microbes, and biological products.

In most cases, the creation of MONPs is accomplished through the utilization of chemical and physical synthesis techniques, also known as bottom-up approaches. This results in the manufacturing of a significant quantity of nanoparticles. There are a number of problems connected with these technologies, including the fact that they are associated with increased costs, the presence of harmful compounds (for example, those that are adsorbed on the surface of the nanoparticles), which may cause adverse effects in biomedical applications, and the requirement for maintaining stability.

A number of different methods of synthesis are included, but are not limited to the following: (i) chemical precipitation; (ii) wet chemical synthesis; (iii) hydrothermal synthesis; (iv) solvothermal synthesis; (v) sol-gel synthesis; (vi) solid-state pyrolytic techniques; (vii) thermal decomposition; and (viii) microwave-assisted synthesis.

In order to address the drawbacks of MONPs produced through conventional methods, which result in negative impacts on biomedical applications, the green synthesis of MONPs, also known as biosynthesis, has garnered a significant amount of interest. This is due to the fact that it makes use of eco-friendly and non-toxic reagents, which reduce harmful effects and enhance biocompatibility. This technique makes use of a number of different biopolymers, plant leaf extracts, algae, and surface-active biosurfactants, all of which contribute to increased specificity, biodegradability, and biocompatibility during the process. When it comes to the manufacturing of metal oxide nanoparticles, a complete investigation into the various synthetic processes that are utilized is presented.

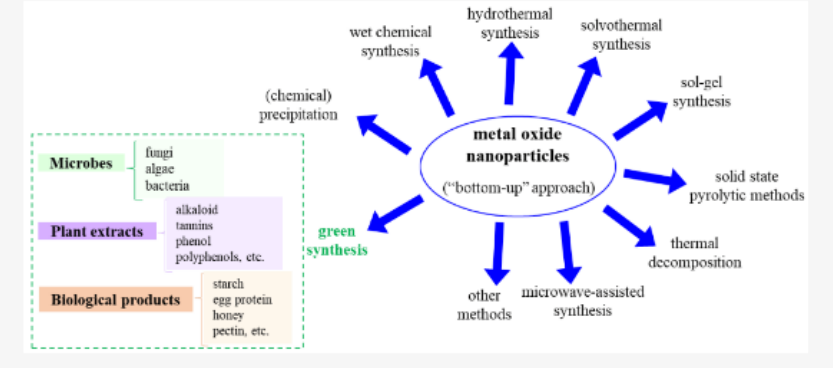

Figure 1 Possible metal oxide nanoparticle synthesis methods ("bottom-up" approach)

In order to produce the desired metal oxide nanoparticles (MONPs), chemical precipitation requires the application of a precipitating reagent (such as sodium hydroxide, ammonium hydroxide, or urea) inside the metal precursor aqueous solution. This is then followed by the annealing of the precipitate that is produced at elevated temperatures. At the same time as this method of synthesis produces nanoparticles that are extremely small, uniformly dispersed, and highly pure, it also has the potential to make nanoparticles that have a

crystallinity that is less than ideal and poses a risk of contamination due to the fact that it involves intermediate manufacture. Within the realm of spontaneous precipitation, the process takes place without the presence of a precipitating agent being introduced. Several other metal oxide nanoparticles, such as zinc oxide (ZnO), cerium oxide (CeO2), iron oxide (Fe2O3), titanium dioxide (TiO2), magnesium oxide (MgO), and nickel oxide (NiO), are precipitated using these processes.In order to stabilize the nanoparticles that are produced, the wet chemical synthesis method utilizes chemical precipitation in conjunction with an additive. For instance, this method is utilized for ZnO nanoparticles, which utilize starch as the stabilizing agent. Additionally, this method is utilized for other metal oxide nanoparticles, such as CeO2, Fe2O3, TiO2, MgO, NiO, ZrO, and CdO.

The hydrothermal synthesis of MONPs is widely used because it allows for great control over the form and size of the particles, reduces the amount of particle aggregation, is compatible with manufacturing on a large scale, and produces good purity. In spite of this, it is necessary to have longer reaction times, and it is vital to have multiple post-processing steps, as will be expounded upon further. When performing the synthesis, it is common practice to place an aqueous solution of a metal precursor inside of a Teflon-lined stainless steel autoclave. This autoclave is then filled with a precipitating agent, such as sodium hydroxide, which has been added drop by drop in order to achieve the desired pH. After the autoclave has been kept at a constant temperature (for example, 80–200 degrees Celsius) for a predetermined amount of time (for example, one to twenty hours), it is then subjected to a series of washing treatments, and finally, it is annealed. Examples of metal oxide nanoparticles (MONPs) that have been produced using hydrothermal synthesis include zinc oxide (ZnO), cerium oxide (CeO2), iron oxide (Fe2O3), titanium dioxide (TiO2), magnesium oxide (MgO), nickel oxide (NiO), and cadmium oxide (CdO).

The solvothermal synthesis is similar to the hydrothermal method, except other solvents are used in place of water. Typically, the reaction vessels or autoclaves are operated in a temperature range of 100 to 1000 °C and a pressure range of 1 to 10,000 bar. The solvents used typically include diethanolamine (ZnO NPs .ethanol (α-Fe_2O_3 NPs), methanol (ZnO NPs) 1,4-butanediol (γ-Fe_2O_3 NPs), toluene (TiO_2 NPs ,NiO) and ethylene glycol (Fe_3O_4 NPs). Nevertheless, in some syntheses, the use of a stabilizer is necessary, and when targeting biomedical applications, it should also be biocompatible (e.g., trisodium citrate).

The sol-gel method is a conventional and industrial method widely used for the synthesis of various NPs , offering, especially, good control over their size, high purity and homogeneity and low temperatures (on the downside, the use of organic solvents, availability of necessary precursors and long reaction times pose challenges). The key lies in the production of a homogeneous sol from the precursors and its conversion into a gel, followed by the removal of the solvent from the gel and subsequent drying. The molecular precursor is usually the corresponding metal alkoxide, which is dissolved in water or alcohol and converted to a gel by heating and stirring by hydrolysis/alcoholysis .Appropriate drying methods are necessary depending on the desired properties and application of the resulting NPs. A noteworthy point is the broad size-distribution of particles obtained via sol-gel processes. Examples of MONPs synthesized by sol-gel include the synthesis of ZnO NPs either by a modified sol-gel method resulting in a 25 nm NPs, which is smaller than with previously reported sol-gel processes ,or by typical sol-gel processes .Additionally, several other MONPs can typically be obtained via the sol-gel method, e.g., α-Fe_2O_3 ,MgO, NiO and CdO .

Solid-state pyrolytic methods are based, as the name suggests, on the pyrolysis of the metal precursor, while the pyrolysis temperature controls the particle size and the additional chemicals and resulting by-products,

and their dissolution can control the NP agglomeration. For example, different sizes of ZnO NPs (8 to 35 nm) were obtained by adjusting the pyrolysis temperature of the reaction mixture.

In order to produce metal-organic nanoparticles (MONPs) through thermal breakdown, it is necessary to raise the temperature of the metal precursor above its decomposition threshold while the solvent has a high boiling point. Despite the fact that these nanoparticles have the advantage of being highly monocrystalline, which eliminates the requirement for post-synthesis annealing, their yield is rather low. In most cases, precursor chemicals are composed of organometallic compounds that are dissolved in organic solvents. These organic solvents also contain surface-stabilizing agents, and the synthesis process takes place at elevated temperatures within an environment that is otherwise inert. It is necessary for a precursor to have a low decomposition temperature in order to achieve a big surface area and a small crystallite size. In addition, elevated temperatures are avoided since they have the potential to cause particle sintering, which in turn prevents the formation of nanoparticles. Modifying the parameters of the reaction, such as the precursor and the temperature, allows for the nanoparticles' dimensions to be altered to satisfy certain requirements. Nanoparticles of zinc oxide, iron oxide, cerium oxide, titanium dioxide, magnesium oxide, nickel oxide, and cadmium oxide are typically produced using this method.

As a result of microwave radiation's ability to facilitate rapid heating of the reaction system, the microwave-assisted synthesis of nanomaterials has made significant progress. This has resulted in a significant increase in the reaction rate (by several orders of magnitude due to the creation of localized reaction sites), which in turn has led to a reduction in the amount of time required for the reaction.Due to the fact that this method of synthesis cannot be scaled up since temperature and pressure cannot be controlled, it is advantageous for further exploration because it has a high nanoparticle yield, minimal agglomeration, and rapid response times. The synthesis of numerous metal oxide nanoparticles (MONPs) was accomplished through the utilization of microwave-assisted techniques. These techniques included microwave polyol synthesis (ZnO, CeO2), microwave heating techniques (α-Fe2O3, β-Fe2O3, Fe3O4, CdO), solid-state microwave irradiation (NiO NPs), microwave-assisted solution-based synthesis (TiO2, NiO), microwave-assisted hydrothermal methods (ZnO), low-power microwave-assisted heating (ZnO), and surfactant-free microwave-assisted mixing (ZnO), amongst others.

Additionally, additional synthesis methods have been reported, and these procedures are often exclusively applicable to particular metal oxides rather than all MONPs. These encompass mechanochemical synthesis (mechanochemical reactions at varying milling durations—Fe2O3 nanoparticles), co-precipitation through flow chemistry (Fe2O3 nanoparticles), continuous flow synthesis (TiO2 or γ-Fe2O3 nanoparticles), successive ionic layer absorption and reaction (NiO), direct chemical synthesis (NiO), anodic arc plasma (NiO), among others.

Due to the utilization of eco-friendly and non-toxic reagents, the green synthesis of MONPs has garnered increased interest. This is in contrast to other wet chemical synthesis methods, which involve hazardous chemicals. These chemicals may subsequently be incorporated into the final products, which can have an impact on the applicability of such nanoparticles in the pharmaceutical industry as well as other medical or biomedical domains. The management of nanoparticle morphology, lower expenditures, and the usefulness of enzymes and proteins contained in the source materials as effective reducing and capping agents are some of the benefits of green synthesis. In addition to the enhanced biocompatibility of the nanoparticles that are produced, green synthesis also produces nanoparticles that are more biocompatible. Microorganisms (fungi, algae, and bacteria), plant extracts from leaves, roots, fruits, or flowers (terpenoids, alkaloids, tannins, phenols, polyphenols, and so on), and a wide variety of biological products (starch, egg protein, honey,

agarose, pectin, and so on) are also exploited in this context.The microbial synthesis of MONPs is advantageous because, in comparison to conventional high-pressure and chemical procedures, it is less hazardous. Additionally, bacteria are able to adapt to the conditions of the synthesis, which is another reason why this method is advantageous. The green synthesis of metal-organic frameworks (MONPs) using plant extracts is dependent on the ability of these extracts to bioreduce metal ions and to generate nanoparticles that are stable. Furthermore, the process is uncomplicated, quick, and environmentally beneficial. In green synthesis, reaction rates are relatively slower than in conventional synthesis, and the variety of nanoparticle forms and sizes is limited. Due to the aforementioned advantages, this synthesis method is widely utilized for the production of metal-organic nanoparticles (MONPs) for use in biomedical applications. The process focuses on a wide range of nanoparticles, including silver and other metal oxides, as described in recent research or reviews (ZnO, Fe2O3, CeO2, TiO2, MgO, CuO, NiO, ZrO, ZrO2, and CdO).

On a final note, for more details with respect to the synthesis approach and methodology (used precursors, additives, stabilizers, or reactions conditions), the following reviews are recommended for the synthesis of various MONPs, i.e., for ZnO, CeO_2 ,Fe_2O_3 ,TiO_2 ,MgO ,CuO ,NiO and CdO NPs.

Additionally, in view of the biological effects (interactions with biofluids, cells, biomolecule, etc.) of such MONPs, these are influenced by a wide range of factors, such as NPs size, aggregation state, morphology and stability and, therefore, the synthesis methods are typically tailored towards achieving control over the NPs morphology, size and stability. For example, the physical and chemical properties of MONPs that have an impact on the interactions with cells are the (i) NPs morphology (shape, size), which controls aspects such as overcoming cell barrier, internalization and toxicity (ii) NPs surface area and surface energy, as this influences the number of active sites and can control reactivity (iii) crystal structure, which together with size, defects, media composition and aggregation, influences the dissolution of the metal ions, which can cause toxic effects (iv) surface chemistry, such as surface charge (zero-point of charge, acidity constant), dispersibility and aggregation, influence surface cascade reactions consequential for healing and subsequent biointegration (v) photocatalytic properties and chemical composition of the MONPs, as some nanoparticles can generate hydroxide or peroxide radicals and, furthermore, can (photo)release metal ions that may either promote adsorption reactions and/or facilitate favorable/unfavorable localized '-cidal' effects

Functionalization of Metal Oxide Nanoparticles for Biomedical Applications

The previous section discussed the various synthesis methods of MONPs, with an overview of ZnO, Fe_2O_3, CeO_2, TiO_2, MgO, NiO, ZrO and CdO NPs. While some of these NPs already present some biological effects in their bare nanoparticulate form, the performance and use of others type of NPs can be maximized by additional modifications. These modifications involve the surface functionalization of NPs such that they can elicit specific responses that may be biologically or chemically more favorable.

It should be noted that most of the synthesis processes result in hydrophobic NPs, as a result of synthesis conditions and due to the use of surfactants. This, in turn, limits the solubility of the NPs in aqueous or biological media. There are many approaches for surface modifications and these include functionalization with drugs, polymers, biopolymers, inorganic materials, or bioconjugation This is achieved by methods such as coating, conjugation strategies, in situ synthesis, self-assembly, surface encapsulation, or the synthesis of core-shell nanoparticles. After the surface modification, the functionalized nanoparticle is compatible with the biological environment, predominantly due to the hydrophilic nature of the coated shell

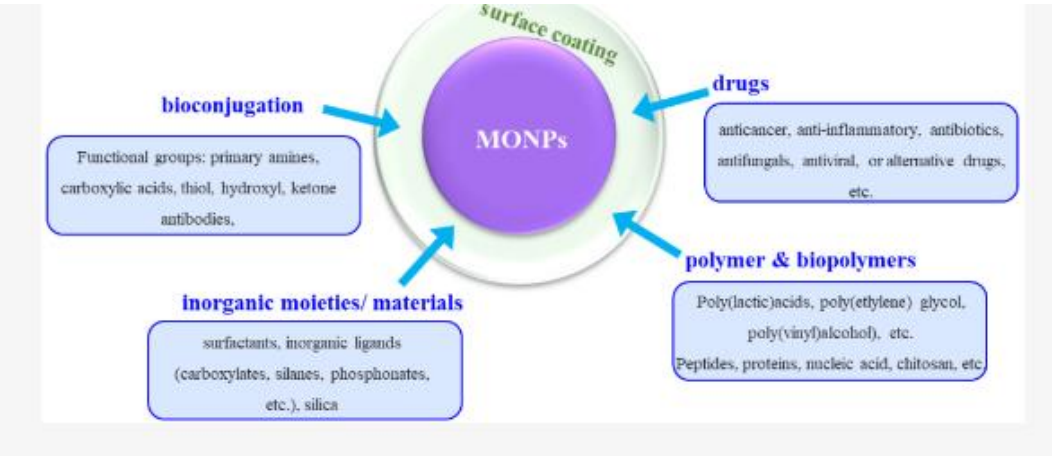

Figure 2 Schematic overview of the different surface modification/functionalization of MONPs usually applied for improving the biological effects

With respect to drug functionalization, the key advantages of using MONPs are twofold. The first is connected to the possibility of localizing the drug to the target cell or area, which significantly increases the potency of the drug, while reducing the dosage and, thus, removing the issue of toxicity to the tissue. Secondly, having a surface coating (shell) on the MONPs can stabilize the nanoparticles, influence the size of the colloid particle and their bio-kinetics and distribution in the body, as well as diminishing their toxicity .A wide range of specific drugs can be employed, such as anticancer, anticonvulsants, immunosuppressants, antibiotics, anti-inflammatory, antiviral, antifungal, or alternative, drugs. The drugs can either be covalently bound to the MONPs surface or via electrostatic interactions or via sequential functionalization such that loading and release kinetics are governed by affinity to binding substrates and localized environments.

MONPs can be modified by polymers and/or biopolymers, which also contributes to nanoparticle stability in physiological conditions, increase their activity towards biological interactions, and can be further used to introduce more diverse functionalities. Such polymer coatings can be achieved by either replacing an initial coating on the MONPs (e.g., legends) or by directly coating the polymer. Typical examples of polymers used include poly(ethylene glycol), poly(lactic-co-glycolic acid), poly(vinyl alcohol), poly(lactic acids), poly(vinylpyrrolidone), poly(alkyl cyanoacrylates), poly(e-caprolactone), poly(methyl methacrylate), poly(ethylene mine) and poly(dopamine). To further tackle the issue of toxicity of polymers at higher concentrations of longer treatment duration, an alternative is represented by using biopolymers such as peptides, proteins, dextrin, chatoyant, heparin, cellulose, lignin, etc.

The use of inorganic moieties or materials such as surfactants, e.g., sodium dodecyl sulfate and sodium oblate, inorganic legends such as carboxylates, silanes, phosphates and so on, or silica, is also widely employed for establishing a coating on the MONPs. For example, silica significantly increases NPs stability, biocompatibility and surface functionality with respect to biomedical applications, and is used for coating the surface of magnetic nanoparticles.

Another approach used for the fictionalization of the MONPs is bioconjugation, which consists of the conjugation of NPs surfaces with bimolecular whose tailored properties evoke favorable interactions in the biological environment. Often, linker molecules are necessary to obtain good adhesion and functionality of

the immobilized bimolecular. These conjugations enable the NPs to reach and effectively interact with site-specific cells.

Furthermore, the fictionalization method also depends on the chemical nature and surface properties of the chosen nanoparticles, and thus there is no universal method valid for all MONPs. For detailed information with respect to specific fictionalization approaches targeting the discussed MONPs of the present review, readers are referred to the following literature reports—ZnO ,Fe_2O_3 ,CeO_2 ,TiO_2 ,MgO NPs, or recent fictionalization approaches (NiO NPs).

Characterization of Metal Oxide Nanoparticles for Biomedical Applications

The typical characterization techniques for MONPs also in view of targeting biomedical applications are based on evaluating the: (a) morphology and composition—scanning electron microscopy (SEM) and transmission electron microscopy (TEM), combined with energy dispersive X-ray (EDX) analysis; (b) crystallographic structure—X-ray diffraction analysis (XRD); (c) molecular groups and chemical bonding—Fourier-transform infrared spectroscopy (FTIR), or time-of-flight secondary ion mass spectrometry (ToF-SIMS); (d) NPs' chemical and compositional properties—X-ray photoelectron spectroscopy (XPS); (e) synthesis mechanism—thermogravimetric analysis and differential thermal analysis (TGA–DTA); and (f) evaluation of the bandgap adsorption peak—UV-Vis spectroscopy. Moreover, other typical characterization techniques include the evaluation of the zeta potential, which is crucial in evaluating the effective electric charge of the nanoparticles (without or with further functionalization of the NPs). The different characterization techniques are also chosen as a function of the MONPs evaluated, due to the specifics of the metal oxide material and/or their further modifications.

MONPs—Morphology Evaluation

In order to evaluate nanostructure and nanoparticle morphology with regard to particle size (mean and distribution), electron microscopy techniques are indispensable. These techniques also provide precise structural information at the atomic level.

There are various MONPs that were synthesized utilizing a variety of techniques, and representative TEM images of these MONPs are provided in This article presents a high-resolution transmission electron microscopy (HRTEM) image that is characteristic of ZnO nanoparticles (NPs) that were manufactured using an environmentally friendly method through the utilization of E. prostrate leaf extract as a capping agent. In addition to displaying a variety of shapes, such as triangular, radial, hexagonal, rod, and rectangular forms, the nanoparticles have an average size of 29 nanometers and a size distribution that ranges from 16 to 85 nanometers each.The selected area electron diffraction (SAED) pattern is presented here, which demonstrates the nanoparticles' high degree of crystalline. A easy wet chemical method was utilized to manufacture the minuscule CeO spherical particles that are depicted in Figure b. These particles have a diameter of around 5 nm and were produced through the use of the HRTEM technique. Through the use of hydrothermal synthesis with E. globules leaf extract, Balaji et al. were able to synthesize biogenic ceria (CeO2) nanoparticles of varied sizes, including 50, 20, 10, and 5 nm. The HRTEM image exhibits the CeO2 nanoparticles with a diameter of 20 nm. Furthermore, the authors successfully validated the formation of CeO2 particles that displayed a fringe space measuring 3.1 Å. This was further corroborated by the XRD results, which indicated that CeO2 nanoparticles were arranged in a (111) plane measuring 3.24 Å or greater. In their study, Rufus and colleagues

have provided evidence that the creation of α-Fe2O3 nanoparticles utilizing guava leaves (Sodium guajava) by an easy precipitation approach is environmentally friendly and causes no harm to the environment. The quasi-spherical morphology of the nanoparticles was confirmed by scanning electron microscopy (SEM). The nanoparticles had diameters ranging from 20 to 48 nanometers, with a mean diameter of 35 nanometers. The EDX analysis revealed that the iron and oxygen weights were 62.55% and 37.45%, respectively. Additionally, transmission electron microscopy (TEM) examination validated the irregular morphology of the nanoparticles. The nanoparticles had an average size of around 38 nanometers and a rhombohedra structure. The lattice fringe width of 0.27 nanometers corresponded to the 104 facets of the rhombohedra configuration.

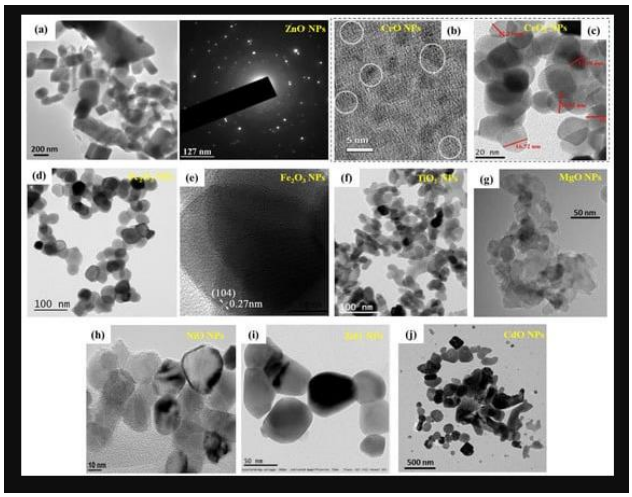

Figure 3 Morphology of MONPS: (**a**) HRTEM image showing green-synthesized ZnO NPs (*E. prostrata* leaf extract) together with the SAED pattern (reprinted from ref (**b**) HRTEM image demonstrating spherical crystalline CeO NPs, diameter ~5 nm, representative crystallites with lattice fringes in white circles (reprinted with permission from ref. Copyright 2021 Elsevier). (**c**) HRTEM image of green-synthesized 20 nm CeO_2 NPs (hydrothermal method mediated by *E. globulus* leaf extract (reprinted from ref. (**d**,**e**) green-synthesized α-Fe_2O_3 nanoparticles (guava leaves, Psidium guajava): (**d**) TEM micrograph, (**e**) HRTEM image of a single nanocrystal showing lattice fringes with a spacing of 0.27 nm (reprinted with permission from ref. Copyright 2016 Royal Society of Chemistry). (**f**) TEM image of TiO_2 NPs obtained by thermal decomposition (reprinted from ref (**g**) HRTEM image of MgO NPs obtained by a sol-gel method (reprinted from ref. Copyright© 2019 Alfaro et al.). (**h**) TEM image of hydrothermal NiO NPs (reprinted from ref. (**i**) HRTEM image of ZrO NPs obtained by a green synthesis (reprinted from ref. Copyright 2017 Elsevier). (**j**) TEM image of CdO NPs obtained by the annealing of formed complexes.

MONPs—Crystallographic Structure Evaluation

The technique of X-ray diffraction is widely used in the field of materials science for the purpose of detecting the crystalline structure of materials. It is also a crucial evaluation tool for metal-organic framework nanoparticles (MONPs). Some synthesis procedures require a phase of annealing or heat treatment, which is referred to as post-synthesis, in order to crystallize the nanoparticles (for example, chemical precipitation).

On the other hand, other methods immediately produce crystalline nanoparticles (for example, hydrothermal synthesis).

In particular, X-ray diffraction (XRD) is an indispensable tool for synthesis techniques that call for the evaluation of the influence that thermal treatment has on the crystallinity of nanoparticles. An X-ray diffraction (XRD) investigation utilizing the Debye–Scherrer equation revealed that the green production of zinc oxide nanoparticles employing cyanobacterium from A. platensis confirmed the presence of a wurtzite structure with an average crystal size of approximately 45 nanometers. Using Procopius fractal leaf extract, the scientists evaluated the effect that temperature had on the green synthesis of CeO2 nanoparticles. They discovered that the CeO2 nanoparticles had a fluorite cubic structure, which was comparable to the structure that was reported when the synthesis was carried out using Elea gnus angustifolia leaves.

X-ray diffraction (XRD) is an indispensable technique for determining crystallinity, lattice parameters, Miller indices, and crystallite size in a wide range of metal-organic frameworks (MONPs) such as zinc oxide (ZnO), iron oxide (Fe2O3), magnesium oxide (MgO), nickel oxide (NiO), zinc oxide (ZrO), and cadmium oxide [CdO].

MONPs—Chemical and Compositional Evaluation

The chemical and compositional structure of the MONPs, without or with further fictionalization, can be evaluated by several complementary techniques. FTIR can be used to identify the chemical bonds and characteristic functional groups, especially in view of functionalized MONPs or biomedical composites. For example, for the green-synthesized ZnO NPs (cyan bacterium from *A. Platens is* of a), the functional groups and chemical structures can be determined by FTIR. Peaks are observed and their assignments were: 3415 cm^{-1}—N–H overlap with a stretching O–H band, 3000 cm^{-1}—stretching CH_2 of asymmetric and symmetric carbohydrates and/or lipids, 1600 cm^{-1}—stretching C=O vibration of proteins or remaining acetate, 1410 cm^{-1}—C–N stretching bond of amino acid, 1341 cm^{-1}—vibration bending of the C–H (absorption wave of CH_2 or CH_3 of proteins), 1025 cm^{-1}—C–O–C ether of polysaccharides, 676 cm^{-1}—C=C bonds and 503 cm^{-1}—Zn–O absorption band. In addition to confirming the formation of the ZnO nanoparticles, data show the role of organic substances present in the *A. platens is* extract in the reduction, capping and stabilization of the biosynthesized ZnO NPs.

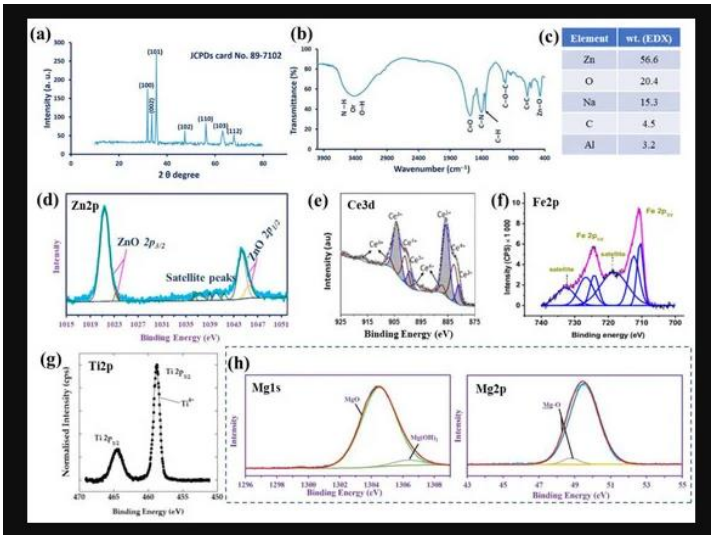

Figure 4 Green-synthesized ZnO NPs in the presence of *A. platens is* (cyan bacterium): (a) XRD patterns, (b) FTIR spectrum, (c) weight percentage from EDX (data from ref.), and (d) high-resolution XPS peak of Zn2p (a,b,d: reprinted from ref.). High-resolution XPS spectra for (e) Ce3d of CeO NPs (reprinted with permission from ref. . Copyright 2021 Elsevier), (f) Fe3d in Fe_2O_3 NPs (solvothermal synthesis in the presence of double capping agents) (reprinted from ref.), (g) Ti2p in TiO_2 NPs synthesized by microwave-assisted method (reprinted from ref.), and (h) Mg1s and Mg2p in MgO NPs (biosynthesis in the presence of metabolites from *Penicillium chrysogenum*) (reprinted from ref.

EDX can be used to evaluate the elemental composition of the NPs, and EDX coupled with TEM provides local chemical composition and mapping of the NPs. Similarly, for the ZnO NPs synthesized via a green route (cyan bacterium from *A. Platens is*) a,b, EDX was employed to evaluate the quantitative elemental structure and confirmed the presence of Zn, O, Na, C and Al with weight percentages of 56.6, 20.4, 15.3, 4.5 and 3.2%, respectively thus, confirming the ZnO NPs formation through the use of the metabolites in the *A. platens is* filtrate

CONCLUSION

In order to conduct an analysis of the physical and chemical properties of nanostructured materials, it is necessary to utilize a variety of characterization methodologies. Nanoparticles have attracted significant interest in a variety of applications due to their potential utility in medicine as well as their compatibility with other materials. When compared to other nanoparticles, biosynthesized nanoparticles are believed to be significantly safer, more sanitary, cost-effective, non-toxic, efficient, and environmentally sustainable. This is because they are created by plants and microbes. There is a growing demand for technologies that can change and characterize nanomaterials in both their raw and finished stages as a result of the widespread use of nanoparticles. Scanning probe microscopy (SPM), near-field scanning optical microscopy (NSOM), confocal microscopy, dynamic light scattering (DLS), and field emission scanning electron microscopy (FESEM) are some of the techniques that are discussed in this article. These techniques are used to analyze the topology of nanostructures. Furthermore, X-ray diffraction (XRD), transmission electron microscopy

(TEM), and magnetic resonance force microscopy (MRFM) are outlined as methods for conducting an examination of the internal structure of a material. There are a number of additional methods for compositional analysis that have been examined. These methods include X-ray Photoelectron Spectroscopy (XPS), Energy Dispersive X-ray Spectroscopy (EDS), Auger Electron Spectroscopy (AES), and Secondary Ion Mass Spectroscopy (SIMS). As a result of the fact that the majority of characterisation methods are non-destructive, make use of direct measuring instruments, are reproducible, and offer superior atomic-scale spatial resolution, these methods are advantageous for the characterization of nanostructures in the biomedical industry. In the coming years, there will be an increased demand for nanoscale characterization of complex structures. In order to meet this demand, a wide variety of techniques and enhanced high-tech sample preparation capabilities will be required. It is dependent on the level of technical innovation whether characterization techniques are sufficiently developed to support them or whether they require protocol design and proper validation for the issues that are currently present. Data processing is essentially statistical, and in-situ localization and placement create substantial obstacles. In addition, the data processing is important. In subsequent research, the special constraints of the physical and chemical testing methods that are currently in use for mixed nanoparticles will be the primary center of attention. It is necessary to conduct additional study in order to investigate the transdisciplinary ways that are not only highly scalable but also cost-effective.

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