

Chapter 11

Nanocharacterization Methods for Different Materials

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1. Introduction

Nanotechnology has emerged as a rapidly advancing field that enables the design and manipulation of materials at dimensions below 100 nm (Madkour, 2019). At this scale, materials exhibit unique physical, chemical, mechanical, and optical properties due to quantum confinement effects, large surface-to-volume ratios, and altered interfacial interactions. These distinctive characteristics make nanomaterials highly valuable in applications such as electronics, energy storage, catalysis, bio-medicine, and advanced materials engineering.

Nano characterization refers to a suite of characterization tools that aid in analyzing the structure, morphology, chemistry, and functionality of materials at the nanoscale. It plays a vital role in understanding the relationship between nanomaterial structure and performance. Considering the complexity involved in studying nanomaterials due to the nanoscale nature of these materials, nano characterization is very important.

Nano characterization techniques provide critical information regarding morphology, crystallinity, surface chemistry, composition, and electronic structure, enabling optimization of synthesis processes and material performance.

Since no single technique can fully characterize a nanomaterial, complementary approaches including microscopy, spectroscopy, and structural analysis are often combined to achieve a comprehensive understanding (Kumar et al., 2019). **Figure 1** presents an overview of the principal techniques employed for characterizing materials at the nanoscale.

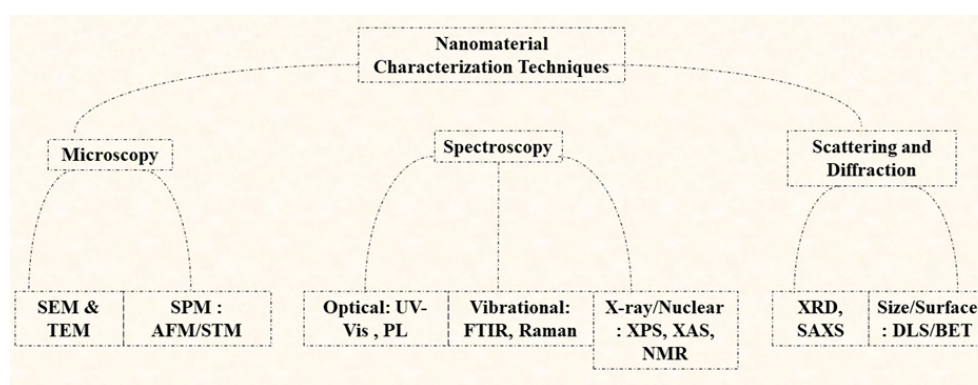


Figure 1. Flowchart of nano characterization methods

Even though significant progress has been made, there are still many challenges in nanoscale characterization because of problems like sample preparation, beam damage, instrument limitations, and difficulty in interpreting data, among others. In fact, for some systems, it is necessary to use in situ or even in situ and real-time characterization techniques (Kong & Ariga, 2025).

This chapter presents an overview of major nanocharacterization techniques, discussing their principles, capabilities, limitations, and applications. It also highlights current challenges and emerging developments to provide a comprehensive understanding of characterization strategies used in modern nanoscience and nanotechnology.

2. Fundamentals of Nanocharacterization

2.1. Nanoscale Regime and Size-Dependent Phenomena

Nanocharacterization examines materials in the 1–100 nm range, where quantum effects and high surface area lead to properties that differ from those of bulk materials. These effects strongly influence optical, electronic, mechanical, and chemical behavior.

Structure–Property Relationships Nanocharacterization helps correlate nanoscale structure with material performance, as small changes in size, morphology, and interfaces can significantly affect functional properties (Balaya, 2008). Understanding these relationships is essential for tailoring materials for specific applications.

2.2. Key Parameters in Nanocharacterization

Nanocharacterization techniques are employed to determine several critical parameters:

Particle Size and Size Distribution: Affects optical, thermal, and reactive properties

Morphology and Topography: Includes shape, porosity, and surface roughness

Surface Properties: Functional groups, surface charge, and interfacial behavior

Chemical Composition: Elemental makeup and bonding environment

Crystallinity and Phase Structure: Degree of order and phase identification

These parameters collectively define the performance of nanomaterials in various applications.

2.3. Chemical and Electronic Structure Analysis

Understanding the chemical environment at the nanoscale is essential for predicting material functionality. This includes identification of elemental composition, oxidation states, and bonding configurations. Electronic structure analysis further provides insight into conductivity, optical behavior, and catalytic efficiency (Gopalakrishnan & Ashokkumar, 2024). Such information

is particularly important for advanced materials such as nanocomposites, semiconductors, and functional polymers.

2.4. Spatial and Temporal Resolution

High spatial resolution is necessary to observe nanoscale features, while temporal resolution enables real-time study of dynamic processes through in situ and operando techniques (Chee et al., 2021). Together, they provide deeper insight into nanoscale transformations and mechanisms.

2.5. Challenges in Nanocharacterization

Despite advancements, several limitations remain:

Sample Preparation Issues: Risk of contamination or structural alteration

Beam-Induced Damage: Especially in electron-based techniques

Resolution Limitations: Vary across different methods

Environmental Sensitivity: Measurements affected by external conditions

These challenges can impact the accuracy and reliability of results.

2.6. Multimodal and Correlative Approaches

Because no single method provides complete information, multiple complementary techniques are often combined to achieve comprehensive nanoscale analysis. Correlative approaches improve accuracy by integrating structural, chemical, and surface information.

2.7. Data Interpretation and Reproducibility

Reliable nano-characterization requires careful data interpretation, standardized protocols, and cross-validation to ensure reproducibility and minimize experimental inconsistencies. Proper analytical practices are essential for generating trustworthy scientific results (Baer et al., 2013).

2.8. Electron Microscopy

Electron microscopy techniques are widely used in nanocharacterization because they provide very high-resolution images by using a beam of electrons. These methods allow detailed analysis

of surface features, internal structure, and even atomic arrangements, making them essential for studying nanomaterials.

2.9. Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) is primarily used to study the surface morphology and topography of materials. It works by scanning a focused electron beam across the sample and detecting emitted signals to form detailed images (Lenthe et al., 2018).

SEM is especially useful for analyzing particle shape, surface roughness, and fracture features, and it provides images with a strong three-dimensional appearance. The electron-beam scanning process and the main components involved in SEM analysis can be seen in Figure 2. When combined with Energy Dispersive X-ray Spectroscopy (EDS), it can also provide elemental composition, making it a versatile technique for both imaging and analysis (Scimeca et al., 2018), as shown in Figure 2.

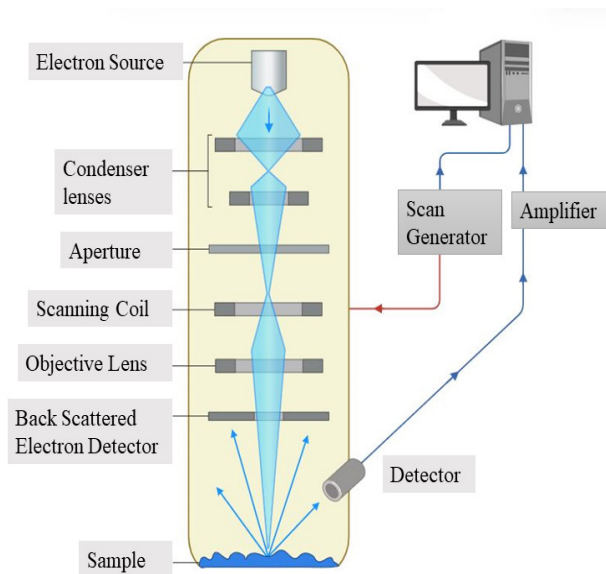


Figure 2. Schematic illustration of scanning electron microscopy

SEM uses a focused electron beam to scan a specimen and generate high-resolution surface images. Secondary electrons provide surface topography, while backscattered electrons reveal compositional contrast. Modern SEM offers nanometer-scale resolution, large depth of field, and can be coupled with EDS for elemental analysis and mapping.

2.10. Transmission Electron Microscopy (TEM)

Transmission Electron Microscopy (TEM) provides information about the internal structure of materials by passing electrons through a very thin sample. It allows direct observation of particle size, shape, and internal features such as grain boundaries and defects. **Figure 3** depicts how electrons pass through a thin specimen to obtain detailed information about its internal structure. TEM offers much higher resolution than SEM and is widely used for detailed structural analysis of nanomaterials. However, it requires careful sample preparation, as samples must be extremely thin (Lyu et al., 2023).

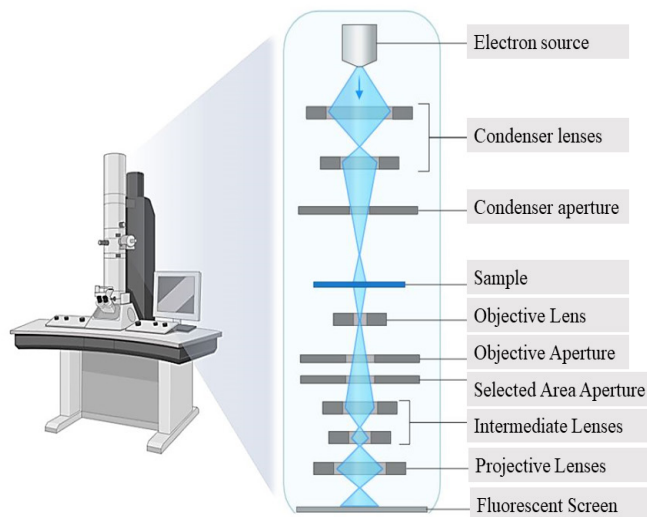


Figure 3. Schematic representation of transmission electron microscopy (TEM)

TEM passes a high-energy electron beam through an ultrathin sample to produce high-resolution images and diffraction patterns. It reveals internal structures such as lattice fringes, grain boundaries, and defects, while SAED provides crystallographic and phase information (Lucuța et al., 1985).

2.11. High-Resolution Transmission Electron Microscopy (HRTEM)

High-Resolution TEM (HRTEM) is an advanced form of TEM that enables imaging at the atomic scale (Wang, 2003). It allows researchers to observe lattice fringes and determine crystallographic structures directly. This makes HRTEM particularly valuable for studying

crystallinity, defects, and nanoscale interfaces in materials. Despite its advantages, it requires precise operating conditions and expertise.

2.12. Energy Dispersive X-ray Spectroscopy (EDS)

Energy Dispersive X-ray Spectroscopy (EDS) is often used alongside SEM or TEM to analyze the elemental composition of materials. It works by detecting X-rays emitted from the sample when it is bombarded with electrons. EDS provides qualitative and semi-quantitative information about the elements present and can also generate elemental maps, helping to understand the distribution of components within a material (Alsmadi & Fox, 2001).

3. Spectroscopic Techniques

Spectroscopy encompasses a variety of methods where electromagnetic radiation or particles interact with the sample, providing information on electronic states, chemical bonds, and composition (Sinha et al., 2023). **Figure 4** highlights the different wavelength regions of the electromagnetic spectrum relevant to nanocharacterization.

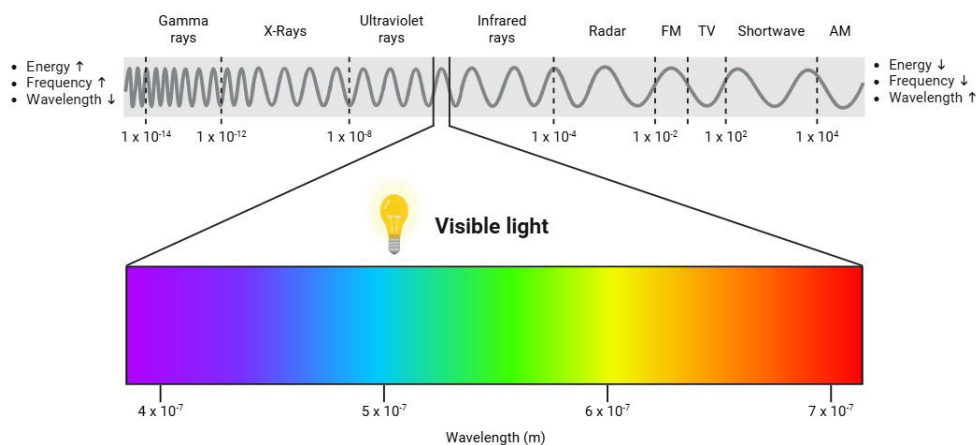


Figure 4. Electromagnetic spectrum showing wavelength regions commonly employed in nanocharacterization and spectroscopic techniques

Spectroscopic techniques are essential in nanocharacterization because they reveal the chemical composition, bonding, and electronic structure of materials. Instead of imaging the surface like microscopy, these methods study how materials interact with light or radiation, providing insight at the molecular and atomic levels. This is particularly important for nanomaterials, where small changes in structure can strongly affect performance.

3.1. Optical Spectroscopy

Optical techniques are widely used to study how nanomaterials absorb and emit light, giving information about their electronic properties.

UV–Visible Spectroscopy (UV–Vis) is commonly used due to its simplicity and speed, especially for metal and semiconductor nanoparticles (Begum et al., 2018). It measures light absorption and is especially useful for tracking nanoparticle formation, estimating band gaps, and studying size-dependent optical behavior. The main components of UV-VIS spectroscopy are presented in Figure 5.

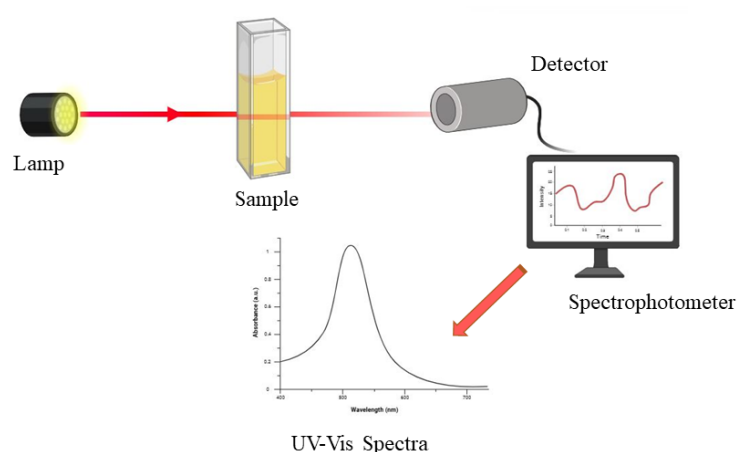


Figure 5. Schematic illustration of the components and workflow of a UV-Vis spectrophotometer system

For example, metal nanoparticles such as gold or silver show characteristic absorption peaks due to surface plasmon resonance (Lee & El-Sayed, 2006).

Similarly, semiconductor quantum dots exhibit size-tunable optical band gaps (Bailey & Nie, 2003); measuring the absorption edge can estimate the particle size distribution. However, it provides limited structural detail.

Photoluminescence Spectroscopy (PL) focuses on light emission after excitation. It is highly sensitive to defects and electronic states, making it valuable for studying semiconductors

and quantum dots (Kilina et al., 2016). While powerful, it only works well for optically active materials and can be affected by environmental conditions.

3.2. Vibrational Spectroscopy

Vibrational techniques help identify chemical bonds and functional groups, which is especially important for polymers and surface-modified nanomaterials.

Fourier Transform Infrared Spectroscopy (FTIR) identifies functional groups ($-\text{OH}$, $-\text{COOH}$, $-\text{CH}$, etc.) present in nanoparticles, surfaces, or ligands (Khan et al., 2018). It detects molecular vibrations and is widely used to confirm chemical structures, surface functionalization, and interactions in nanocomposites. It is simple and versatile but has limited spatial resolution. **Figure 6** provides a visual representation of the FTIR spectral regions associated with different types of chemical bonds.

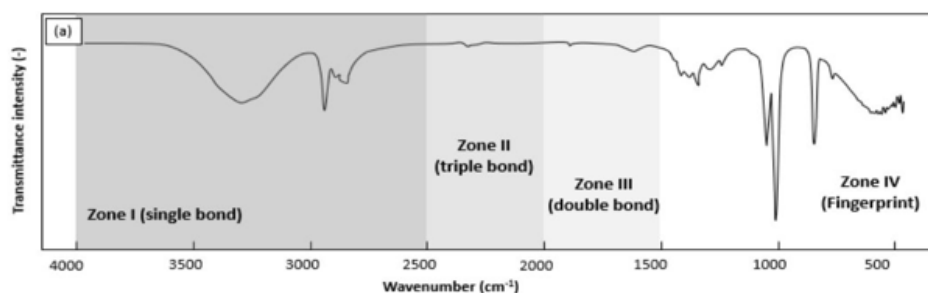


Figure 6. Overview of FTIR spectroscopy spectral regions demonstrating the classification of chemical bonds into four major zones based on wavelength

Raman Spectroscopy: It provides complementary information based on light scattering. It is particularly useful for studying carbon-based materials, crystallinity, and structural changes. Raman requires minimal preparation, but signals can be weak and sometimes affected by fluorescence (Wei et al., 2015).

4.3 Surface and Chemical Analysis: Surface-sensitive techniques are important because nanomaterial properties are often dominated by surface atoms.

X-ray Photoelectron Spectroscopy (XPS) : XPS is widely used to analyze surface composition and chemical states (Aziz & Ismail, 2017). It provides information about elements, oxidation states, and bonding within a few nanometers of the surface. Its main limitation is the need for vacuum conditions and shallow depth analysis.

X-ray Absorption Spectroscopy (XAS) : XAS offers deeper insight into local atomic structure and electronic states (Chen et al., 2024). It is especially useful in catalysis and advanced materials research, though it typically requires access to specialized facilities like synchrotrons.

Nuclear Magnetic Resonance (NMR) : NMR provides detailed information about molecular structure and dynamics. It is particularly valuable for polymers and organic nanomaterials. While highly informative and non-destructive, it generally requires larger sample amounts and has lower sensitivity compared to other techniques (Gerothanassis et al., 2002).

3.3. Combined Use of Techniques

No single spectroscopic method can fully describe a nanomaterial. In practice, multiple techniques are used together for example, FTIR and Raman for bonding analysis, or XPS and XAS for surface and electronic structure. This combined approach provides more reliable and complete information.

4. Structural Characterization Techniques

Structural characterization focuses on understanding how atoms and phases are arranged within a material. At the nanoscale, these structural features such as crystallinity, lattice structure, and phase composition have a strong impact on properties like strength, conductivity, and thermal behavior. Even small structural variations can significantly influence performance, making accurate analysis essential (Padmanabhan, 2001).

4.1. X-ray Diffraction (XRD)

X-ray Diffraction (XRD) is one of the most widely used techniques for studying the structure of nano materials. It works by analyzing how X-rays scatter from atomic planes, producing a pattern that reflects the material's crystal structure. The diffraction pattern presented in Figure 7 demonstrates the characteristic peaks used to assess the crystalline nature of the material.

XRD is commonly used to identify phases and determine the degree of crystallinity. In nanomaterials, diffraction peaks often appear broadened due to small crystallite size and lattice strain (Uzun, 2023). This broadening can be used to estimate crystallite size, making XRD especially useful at the nanoscale.

The technique is widely applied to nanoparticles, thin films, and nanocomposites, including polymer systems where it helps distinguish between crystalline and amorphous regions. However, it is less effective for fully amorphous materials or highly complex mixtures.

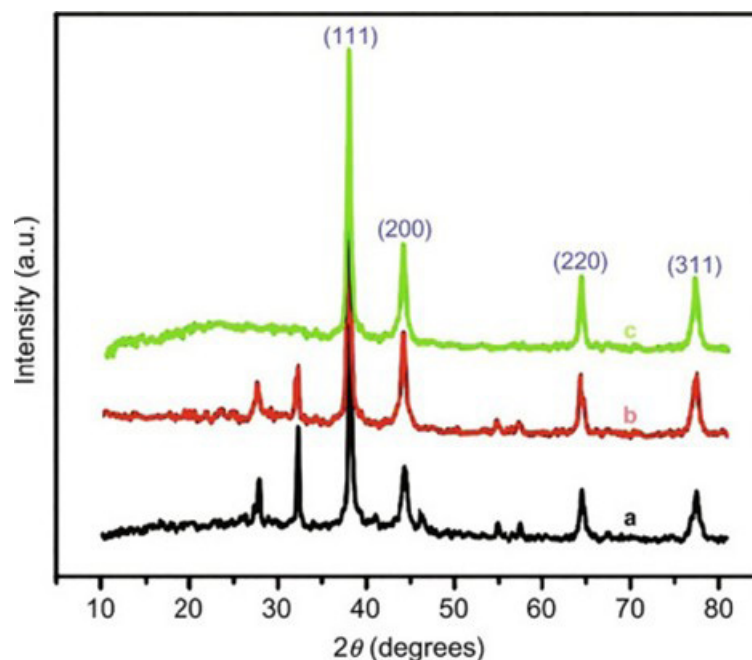


Figure 7. X-ray diffraction(XRD) patterns of samples a(black), b(red), and c(green). The sharp diffraction peaks at 2 values corresponding to the (111), (200), (220), and (311) planes confirm the face centered cubic(FCC) crystalline structure of the material (Raja et al., 2022)

4.2. Small-Angle X-ray Scattering (SAXS)

Small-Angle X-ray Scattering (SAXS) is used to study structures in the range of 1–100 nm, focusing on features such as particle size, shape, and distribution. Unlike XRD, which probes atomic structure, SAXS provides information about larger nanoscale organization.

It is particularly useful for analyzing nanoparticle dispersion and aggregation in systems like polymers, colloids, and gels. SAXS can be applied to samples in different states with minimal preparation, which adds to its versatility (Dyer et al., 2013).

However, SAXS does not produce direct images and relies on data modeling for interpretation, which can make analysis more complex.

4.3. Crystallinity and Phase Analysis

Crystallinity describes the level of atomic order in a material and plays a key role in determining mechanical and thermal properties. Phase analysis involves identifying the different structural forms present, which is especially important in multiphase systems such as nanocomposites (Chung & Cho, 2019).

At the nanoscale, phase behavior can change more easily due to high surface energy, making accurate identification important. Techniques like XRD are typically used for this purpose, often alongside other methods for better reliability.

5. Particle Size and Surface Area Analysis

Particle size and surface area are two of the most important parameters in nanomaterials, as they directly influence properties such as reactivity, stability, and interaction with the surrounding environment. Because nanomaterials have a very high surface-to-volume ratio, even small changes in size or surface area can significantly affect their performance (Asha & Narain, 2020). Techniques used in this area help quantify these features in a reliable and practical way.

5.1. Dynamic Light Scattering (DLS)

Dynamic Light Scattering (DLS) is a widely used technique for measuring the size of nanoparticles in suspension. It works by analyzing fluctuations in scattered light caused by the random motion of particles (Brownian motion) (Hassan et al., 2015). From this, the hydrodynamic diameter and size distribution can be determined.

DLS is popular because it is quick, easy to use, and requires minimal sample preparation. It is especially useful for studying colloidal systems, polymer nanoparticles, and dispersions. However, the technique provides an average size rather than detailed shape information, and results can be affected by particle aggregation or the presence of larger particles in the sample (Falke & Betzel, 2019), as shown in **Figure 8**.

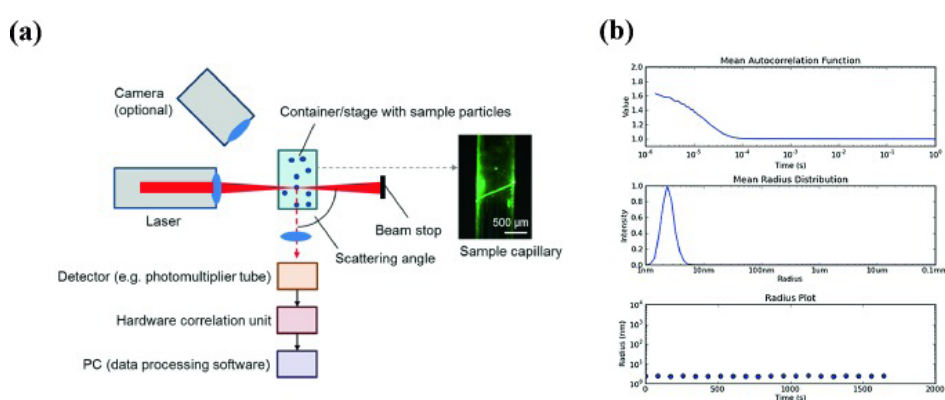


Figure 8. (a) Dynamic Light Scattering setup (DLS) (b)corresponding particle size analysis plots

5.2. Surface Area Analysis (BET Method)

Surface area is another key parameter, particularly in applications such as catalysis, adsorption, and energy storage. The Brunauer-Emmett-Teller (BET) method is commonly used to measure

the specific surface area of materials by analyzing gas adsorption behavior (Irwansyah et al., 2024). In this technique, an inert gas (usually nitrogen) is adsorbed onto the surface of a material, and the amount of gas adsorbed is used to calculate the surface area. Materials with smaller particle sizes or porous structures typically show higher surface areas. BET analysis is widely applied to nanoporous materials, catalysts, and nanocomposites. However, it requires careful sample preparation, and the results depend on assumptions in the adsorption model, which may not always perfectly represent real systems.

6. Thermal and Mechanical Characterization

Thermal and mechanical properties are critical for understanding how nanomaterials behave under different conditions, especially in real-world applications (Radhakrishnan et al., 2024). These properties influence stability, durability, and performance in areas such as coatings, polymers, and energy systems. At the nanoscale, these behaviors can differ significantly from bulk materials due to size effects and strong interfacial interactions.

6.1. Thermo gravimetric Analysis (TGA)

Thermo gravimetric Analysis (TGA) is used to study how a material's weight changes with temperature. As the sample is heated, processes such as decomposition, oxidation, or loss of volatile components can be observed.

TGA is particularly useful for evaluating thermal stability, composition, and degradation behavior. In nanomaterials and polymer-based systems, it helps determine how additives, fillers, or surface modifications affect stability (Galko & Sajdak, 2022). While it provides clear information about weight changes, it does not directly identify the chemical nature of the processes occurring.

6.2. Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetry (DSC) measures heat flow associated with thermal transitions in a material, such as melting, crystallization, and glass transition. This technique is widely used to study phase transitions and thermal behavior in polymers and nanocomposites (Corcione & Frigione, 2012). It provides valuable insight into crystallinity and molecular mobility, which are important for understanding mechanical performance. However, interpretation of overlapping thermal events can sometimes be challenging.

6.3. Nano indentation

Nano indentation refers to a testing method whereby mechanical properties like hardness and elastic modulus of material are determined at very small scales (Volinsky & Gerberich, 2003). It works by pressing a sharp tip into the material's surface and recording the response.

The technique is highly suitable for studying thin films, coatings, and nanomaterials, which cannot otherwise be analyzed by conventional testing methods. The ability to measure mechanical properties locally is significant for heterogeneous materials. Nonetheless, surface topography may affect the outcomes of the test.

7. Advanced and Emerging Techniques

In view of increasingly complicated structures of nanomaterials, innovative techniques have been created that make it possible to analyze such materials more efficiently and under realistic conditions (Adams et al., 2005).

7.1. Cryo-Electron Microscopy (Cryo-EM)

Cryo-electron microscopy involves the rapid freezing of the samples followed by imaging under extremely low temperature conditions. This facilitates the preservation of the native structure of the samples (Weissenberger et al., 2021). Cryo-EM is extensively applied to biological and hydrated samples.

7.2. In Situ and Operando Techniques

In situ and operando techniques make it possible to study materials in the process of their work, for example, when being heated or during any chemical processes (Roldán Cuenya & Bañares, 2024). Thus, it will be easier to monitor the ongoing changes. Such experiments are especially useful for catalysis and energy materials. However, such setups are usually complicated.

7.3. Synchrotron-Based Techniques

Synchrotron-based methods employ high-energy X-rays that make it possible to obtain very detailed data regarding the structure and electronic properties of a material (Baruchel et al., 2008). They are rather specific, but highly sensitive and useful for advanced investigations.

7.4. Correlative Approaches

The correlative or multimodal approach uses multiple tools to analyze the same sample simultaneously. This is beneficial because it enables integration of structure, chemistry, and functionality of the materials under investigation.

8. Applications of nanocharacterization

There are various applications of nanocharacterization techniques in different scientific areas where understanding and designing new materials on a nanoscale is important (Kumar et al., 2019). By providing detailed information about structure, composition, and properties, these techniques help link material design to performance in practical applications.

8.1. Nanomaterials and nanocomposites

In nanomaterials research, characterization techniques are essential for analyzing particle size, morphology, dispersion, and interfacial interactions. For nanocomposites, especially polymer-based systems, they help evaluate how well nanoparticles are distributed within the matrix and how this affects mechanical, thermal, and functional properties (Hiremath et al., 2021). The broad range of scientific and technological applications of nano characterization is illustrated in Figure 9.

8.2. Biomedical Applications

In biomedical fields, nanocharacterization is used to study drug delivery systems, nanoparticles, and biomaterials (Bhatia, 2016). Techniques help determine particle size, surface chemistry, and biocompatibility, which are critical for ensuring safe and effective performance in medical applications.

8.3. Energy Storage and Conversion

Nanocharacterization plays an important role in energy-related applications such as batteries, supercapacitors, and solar cells. It helps analyze electrode materials, surface reactions, and structural stability, which are key factors in improving efficiency and lifespan (Saleh & Hassan, 2023).

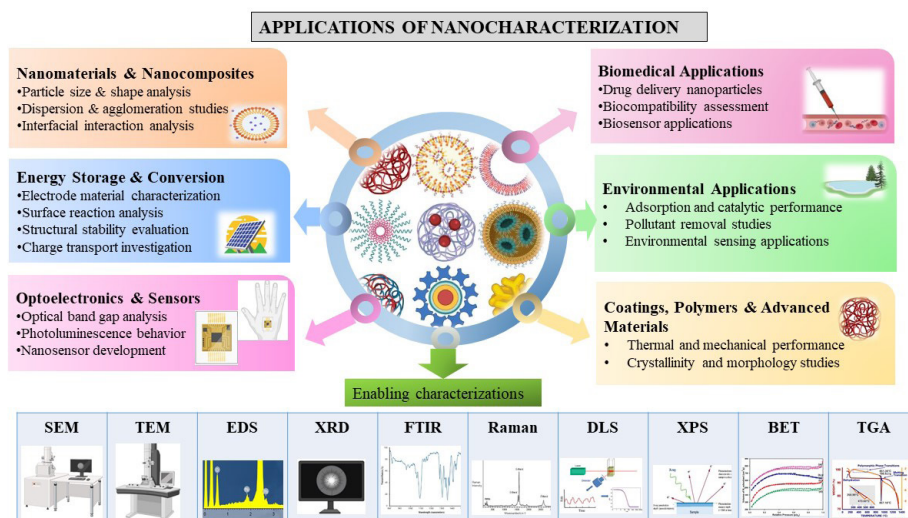


Figure 9. Schematic illustration of applications of nanocharacterization

8.4. Environmental Applications

In environmental science, these techniques are used to study nanomaterials for water purification, pollutant removal, and sensing applications. Characterization helps understand adsorption behavior, surface interactions, and long-term stability.

9. Challenges and Limitations

Nanocharacterization, while powerful, comes with several challenges. Sample preparation is often complex and can sometimes alter the material, especially when coatings or thinning are required (Nadkarni et al., 2024). In addition, high-energy probes like electron beams may damage sensitive nanostructures during analysis. Another key limitation is that no single technique provides complete information. Multiple methods are usually needed to fully understand a material, which increases both complexity and cost. Data interpretation can also be difficult, as nanoscale signals are often weak and influenced by experimental conditions (Yue & Wang, 2012). This can affect reproducibility and consistency across different studies. Moreover, access to advanced techniques may be limited due to expensive instrumentation and infrastructure requirements.

10. Conclusion and future perspectives

Nanocharacterization is continuously evolving as materials become more complex and application demands increase. Future developments are likely to focus on **higher resolution, faster analysis, and real-time monitoring** of materials under working conditions. Techniques such as in situ characterization and correlative approaches are expected to become more common, allowing researchers to study materials more realistically and in greater detail (Robertson et al., 2011).

Another important direction in the development of characterization techniques is the inclusion of data analytics methods and artificial intelligence and machine learning (Strielkowski et al., 2023). This approach will assist in identifying patterns, saving time, and increasing accuracy in the process, particularly when dealing with complicated nanosystems.

At the same time, efforts are being made to simplify the use of modern methods by reducing the costs associated with the equipment used. In conclusion, characterization methods for nanomaterials are crucial when it comes to studying the unique properties of nanoparticles. These methods can be applied not only during research but in various practical areas as well. While challenges remain, continued advancements in techniques and data analysis are expected to further strengthen the field and support future innovations in nanotechnology.

References

- Adams, F., Van Vaeck, L., & Barrett, R. (2005). Advanced analytical techniques: Platform for nano materials science. *Spectrochimica Acta Part B: Atomic Spectroscopy*, 60(1), 13–26. <https://doi.org/10.1016/j.sab.2004.10.003>
- Alsmadi, B. M., & Fox, P. (2001). Semi-quantitative analysis of changes in soil coatings by scanning electron microscope and energy dispersive X-ray mapping. *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, 194(1–3), 249–261. [https://doi.org/10.1016/S0927-7757\(01\)00814-7](https://doi.org/10.1016/S0927-7757(01)00814-7)
- Asha, A. B., & Narain, R. (2020). Nanomaterials properties. In R. Narain (Ed.), *Polymer science and nanotechnology: Fundamentals and applications* (pp. 343–359). Elsevier. <https://doi.org/10.1016/B978-0-12-816806-6.00015-7>
- Aziz, M., & Ismail, A. F. (2017). X-ray photoelectron spectroscopy (XPS). In N. Hilal, A. F. Ismail, T. Matsuura, & D. Oatley-Radcliffe (Eds.), *Membrane characterization* (pp. 81–93). Elsevier. <https://doi.org/10.1016/B978-0-444-63776-5.00005-X>
- Baer, D. R., Engelhard, M. H., Johnson, G. E., Laskin, J., Lai, J., Mueller, K., Munusamy, P., Thevuthasan, S., Wang, H., Washton, N., Elder, A., Baisch, B. L., Karakoti, A., Kuchibhatla, S. V. N. T., & Moon, D. (2013). Surface characterization of nanomaterials and nanoparticles: Important needs and challenging opportunities. *Journal of Vacuum Science & Technology A*, 31(5), Article 050820. <https://doi.org/10.1116/1.4818423>
- Bailey, R. E., & Nie, S. (2003). Alloyed semiconductor quantum dots: Tuning the optical properties without changing the particle size. *Journal of the American Chemical Society*, 125(23), 7100–7106. <https://doi.org/10.1021/ja035000o>
- Balaya, P. (2008). Size effects and nanostructured materials for energy applications. *Energy & Environmental Science*, 1(6), 645–654. <https://doi.org/10.1039/B809078P>
- Baruchel, J., Bleuet, P., Bravin, A., Coan, P., Lima, E., Madsen, A., Ludwig, W., Pernot, P., & Susini, J. (2008). Advances in synchrotron hard X-ray based imaging. *Comptes Rendus Physique*, 9(5–6), 624–641. <https://doi.org/10.1016/j.crhy.2007.08.003>
- Begum, R., Farooqi, Z. H., Naseem, K., Ali, F., Batool, M., Xiao, J., & Irfan, A. (2018). Applications of UV/Vis spectroscopy in characterization and catalytic activity of noble metal nanoparticles fabricated in responsive polymer microgels: A review. *Critical Reviews in Analytical Chemistry*, 48(6), 503–516. <https://doi.org/10.1080/10408347.2018.1451299>
- Bhatia, S. (2016). Nanoparticles types, classification, characterization, fabrication methods and drug delivery applications. In *Natural polymer drug delivery systems: Nanoparticles, plants, and algae* (pp. 33–93). Springer. https://doi.org/10.1007/978-3-319-41129-3_2
- Chee, S. W., Lunkenbein, T., Schlögl, R., & Roldan Cuenya, B. (2021). In situ and operando electron microscopy in heterogeneous catalysis—Insights into multi-scale chemical

- dynamics. *Journal of Physics: Condensed Matter*, 33(15), Article 153001. <https://doi.org/10.1088/1361-648X/abddfd>
- Chen, Z., Walsh, A. G., & Zhang, P. (2024). Structural analysis of single-atom catalysts by X-ray absorption spectroscopy. *Accounts of Chemical Research*, 57(4), 521–532. <https://doi.org/10.1021/acs.accounts.3c00693>
- Chung, I., & Cho, M. (2019). Recent studies on the multiscale analysis of polymer nanocomposites. *Multiscale Science and Engineering*, 1(3), 167–195. <https://doi.org/10.1007/s42493-019-00022-4>
- Corcione, C. E., & Frigione, M. (2012). Characterization of nanocomposites by thermal analysis. *Materials*, 5(12), 2960–2980. <https://doi.org/10.3390/ma5122960>
- Dyer, K. N., Hammel, M., Rambo, R. P., Tsutakawa, S. E., Rodic, I., Classen, S., Tainer, J. A., & Hura, G. L. (2014). High-throughput SAXS for the characterization of biomolecules in solution: A practical approach. In *Structural genomics: General applications* (pp. 245–258). Humana Press. https://doi.org/10.1007/978-1-62703-691-7_18
- Falke, S., & Betzel, C. (2019). Dynamic light scattering (DLS): Principles, perspectives, applications to biological samples. In A. S. Pereira, P. Tavares, & P. Limão-Vieira (Eds.), *Radiation in bioanalysis: Spectroscopic techniques and theoretical methods* (pp. 173–193). Springer. https://doi.org/10.1007/978-3-030-28247-9_6
- Galko, G., & Sajdak, M. (2022). Trends for the thermal degradation of polymeric materials: Analysis of available techniques, issues, and opportunities. *Applied Sciences*, 12(18), Article 9138. <https://doi.org/10.3390/app12189138>
- Gerothanassis, I. P., Troganis, A., Exarchou, V., & Barbarossou, K. (2002). Nuclear magnetic resonance (NMR) spectroscopy: Basic principles and phenomena, and their applications to chemistry, biology and medicine. *Chemistry Education Research and Practice*, 3(2), 229–252. <https://doi.org/10.1039/B2RP90018A>
- Gopalakrishnan, R., & Ashokkumar, M. (2024). Comparative assessment of transition metals doping effects on structural, optical, optical conductivity, and photocatalytic features of ZnO nanoparticles. *Journal of Materials Science: Materials in Electronics*, 35, Article 1614. <https://doi.org/10.1007/s10854-024-13395-6>
- Hassan, P. A., Rana, S., & Verma, G. (2015). Making sense of Brownian motion: Colloid characterization by dynamic light scattering. *Langmuir*, 31(1), 3–12. <https://doi.org/10.1021/la501789z>
- Hiremath, A., Murthy, A. A., Thipperudrappa, S., & Bharath, K. N. (2021). Nanoparticles filled polymer nanocomposites: A technological review. *Cogent Engineering*, 8(1), Article 1991229. <https://doi.org/10.1080/23311916.2021.1991229>
- Irwansyah, F. S., Amal, A. I., Diyanthi, E. W., Hadisantoso, E. P., Noviyanti, A. R., Eddy, D. R., & Risdiana, R. (2024). How to read and determine the specific surface area of inorganic materials using the Brunauer–Emmett–Teller (BET) method. *ASEAN Journal of Science and Engineering*, 4(1), 61–70. <https://doi.org/10.17509/ajse.v4i1.60748>

- Khan, S. A., Khan, S. B., Khan, L. U., Farooq, A., Akhtar, K., & Asiri, A. M. (2018). Fourier transform infrared spectroscopy: Fundamentals and application in functional groups and nanomaterials characterization. In S. K. Sharma (Ed.), *Handbook of materials characterization* (pp. 317–344). Springer. https://doi.org/10.1007/978-3-319-92955-2_9
- Kilina, S. V., Tamukong, P. K., & Kilin, D. S. (2016). Surface chemistry of semiconducting quantum dots: Theoretical perspectives. *Accounts of Chemical Research*, 49(10), 2127–2135. <https://doi.org/10.1021/acs.accounts.6b00196>
- Kong, N., & Ariga, K. (2025). Confined nanoarchitectonics for nano-reactors: In situ characterization and tracking systems at the nanoscale. *Nanoscale Horizons*, 10(9), 1882–1904. <https://doi.org/10.1039/D5NH00252D>
- Kumar, P. S., Pavithra, K. G., & Naushad, M. (2019). Characterization techniques for nanomaterials. In S. Thomas, E. H. M. Sakho, N. Kalarikkal, S. O. Oluwafemi, & J. Wu (Eds.), *Nanomaterials for solar cell applications* (pp. 97–124). Elsevier. <https://doi.org/10.1016/B978-0-12-813337-8.00004-7>
- Lee, K.-S., & El-Sayed, M. A. (2006). Gold and silver nanoparticles in sensing and imaging: Sensitivity of plasmon response to size, shape, and metal composition. *The Journal of Physical Chemistry B*, 110(39), 19220–19225. <https://doi.org/10.1021/jp062536y>
- Lenthe, W. C., Stinville, J.-C., Echlin, M. P., Chen, Z., Daly, S., & Pollock, T. M. (2018). Advanced detector signal acquisition and electron beam scanning for high resolution SEM imaging. *Ultramicroscopy*, 195, 93–100. <https://doi.org/10.1016/j.ultramic.2018.08.025>
- Lucușă, P. G., Teodorescu, V., & Vasiliu, F. (1985). SEM, SAED, and TEM investigations of domain structure in PZT ceramics at morphotropic phase boundary. *Applied Physics A*, 37(4), 237–242. <https://doi.org/10.1007/BF00614823>
- Lyu, Z., Yao, L., Chen, W., Kalutantirige, F. C., & Chen, Q. (2023). Electron microscopy studies of soft nanomaterials. *Chemical Reviews*, 123(7), 4051–4145. <https://doi.org/10.1021/acs.chemrev.2c00461>
- Madkour, L. H. (2019). Introduction to nanotechnology (NT) and nanomaterials (NMs). In *Nanoelectronic materials: Fundamentals and applications* (pp. 1–47). Springer. https://doi.org/10.1007/978-3-030-21621-4_1
- Nadkarni, A., Rana, D., Desai, N., Benival, D., Joshi, V., Salave, S., & Khunt, D. (2024). Advanced characterization and sample preparation strategies for nanoformulations. *Journal of Nanotheranostics*, 5(3), 104–127. <https://doi.org/10.3390/jnt5030008>
- Nandiyanto, A. B. D., Ragadhita, R., & Fiandini, M. (2023). Interpretation of Fourier transform infrared spectra (FTIR): A practical approach in the polymer/plastic thermal decomposition. *Indonesian Journal of Science and Technology*, 8(1), 113–126. <https://doi.org/10.17509/ijost.v8i1.53297>
- Padmanabhan, K. A. (2001). Mechanical properties of nanostructured materials. *Materials Science and Engineering: A*, 304–306, 200–205. [https://doi.org/10.1016/S0921-5093\(00\)01437-4](https://doi.org/10.1016/S0921-5093(00)01437-4)
- Radhakrishnan, S., Das, P. P., Alam, A., Dwivedi, S. P., & Chaudhary, V. (2024). Mechanical, thermal, and electrical properties of 2D nanomaterials for advanced applications. *Proceedings*

- of the Institution of Mechanical Engineers, Part C: Journal of Mechanical Engineering Science, 238(17), 8739–8755. <https://doi.org/10.1177/09544062241245018>
- Raja, P. B., Munusamy, K. R., Perumal, V., & Ibrahim, M. N. M. (2022). Characterization of nanomaterial used in nanobioremediation. In H. M. N. Iqbal, M. Bilal, & T. A. Nguyen (Eds.), *Nano-bioremediation: Fundamentals and applications* (pp. 57–83). Elsevier. <https://doi.org/10.1016/B978-0-12-823962-9.00037-4>
- Robertson, I. M., Schuh, C. A., Vetrano, J. S., Browning, N. D., Field, D. P., Jensen, D. J., Miller, M. K., Baker, I., Dunand, D. C., & Dunin-Borkowski, R. E. (2011). Towards an integrated materials characterization toolbox. *Journal of Materials Research*, 26(11), 1341–1383. <https://doi.org/10.1557/jmr.2011.41>
- Roldán Cuenya, B., & Bañares, M. A. (2024). Introduction: Operando and in situ studies in catalysis and electrocatalysis. *Chemical Reviews*, 124(13), 8011–8013. <https://doi.org/10.1021/acs.chemrev.4c00184>
- Saleh, H. M., & Hassan, A. I. (2023). Synthesis and characterization of nanomaterials for application in cost-effective electrochemical devices. *Sustainability*, 15(14), Article 10891. <https://doi.org/10.3390/su151410891>
- Scimeca, M., Bischetti, S., Lamsira, H. K., Bonfiglio, R., & Bonanno, E. (2018). Energy dispersive X-ray (EDX) microanalysis: A powerful tool in biomedical research and diagnosis. *European Journal of Histochemistry*, 62(1), Article 2841. <https://doi.org/10.4081/ejh.2018.2841>
- Sinha, S., Jeyaseelan, C., Singh, G., Munjal, T., & Paul, D. (2023). Spectroscopy—Principle, types, and applications. In A. K. Bhatt, R. K. Bhatia, & T. C. Bhalla (Eds.), *Basic biotechniques for bioprocess and bioentrepreneurship* (pp. 145–164). Academic Press. <https://doi.org/10.1016/B978-0-12-816109-8.00008-8>
- Strielkowski, W., Vlasov, A., Selivanov, K., Muraviev, K., & Shakhnov, V. (2023). Prospects and challenges of the machine learning and data-driven methods for the predictive analysis of power systems: A review. *Energies*, 16(10), Article 4025. <https://doi.org/10.3390/en16104025>
- Uzun, İ. (2023). Methods of determining the degree of crystallinity of polymers with X-ray diffraction: A review. *Journal of Polymer Research*, 30(10), Article 394. <https://doi.org/10.1007/s10965-023-03744-0>
- Volinsky, A. A., & Gerberich, W. W. (2003). Nanoindentation techniques for assessing mechanical reliability at the nanoscale. *Microelectronic Engineering*, 69(2–4), 519–527. [https://doi.org/10.1016/S0167-9317\(03\)00341-1](https://doi.org/10.1016/S0167-9317(03)00341-1)
- Wang, Z. L. (2003). New developments in transmission electron microscopy for nanotechnology. *Advanced Materials*, 15(18), 1497–1514. <https://doi.org/10.1002/adma.200300384>
- Wei, D., Chen, S., & Liu, Q. (2015). Review of fluorescence suppression techniques in Raman spectroscopy. *Applied Spectroscopy Reviews*, 50(5), 387–406. <https://doi.org/10.1080/05704928.2014.999936>

- Weissenberger, G., Henderikx, R. J. M., & Peters, P. J. (2021). Understanding the invisible hands of sample preparation for cryo-EM. *Nature Methods*, 18(5), 463–471. <https://doi.org/10.1038/s41592-021-01130-6>
- Yue, Y., & Wang, X. (2012). Nanoscale thermal probing. *Nano Reviews*, 3(1), Article 11586. <https://doi.org/10.3402/nano.v3i0.11586>

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