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Various characterization techniques of nanoparticles used in numerous fields such as dentistry, medicine and pharmaceutical industry: a review article

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Abstract

Characterizing nanoparticles is crucial for grasping their distinct properties and behaviours, which influence their applications in fields such as medicine, electronics, and environmental science. This process employs a range of techniques designed to uncover the physical, chemical, and structural characteristics of nanoparticles. Methods like Transmission Electron Microscopy (TEM), Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM), and Dynamic Light Scattering (DLS) reveal information about size, shape, morphology, and surface properties. Spectroscopic techniques, such as X-ray Diffraction (XRD) and Fourier-Transform Infrared Spectroscopy (FTIR), provide insights into crystallinity and chemical composition. Each technique has its own advantages and limitations, often necessitating a combination of methods for thorough characterization. Understanding these techniques and their applications is vital for advancing nanoparticle technology and enhancing their performance across various domains. This article explores the different techniques, their necessity, and the challenges associated with nanoparticle characterization.

Keywords: Nanoparticle Characterization, Transmission Electron Microscopy, Scanning Electron Microscopy, Dynamic Light Scattering, X-ray Diffraction, Fourier- Transform Infrared Spectroscopy.

Introduction

The Greek word nanos, which means "a dwarf," is the source of the prefix nano. The prefix nano was formally accepted in 1947 during the 14th session of the International Union of Pure and Applied Chemistry (IUPAC) to denote the one-billionth part (10^{-9}) of a unit. In various branches of contemporary research, the prefix "nano" has become a common designation in scientific writing to refer to small objects and processes. Nanoscience, nanotechnology, nanorobots, nanomagnets, nanoelectronics, nanoencapsulation, and other terminology are among them, although they are not the only ones. The prefix nano is used in each of these instances to refer to "extremely small" objects or processes, most frequently at the nanometre scale.(Joudeh & Linke, 2022)

Characterization involves the analysis and comprehension of a material's properties and behaviour. In the realm of nanoparticles, this process entails employing diverse analytical techniques to examine and ascertain the physical, chemical, and structural attributes of nanoparticles, including size, shape, composition, crystal structure, surface chemistry, and behaviour in different environments. (Ealias & Saravanakumar, 2017) . These properties are illustrated in detail in the picture below (Figure 1)

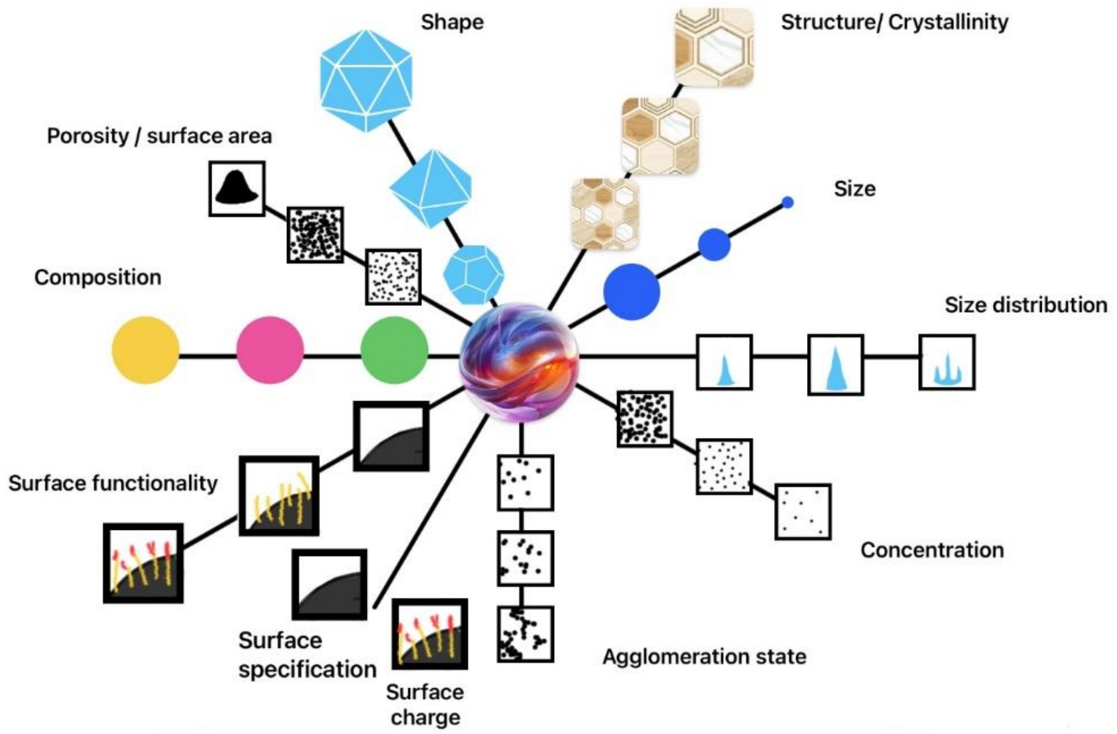

Figure 1. various properties and behaviours of nanoparticles involved in characterization

The following factors make nanoparticle characterisation critically important:

1. Recognizing Unique Features: Because of their small size and high surface area to volume ratio, nanoparticles frequently have unique features. Understanding and using these features for a variety of purposes is made easier with characterization.

2. Quality Assurance: Characterization guarantees the uniformity and superiority of synthesized nanoparticles, allowing scientists to verify the achievement of the desired characteristics.

3. Expanding Use: By gaining a thorough grasp of a nanoparticle's characteristics, scientists can modify them for use in medication administration, catalysis, imaging, and nanoelectronics, among the other fields.

4. Impact on the Environment and Biological Systems: Characterization helps to understand how nanoparticles interact with the environment and biological systems, providing information on their safety and possible impact.

Discussion

This article focuses on the various characterization methods involved in regard to nanoparticles. Nanoparticle characterization can be broadly divided into X-ray based techniques, other additional techniques involved, microscopic techniques and characterization techniques involving magnetic nanoparticles. X-ray based techniques involves techniques such as X-ray Diffraction (XRD) and X-ray Absorption Spectroscopy (XAS). Additional techniques include, other techniques except for X ray techniques such as Differential Scanning Calorimetry (DSC), Quartz Crystal Microbalance (QCM), Fourier-Transform Infrared Spectroscopy (FTIR), Electrophoretic Mobility (EPM) among other techniques. In characterization techniques for magnetic nanoparticles X-ray Magnetic Circular Dichroism (XMCD), Ferromagnetic Resonance (FMR) etc are few of the techniques used. Apart from the techniques mentioned above, microscopic techniques like Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM) etc are also used to characterize different properties of nanoparticles which have been discussed in detail in this article.

Types of nanoparticle characterization

X RAY BASED TECHNIQUES

X-ray diffraction (XRD) and X-ray absorption spectroscopy (XAS) are key techniques for nanoparticle characterization. XRD reveals crystal structure, phase, lattice parameters, and grain size by analyzing diffraction patterns from X-ray interactions with the sample. (Thanh et al., 2024) XAS, including Extended X-ray absorption fine structure (EXAFS) and X-ray Absorption Near-Edge Structure (XANES), offers insights into the local electronic and atomic structure, interatomic distances, and chemical states. EXAFS focuses on atomic structure and coordination, while XANES probes electronic structure and oxidation states.

Small-Angle X-ray scattering (SAXS) complements these methods by providing details on nanoparticle size, shape, and internal structure through scattering patterns. Together, these techniques advance nanoscience and technology. (Pugsley et al., 2011)

ADDITIONAL TECHNIQUES FOR THE CHARECTERIZATION OF THE STRUCTURE COMPOSITION AND OTHER MAIN NP PROPERTIES

Differential Scanning Calorimetry (DSC) is a thermoanalytical method that measures the difference in heat required to raise the temperature of a sample compared to a reference. (Badia et al., 1997)

Quartz Crystal Microbalance (QCM) measures the mass of nanoparticles adsorbed onto a quartz surface, offering real-time information on adsorption behaviour.(Burg et al., 2007)

Fourier-Transform Infrared Spectroscopy (FTIR) is a key technique for analyzing the chemical composition and functional groups of nanoparticles. FTIR generates an absorption spectrum that reveals details about surface chemistry, functionalization, and interactions with molecules, by directing infrared radiation at a sample. It's useful for understanding the surface chemistry and adsorption behaviours of nanoparticles. (Klasovsky et al., 2008)

Electrophoretic Mobility (EPM) is used to assess the surface charge of nanomaterials. Studies have examined how the aggregation and disaggregation of iron oxide nanoparticles (NPs) vary with NP concentration, pH, and natural organic matter. Low EPM values were linked to the formation of large aggregates, while very high EPM values were observed in highly stable NPs over extended periods.(Baalousha, 2009)

Measurements of the mass-to-charge ratio of ions and the emission of secondary ions, respectively, are used in Mass Spectrometry (MS) and Secondary Ion Mass Spectrometry (SIMS) to examine nanoparticles. These techniques offer vital insights on the elemental composition and surface chemistry of the nanoparticles. (Harkness et al., 2010)

Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) quantifies trace elements in nanoparticles, aiding in environmental and materials science research.(Elzey et al., 2012)

Dynamic Light Scattering (DLS) measures the Brownian motion of nanoparticles in solution to determine their size and distribution, though it may struggle with non-spherical or polydisperse samples.(Lim et al., 2013)

Nanoparticle Tracking Analysis (NTA) tracks individual nanoparticles to provide size distribution and concentration data, particularly useful for bimodal samples.(Hole et al., 2013)

Low-Energy Ion Scattering (LEIS) and its High-Sensitivity variant (HS-LEIS) analyze surface composition and thickness by measuring the scattering of low-energy ions from a sample. LEIS is effective for characterizing surface layers and coatings on nanoparticles.(Rafati et al., 2013)

For molecular analysis, Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS) offers excellent chemical sensitivity, but its resolution is only able to measure hundreds of nanometers. (Yang et al., 2013)

Elliptically Polarized Light Scattering (EPLS) provides detailed information on nanoparticle agglomerate's size, shape, and distribution, useful for various research applications. (Jiménez-Pérez et al., 2013)

Ultraviolet-Visible spectroscopy (UV-Vis) measures the absorption of UV and visible light, providing information on electronic structure, size, shape, and concentration of nanoparticles, especially useful for studying Localized Surface Plasmon Resonance (LSPR) in metallic nanoparticles.(Hendel et al., 2014)

Photoluminescence (PL) analyzes light emitted from nanoparticles after excitation, revealing information about their optical properties and electronic structure. It is useful for studying various nanoparticle types but may be limited by impurities or defects.(Zhang et al., 2014)

Thermogravimetric Analysis (TGA) tracks weight changes as a sample is heated, offering insights into thermal stability, composition, and the behaviour of organic ligands or coatings. TGA is essential for evaluating the stability and compositional characteristics of nanoparticles under thermal stress.(Sebby & Mansfield, 2015)

Resonant Mass Measurement Microelectromechanical System (RMM-MEMS) detects and measures sub-micron particles by analyzing changes in the resonating frequency of a cantilever. It is useful for various applications including environmental monitoring.(Patton et al., 2008)

Zeta Potential (ZP) and Electrophoretic Mobility (EPM) measure the surface charge and stability of nanoparticles in solution. Zeta potential assesses colloidal stability, while EPM evaluates particle movement in an electric field.(Baldassarre et al., 2015)

Gel Permeation Chromatography (GPC) separates molecules by size, providing data on polymer adsorption to nanoparticles and molecular weight distribution. Advanced detection systems integrated with Gel Permeation Chromatography (GPC) can provide detailed information about polymers, including molecular weight (MW) distribution, average molecular mass, and degree of branching.(Naden et al., 2015)

Nuclear Magnetic Resonance (NMR) spectroscopy offers valuable information about the structure, composition and surface characteristics of nanoparticles. NMR spectroscopy examines surface ligands, interactions, and molecular dynamics, by inducing nuclei to resonate within a magnetic field making it crucial for understanding the surfaces and behavior of nanoparticles.(Mourdikoudis et al., 2018)

The Brunauer–Emmett–Teller (BET) technique is employed to characterize nanoscale materials by measuring the physical adsorption of a gas onto a solid surface. This technique is named after its developers Brunauer, Emmett and Teller. This method is commonly used to determine the surface area of nanostructures due to its accuracy, speed, and simplicity.(Mourdikoudis et al., 2018)

Electrospray-Differential Mobility Analysis (ES-DMA) determines nanoparticle concentration with high resolution, complementing other size measurement techniques.(Mourdikoudis et al., 2018)

CHARACTERIZATION METHODS FOR MAGNETIC NANOSTRUCTURES

X-ray Magnetic Circular Dichroism (XMCD) examines magnetic moments and site symmetry of transition metal ions, offering detailed insights into the magnetic structure and contributions of specific ions within nanoparticles.(Brice-Profeta et al., 2005)

Ferromagnetic Resonance (FMR) probes magnetization in ferromagnetic materials, assessing particle shape, size, and magnetic properties. It helps quantify superparamagnetic nanoparticles in biological samples and study magnetic states in nanoparticle arrays.(Duraia & Abdullin, 2009)

Superparamagnetic Relaxometry (SPMR) utilizes the superparamagnetic properties of nanoparticles and sensitive sensors to study magnetization dynamics. It is used in fields like cancer research, where nanoparticles can be functionalized for targeted therapy.(Ludwig et al., 2009)

Electron Energy Loss Spectroscopy (EELS) identifies the atomic structure and chemical properties of samples, including atom types, chemical states, and interactions. It is effective for studying nanoparticle plasmon modes and distinguishing core/shell structures, though it is sensitive to electron beam damage, which can be minimized with careful sample preparation.(Schaffer et al., 2010)

Magnetic Susceptibility (MS) measures how a material's magnetization responds to an applied magnetic field, revealing whether it is attracted or repelled. It helps characterize nanoparticle’s magnetic properties, like superparamagnetism or ferromagnetism, and quantify their concentration in different matrices. This technique also assesses relaxation times in carrier liquids or immobilized particles, providing insights into their behaviour for applications in biomedicine, environmental science, and materials science.(Herrera et al., 2010)

An extremely sensitive method for determining the magnetic characteristics of nanoscale materials, such as blocking temperature, remanence and magnetization saturation is Superconducting Quantum Interference Device (SQUID) magnetometry. Through a superconducting loop, it senses magnetic flux and can distinguish moments as small as 10^{-8} emu. SQUID is adaptable and non-destructive, making it appropriate for a variety of settings, including biological fluids. An improved version, called NanoSQUID has the capacity to measure individual molecules and has better sensitivity. Deep sub-micron Josephson junctions are utilised, and it works well for researching the magnetic characteristics of nanoparticles, such as iron oxides. (Russo et al., 2012)

Mössbauer Spectroscopy uses hyperfine interactions to explore the local environments of isotopes like 57Fe, providing insights into iron oxidation states, magnetic anisotropy, and cation distribution in nanoparticles.(Xiao et al., 2013)

Vibrating Sample Magnetometry (VSM) analyses magnetic nanomaterials by recording M–H loops, revealing parameters like magnetic saturation and remanence. It has been used to study superparamagnetic nanoparticles and their electromagnetic wave absorption properties.(Wang et al., 2016)

MICROSCOPY TECHNIQUES FOR NP CHARACTERIZATION

Atomic Force Microscopy (AFM) creates 3D images of nanoparticle surfaces, measuring topography and mechanical properties. It can be used in contact, non-contact, or tapping modes and is useful for Surface-Enhanced Raman Spectroscopy (SERS) studies and assessing subcellular structure changes.(Binnig et al., 1986)

Electron Diffraction (ED) determines crystal structures by measuring electron diffraction patterns, providing insights into lattice parameters and nanoparticle orientations, though it requires careful analysis to account for scattering effects.(Buffat, 2003)

One popular way to get detailed images of surfaces at the nanoscale is through Scanning Electron Microscopy (SEM). Unlike light microscopes that use visible light, SEM uses electrons to create images. To study how gold nanoparticles interact with certain compounds, Mazzaglia and colleagues used both X-ray photoelectron spectroscopy (XPS) and field-emission SEM (FE-SEM). These methods helped them understand the shape and interaction of two types of carbon chains, thiohexyl and thiohexadecyl, with gold nanoparticles on a silicon surface.(Mazzaglia et al., 2009)

Transmission Electron Microscopy (TEM) provides high-resolution images of nanoparticles, revealing size, shape, crystal structure, and defects. It also allows for elemental analysis through Energy Dispersive X-ray (EDX) or Electron Energy Loss Spectroscopy (EELS) but requires careful sample preparation and sample might get damaged by electron beam. (Schaffer et al., 2010)

Magnetic Force Microscopy (MFM) uses a magnetic probe to scan and study the magnetic properties of nanoparticles, including distinguishing magnetic from non-magnetic types and analyzing superparamagnetic behaviour.(Neves et al., 2010)

High-Resolution Transmission Electron Microscopy (HRTEM) delivers phase-contrast imaging with the highest resolution, revealing atomic arrangements and internal structures. It distinguishes between single crystal and polycrystalline nanoparticles and examines ligand effects. (Axet et al., 2011)

High-Angle Annular Dark-Field Scanning Transmission Electron Microscopy (HAADF-STEM) provides high-resolution imaging and reveals internal structures and chemical variations within nanoparticles, useful for structure characterization and composition measurement. (Akita et al., 2011)

High-Resolution Scanning Electron Microscopy (HRSEM) offers high-resolution surface imaging, useful for studying morphology, size distribution, and surface characteristics, with capabilities for elemental analysis through Energy-dispersive X-ray spectroscopy (EDS). (Rades et al., 2014)

Aberration-corrected electron microscopy improves resolution by correcting optical aberrations, aiding in atomic structure analysis and mapping nanoparticle reactions and metastability. (Wells et al., 2015)

Challenges in characterization of nanoparticles

There are several challenges in the characterization of nanoparticles that have not been fully recognized by the research community. These challenges include:

1. Importance of Surfaces and Surface Characterization: The document emphasizes the significance of surfaces and interfaces in controlling the properties of nanostructured materials. It points out that efforts to understand the chemical and physical nature of these surfaces and interfaces are often not reported in the literature, and sometimes not measured. The rigorous application of surface studies is needed to understand and control the properties of nanoparticles, a concept referred to as nanosurface analysis.

2. Nanoparticles are not created equal: Subtle differences in synthesis and processing can have significant impacts on nanoparticles. This variability poses challenges in characterizing and understanding the properties of nanoparticles.

3.Properties of nanoparticles vary with time: Nanoparticles frequently change with time, which has implications for product lifetime and complicates the understanding of health and safety impacts. Understanding the time-dependent behaviour of nanoparticles is crucial for their effective utilization.

4. Nanoparticles respond to their environment: Nanoparticles are highly sensitive to their environment, which complicates their characterization and applications in various ways. This sensitivity adds another layer of complexity to the characterization process.

5. Nanoparticles are often unstable and easily altered: Nanoparticles are highly unstable and easily altered (damaged) during analysis. This instability poses challenges in accurately characterizing nanoparticles without altering their properties.

These challenges underscore the critical need for comprehensive and accurate characterization of nanoparticles to ensure their safe and effective use in various applications. Addressing these challenges is essential for advancing the understanding of nanoparticle.(Baer, 2011)

Conclusion

Understanding the complicated behaviours and properties of nanoparticles is essential for a wide range of scientific and technological applications. Various techniques provide distinct insights into the size, shape, structure, and magnetic properties of nanoparticles. These techniques include Transmission Electron Microscopy (TEM), High-Resolution TEM (HRTEM), Magnetic Force Microscopy (MFM), Atomic Force Microscopy (AFM), High-Angle Annular Dark-Field Scanning TEM (HAADF-STEM), Electron Diffraction (ED), Aberration-Corrected Microscopy, and High-Resolution Scanning Electron Microscopy (HRSEM). Through the provision of crucial information on atomic configurations, surface properties, and chemical changes, these techniques further the science of nanoparticles and facilitate the creation of novel materials and applications in a wide range of industries.

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