

Chapter 12

Nanomaterials Characterization

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ABSTRACT

Nanoparticles (NPs) are materials with size range of 1 to 100 nm. Due to their wide range of uses, nanostructures, a rapidly expanding class of materials, have drawn intense attention. Because of their small extent, NPs show increased attributes including high stability, sensitivity, strength, surface area and reactivity. These properties make them attractive candidates for a range of household and commercial uses, including as energy-based research, imaging, medicinal, catalysis, and environmental applications. Numerous methods have been used to describe the morphological, optical, structural, dimensions elemental makeup and several other physical characteristics of NPs. The main objective of NPs characterizations is to offer a summary of current understanding of that several experimental techniques accessible for NPs characterization. Numerous characterization methods that allow for accurate determination of these essential NPs characteristics are described as transmission electron microscope (TEM), scanning electron microscope (SEM), Atomic Force Microscopy (AFM), High-resolution transmission electron microscope (HR-TEM), Energy dispersive X-ray spectroscopy (EDX), Ultraviolet-visible diffuse reflectance spectrometer (UV-Vis DRS), Ultraviolet-visible (UV) Photoluminescence Spectroscopy (PL), Fourier-transform infrared spectroscopy (FT-IR), Raman spectroscopy, Brunauer-Emmett-Teller (BET), X-ray diffractometer (XRD), and X-ray photoelectron spectroscopy (XPS).

KEYWORDS

Nanoparticles, Characterizations, SEM, TEM, XRD, XPS, FTIR

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INTRODUCTION

The word "nano" comes from Greek word "nanos," which means "a dwarf." During 14th Congress of the International Union of Pure and Applied Chemistry (IUPAC) in 1947, term "nano" was legally recognized to denote one-billionth part (10^{-9}) of a unit (Joudeh et al., 2022). Nanomaterials are defined as materials containing at least 50% of nanoparticles (NPs) having one or more external dimensions between 1 and 100 nm (Schellau, 2019). When NPs are less than one nanometer, the phrase "atom clusters" are typically used. NPs can be crystallized or amorphous, containing either one or several crystals solids and may exist in aggregated or loose forms (Machado et al., 2015). In worldwide, nanotechnology is a concentrated field of contemporary study, endeavor for science and technology in this century. Nanotechnology is based on sophisticated production techniques and advanced nanomaterials (Wang, 2003b). Recent advancements in quickly expanding field of nanotechnology have produced wide variety of unique NPs (NP) with unique size-dependent characteristics that differ greatly from those of their bulk material (Fischer et al., 2007; Jong et al., 2008). These materials can have general forms that are 0D, 1D, 2D, or 3D (Tiwari et al., 2012). The significance of these NPs noticed when researchers found that substance's size may have an impact on its physiochemical characteristics such as visual qualities (Khan et al., 2019). NPs are not simple molecules and are composed of three layers. (a) The surface is initial layer, and it may be modified with variety of metal ions, small molecules, polymers and surfactants (b) The shell layer, which is chemically completely different from the core; and (c) The term "core" usually refers to NPs themselves and is essentially the center of NP (Shin et al.2016). Because of these remarkable qualities, researchers in multidisciplinary fields have shown

great deal of interest in these materials (Khan et al., 2019).

More varieties and larger volumes of nanomaterials being synthesized nowadays, than they were only a decade ago. It is necessary to develop more precise and reliable techniques for NPs characterization. Meanwhile, such characterization techniques aren't always precise. This is due to inherent difficulties in precisely analyzing materials at the nanoscale in comparison to bulk materials (such as their tiny size and, in certain situations, limited quantity after manufacture on a laboratory scale) (Mourdikoudis et al. 2018). Here, we describe in detail how different techniques are used to analyze nanostructures. Sometimes various techniques are used exclusively for studying particular characteristics, and often they are combined (Kim et al. 2004). Certain microscopy-based methods (as AFM, TEM, and HR-TEM; complete titles of aforementioned technique are afterwards in the passage when describing all of them together) present details on crystal structure morphology, and size of nanomaterials. A plethora of different approaches provide further details on optical characteristics, structure, elemental composition, and various common and more specific actual aspects of NPs samples. Among these approaches are scattering techniques and X-ray spectroscopy (Mourdikoudis et al., 2018).

This chapter is divided into many sections that provide general summary of present knowledge and latest advances for different NPs characterization techniques with respect to characteristics being study. We will then discuss the practical benefits and critical review of their underlying principles of those specialized techniques currently used for NPs evaluation. Future aspects and recommendations are also included in the last part.

Characterization Techniques for NPs

Due to inherent characteristics of NPs, such as their low ligand concentration, heterogeneity, nanoscale size, surface curvature, and characterizing NPs surfaces may be challenging. Furthermore, organic ligands or capping agents are frequently present on surfaces of nanomaterial. As a result, characterizations at both material and molecular levels are required (Jayawardena et al. 2021). It is necessary to thoroughly characterize NPs properties such as size, surface morphology, crystalline nature, shape, and light absorption by utilizing relevant characterization approaches (Chanderiya et al., 2024). For analysis of various physical and chemical properties of NPs various portrayal techniques have been used as presented in Fig. 1. These techniques including infrared (IR), SEM, TEM, X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), Brunauer–Emmett–Teller (BET) and particle size analysis (Khan et al., 2019).

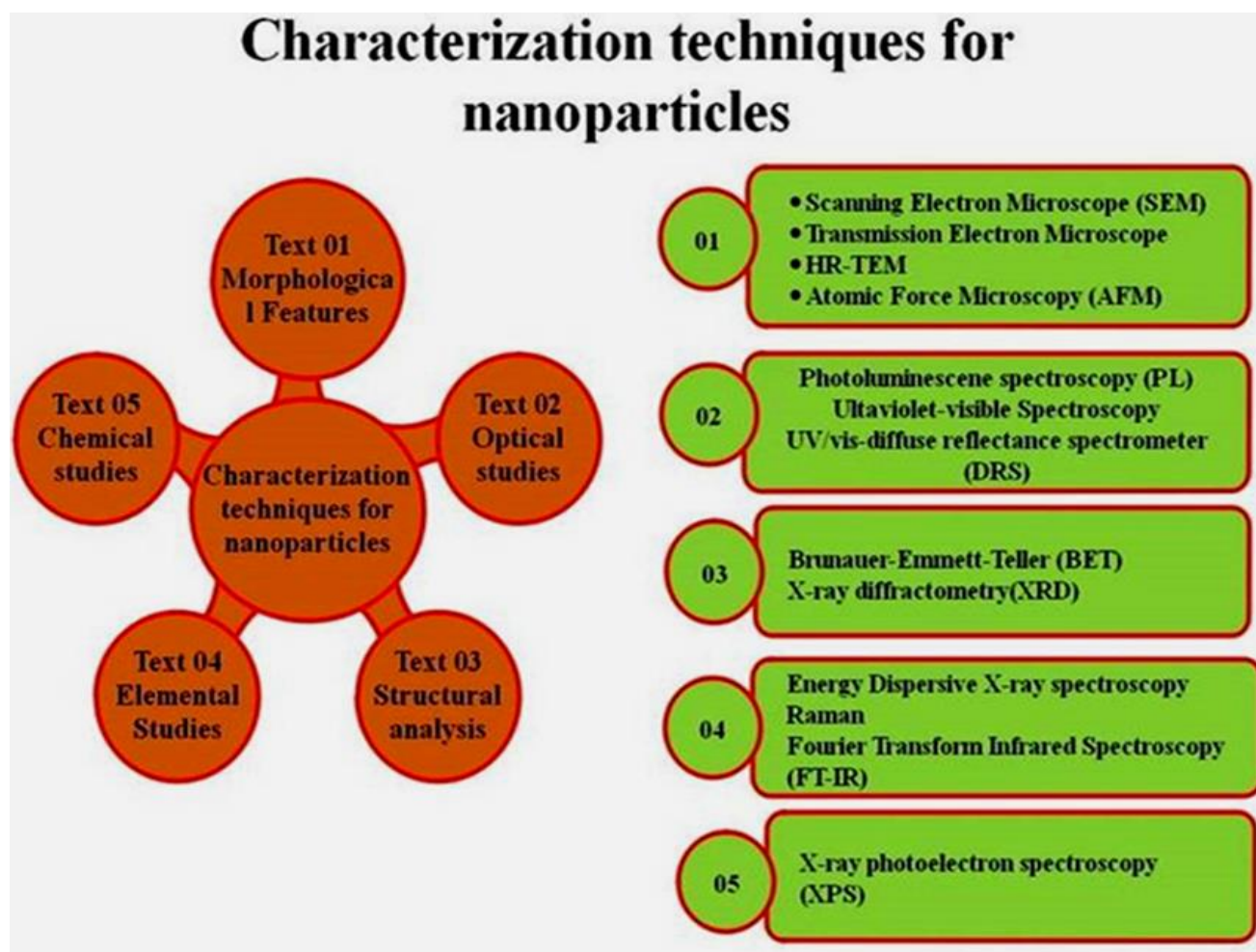


Fig. 1: Characterization techniques for nanoparticles.

Morphological Features

Scanning Electron Microscope

SEM allows revelation of details and complexity that are not visible with light microscopy. Many fields throughout the globe have used scanning electron microscopy (SEM), often classify as SEM analysis or SEM technique. SEM provides a means of glimpse to invisible world of nano and micro-space as presented in Fig. 2. The apparatus has variable pressure system that can accommodate any kind of sample, including ones that need little pretreatment or are moist. The apparatus can analyze samples up to 200 mm in diameter and 80 mm in height. The analysis will be carried out by using a high-energy electron beam with an electron voltage between 100 and 30,000 volts. For electron emission, heat source is typically utilized. It is not possible to make a crisp image with gun's spot size. The way scan coils move determines how specimen's picture is created point by point. The SEM picture that is somewhat three-dimensional is dependent on how sample's topography is visualized in terms of form, size, and surface texture (Mohammed and Abdullah 2018). On the nanometer to micrometer (μm) range, SEM may be considered as an efficient technique for analyzing both inorganic and organic nanomaterials. SEM produces extremely exact pictures of broad range of components up to 300,000x and even 1,000,000 times magnification (in certain modern versions) (Mohammed and Abdullah, 2018). SEM may offer details on crystalline structure, chemical makeup, electrical behavior, and surface topography of top 1 μm or so the specimen (Vernon-Parry, 2000).

SEM's advantages of high depth of field enable for simultaneous focus on most of specimen surface, regardless of surface roughness. It is possible to obtain far greater magnification (up to 1,000,000x) while maintaining an ultimate 1 nm resolution. It is possible to obtain more knowledge beyond surface topography, i.e. electrical properties, chemical constitution, and crystal structure. Extremely brief sample preparing time (possibly only a few seconds) while the sample is fastened to a "stub" (specimen holder) (Vernon-Parry, 2000).

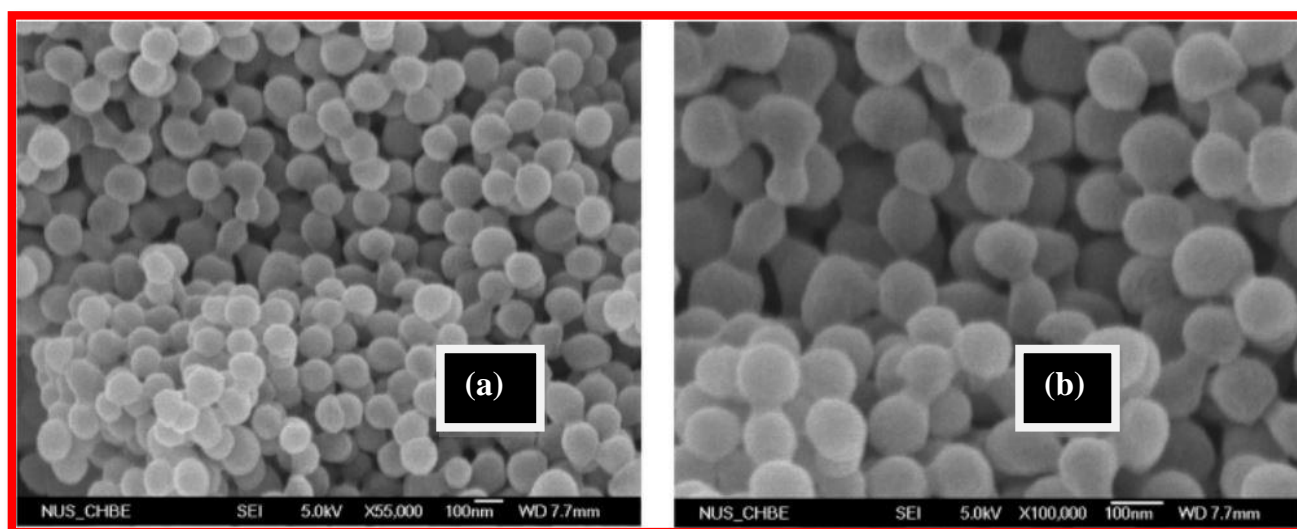


Fig. 2: (a) PLA-Tween 80-20, 55, 000 magnification; and (b) PLA-Tween 80-20, 100, 000 magnification (Zhang and Feng 2006).

Transmission Electron Microscope

TEM stands for transmission electron microscope is certainly among most significant technique for NPs characterization. By concentrating an electron beam on narrow sample (often less than 200 nm), TEM produces micrographs of nanoscale materials with great lateral spatial resolution (Williams et al., 2009; Surrey et al. 2012). A portion of electrons are transferred when electron beam hits the material, while, remaining electrons are elastically or in elastically dispersed (Kohl et al., 2008). The specimens must also be prepared and handled with utmost care because they are very thin and easily bent or broken. Thinning specimens to an electron transparent thickness is a necessary step in the production of TEM specimens. After that, sample is viewed under a table light to confirm the level of polishing without taking it out of holder. After that, procedure can be continued by replacing sample holder in electrolyte. It should just take a few minutes to complete this process. After quickly turning off power supply, sample holder is removed from electrolyte and carefully immersed in a beaker of methanol. The table lamp's light may be used to examine disc's perforation. Utilizing forceps, sample is extracted from holder, cleaned with methanol, and then relocated to a new petri dish filled with methanol. This procedure will remove the sample's electrolyte layer, if any exists. Next, a filter paper is used to dry the sample. In TEM, it may be briefly inspected (Rao et al., 2010). Several parameters, including size, elemental composition, and sample density, influence the interaction's strength. The final picture is constructed by the data obtained from transmitted electrons (Mourdikoudis et al., 2018).

TEM is most widely used approach for analyzing the shape and size of NPs because it offers not only direct images of the specimen but also the most precise estimate for homogeneity of each NP (Mourdikoudis et al., 2018). TEM is a technique for examining the synthesis of different super lattice nano-composites materials, which may be iso-structural to

several systems of atomic crystals (Shevchenko et al., 2006). TEM is usually used to see a single NP because of its high resolution (Ostrowski et al., 2015). Another benefit of TEM, is the ability to evaluate alterations in sub cellular structures brought on by NPs (Mourdikoudis et al., 2018).

High-resolution Transmission Electron Microscopy

A very effective technique for analyzing nanomaterials is electron microscopy with high resolution (HR-TEM), which is essential tool of nanotechnology (Wang, 2003a). A TEM imaging mode called HR-TEM (High-resolution transmission electron microscopy) can image both transmitted and scattered electrons (Andujar et al., 2016). HR-TEM requires larger objective aperture than conventional TEM in order to exploit the scattered electrons for imaging (Mourdikoudis et al., 2018).

While traditional electron microscope may offer an analysis of statistics evaluation of NPs form, single particle crystal structure cannot be seen because of their lack of resolution. This makes HR-TEM a valuable tool for learning about structure of NPs. As a result, most effective technique for characterizing NPs internal structure is now HR-TEM. Additionally, impact of materials on characteristics of metallic NPs has also been clarified by using HR-TEM (Mourdikoudis et al., 2018). HR-TEM is able to differentiate between anisotropic polycrystalline and single crystal Au NPs with comparable optical characteristics (Pallares et al., 2016). HR-TEM has been traditionally used primarily for solid material imaging, diffraction, and chemical analysis (Wagner, 1993).

Atomic Force Microscopy

AFMs, a kind of probe microscopes which employ tiny probes instead of electrons or light beams to examine a surface. Three-dimensional views of surfaces may be obtained using this kind of microscope as depicted in Fig. 3. AFM contains a tip that can be adjusted in numerous ways to examine surface qualities; as such, it is more evolved version of STM which is capable of imaging practically any form of surfaces at micro sizes (Vahabi et al., 2013). In 1986 at IBM AFM was invented by Heinrich Rohrer and Gerard Binnig (Binnig et al., 1986). The degree of closeness between sample and probe will determine, AFM may scan in 3 distinct modes: tapping mode, non-contact and contact mode (sometimes referred to as intermediate or in an oscillating form) (Clemente et al., 2008).

One benefit of AFM is that it doesn't require surface modification or coating before imaging. Therefore, AFM has been used without any specific treatment to perform topological description of tiny NPs (less than 6 nm), like ion-doped Yttrium oxide (Y_2O_3) (Patel- et al., 2007). Over last ten years, AFM has become highly effective instrument for obtaining biomechanical characteristics and nanostructural information of biological materials, such as cells and biomolecules. (Charrasand Horton, 2002; Horber et al., 2003; Pelling et al., 2004; Greenleaf et al., 2007). The primary benefit of AFM method in biology is its ability to examine biological samples in their native habitat. This is particularly useful for examining biological samples in buffer solutions *in-vitro*, *in-situ*, or even *in-vivo* a technique that used to require a lot of time to prepare (Yang et al., 1999; Sokolov, 2007;). In realm of cell biology, it can also detect live cells' surfaces down to single molecules (Hoh et al., 1994; Mathur et al., 2000; Berdyeva et al., 2004; Rabinovich et al., 2005; Zhao et al., 2005). Researching nanoscale, *in-situ* Deoxyribonucleic acid (DNA) structures using AFM has shown interest in creation of more potent gene delivery vehicles (Vahabi et al., 2013).

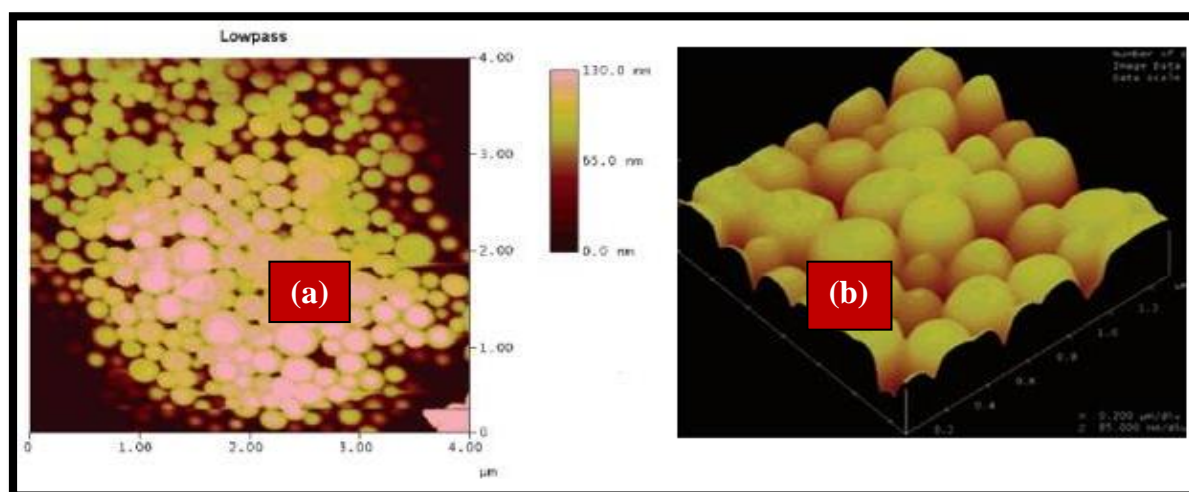


Fig. 3: AFM pictures of PLA-Tween 80-10 nanoparticles loaded with paclitaxel (a) 2D picture of 4 μm by 4 μm and (b) a 3D picture magnified (Zhang and Feng 2006).

Optical studies

Photoluminescence Spectroscopy

PL spectroscopy is an additional method for studying materials at nanoscale; it tracks light released by atoms or molecules that have taken up photons. For characterization of metal nano-clusters and fluorescent NPs like quantum dots,

PL is usually helpful. Depending on the type of study, possible PL spectrum is recorded as absorption or emission (Khan et al., 2019). Inherent PL of metallic NPs has attracted a lot of attention recently (Mourdikoudis et al., 2018).

Additionally, PL views this technique as useful for researching the visual characteristics of photosensitive NPs and different NPs. Furthermore, this method is effective in determining layer thickness (Lin et al., 2015), material doping amount (Huang et al., 2012; Gupta et al., 2013) and defects/oxygen vacancies (Torchynska et al., 2016) of NPs.

Ultraviolet-visible Spectroscopy

One rapid analytical technique to determine a light transmittance or absorbance is UV-vis spectroscopy (García et al., 2007). The feasible range for UV-vis spectroscopy is 200–800 nm range; wavelength underneath 200 nm is considered as vacuum UV while wavelength more than 800 nm is considered infrared (Rocha et al., 2018).

Chromophores are stimulated when UV-vis light strikes them; this process is known as electron-excitation. On other hand, auxochromes are electron-donating substances that may alter the color of chromophores without changing their own color. A great medium for UV-visible spectroscopy is alcohols and water generally they are transparent and they don't absorb in the UV-vis spectrum (Gürses et al., 2016). Using UV-vis spectrophotometer, light is passed through a specimen and on other side transmitted light is recorded by detector as depicted in Fig. 4. Transitions from one band to another, either longer wavelengths (red shifts) or shorter wavelengths (blue shifts), are known as bathochromic shifts and hypochromic. The terms hyperchromism and hypochromism refer to variations in the peak intensity of an absorption band (Robinson et al., 2005). The transmittance indicates amount of light absorbed at every spectrum, primarily consider greatest peak as λ_{max} . UV-visible spectroscopy is based on electronic transitions of organic molecules that excite electrons from lower energy orbital (highest occupied molecular orbital, or HOMO) to higher energy unoccupied orbital (lowest unoccupied molecular orbital, or LUMO) upon absorption of light (Rocha et al., 2018).

Applications in chemical engineering includes waste-water treatment, (Halim et al., 2016; Farias et al., 2017; Xiao et al., 2017; Quinlan et al., 2017; Zhou et al., 2017; Rocha et al., 2018) degradation of dyes, (Fan et al., 2017; Liu et al., 2017) and characterization of silver colloidal NPs, (Nogueira et al., 2016; Chen et al., 2017) copper (Das and Srivastava, 2016; Kalidhasan et al., 2017) and gold, (Haiss et al., 2007). The semiconductor band separation is deliberate using UV-vis diffuse reflectance spectroscopy, which is very helpful in photo catalysis research (Rocha et al., 2018).

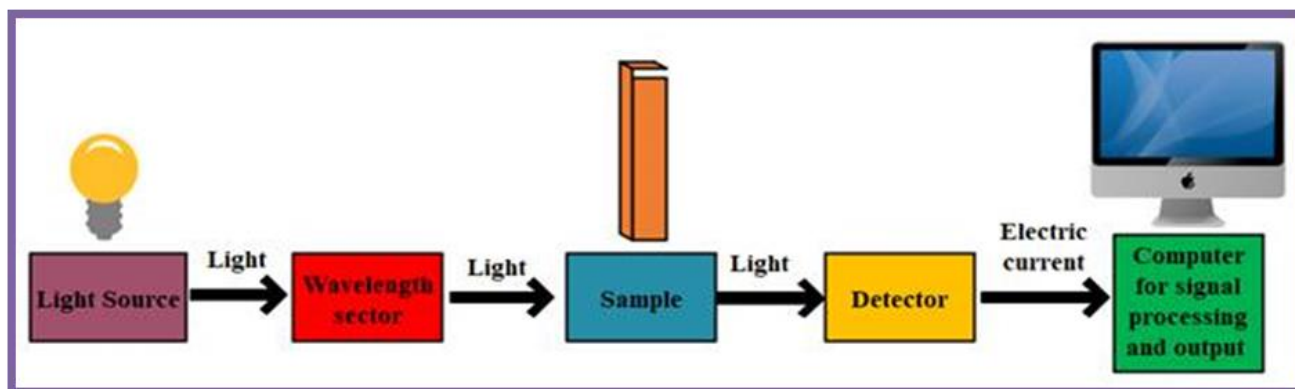


Fig. 4: Principle of Ultra visible spectroscopy

Ultraviolet-visible Diffuse Reflectance Spectrometer

UV-vis diffuse reflectance spectrometer (DRS) is a comprehensive tool for deliberate transmittance, reflectance and optical absorption (Khan et al., 2019). DRS spectroscopy is a type of spectroscopy that relies on powdered sample reflecting light in visible, near-infrared, and ultraviolet (vis) regions. The ratio of scattered light from an infinitely thick layer to scattered light from perfect non-absorbing reference sample is calculated as function of wavelength in DRS spectrum. Diffuse illumination of powdered samples is produced when incoming radiation illuminates them. Part of incident light is dispersed and partially absorbed. The sample's dispersed radiation is gathered in an integration sphere and detected (Weckhuysen and Schoonheydt, 1999). The bandgap is computed first by determining electronic state of sample by resultant diffuse reflectance spectra. The technique works incredibly well for determining out band gaps of a variety of NPs and other nanomaterials (Khan et al., 2019). Determining the bandgap is essential for evaluating conductance and photocatalytic characteristics, particularly for semiconductor NPs (Zhang et al., 2014). Comparably, this technique is also used to observe shift in absorbance when doping, composite formation, or heterostructured NPs materials are present (Zhang et al., 2014).

Structural Analysis

Brunauer-Emmett-Teller

The most efficient technique BET is used for measuring out NPs surface area. The substantial surface area of NPs provides infinite space intended for different uses. This method relies on Brunauer-Emmett-Teller (BET) theorem and

desorption and adsorption principle. For this, nitrogen gas (N₂) is often utilized (Khan et al., 2019). Since, there is little contact between the solid and gaseous phases, a partial vacuum is established during BET analysis in order to induce adsorption between sample and liquid N₂. This allows for surface to be cooled and observable levels of adsorption to be obtained.

At which point the amount of adsorption mono layers is formed the sample is taken out of N₂ environment and heated to induce the material adsorbed N₂ to be release (desorbs), after which the amount is measured. Isotherms (graphs that show how much N₂ is adsorbed function as a relative pressure on fixed heat) are used to portray gathered data. The surface area of sample is calculated using the data, which is shown in five isotherms (Naderi, 2015; Khan et al., 2019).

X-ray Diffraction

The most important characterization techniques for enlightening the structure properties of NPs are XRD. It provides sufficient information on phase and crystalline nature of NPs. Moreover, it offers a general notion size of particles via Debye-Scherer formula (Khan et al., 2017; Khan et al., 2017; Khan et al., 2017).

$$D = K\lambda / \beta \cos\theta$$

Where D is nanoparticles crystalline size, K represents Scherer constant (0.98), λ denotes wavelength (1.54), β denotes full width at half maximum (FWHM).

XRD methods are based on ability of crystals to diffract X-rays in a characteristic manner allowing precise study of the structure of crystalline phases. Recorded diffraction patterns contain additive contributions of several micro- and macro structural features of a sample as elaborated in Fig 5 (Epp, 2016). This method is well performed for identifying NPs both as solitary and multiphase processes (Emery et al., 2016). This method is based on incident X-rays being applied to a material and then measuring the X-rays' scattering angles and intensities as they exit from substance (Epp, 2016). Based on peak intensity, information about crystal structure (atomic positions, temperature factor, or occupancy) as well as texture and quantitative phase analyses can be obtained. Finally, peak shape gives information about sample broadening contributions (micro strains and crystallite size) (Dinnebier and Billinge, 2008). However, when samples exhibit highly amorphous properties with varying inter-atomic distance or when nanomaterials are less than few hundred of atoms, resolution and accuracy of XRD may be reduced (Khan et al., 2019).

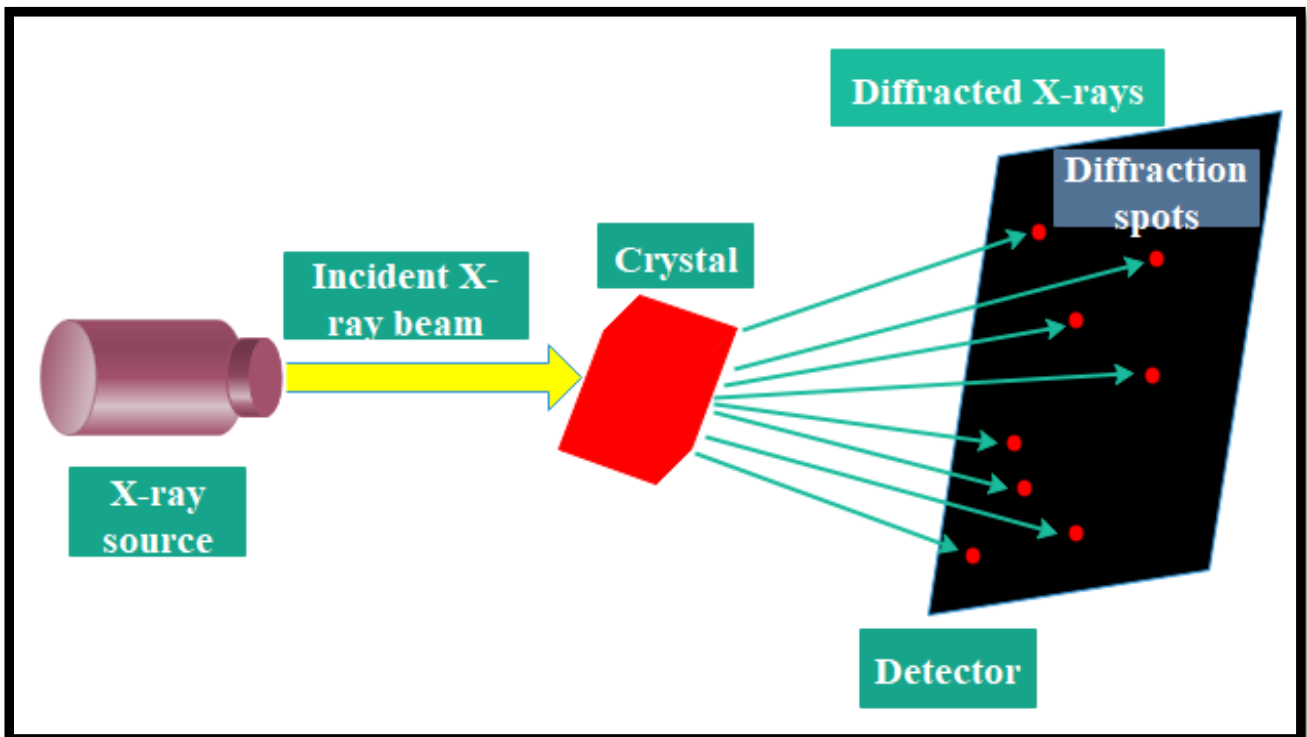


Fig. 5: An X-ray diffraction experiment's basic plan. A detector records the diffraction pattern, or "diffraction spots," created by the diffracted rays of an incoming X-ray beam entering the crystal.

Elemental Studies

Energy Dispersive X-ray Spectroscopy

Energy dispersive X-ray spectroscopy (EDX) is a non-destructive method for element classification by using scanning electron microscopy (SEM). A uniform energy of electron beam stimulates atoms in the sample, producing X-ray with distinct energy for every component which means energies of radiation released reveals elemental composition of specimen (Goldstein et al., 2017) as depicted in Fig 6. Electrons from inside shells of atoms with in specimens are removed

during contact by incoming beam of electrons, while outer shells orbits are replaced by vacancies. The energy released in the form of X-rays is a result of two shells' different energy levels. The chemical structure of sample may establish since X-ray intensity are intensities of the constituent components. Furthermore, concentration of that element in electron beams path determines the signal's strength of X-ray at any given energy intensity (Al-Fulaij et al., 2006) (Scoutaris et al., 2014).

EDX has been used in past for phase distribution and identification (Inman et al., 2007; Chen et al., 2013) enterically coated tablet dissolution (Liu et al., 2009) and composition of hydroxyapatite biomimetic coatings (Nie et al., 2000) examination of tiny size components (Doménech-Carbó et al., 2001; Iannuccelli et al., 2013; Gao et al., 2014) identification of alloys, and examination of foreign materials (Vilhelmsen et al., 2005; Chen et al., 2012). Using SEM images, EDX mapping may be utilized to evaluate the whole distribution of molecules on surfaces.

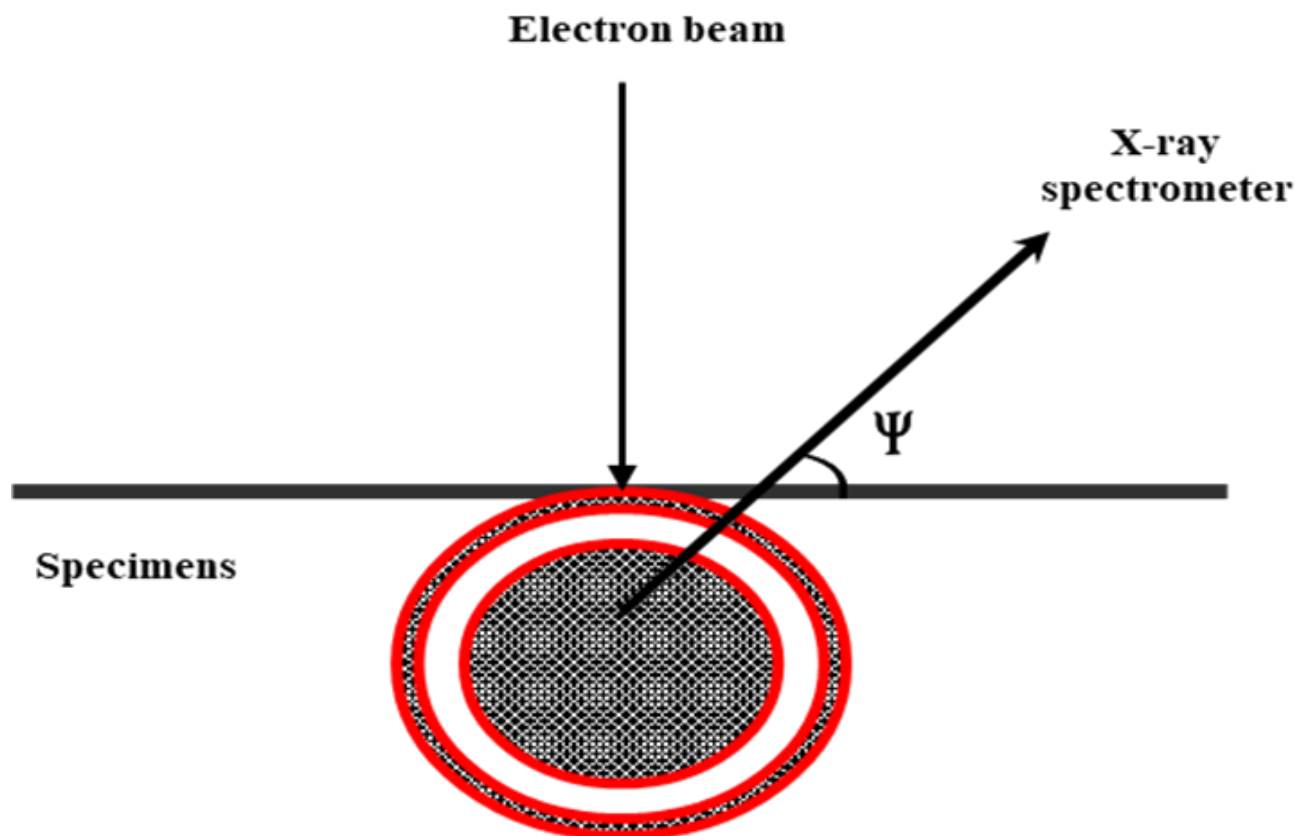


Fig. 6: X-ray source region where (X-ray path to the spectrometer, is represented by ψ being the take-off angle)

Raman Spectroscopy

The use of Raman spectroscopy has not regarded as valuable systematic technique for many years due to extremely poor competence of "typical" scattering of Raman. A normal molecule's overall Raman scattering of sample is around 10^{-29} cm^2 , while an infrared and ultraviolet molecule's typical cross-sections are approximately 10^{-18} and 10^{-21} cm^2 , respectively (Aroca, 2006). Cross sections Raman scattering may be significantly greater than before, for example, to 2×10^{-14} cm^2 for every element (Michaels et al., 1999) by using special resonators made of metal nano-clusters. This is approximately greater than 15 orders of magnitude rather than normal Raman scattering, and it allows for even detection of single molecule's Raman spectrum (Kneipp et al., 1997; Michaels et al., 1999; S. Nie and Emory, 1997). Analysis of sample portions not exposed at the surface can be performed when specimens is semi-transparent (or transparent) to dispersed and stimulating radiation (Matousek et al., 2005; Eliasson and Matousek, 2007; Welter et al., 2007).

Raman spectrum a useful technique that may be regarded as fingerprint of compound for identification of many compounds (Kudelski, 2008). Non-destructive Raman examination of different living specimens is very important from an operational perspective (Schrader et al., 2005; Reitzenstein et al., 2007). The Raman mapping approach may also be used to provide comprehensive information on the distribution of certain molecules, such as secondary metabolites (Baranska et al., 2004; Baranska et al., 2005; Schrader et al., 2005). Raman spectroscopy has several biological applications, including detection of precancerous cells and differentiation of malignant from normal breast tissue. Demonstrated that Raman spectroscopy may distinguish malignant tumors from healthy breast tissue, and in mouse model, it may detect early neoplastic alterations (Baranska et al., 2005). Jess et al. demonstrated the potential of Raman spectroscopy a valuable instrument for early identification of human papillomavirus (HPV)-exposed cells (Jess et al., 2007). Analytical science will employ Raman spectroscopy much more as long as technology advances spectrometers as well as lasers are smaller, less costly, more dependable, and easier to operate (Kudelski, 2008).

Fourier Transform Infrared (FTIR) Spectroscopy

The oscillatory properties of co-factors and amino acids are investigated using Fourier transform infrared (FTIR) spectroscopy that is delicate to even smallest structure alterations. With Fourier transform infrared (FTIR) spectroscopy, reducing agents that are in charge of stabilizing, reducing, and capping metal nano materials might be found as depicted in mechanistic illustration shown in Fig. 7. It is known from studying FTIR spectra that functional groups adhere the surface of biosynthetic non-metals include $-C = O-$, $-NH_2$, and $-SH-$ groups (Huq, 2020). If molecule's dipole moment changes during typical vibration mode, mode turns on infrared activation, meaning incident infrared light absorbs by it. For this reason, symmetric vibrations are typically undetectable in infrared. Specifically, all vibrations that are symmetrical with regard to center of a molecule are infrared inactive. All molecules, on the other hand, exhibit asymmetric vibrations. Since it is less selective than other spectroscopic techniques, we are able to examine the characteristics of practically each functional groups of a single specimen, particularly those of water molecules and amino acids. Between $4,000$ and $1,000\text{ cm}^{-1}$ in the mid-infrared, two primary vibrational type are detected: which are oscillations along chemical bond involving bond length changes is stretching vibration (m); and vibration involving change in bond angles, particularly bending vibrations (p—out of plane, d—in plane).

Applications for Fourier transform infrared (FTIR) spectroscopy or infrared (IR) are many and include examination of tiny molecules, chemical complexes, cells, and tissues (Berthomieu and Hienerwadel, 2009). Additionally, the study of proteins has made greater use of FTIR spectroscopy. During enzyme reactions, protein folding, conformation, and molecular characteristics from protein active sites are examined using reaction-induced FTIR difference spectroscopy (Siebert and Hildebrandt, 2008).

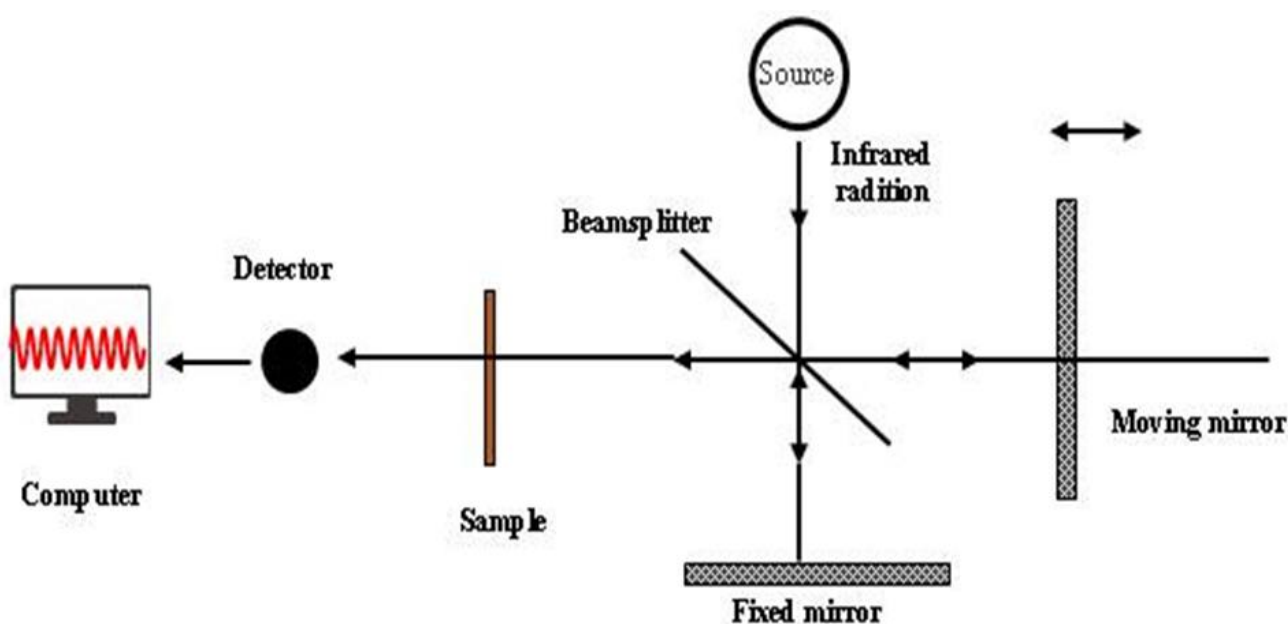


Fig. 7: Michelson interferometer's basic graphic, from Leng "Materials Characterization"

Chemical Studies

X-ray Photoelectron Spectroscopy

Among most effective methods for characterizing surface particularly assessment of corrosion, is X-ray photoelectron spectroscopy (XPS), this is capable of figuring out chemical makeup of various material surfaces within 10 nm . When examining surface reactions in vacuum, with relation to monolayer, XPS is very useful instrument. Tools for exploring nanoscale surface layers are crucial since layer on the surface involved in first surface reaction in relationship to surroundings is just a few nanometer thick as depicted in Fig. 8 (Krishna and Philip, 2022). Many different types of nanostructures are now regularly examined with XPS. The structure and shape of NPs may be crucial in accurately interpreting XPS (and relevant) information of any nanoscale-sized substance even though most XPS research is carried out under assumption that specimen has layer of uniformly smooth surface (Baer et al., 2010).

For understanding various crucial characteristics of nanostructure in natural and synthetic materials that are difficult to get through other methods, XPS is a valuable, well-established, and frequently indispensable instrument (Baer and Engelhard, 2010). Surface evaluation using XPS has recently, applied in number of domains, including mineral processing, biomedicine, electronics, corrosion, catalysis, nanomaterials, automotive, and aerospace etc (Krishna and Philip, 2022). The method is most frequently employed to determine nanomaterials' surface functionalization, element composition, coating thickness, adsorbents and in few situations, particle dimensions. For instance, core-shell structure and element composition of iron oxide NPs (Fe_3O_4 Fe_2O_3), as well as shell thickness at less than 1 nm , were effectively estimated using XPS (Anushree et al., 2020). X-ray photoelectron spectroscopy (XPS) is a tool use in material science to verify true

substance makeup as well as composition of surfaces and interfaces. In addition to these characteristics, XPS is also able to reveal the following: (i) distribution of element on shell; (ii) structure and thickness of surface layers; (iii) molecular orientation deposited on surface; (iv) coating material composition; and (v) size of NPs (1-20 nm).

In domain of organic materials and in biology, XPS offers knowledge that is comparable to that of material science. The plane chemistry of microorganisms, development of biocompatible materials, biofilms, and medicinal resources are all studied using XPS. Similar to this, in medicine, the surface characteristics of pharmacological medicines and biomaterials determine how they interact with the host body (Krishna and Philip, 2022).

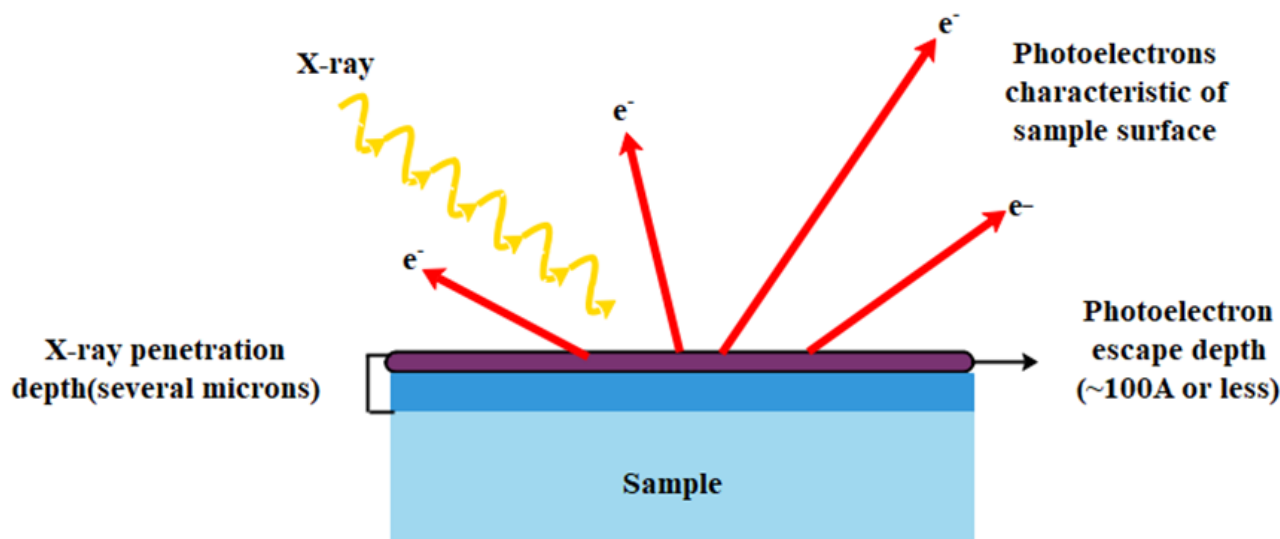


Fig. 8: X-ray photoelectron spectroscopy

Future Recommendation:

Naturally, there are barriers in scientific community that prevent accuracy and resolution of certain approaches from being further improved. As temperature, pressure, time, and pH may all have substantial impact on structure and morphology of NPs materials and produce novel products, future study should concentrate on optimizing these factors. Furthermore, certain characterization techniques have to be used for positive consequences and characteristics. It is increasingly important to think about environmental concerns before using these materials for any purpose, especially when it comes to heavy metals, which may negatively affect both environment and human health.

Conclusion

Nanotechnology, a key research area, focuses on advanced nanomaterials and manufacturing, leading to unique NPs properties. An overview of many important NPs characterization techniques is provided in this chapter. One of main areas of modern research is nanotechnology. A number of techniques for characterizing NPs are designed to help to explain their size, shape, optical, structural, elemental, and chemical characteristics. Every attribute may be obtained from various machines and through various methods. NPs characterization is carried out using different techniques, including SEM, TEM, HR-TEM, AFM, PL, UV, DRS, EDX, Raman, FTIR, BET, XPS and XRD.

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