# STUDY ON CHARACTERIZATION TECHNIQUES OF NANOPARTICLES

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*ABSTRACT*

*There are a number of different classifications that can be used to the properties, shapes, and sizes of nanoparticles (NPs), which are very small materials that have dimensions that range from one to one hundred nanometers. The underlying material of nanoparticles may be used to classify them into many categories. These categories include metal, carbon, semiconductor, ceramic, polymeric, and lipid examples. In order to provide a description of the nanoparticles, the four primary factors that are used are size, optical qualities, surface charge, and shape. In order to explore the topography and morphology of nanoparticles, many microscopy methods are used. These techniques include scanning tunneling microscopy (STM), tip-enhanced Raman spectroscopy (TERS), environmental scanning electron microscopy (ESEM), and transmission electron microscopy (TEM). Through the use of spectral analysis, optical properties can be confirmed, and the fundamental properties of nanoparticles (NPs) can be learned through the utilization of X-ray crystallography (XRD), energy-dispersive X-ray spectroscopy (EDX), Fourier transform infrared spectroscopy (FTIR), and fluorescence correlation spectroscopy (FCS).*

*Keywords: - nanoparticles, polymeric nanoparticles, characterizing technique.*

## INTRODUCTION

The use of nanoparticles is rapidly becoming the norm in the field of current medicine. There has been a significant increase in interest in particles with a size range of 1-100 nm over the course of the last decade. This is due to the many possible applications for these very small particles in a wide variety of sectors, including the delivery of medication and the detection and treatment of cancer. As the size, structure, and morphology of nanoparticles diverge from those of bulk materials, the catalytic properties of nanoparticles become more pronounced. Nanoparticles that are composed of metals, such as gold, silver, alloys, magnetic minerals, and so on, have a wide range of applications across a variety of industries (Fig. 1). Both the top-down and bottom-up strategies are considered to be the two primary approaches to the production of nanoparticles. The transformation of metal ions into metal nanoparticles is an example of a chemical process, while the ball milling mechanism is an example of a physical activity. Coreduction, electrochemistry, micelle synthesis, reduction, sonochemical coreduction, sol-gel, laser irradiation, and microwave irradiation are some of the various methods that may be used in the synthesis process. Through their ability to make medical and technical procedures more accessible, less intrusive, less costly, and more portable, nanoparticles are revolutionizing their respective fields. Some of the numerous sectors that find use for nanoparticles include biological labeling, the treatment of certain cancers, optical devices, sensor

technologies, electronics, and a broad variety of industrial applications. These are only some of the possible applications. Nanoparticles have garnered a lot of attention in recent years owing to the fact that they possess a number of distinctive characteristics, including the capacity to withstand high temperatures and oxidation, as well as the antibacterial effectiveness that they possess.
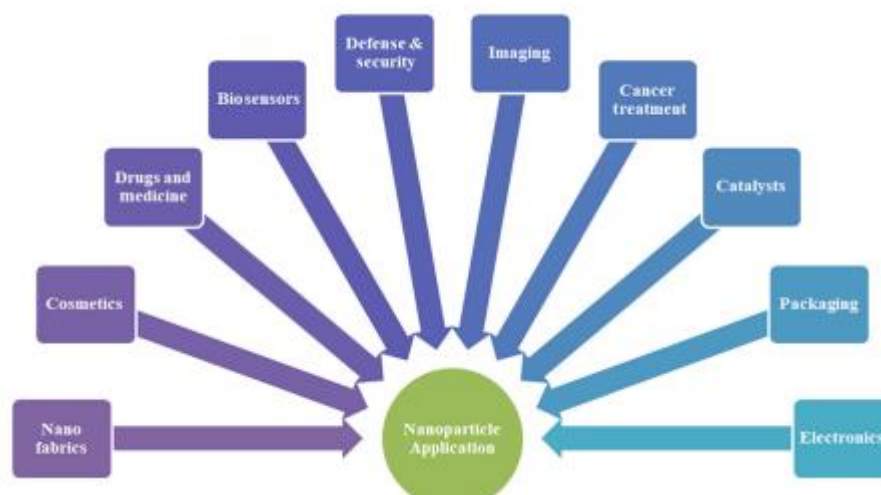


**Fig. 1 Applications of nanoparticles.**

It is also possible for biological activities to result in the production of nanoparticles. Biological methods are primarily concerned with the processes that include microbes and green synthesis. Relying on microbes for synthesis is a hard and sensitive process. Furthermore, the secondary metabolites of the plant extract, which perform the role of a reducing agent, contribute to the acceleration of the process. A number of publications have shown that the manufacture of extracts utilizing almost every component of the plant has been successful.

In addition, characterisation is required in order to confirm that the particles that were created are nanoscale. Within the realm of materials science, the term "characterization" is used to refer to the comprehensive and general techniques that are used in order to acquire knowledge on the structure and properties of a material. This fundamental method is necessary in order to get a complete understanding of the topic from a scientific point of view. characterisation encompasses a wide range of processes, including but not limited to mechanical testing, thermal analysis, density calculation, and any other operation that involves material analysis. Additionally, it encompasses the methods that are required to analyze microscopic structures and properties. Alongside the techniques of characterization that have been used for many generations, there are also strategies that are always being introduced that are more advanced and more recent. Characterization is a technique that may help improve understanding of the composition and structure of materials. Characterization also offers information about the effectiveness of the procedure. There are two methodological approaches: qualitative and quantitative.

**Microscopy-based nanoparticle characterization**

Through the use of optical microscopy, it is possible to see materials with a high degree of resolution at the micron level. When using optical microscopes, it is difficult to get further resolution due to the

characteristics of aberrations and the restrictions of wavelength. As a consequence of this, technology has made it possible to create alternative imaging techniques, such as atomic force microscopy (AFM), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and scanning transmission microscopy (STM), which are able to identify materials with a size that is less than one micron. The image that is produced by these methodologies is greatly expanded, despite the fact that the fundamental notions that underlie them are different. Nanoparticles may be seen via the use of these imaging techniques. The following material provides in-depth descriptions of the underlying operating concepts that underlie the various techniques.

**Scanning electron microscope mechanism and principles**

When compared to an optical microscope, scanning electron microscopy (SEM) is distinguished by the fact that it employs an electron beam rather than light in order to study objects. An electron cannon is housed inside the equipment at its highest point. When ignited, the electron cannon generates a concentrated stream of electrons. Both field emission guns and thermionic guns are examples of the two types of electron guns that are most often used. Electrons are extracted from atoms by field emission guns by the application of a high electric field, while thermionic guns accomplish the same thing through the heating of the filament. The scanning electron microscope (SEM) is a device that analyzes the surface of the sample by using electron beams of high intensity. Therefore, scanning electron microscopes are unique from conventional light microscopes due to the fact that they produce a magnified image by using light waves. The interaction between a beam of electrons from a scanning electron microscope (SEM) and a specimen occurs when the beam of electrons strikes the specimen.

Backscattered electrons, also known as primary electrons, secondary electrons, and Auger electrons are the types of electrons that are emitted by the specimen when it is struck by the input electron beam. The scanning electron microscope (SEM) makes use of both primary electrons, also known as backscattered electrons, and secondary electrons, also known as incoming electrons. Secondary electron microscopy (SEM) is a technique that provides high-resolution images that may display details as tiny as 1-5 nanometers. Electron microscopy, often known as EDX, is a technique that makes use of distinctive X-rays in order to determine the elemental compositions of a sample. In order to construct the image, this approach also takes use of the electrons that have been backscattered. This is a schematic representation of how a scanning electron microscope works, which can be seen in Figure 3. The electron beam is obtained by a final lens after it has been transmitted via the scanning coils that are included inside the electron column. Through this process, the beam is redirected in both the vertical and horizontal directions, which allows for raster scanning to be performed throughout the rectangular area of the surface. Cathode ray tubes are used for the purpose of displaying the images that are generated by electrical apparatus that is capable of detecting and amplifying signals. Alongside one another, microscopy and raster scanning are carried out. In the image that is being provided, the signal intensity distribution map that was obtained from the scanned area of the specimen is displayed.
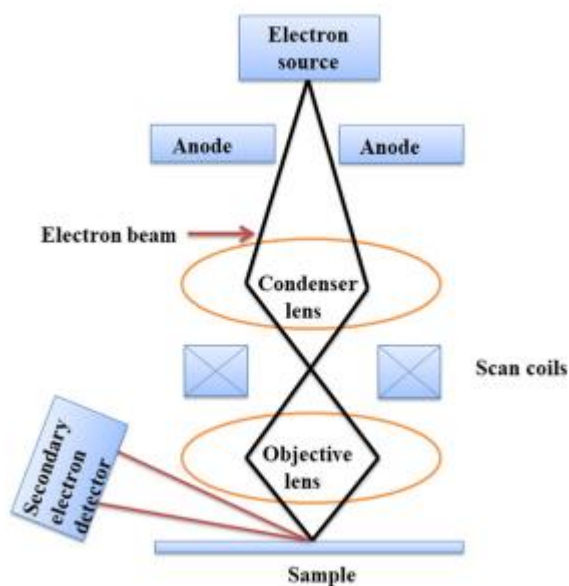
**FIG. 3 Working of SEM.**

## X-ray-based techniques

X-ray diffraction (XRD) is one of the many techniques that are used in the process of characterizing nanoparticles (NPs). X-ray diffraction (XRD) is often used to shed light on crystalline structure, as well as crystallite size, phase type, and lattice parameters. The Scherrer equation, which takes into consideration the sample's distinctive peak broadening, is used to estimate the second parameter. This is accomplished by fitting the XRD data of the sample to the equation. The results obtained by X-ray diffraction (XRD) techniques, which are often applied to powder samples after the colloidal solutions have been dried, are favorable due to the fact that they yield volume-averaged values that are statistically representative. It is possible to determine the particle composition by comparing the peak locations and intensities with reference patterns that may be obtained in the database maintained by the International Centre for Diffraction Data (ICDD), which was previously known as the JCPDS. Due to the fact that the peaks of the XRD waveform are overly broad for particles that are smaller than 3 nm, this technique is not suitable for amorphous materials.

Through the use of X-ray line broadening, Upadhyay and colleagues were able to ascertain that the average size of magnetite nanoparticle crystallites ranged from 9 to 53 nanometers. Instrumental widening was not the main cause of the XRD peak broadening; rather, the size of the particles or crystallites and the tensions in the lattice were the most responsible factors. Even in a single-domain particle, the XRD-derived size is often greater than the so-called magnetic size. This is due to the fact that smaller domains exist inside a particle that has all of its moments orientated in the same direction with the same orientation. The size that was derived using the transmission electron microscope (TEM) was greater than the size that was computed using X-ray diffraction (XRD). In actuality, samples that had particles that were bigger than 50 nanometers had more than one crystal boundary on their surface. Due to the fact that XRD is unable to discern between the two boundaries, the Scherrer formula's TEM size of 50-55 nm could not accurately represent the size of some materials. Due to the fact that the peak that corresponds to the ⟨111⟩ direction was much more intense

in their XRD measurement, it is likely that Dai and his colleagues manufactured ultra-small Au NPs that were more developed along that direction as opposed to the ⟨220⟩ direction during their experiment. In a similar vein, Li and colleagues discovered that the relative intensities between the various XRD peaks altered with particle shape after constructing copper telluride nanostructures of various shapes (for example, cubes, plates, and rods). This happened after they created the nanostructures at different sizes.

Both extended X-ray absorption near edge structure (also known as NEXAFS) and X-ray absorption fine structure (also known as EXAFS) are components of X-ray absorption spectroscopy (also known as XAS). The X-ray absorption coefficient of a material may be determined using X-ray absorption spectroscopy (XAS) via energy. In X-ray diffraction (XAS), the selectivity of elements is determined by the fact that every element has its own distinct collection of absorption edges that correspond to the binding energies of its electrons. Even when the concentrations of the species are very low, it is simple to detect the chemical state of the species using EXAFS since it is a highly sensitive approach. This technique is neither widely used nor readily available due to the fact that XAS spectra often need chromium. An investigation of the density of states of empty and partly filled electronic states is carried out by XANES. This is accomplished by taking into consideration the excitation of an electron in the inner shell to those states that are permitted by dipole selection criteria. Using in situ XAS, Pugsley et al. studied the rate of germanium NP formation as well as the process involved in producing germanium NP by reacting Mg2Ge and GeCl4.

The results obtained from TEM and EXAFS demonstrated that GeO2 nanoparticles were generated at the same time as Ge nanoparticles. For the EXAFS analysis, the first-neighbor Ge-Ge distance was found to be 2.45 Å, which is in agreement with the XRD findings. In addition, Chen et al. used in situ EXAFS in order to investigate the structural changes that would occur around germanium atoms in GeO2 nanoparticles. They discovered that germanium dioxide underwent a complete transformation when subjected to high temperatures in the presence of a sulfur source, which resulted in the formation of GeS2. This discovered was rather intriguing. Researchers Requejo and colleagues have been investigating the structural and electrical properties of alkyl thiol-capped palladium nanoparticles (NPs) in relation to the interaction between sulfur and palladium. The surface and bulk sulfidation of Pd clusters that were formed by the capping thiol molecules were disclosed by the atomic structure and electronic property investigations of these nanoparticles (NPs) that were conducted using XANES and EXAFS measurements.

**Additional techniques for the characterization of the structure, composition and other main NP properties**

A number of other methods are available for use in elucidating the NPs' fundamental characteristics, such as their structure, composition, size, and so on. The measurement of electromagnetic radiation absorption in the mid-infrared region (4000-400 $cm^{-1}$) is the basis of Fourier transform infrared spectroscopy (FTIR). A molecule may become IR active by absorbing infrared light, which modifies its dipole moment. By revealing the locations of bands associated with bond strengths and types as well as certain functional groups, recorded spectra provide light on molecular structures and interactions. Feliu and colleagues investigated the effects of Pt nanostructures on ethanol oxidation by using a hybrid method including in situ ATR-FTIR and differential electrochemical mass spectroscopy (DEMS). The use of these methods allowed for the electrochemical probing of adsorbates and the detection of volatile reaction products. The results corroborated earlier research indicating that COads generation on (100) domains and acetaldehyde/acetic

acid formation on (111) domains were the favored breakdown products, both of which were associated with surface features. The CO oxidation reaction was seen using DRIFTS and quadrupole mass spectrometry (QMS) in a separate study that employed carbon-supported platinum NPs (3-8 nm size). In line with the findings from QMS, the FTIR measurements of adsorbed CO verified the changes in COad and Oad at different experimental phases; moreover, changes in the distribution of CO across distinct kinds of Pt surface sites were also noted. When it came to testing the surface structure of Pt NPs in real-world settings, DRIFTS was generally considered a crucial instrument.

The surfactant bonding to FePt NPs produced in the presence of oleic acid and oleylamine was investigated by Shukla et al. in a work dedicated to FTIR analysis. The former ligand was shown to establish monodentate and bidentate bonds with FePt NPs, while oleylamine molecularly bound to FePt while preserving the NH2 group. In addition, two-phase synthesis methods in water/toluene combinations were used to create Au/Ag bimetallic NPs stabilized with dodecanethiol and soluble in nonpolar solvents. The primary finding from the XPS and FTIR analyses was that, in contrast to the Au atoms, the outermost region of the alloy clusters had more Ag atoms. A different study used ATR-FTIR to assess how the amount of Ag NPs affected the photocatalytic degradation of oxalic acid adsorbed on TiO2 NPs. After experimenting with different quantities of Ag NPs, it was found that even a modest amount (2% by weight) significantly improved the photocatalytic performance of TiO2 NPs.

Composite NP films were characterized topographically and chemically by means of AFM and XPS. By reducing nickel stearate in the presence of PEG, oleic acid, and oleylamine, Tzitzios et al. were able to manufacture Ni NPs with a hexagonal crystal structure ranging in size from 13 to 25 nm. In addition to studying the ligand binding modes onto the NP surface, FTIR spectra revealed the existence of distinctive groups at the NP surface, such as the -HCvCH- arrangement in OAc and OAm. Haram and colleagues used a hot-injection procedure to create copper zinc tin sulpho-selenide (CZTSxSe1\x) nanocrystals. At a temperature more than 200 °C, the precursors were dissolved in OAm and then subjected to synthesis. FTIR analysis revealed that OAm had been adsorbed onto the particles' surfaces. Its effective coordination with the NPs was shown by the presence of characteristic bands originating from the moieties present at the OAm molecule.

**CONCLUSION**

Nanotechnology, an important field of study, spans a broad variety of fields, such as the cleanup of the environment, the creation of energy, and several applications in the field of bioscience. The fundamental processes that are at the heart of nanotechnology include synthetic, industrial, and manipulative activities. Nanoparticles, which have emerged as the principal focus of attention in the present day, are what are necessary for the development of innovative and cutting-edge technologies. Understanding the characterisation of nanoparticles is equally important when it comes to the controlled synthesis of nanoparticles and the uses of these particles. Nanotechnology as a discipline has a tremendous deal of potential for the future; nevertheless, it is limited by limits such as the need for techniques of material characterization that are more effective, less complicated, and accomplished in a shorter amount of time. Combining a number of different approaches is the only way to get a more comprehensive understanding of particles and the characteristics they possess.

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