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(Review Article)

Advanced characterization techniques for organic and inorganic materials: Emerging trends, innovations, and multidisciplinary applications

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Abstract

Advanced material characterization is credited with unraveling the complex structures and properties of organic and inorganic materials, thereby supporting innovations in energy, electronics, catalysis, and the environment. This review comprehensively analyzes modern characterization techniques, classifying them into spectroscopic, microscopic, chromatographic, thermal, optical, and specialized advanced methods. Specific emphasis is given to how the techniques respond to the unique demands of organic and inorganic systems. The review also discusses emerging trends such as in-situ and operando analysis, multimodal approaches, high-throughput procedures, and the integration of machine learning for data interpretation. Applications in organic electronics, nanomaterials, catalysis, energy storage, environmental monitoring, and quantum technologies are discussed. Challenges and future outlook are critically discussed to guide the creation of more efficient and automated platforms for next-generation materials research.

Keywords: Advanced Characterization; Organic Materials; Inorganic Materials; In-Situ Techniques; Multimodal Analysis; Machine Learning.

1. Introduction

The subject of materials science has seen a remarkable transformation due to the development of characterization techniques [1]. These techniques have progressed from simple mechanical testing to highly complex approaches that investigate materials on an atomic and molecular level. In materials research, characterization methods are pivotal in elucidating organic and inorganic materials' structure, content, and characteristics [2]. These methods are essential for research and real-world applications because they show how organic and inorganic materials behave under various situations. Thermal analysis, microscopy, spectroscopy, and other characterization tools allow scientists to study organic and inorganic materials thermal, mechanical, chemical, and physical characteristics. The intrinsic structure of

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materials, including crystal phases, grain boundaries, and microstructural characteristics, maybe revealed using advanced techniques like SEM and XRD. As a result, organic and inorganic materials designers can better connect a material's structure and its mechanical and electrical characteristics [3-5].

Characterization is crucial to confirm organic and inorganic materials' chemical structure, composition, thermal, optical, electrical, magnetic, mechanical properties, etc [6]. The industrial sector relies on heat analysis, tensile strength measures, and hardness testing to verify that materials are up to par. Characterization methods aid in the quest for novel materials with targetable characteristics in research and development (R&D). Materials failure in applications can be better understood using characterization techniques. Fatigue, corrosion, or incorrect processing are reasons why materials degrade; techniques like thermal analysis, fractography, and energy-dispersive X-ray spectroscopy (EDX) help pinpoint these issues [7].

Therefore, this review aims to provide an updated, structured, and comparative insight into the advanced characterization methods tailored for organic and inorganic materials. It not only categorizes the tools but also contextualizes them within current technological demands, emerging innovations, and multidisciplinary applications.

Over the past decade, several reviews have addressed material characterization techniques. However, this review differentiates itself by integrating a comprehensive, side-by-side exploration of organic and inorganic materials, emerging technologies, and cross-disciplinary applications, while also addressing AI and automation integration, which many previous reviews overlook.

The review begins by differentiating organic and inorganic materials, followed by a critical assessment of traditional and modern characterization methods. It explores both established and emerging techniques, highlighting novel instrumentation and data-driven approaches. Finally, it presents current and potential applications across diverse scientific domains, discusses present challenges, and outlines future perspectives.

1.1. Fundamentals of Organic and Inorganic Materials and the Role of Characterization

Understanding the nature of materials—whether organic or inorganic is essential for the selection of appropriate characterization techniques. The structure, bonding, and properties of these materials define their performance in diverse applications, and precise characterization is central to optimizing material development.

1.1.1. Comparison of Organic and Inorganic Materials

Organic materials are primarily carbon-based compounds that include polymers, biomolecules, and organic semiconductors. These materials exhibit covalent bonding and often feature complex molecular architectures with varying degrees of flexibility and functionality. Common organic materials include polyethylene, polystyrene, polythiophene, and small-molecule dyes [8].Organic materials are typically lightweight, flexible, and chemically tunable, making them ideal for applications in soft electronics, packaging, drug delivery, and photovoltaics. For example, organic light-emitting diodes (OLEDs) and organic solar cells (OSCs) are key technologies relying on the unique optoelectronic properties of organic compounds [9].

Inorganic materials on the other hand are composed of metals, ceramics, oxides, and salts, generally characterized by ionic or metallic bonding [10]. These materials tend to exhibit high thermal and chemical stability, hardness, and electrical conductivity. Common inorganic materials include silicon, titanium dioxide, copper oxides, and various metal alloys. Their robust mechanical and thermal properties make inorganic materials indispensable in structural applications, catalysis, energy storage, and semiconductors. For instance, silicon is the backbone of the microelectronics industry, while metal oxides serve as catalysts in industrial chemical processes [11].

The key distinctions between organic and inorganic materials lie in their bonding types, structural flexibility, and functional applications. Organic materials offer design flexibility and biocompatibility, while inorganic materials provide thermal stability and durability. These differences necessitate tailored characterization strategies. For example, while UV–Vis and FTIR spectroscopy are highly effective for probing organic systems, X-ray diffraction (XRD) and electron microscopy are more suited to inorganic crystals [12].

1.1.2. Need for Advanced Characterization Approaches

Characterization is a critical component of materials research, offering insights into the structure–property relationships that govern performance [1]. It provides the data needed to validate material synthesis, understand degradation mechanisms, and inform processing decisions. Effective characterization allows researchers to correlate microscopic features with macroscopic behavior, enabling rational design and optimization of materials. Techniques like scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS), and thermal analysis provide detailed insights into morphology, composition, and stability. Such information is vital for quality control, failure analysis, and innovation in materials science [13].

Conventional techniques include optical microscopy, classical spectroscopy, and thermal gravimetric analysis. These methods have been foundational in material research, offering accessible tools to measure particle size, functional groups, thermal transitions, and optical properties. XRD has long served as the benchmark for crystallinity and phase identification in inorganic compounds. Despite their usefulness, traditional methods often lack the resolution, sensitivity, or specificity required for advanced materials. Optical microscopy, for instance, cannot resolve features below the diffraction limit (~200 nm), and bulk thermal techniques do not capture nanoscale heterogeneity. Furthermore, many conventional techniques are ex situ, failing to reveal dynamic behavior during real-world operation [14].

However, the emergence of complex materials systems such as nanostructured composites, hybrid interfaces, and quantum materials demands advanced techniques capable of high spatial, temporal, and spectral resolution. In situ and operando analyses, super-resolution microscopy, and AI-assisted data interpretation represent transformative innovations. These approaches not only overcome the limitations of classical techniques but also provide multidimensional data that drive materials-by-design initiatives [15].

2. Advanced Characterization Techniques for Organic and Inorganic Materials

Additional in-depth examination of the minute details of organic and inorganic materials, chemicals, or biological beings can be accomplished using advanced characterization techniques. These methods go beyond simple analysis by providing a more complex knowledge of characteristics and behaviors. For enhanced characterization, tools like GCMS, Auger electron spectroscopy, inductively coupled plasma, and electron spin resonance are used in organic and inorganic materials research. These techniques provide exact access to crystallography, elemental composition, and atomic and molecular structures of organic and inorganic materials for scientific investigation [16]. Table 1 is an overview of advanced characterization techniques for organic and inorganic materials

2.1. Spectroscopic Techniques

NMR, FTIR, Raman spectroscopy, and UV/Vis are spectroscopic techniques vital to material science because they reveal intricate information about materials' physical, chemical, and structural characteristics.

2.1.1. Nuclear Magnetic Resonance (NMR) Spectroscopy

Nuclear magnetic resonance (NMR) spectroscopy is very useful to utilize radio wave range (wavelengths). Wave range is one of the most quantitative analytical methods for determining the structural information of organic and inorganic materials. The magnetic field involves the interaction of radio waves with nuclei that contain odd quantities of neutrons or protons that show evidence of the intrinsic magnetic moment, which can be able to possess resonant condition or flipping condition of spin state. Moreover, the resonant condition mainly depends upon the nuclei, chemical bonds, and functional groups. In addition, organic and inorganic compounds in both forms such as solid and liquid can be determined and also used for the synthesis of novel compounds and determination of chemical structures of biomolecules and polymers [17].

NMR spectroscopy aims to study and understand the physical process by which atomic nuclei with nonzero spin absorb and re-emit electromagnetic radiation in reaction to a magnetic field. By monitoring the resonance frequencies of specific nuclei (such as ^1H and ^{13}C), NMR spectroscopy reveals essential information on molecules' dynamics, bonding, and structural makeup. This method can learn a lot about the molecular surroundings of different compounds and applies to a wide range of elements and isotopes [18]. Atomic nuclei with nonzero spin interacting with an externally applied magnetic field are the basis of the working principle of NMR spectroscopy (Figure 1). Nuclei composed of an uneven number of protons and neutrons can align with or against the magnetic field, resulting in different energy states due to their magnetic moment and angular momentum. The nuclei absorb the energy difference between these states and transition between them when radiofrequency radiation is supplied at a frequency corresponding to it. The nuclei release radiofrequency signals as they relax, and these signals are picked up and stored. Each nucleus's electrical environment affects its resonance frequency, revealing information about the molecule's chemical makeup, bonding, and atomic arrangement.

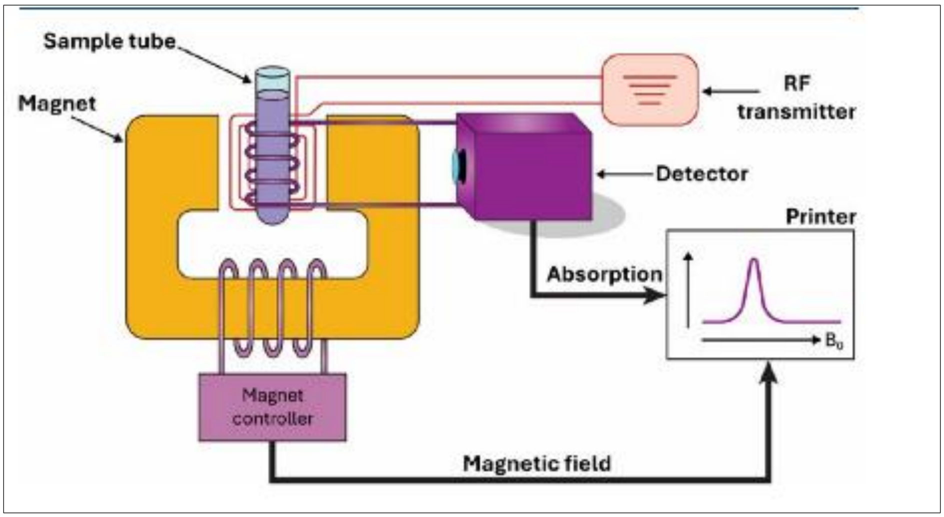

Figure 1 Instrumentation of NMR spectroscopy [19]

2.1.2. Infrared (IR) Spectroscopy

Infrared spectroscopy (IR) is a non-destructive technique; it provides electronics and structural information of organic and inorganic materials with high spatial resolution and high spectra (vibrational and rotational). IR spectroscopy directly involving in vibrational and rotational transitions ranges between 4000 and 400cm^{-1}; this range facilitates the permanent change of dipole moment. Subsequently, IR and Raman gave correlative molecular data, as regularly IR-inactive modes are Raman-active modes and VV [20].

With regard to inorganic materials characterization, Fourier transform infrared (FT-IR) spectroscopy is basically used to examine the surface modification such as “ligand attachment” of nanomaterials. Specifically, organic molecules involved in characteristic adsorption of IR spectra due to the “fingerprint region” range from 10 to 25mm. This technique provides bands of different chemical bonds present in ligands and complex molecules. This also provides a clue to the coordination mode of ligands and coordination bands at a near-low wave number (400–500 cm^{-1}). Henceforth, these types of organic nanomaterials are involved in the drug delivery system, e.g. nanoliposomes, and also used to the modification of nanoparticles with proteins [21]. This technique is also helpful in determining surface modifications in nanoparticles. Functional groups on the surface of zinc nanoparticles are identified by FT-IR spectroscopy [22]. Moreover, IR spectra involve in situ measurement for matrix molecules like water. Water is a highly strong IR absorber. In this strategy, these types of interfaces possess to minimize the usage of specific probing methods such as infrared attenuated total reflection infrared spectroscopy (IR-ATR) provided sur face sensitivity and a signal limited to the diffusion depth of the transitory field [23].

One of materials science’s most important analytical tools, FTIR spectroscopy, may reveal a material’s molecular makeup and structure. FTIR creates a distinct chemical fingerprint for each substance by measuring the absorption and transmission of infrared energy as the radiation passes through the sample. For qualitative investigation, FTIR works wonders, enabling exact material identification, as no two substances produce identical IR spectra. Furthermore, FTIR is useful for quantitative analysis since the magnitude of the peaks in the spectrum produced is proportional to the concentration of different components. This method improves over previous dispersive approaches; it uses state-of-the-art software algorithms to give rapid, accurate analysis over a broad frequency spectrum. Materials’ chemical makeup, structural characteristics, and molecular interactions may be better understood using FTIR, which has important scientific and practical implications [24].

Using an interferometer to detect all infrared frequencies concurrently, FTIR considerably speeds up the process compared to standard dispersive IR instruments, which scan each frequency separately. The interferometer collects infrared light from a source and reflects it from two mirrors, one of which is stationary and the other of which is mobile. When the two beams’ different route lengths cause an interference pattern, an interferogram containing all the encoded infrared frequencies is produced. On the other hand, this interferogram defies direct interpretation. The individual frequencies are decoded using a mathematical procedure called Fourier transformation (Figure 2) to get a spectrum out of an interferogram. Spectra like this show how a material absorbs infrared light at different frequencies related to the vibrations of its chemical bonds [25].

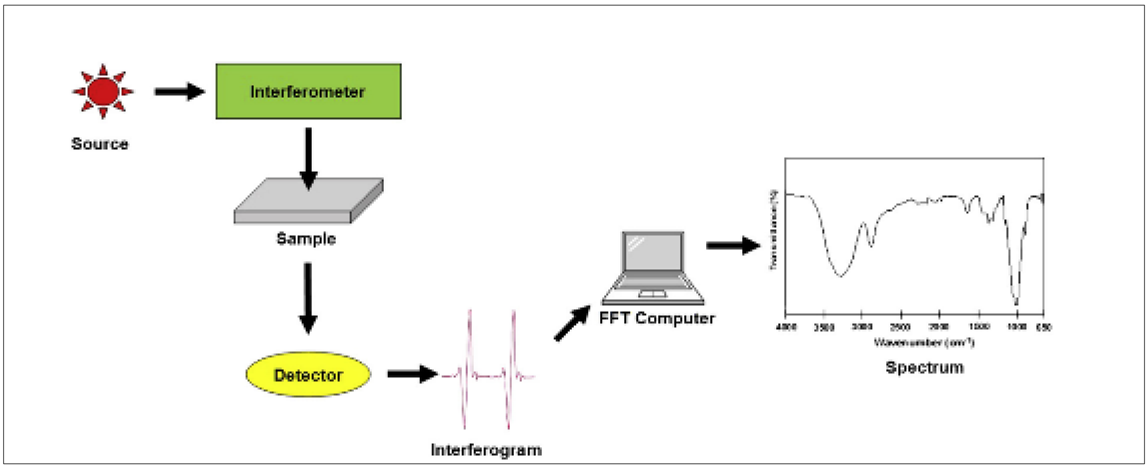

Figure 2 Instrumentation of FTIR spectroscopy [26]

2.1.3. Ultraviolet-visible (UV-Vis) Spectroscopy

Ultraviolet-visible (UV-Vis) spectroscopy involves the spectroscopy of photons in the UV-Vis region to study the optical properties. Absorption of energies in the range 104–105 cm^{-1} by a molecule leads to the change in the electronic energy of the molecule due to the transition of valence electrons from occupied molecular orbitals. UV-Vis spectrum is basically a graph of light absorbance versus wavelength in a range of UV or visible regions [27]. Such a spectrum can often be produced directly by using a UV-Vis spectrophotometer. It is based on Beer–Lambert law stating that the absorbance (A) of a solution is directly proportional to the concentration (c) of the absorbing species present in the solution of the path length (l). According to Beer–Lambert's law:

$$A = -log_{10}\left(\frac{I_t}{I_O}\right) = \epsilon cl$$

where I_0 and I_t are the intensity of the incident light and the transmitted light, respectively, and ε is a constant of proportionality, called the absorptivity. Therefore, following Beer–Lambert equation, concentration of the absorber present in the solution can be determined for a fixed path length. The perceived color of the absorbing species directly depends on the absorption wavelength in the visible range. UV spectrum is normally recorded with sample dissolved in solvents like water, ethanol, or hexane which are transparent within the wavelength range used in the study of organic compounds [28].

In organic materials, the absorption maximum of complexes can be determined using UV/Vis spectroscopy, and complete spectra provide information on electronic transitions in complexes, ligand field effects, and metal oxidation states. In the case of metal nanoparticles (MNPs), the UV–visible spectrum is useful in determining the concentration and size of nanoparticles. The optical properties and surface plasmon resonance (SPR) of copper nanoparticles are determined by UV–visible spectroscopy [29].

2.1.4. Raman Spectroscopy

Raman spectroscopy is a fast, nondestructive technique used to study vibrational, rotational, and other low-frequency modes in a system. Raman scattering is the inelastic scattering of a photon at the electron mist of the particle requiring an adjustment in polarizability, which brings about a Raman shift and vibrational/rotational excitation. It was discovered by Sir C.V. Raman and later by Grigory Landsberg and Leonid Mandelstam. It is an analytical tool that gives the structural information producing a precise spectral fingerprint, unique to a molecule or indeed and individual molecular structure. The spectrum is plotted as intensity vs Raman shift in wavenumbers (cm^{-1}), shifted from the absolute frequency (in cm^{-1}) of the excitation laser. This technique can provide structural, physical, and chemical information (crystalline phase, polymorphic forms, intrinsic stress, etc.) [30]. Integrally, Raman spectroscopy depends on scattering processes with the small fraction of the occurrence of photons prompting vibrational modes through inelastic processes able to make stoke lines (e.g. less energy of emitted photons compared with incident photons) and anti-stoke (e.g. high energy of emitted photons compared with incident photons). Furthermore, Raman spectra having only one primary photon range from 106 to 107, the remaining signal scales are an incident photon, and lasers are required to the light source [31].

Raman spectroscopy deduces information about a molecule's electronic environment, composition, and symmetry from its inelastic light scattering. Because of this, it is an excellent tool for quantitative and qualitative investigation, allowing scientists to learn about phase transitions, identify chemicals, and comprehend molecular interactions in different materials. It finds extensive application in the characterization of semiconductors, polymers, nanomaterials, and many more [32]. Figure 3 displays the instrumentation of Raman spectroscopy.

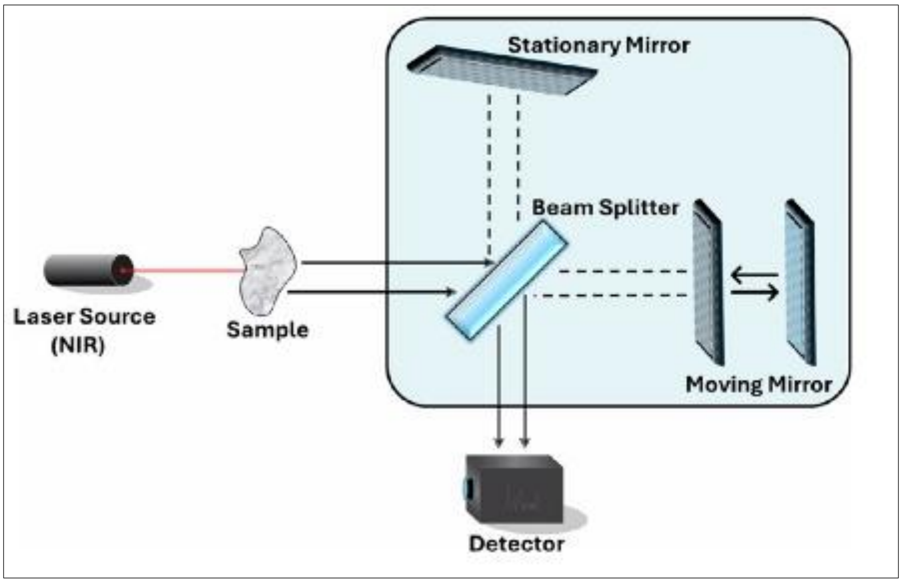

Figure 3 Instrumentation of Raman spectroscopy [19]

2.2. Microscopic Techniques

Materials science extensively uses optical microscopy for microscale material imaging and characterization. One way to learn about a material's makeup, structure, and characteristics is by examining its surface under a microscope. Features like surface morphology, defects, grain structure, and phase boundaries are frequently studied using this method. Optical microscopy's use for investigating micron and submicron scales in several fields has grown exponentially during the past decade [33]. Digital video enables optical microscopy to capture images of fragile optical sections, and confocal optical systems are already operational at most prominent research institutes. When an electron beam is used to illumine the specimen to generate a magnified image, it is known as electron microscopy (EM) [34]. Various types of EM techniques are discussed here

2.2.1. Scanning Electron Microscopy (SEM)

The scanning electron microscope (SEM) can acquire surface morphological, topographical, and compositional data of the constituent metal ions present in the materials [35]. SEM technique provides highly resolute three-dimensional images of conducting as well as non-conducting materials. The features of organic and inorganic materials which can be investigated through SEM are surface crack and other qualitative measures including contaminations [36].

The working principle of SEM is similar to the reflecting light microscope and is based on the reflection of the secondary electron from the surface of the material. The electrons coming out after the interaction of incident electron with the surface of the sample are known as backscattered electrons and maps the surface of the scanned sample by detecting the contrast area between different chemical compositions. The higher contrast region or brighter image in the backscattered electron images is observed due to the higher atomic number of atoms. The observed backscattered electron in the detector determines the relative intensity in the obtained scanned image. This also gives information about the quantitative part of the sample from the X-rays obtained by the interaction of electron beam with sample results from the electron transition from higher energy level to the lower energy levels [37].

SEM is especially constructive to visualize nanomaterials. Most of the conventional microscopy is used in glass lenses for magnifying image but EM is used in the electrons. SEM produces highly magnified, three-dimensional (3D) image. Also, this is impossible for light microscopy. When an electron ray with an increase of velocity of (3–30keV) is engaged in the sample, the actuated auxiliary and backscattered electrons are utilized for imaging. Because of inelastic scattering, secondary electrons (SEs) with low-level energy were generated. Also, SE produces signal to escape depth from the surface. Moreover, SEM is generally manufactured with advanced electron optics and field emission (FE). In this strategy, which make to avoid the chromatic and spherical aberration nature of nanomaterials in the resolution of the nanometer (nm). Utilizing low-voltage SEM improves the surface affectability, decreases harm of the imaged nanomaterials, and essentially decreases the depth level of electron penetration in spatial resolution ranging from 0.4 to 1.6nm. Furthermore, low-voltage high-resolution SEM is used for the determination of morphology, size, and structure of 2D nanomaterials such as metallic, semi-metallic, and inorganic compounds. Besides, non-conductive materials such as C or Pt are also applied (Figure 4) [38].

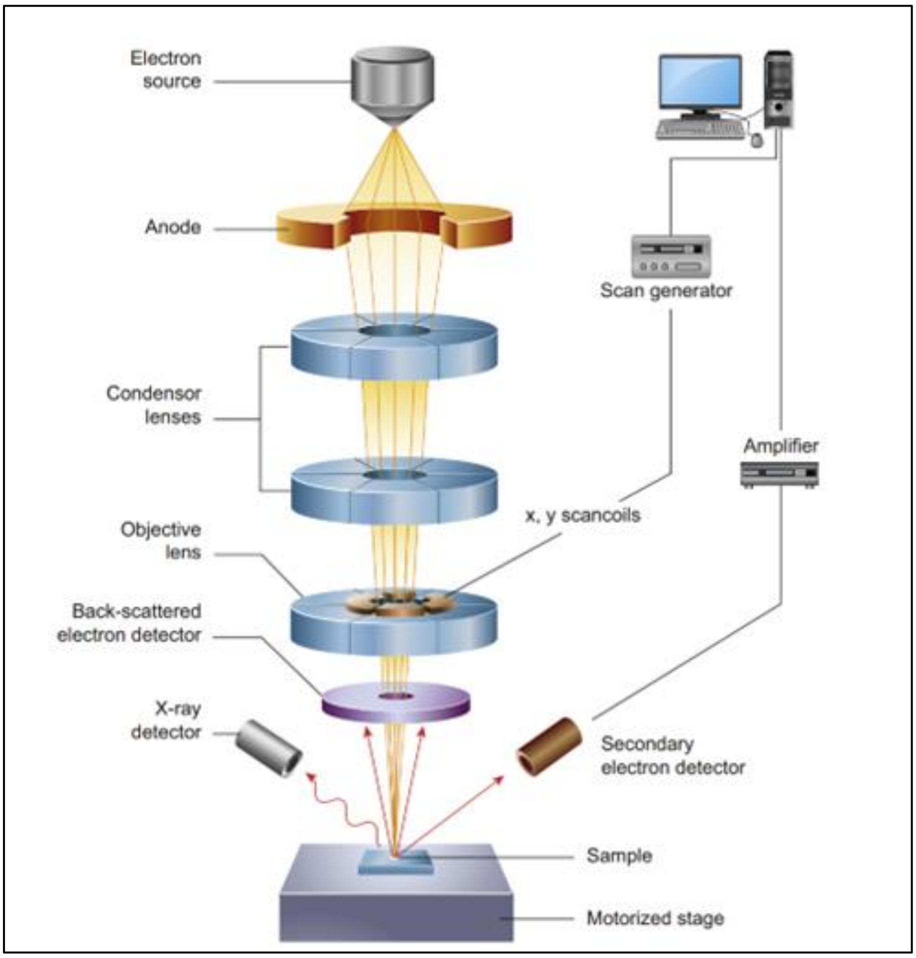

Figure 4 Schematic diagram of the core components of an SEM microscope [39]

2.2.2. Transmission Electron Microscopy (TEM)

TEM is one of the microscopy methods for the characterization of inorganic materials. TEM can be capable of providing direct high-resolution images and in-depth quantitative and quantitative chemical details for materials along with the spatial resolution downward to atomic dimensions range (<1nm) [40]. This technique is particularly important for nanoparticle characterization and provides information about morphological and structural aspect of materials science and inorganic chemistry. It assists in understanding the microstructural examination through high-resolution imaging of resolution 0.1–0.2 nm to identify ultrastructure. It provides results for understanding shapes, sizes, and uniformity in size/shapes of prepared materials. The use of suitable microscopic lenses combination along with replacing objective lens aperture by diffraction aperture, TEM can be utilized as an electron diffraction camera. In this, the electron is used for determining the orientation and shape of the crystal. It is also used to calculate the size of the crystallite by examining the pattern obtained from the TEM images [39].

High-resolution transmission electron microscopy (HR-TEM) mode is mainly involved in the determination of the crystallographic structure of the nanomaterials in “atomic level.” HR-TEM is an authoritative device to study the properties of materials on the atomic scale and the revelation of crystal structures. Also, it is used to analyze the nanoparticles crystals, arrangements, nanocrystalline texture in amorphous-based films, nanofibers, and their arrangement and absorbent materials [41, 42]. Figure 5 shows a schematic representation of TEM

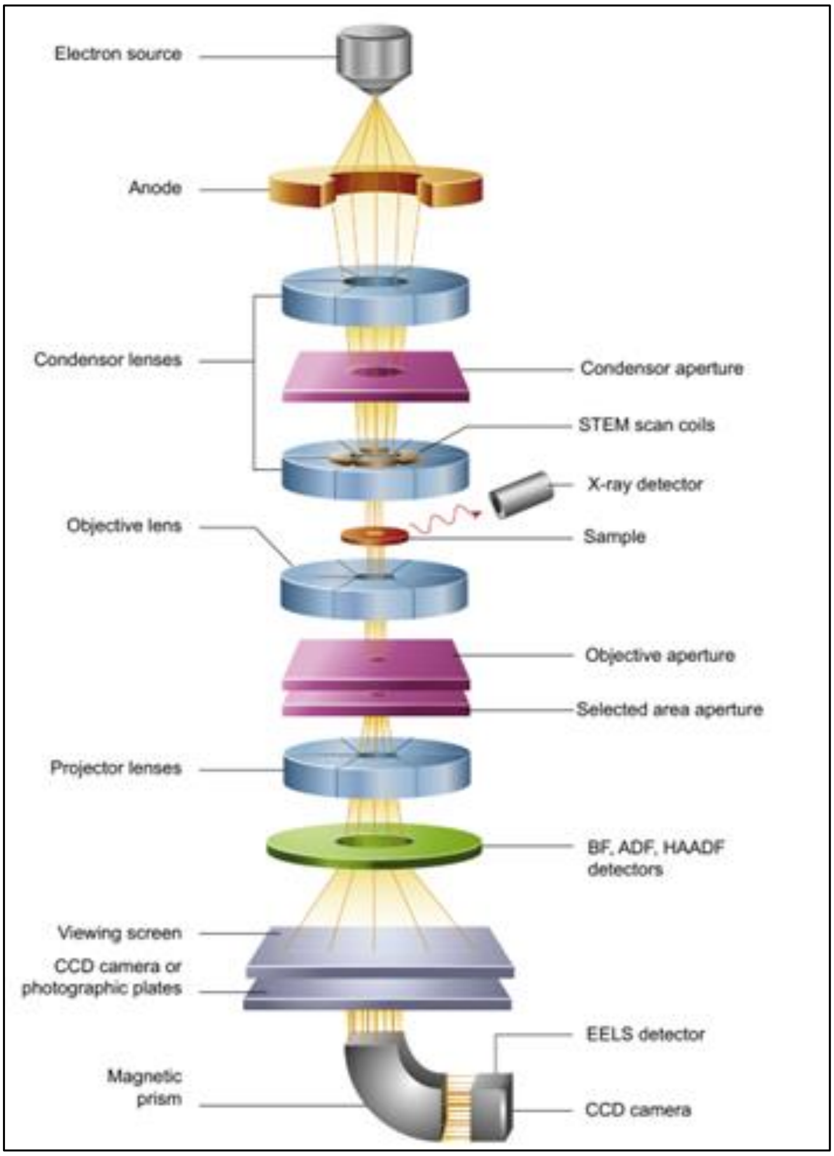

Figure 5 Schematic diagram of the Components of TEM technique [39]

2.2.3. Scanning Transmission Electron Microscopy (STEM)

STEM has proved very effective in measuring compositional changes at buried interfaces and the electronic structure and bonding relevant to a device's mechanical and transport properties [43]. Detection of single dopant atoms with STEM, both on free surfaces and buried inside devices, has proved helpful in studies ranging from characterizing catalysts to understanding the material limits for transistor scaling. It is also possible to detect and image the spatial distribution of single vacancies either directly, by their strain fields, or spectroscopically from their electronic fingerprints on the local densities of states [44].

Similar in principle to SEM, STEM is involved in the determination of sample by using high-energy electron beam, the electron beam range around 100–300KeV. Although the thin layer of the specimen was captured as an image, the remaining part of the scattered electron was collected by "transmission mode". The SEM is regularly operated as STEM, due to the presence of a transmission detector. As well as, the acceleration of voltage was in a limited range, around 30KeV [45].

The basic idea behind STEM characterization is to create a 2D map of a thin sample by directing a high-energy electron beam onto it and then scanning it over it. By detecting scattered electrons and signals from ionized atoms when the beam interacts with the sample, precise information on the sample's structure and composition may be derived. At the atomic level, information about bonds and chemicals may be derived from measurements of the energy lost by transmitted electrons due to core and valence excitations. The capacity to concentrate the electron beam into a tiny area while keeping enough beam current dictates the resolution and efficiency of STEM. Recent developments in correcting lens aberrations have greatly enhanced spatial resolution, paving the way for atomic-resolution imaging and sub-angstrom beam sizes.

TEM also has a good capability for using STEM mode, because of the existence of scanning coils. The resolution into STEM mode mainly depended on the electron probe in dimension. In addition, high electrons are collected by the "bright field detector." These electrons relation between low angle vs. axis. As well as transmitted electrons are collected by "dark field detector," these electrons relatively high angle vs. optical axis, while inner angle of dark field detector is set as the large value, for this purpose collecting only scattered electrons. It is called as "Z-Contrast"/high-angle angular dark field (HAADR) imaging because of the enhanced atomic number (Z), which primarily depends on the contrast of the image [46].

2.2.4. Atomic Force Microscopy (AFM)

AFM is an advanced technique for studying the properties of materials at an atomic scale. The method has significantly influenced material science, molecular biology, and solid-state physics. AFM was invented in 1986 by G. Binning, C. F. Quate, and Ch. Gerber [47]. AFM operates based on nanoscale surface interaction employing a sharp, probing tip connected to a spring-like cantilever. Attractive or repulsive forces develop between the tip and the sample as it gets closer. The cantilever is pushed away from the sample by repulsive forces and drawn toward it by attractive forces; the Pauli exclusion principal accounts for this behavior. A very sensitive position-sensitive detector records the changes in the direction of the laser beam as it reflects off the surface of the cantilever; even small deflections may be detected in this way. Scanner cantilever deflection is controlled thanks to a feedback loop that keeps a preset set point. A piezoelectric XYZ-scanner accurately controls the tip and sample's x, y, and z movements, enabling high-resolution 3D imaging of the sample's topography [48]. The probe depends on the flexible cantilever, which makes able to produce force by using "Hook's law" and can be expressed as:

$$F = K_X$$

Where F is force; K is constant of the cantilever (probe mounted) and x is the deflection of the cantilever

Generally, AFM uses various types of the probe, cantilever length, and weight due to the different types of materials. However, the force was prohibited by the "feedback mechanism." As well as, the short distance between the sample surface and probe was determined by "van der Waal's force". In addition, various modes of AFM operations are possible using AFM due to van der Waal's force. In addition, AFM has a good capability of generating force curves, which make it useful for the determination of mechanical and electrical properties of materials [49]. Figure 6 shows the schematic diagram and mechanism of AFM technique

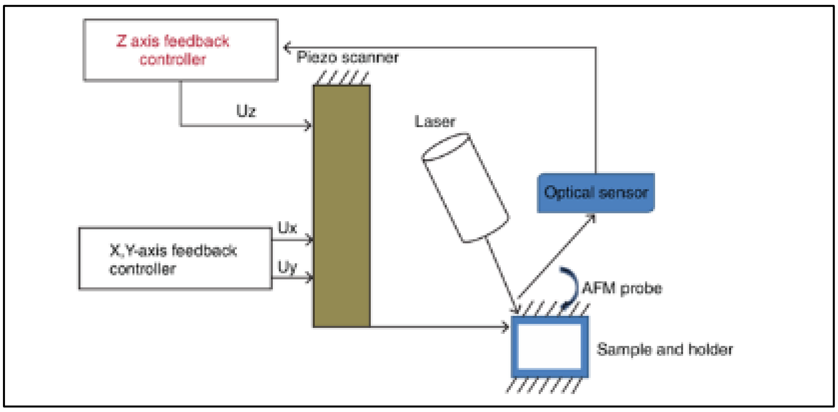

Figure 6 Schematic diagram of AFM technique [17]

2.3. Chromatographic Techniques

Chromatographic methods are used in materials science to efficiently isolate and measure the different elements present in complicated mixes of materials. These methods rely on chemical composition analysis, impurity detection, molecular structure, and interaction comprehension. By isolating individual molecules using chromatography, scientists may learn more about the chemical behavior and features of many materials, such as polymers, ceramics, and metals. Optimal material performance, new material development, and quality control rely on chromatographic techniques to accurately detect additives, pollutants, and byproducts. In fields as diverse as environmental science, medicines, and electronics, these methods also bolster studies to enhance the performance, longevity, and use of materials [50].

2.3.1. High-Performance Liquid Chromatography (HPLC)

High-Performance Liquid Chromatography (HPLC) is a sophisticated analytical technique extensively used for the separation, identification, and quantification of individual components within complex mixtures. The principle of HPLC is grounded in the differential interaction of analyte molecules with a stationary phase and a liquid mobile phase. As the mobile phase carries the sample through a packed column, the various components of the mixture elute at different rates depending on their affinity for the stationary phase, thereby achieving separation [51].

An HPLC system is composed of several essential components: a solvent reservoir that holds the mobile phase, a high-pressure pump that ensures continuous flow, an injector for introducing the sample, a chromatographic column packed with a stationary phase (often silica-based materials), a detector, and a data acquisition system. Upon injection, analyte molecules travel with the mobile phase through the column, where the separation occurs. Detectors such as UV-Visible spectrophotometers, fluorescence detectors, or mass spectrometers are employed to monitor the eluting compounds, enabling their detection and quantification [52].

HPLC plays a vital role in the characterization of organic materials, particularly in the fields of pharmaceuticals, polymer chemistry, and nanotechnology. It is instrumental in assessing the purity of synthesized compounds, analyzing the degradation products in stability studies, and evaluating the molecular weight distribution of organic polymers. In material science, HPLC has been employed to study organic ligands in hybrid systems such as metal-organic frameworks (MOFs) and to characterize surface-modified nanoparticles. The technique is prized for its high resolution, reproducibility, and versatility, offering excellent performance for thermally labile or non-volatile compounds [53].

However, HPLC is not without its limitations. It requires the analytes to be soluble and chemically stable in the mobile phase, which restricts its use with certain types of materials. Additionally, the instrumentation can be costly and maintenance-intensive. Despite these drawbacks, HPLC remains an indispensable tool in modern analytical laboratories, especially where precision and sensitivity are paramount [53].

2.3.2. Gas Chromatography-Mass Spectrometry (GC-MS)

Gas Chromatography-Mass Spectrometry (GC-MS) represents a powerful analytical technique that synergistically combines the separating efficiency of gas chromatography (GC) with the molecular identification capabilities of mass spectrometry (MS). This hyphenated method is widely recognized as a gold standard for the analysis of volatile and semi-volatile organic compounds in diverse disciplines, including material science, environmental chemistry, forensic analysis, and pharmaceuticals [54].

In GC-MS, the sample is first vaporized and transported by an inert carrier gas, typically helium or nitrogen, through a chromatographic column coated with a stationary phase. The components of the sample separate within the column based on their volatility and interaction with the stationary phase. As each component elutes from the column, it enters the mass spectrometer, where it undergoes ionization—most commonly through electron impact. The resulting ions are then sorted and detected based on their mass-to-charge (m/z) ratios, producing a characteristic mass spectrum that can be used for identification and quantification [55]. (Figure 11(c)).

The utility of GC-MS in material characterization is profound. It is particularly valuable for analyzing volatile organic compounds (VOCs) in polymer matrices, nanocomposites, and other organic-inorganic hybrid materials. Moreover, GC-MS facilitates the identification of decomposition products in thermal stability studies, surface-bound organic species on functionalized inorganic materials, and residual solvents or monomers in processed materials. This makes it a critical tool for quality assurance, regulatory compliance, and understanding the degradation pathways of advanced materials [55].

GC-MS offers unparalleled sensitivity and selectivity, capable of detecting trace-level compounds with high accuracy. It also allows for the identification of unknown substances through comparison with extensive spectral libraries, thereby enhancing its utility in exploratory research. Nonetheless, the technique is limited by its applicability primarily to volatile and thermally stable compounds. High-molecular-weight or polar, non-volatile substances often require derivatization to render them amenable to GC-MS analysis, which can introduce additional steps and complexity to the workflow. Despite these challenges, GC-MS remains an indispensable characterization tool, particularly in contexts where detailed compositional analysis of volatile organic substances is required. Its ability to provide both qualitative and quantitative data makes it a cornerstone technique in modern analytical chemistry [56].

2.4. Thermal Analysis

2.4.1. Differential Scanning Calorimetry (DSC)

Instruments used in DSC attempt to evaluate materials' thermal characteristics precisely by monitoring heat flow, temperature, and time. Research into phase transitions, including melting, crystallization, and glass transitions, can be facilitated by DSC, offering valuable insights into the thermal stability and behavior of substances. DSC is helpful in many areas, including polymers, materials science, medicines, and quality control, since it measures the heat of these transitions. Because it allows for the creation of novel materials and improves our knowledge of their thermal properties, DSC is an essential tool in R&D [1]. Using DSC, scientists may characterize materials and understand their thermal behavior by examining thermograms, which reveal critical thermal parameters, including melting points, heat of fusion, and glass transition temperatures [57].

A DSC device compares the heat flow of a reference material and a sample under controlled temperature changes to determine the rate of thermal transitions in the reference material. Within the DSC cell, a small portion of the sample is put into a sample pan, and an inert reference material is placed in another identical pan. Thermal events like melting, crystallization, or glass transitions can be detected by measuring the difference in heat flow between the sample and the reference, which is made possible by the instrument's finely regulated temperature, which usually ranges from 0.1 to 100 °C/min. A thermogram is generated by plotting the data using heat flow vs temperature. This graph shows peaks for different thermal transitions, and the area under the peaks corresponds to the enthalpy changes that occur during these transitions [58].

2.4.2. Thermogravimetric Analysis (TGA)

TGA is a method for studying a sample's thermal stability and chemical composition by measuring and analyzing its mass changes as a function of temperature or time. TGA involves methodically monitoring weight loss or increase in controlled atmospheric settings to understand temperature transitions, breakdown processes, and reaction dynamics. A vital material science and engineering technique for characterizing materials and improving thermal processes, TGA and derivative thermogram (DTG) curves provide visual representations that help understand thermal events [59].

TGA relies on several critical components that function together as an instrumentation concept to quantify the mass changes in a sample when it is exposed to temperature fluctuations. Crucibles consisting of materials that can withstand high temperatures, such as alumina or platinum, are held at the center by a precise balance. The crucible is housed in a furnace that regulates the temperature so that the sample can be heated or kept at a steady temperature according to predetermined heating profiles. A load cell, which transforms mechanical displacements into electrical impulses, allows the electronic balance to detect minute weight variations with high sensitivity, even when the temperature sample. Using gas flow controls also allows for the conduct of TGA under various atmospheric conditions, including inert, oxidizing, and reactive states. A data acquisition system processes the measured mass data, resulting in TGA curves showing the relationship between mass and either temperature or time and DTG curves showing how the mass changes over time [60].

2.5. Optical Techniques

Optical techniques are developing the beam of light scatter to acquire the analytical information on materials. They are versatile tools for studying and characterizing both organic and inorganic materials, providing valuable information about their optical properties and electronic structures [61].

2.5.1. Dynamic Light Scattering (DLS)

Dynamic light scattering (DLS) is one of the important methods for characterizing nanoparticles for particle size distribution regularly in "colloidal solution" [62]. Numerically appropriate the auto-correlation method communicates to the time-dependent fluctuations of the light scattered obtained from their Brownian motion to the particle speed. For this reason, it's depending on the particle size including their distribution of particle size. Moreover, the ISO certified that DLS techniques give good sensitive and reproducible methods. As well as DLS does not allow distinguishing between particles and aggregates [63].

If the molecule size is smaller than the frequency of light conceded through the colloid, then it is called Raleigh scattering. In Raleigh scattering, the power of dissipated radiation is uniform in all directions. When the molecule size is bigger than the wavelength, it is known as an elastic scattering of light or Mie dispersing scattering and it is mainly involved in the rotational and translational degrees of the individual particle [64]. The intensity of light scattered depends on individual particle and dependent angle; in this strategy, the equation is shown below:

$$I = I_0 1 + Cos^2\theta/2r^2\left(2\pi/\lambda)^4\left(\frac{n^2-1}{n^2+2}\right)\right)(\frac{d^2}{6})$$

Where Io is the intensity; λ is the wavelength; r is the distance of scattering center; θ is the angle; n is the refractive index (RI) and d is the diameter size of the particle

The fundamental supposition of DLS is that the nanoparticles suspended in a liquid are moving in the Brownian movement, which is otherwise called the “Random Walk” of the nanoparticles. This development is an aftereffect of the irregular impacts of the particles with the dissolvable atoms that cause the particles to diffuse through the medium. In this strategy, autocorrelation is described by exponential decay function, which is associated with the D (diffusion function) and K (measurement vector). Then, the translational diffusion/hydrodynamic diameter is calculated by using Stoke’s equation [65]. Figure 7 shows a schematic representative of DLS instrumentation. The equation is shown below:

$$R_{h^{1/4}} = kt/6\pi nd$$

where Rh is the hydrodynamic radius of the particle; K is Boltzmann constant; T is the temperature in Kelvin; n is viscosity medium and D is the translational diffusion coefficient

The value of D (translational diffusion coefficient medium) depends on the particle movement of the medium. So, the determination of diameter obtained by DLS depends not only on size but also on shape, structure, and solvent, level of concentration, and various types of electrolytes (medium). If any change occurs from the surface area of the particle, it may affect the diffusion speed and obvious size of the particle [20].

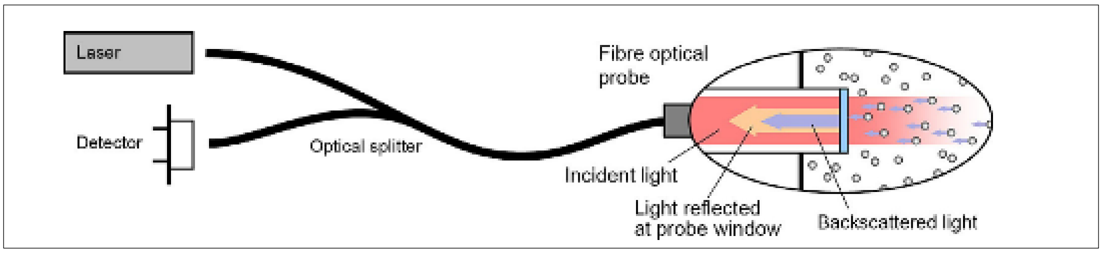

Figure 7 Schematic diagram of DLC techniques [66]

2.6. Other Advanced Characterization Techniques

Inorganic materials are characterized by their melting points and by instrumental techniques like elemental analysis, UV–visible spectroscopy, FT-IR, NMR, ESR, SERS, mass spectrometry, X-rays, and SEM, as well as thermal analysis, electrochemical analysis, and magnetic susceptibility measurements [67].

2.6.1. X-ray Diffraction (XRD)

A significant analytical tool, X-ray diffraction (XRD) is utilized for identifying the phase of a crystalline material and also provides the information about unit cell dimensions as well as other related parameters. It is based on the constructive interference of monochromatic X-rays with the interplane of a crystalline solid sample [68]. It is usually produced by sealed tubes or revolving anodes, and then seeing how the X-rays interact with the material’s ordered atomic planes (Figure 8).

Atoms in the crystal scatter constructive interference when the requirements outlined by Bragg’s Law are satisfied. This pattern, comprised of peaks at certain angles, details the material’s crystallite size, phase composition, and lattice structure. The XRD process usually involves using the K a characteristic radiation and filtering out any other radiation that is not needed. Critical structural factors like atomic plane spacing, crystallographic orientation, strain, and defect existence may be derived from the diffraction data. XRD has several important scientific and industrial uses, including crystal structure determination, phase identification, and the investigation of many material characteristics [69].

It determines the three-dimensional structure of crystalline materials by analyzing X-ray diffraction patterns and provides precise atomic positions and bond lengths in metal complexes. In the case of metal nanoparticles, it is useful to understand the chemical environment of metal atoms in nanoparticle structure as well as to study absorbed species and coatings on the nanoparticles [70].

XRD basically gives the information about the phase purity, crystal structure, and particle size. The obtained PXRD patterns can be used to (1) differentiate between crystalline and amorphous materials; (2) identify the phases formed, as each crystalline substance has its own characteristic powder diffraction pattern [Standard patterns are given in the powder diffraction file (JCPDS File)]; (3) determine the structure of crystalline materials (crystal axes, size, and shape of the unit cell, positions of the atoms in the unit cell); (4) determine the electron distribution within the unit cell; (5) identify the crystalline phases and measure their relative proportions; (6) measure the various kind of randomness, disorder, and imperfections in crystals; and (7) determine the radial distribution functions for amorphous solids and liquids. The obtained data when coupled with the refinement procedures, such as Le Bail and Rietveld refinement, provide structural information for the materials [71].

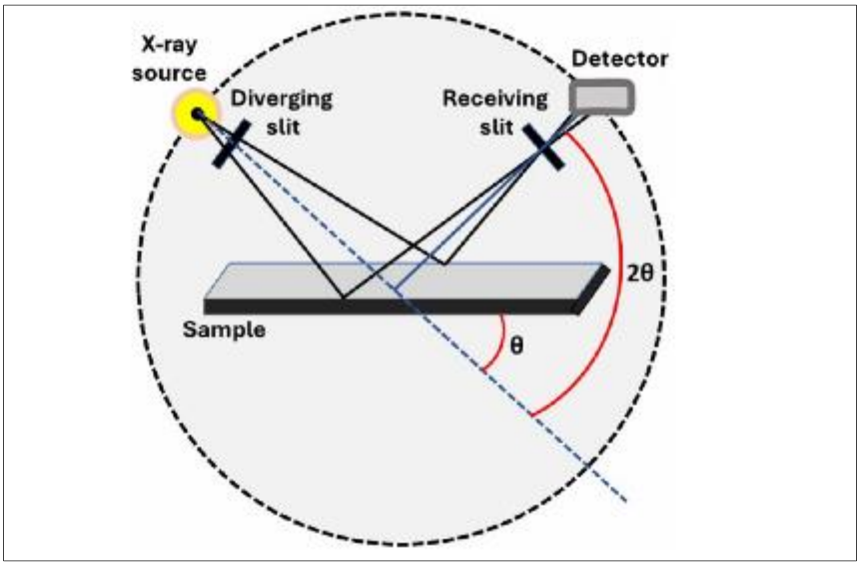

Figure 8 Instrumentation of XRD [19].

2.6.2. X-ray Photoelectron Spectroscopy (XPS)

X-ray Photoelectron Spectroscopy (XPS) is a non-destructive technique using X-rays in an ultrahigh vacuum atmosphere providing quantitative and qualitative information. XPS is a quantitative spectroscopic technique that measures the elemental composition, empirical formula, chemical state, and electronic state of the elements present in a material. XPS is also known as electron spectroscopy for chemical analysis. XPS uses X-rays at the sample's surface to eject photoelectron from the inner electronic shell of atom. An electron from a higher energy level then falls to fill the hole left behind and its emitted radiation energy is used to eject an Auger electron. Thus, XPS emits both photoelectrons and Auger electrons which can be seen in the spectrum [72]. Those photoelectrons are collected by a detector and their energies are measured. Figure 9 The XPS analytical methods depends on the kinetic energy and binding energy expressed with the following Einstein's relation:

$$E_{B=} hv - E_k - \emptyset$$

EB is binding energy of the ejected electron, hν is the energy of the incident photon, EK is the measured kinetic energy of the ejected electron, and is the work function which relates to the minimum energy required to eject an electron from the solid matrix to the vacuum level. Subtracting the measured kinetic energies of the photoelectrons from the energy of X-rays gives the binding energies of the electrons. Binding energies are specific to specific atom for its specific energy level, and elements besides hydrogen can be detected and their relative amounts can be easily determined. XPS can even give information about the oxidation states of those atoms [73].

XPS spectrum represents the number of distinguished electrons as an element of the binding energy inside a material when it is bare to an X-ray beam. It is called as "Photoelectric effect." The composition of elements can be identified within materials because each material has its characteristic peak binding energy. These peaks of individual elements are very sensitive for the electron configuration process due to their location and shape. In this strategy, it is extremely viable to investigate the electron state, chemical composition, and bonding of atoms of the nanomaterials. XPS is generally a high-sensitive method, so it can able to analyze the nanomaterials surface within 10nm. Moreover, it is used to the quantitative determination of the chemical com position of within materials and few XPS quantitative material measurements obtained accurate as ±10% and can be expressed as:

$$I_i = N_i \sigma_i \lambda_i K$$

Where I_i is the intensity of photoelectron peak (p) for the element (i); N_i is the average atomic concentration of element (i) in the surface under analysis; σ_i is photoelectron cross-section for the element (i) expressed by peak (p) or Scofield factor ; λ_i is the inelastic mean free path of a photoelectron from element (i) expressed by peak (p) and K is other factor related to the quantitative detection of a signal [74].

This technique is also useful to differentiate between different oxidation states of metals present in complexes; it can distinguish between Fe (II) and Fe (III) in iron complexes. It also provides information about the nature of metal–ligand bonds in complexes and identifies and quantifies the elements of the surface of nanoparticles. XPS has also been used to understand the surface chemistry of palladium and other metal nanoparticles [75].

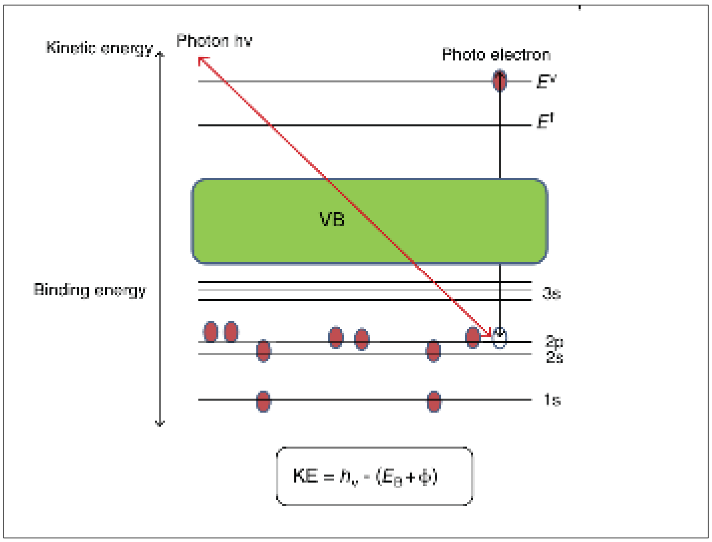

Figure 9 Schematic diagram of XPS techniques [17]

2.6.3. Energy Dispersive X-ray Spectroscopy (EDX)

The goal of EDX in materials science is to conduct very accurate analyses of the chemical properties and elemental composition of materials. The resolution of EDX systems has been dramatically enhanced by recent developments in semi-conductor-based spectrometers, allowing for more precise and thorough examination. The emphasis is moving from improving system resolution to creating creative applications customized to specific analytical demands as this technology approaches its realistic resolution limitations. Various fields within materials science are currently using EDX to improve research and industrial operations by gaining crucial insights into materials' structure, content, and characteristics [76].

2.6.4. Neutron Diffraction

One of the most effective methods for studying atomic and magnetic structures of materials is neutron diffraction. Diffraction analysis is achieved by focusing a neutron beam on a sample and observing the resulting diffraction pattern on the sample's atomic nuclei. A material's crystal structure, such as its atomic arrangement, interatomic distances, and lattice symmetry, may be inferred from the diffraction pattern. Light elements, such as hydrogen, are notoriously difficult to investigate using XRD, but this method is ideal for the job. Neutron diffraction is a tool for studying magnetic structures by seeing how neutrons interact with atomic magnetic moments. When researching crystalline and magnetic materials, as well as identifying phases and analyzing residual stresses, this method finds extensive use in materials science [77].

Neutron diffraction interacts with atomic nuclei in a material and operates based on neutrons' wave-like nature. A diffraction pattern tells us something about the crystal structure of a material when we point a neutron beam at it and the atomic nuclei scatter the neutrons. Schematic diagram of neutron diffraction is illustrated in Figure 10

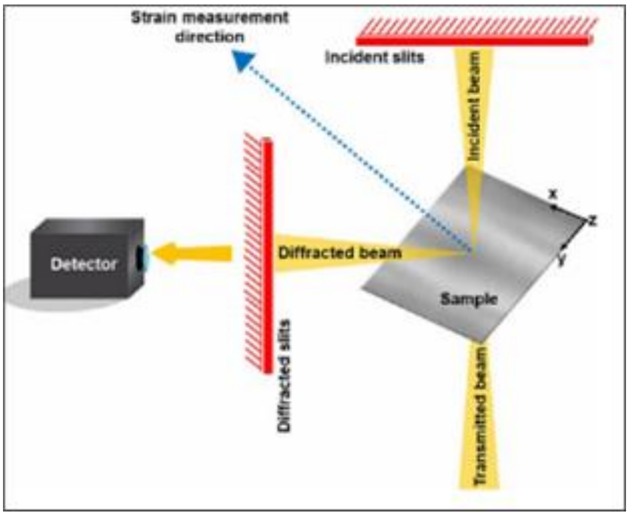

Figure 10 Schematic diagram of neutron diffraction [77]

2.6.5. Auger Electron Spectroscopy (AES)

Measurements of the energy of Auger electrons ejected from a material after being stimulated by an external energy source, such as an electron beam, allow AES to deduce different materials' surface composition and chemical states. Surface characteristics, contamination, and thin film structures may all be better understood with the help of AES, which offers highly surface sensitive data on the chemical bonding and elemental composition of a material's exterior thin layers. Applications in materials research, nanotechnology, and corrosion investigations are extensive [78].

The basis of AES is the Auger effect, which is the process by which a high-energy electron beam energizes a material, and then electrons are ejected from it. A vacancy, or core hole, can be created when an electron beam hits a sample and knocks out an electron from an atom's inner shell. Afterward, a higher energy electron takes its place in the vacant space, and the energy released during the transition is passed on to another electron, known as an Auger electron, which is subsequently in the material may be inferred from the kinetic energy of the released Auger electrons, which are collected and studied. Researchers may examine the electrical characteristics and chemical makeup of materials' topmost layers with this method's surface-sensitive examination. When analyzing the elements and chemicals of inorganic solid surfaces, Auger spectroscopy (AES) is a crucial method [79]. Figure 11(d) shows the pictorial representation of AES

2.6.6. Inductively Coupled Plasma Mass Spectrometry (ICP-MS)

ICP is an atmospheric pressure ionization technique that serves as a hard ionization process, leading to the sample's whole atomization during ionization. ICP source is made up of a sample introduction system, an ICP torch, a radio frequency coil for the production of argon plasma, which serves as the ion fount, and an interface that joins the mass spectrometer and the source as shown in Figure 11(a) [80].

In materials science, ICP is used to precisely analyze the chemical composition of various materials by detecting and quantifying trace and significant components. For the advancement, quality assurance, and investigation of novel materials, this method is vital for deducing the elemental makeup of many materials, including metals, ceramics, polymers, and more. In situations where even little changes in the elements can majorly impact the material's characteristics, ICP is invaluable for detecting tiny contaminants and determining the material's purity. ICP improves industrial operations and research in materials science by allowing for the accurate quantification of many elements and high sensitivity, which in turn allows for the manufacture of high-quality materials with required properties [81].

2.6.7. Electron Spin Resonance Spectroscopy

Analyzing chemical species or materials with one or more unpaired electrons can be effectively accomplished through electron para magnetic resonance or ESR [82]. An ESR spectrum is often generated by altering the magnetic field intensity at a constant microwave frequency, as shown in Figure 11(b).

This technique is used to detect unpaired electrons in metal complexes as well as evaluate the magnetic properties of complexes. ESR is a powerful tool for characterizing metal nanoparticles, especially those containing paramagnetic metal centers. It provides detailed information on the electronic structure, local environments, dynamic behavior, and interactions of metal nanoparticles [83].

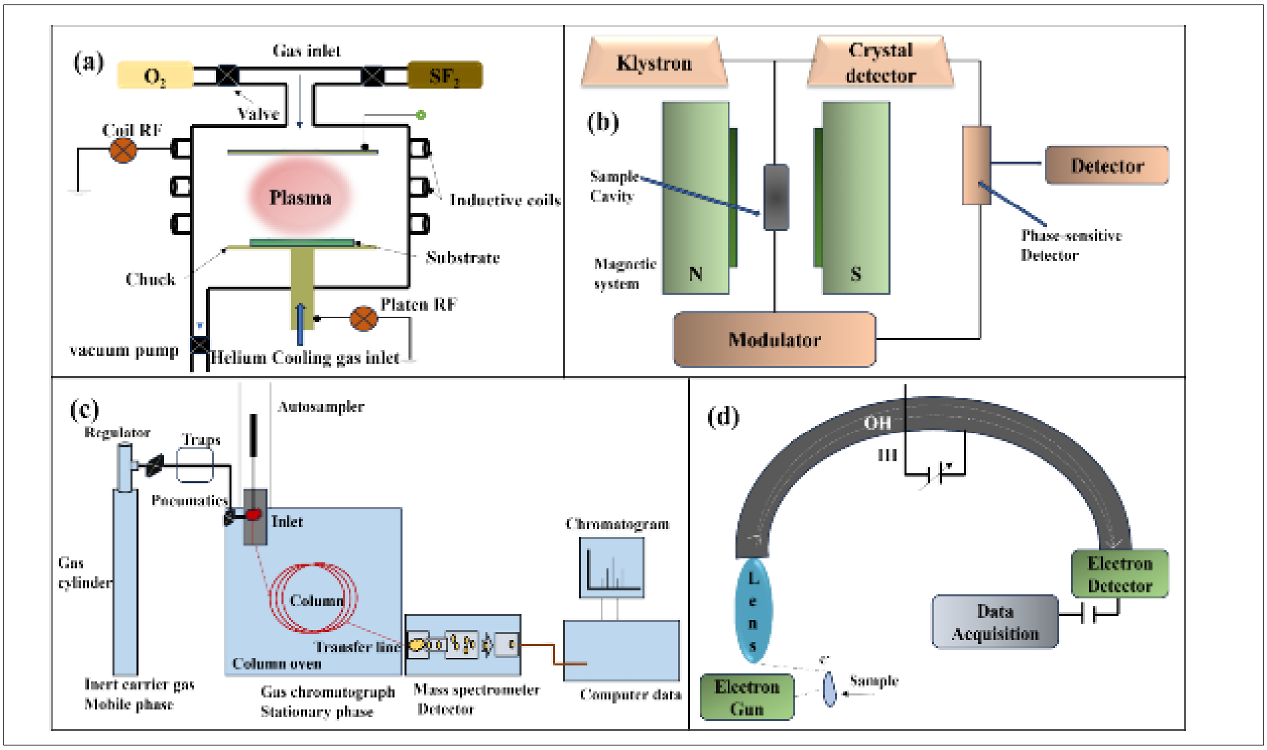

Figure 11 (a) Inductively coupled plasma, (b)Electron spin resonance, (c) Gas chromatography- Mass spectrometry, (d) Auger electron spectroscopy [16]

2.6.8. Synchrotron Radiation X-ray Diffraction

For more precise and quick investigation of even thick or complicated materials, synchrotron radiation is utilized in advanced applications. Synchrotron radiation sources are characterized by great coherence, high brightness, and efficient collimation throughout a wide range of wavelengths. Characterization methods based on synchrotron radiation have lately had a big impact in solving important scientific problems in the energy conversion area. Importantly, they have shed light on the ways in which atomic and electronic structures evolve at solid– liquid interfaces, offering vital experimental proof for comprehending processes of energy conversion. Furthermore, the identification of actual active phases and active sites is made possible by in situ and operando technologies, which capture the kinetic progression of catalytic processes. SRXRD provides better capabilities for studying long-range ordered structures and crystal phase measurements compared to traditional XRD, which is useful for examining bulk crystal phase structures. The superior signal quality achieved by SRXRD makes it a more potent instrument when contrasted with traditional XRD [84].

SRXRD is an advanced diffraction technique that utilizes highly intense and tunable X-rays generated by a synchrotron source. In this process, electrons are accelerated to near-light speeds in a storage ring and guided along a curved path by magnetic fields, emitting high-brightness, collimated X-ray beams as they change direction. These X-rays are then monochromatized using silicon crystal optics to select specific wavelengths, while mirrors or zone plates focus the beam onto the sample. When the monochromatic X-ray beam interacts with the sample, diffraction occurs according to Bragg's law, and the resulting diffraction pattern is recorded using high resolution detectors. This allows for precise structural analysis, including phase identification, strain mapping, and defect characterization. The superior properties of synchrotron X rays, such as higher intensity, better collimation, and energy tunability, enable SRXRD to achieve significantly higher resolution than conventional XRD. Additionally, its ability to perform in situ and operando studies makes it a powerful tool for real-time monitoring of structural changes under varying environmental conditions, such as temperature, pressure, and chemical reactions. Figure 12 displays a general schematic representation of SRXRD.

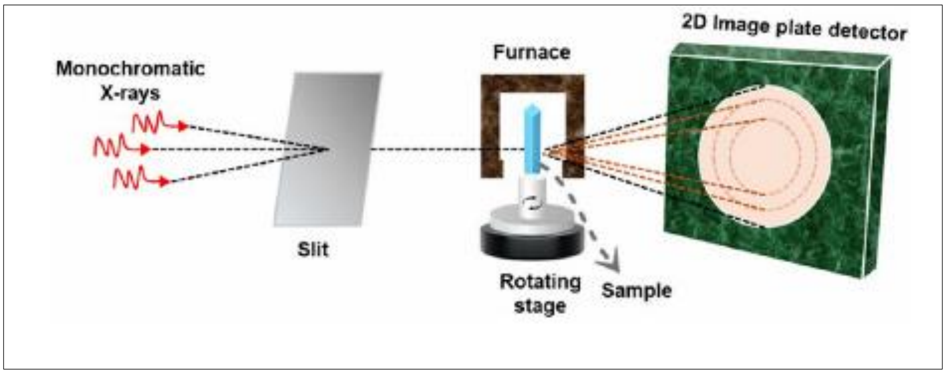

Figure 12 Schematic of SRXRD [19]

With the growth in synchrotron radiation (SR) facilities, synchrotron-based analytical techniques have become outstanding tools that are used in many areas of research, including physics, chemistry, biology, materials, environment, and the nanoscience. For instance, synchrotron radiation X-ray fluorescence (XRF) analysis is particularly well suited to investigating the micro-distributions of trace elements in biological samples, because it has high spatial resolution, the capacity to analyze several elements simultaneously, quantitatively and nondestructively, and its detection limit can reach down to the 50–100 ng/g range for many elements [85]. Synchrotron radiation X-ray absorption spectroscopy (XAS) is very sensitive to the local chemical environment of the element of interest, and can be used to acquire information on its oxidation state, coordination number, the identities of its nearest neighbors, and bond lengths, which are crucial for understanding the interaction of nanoparticles with biomolecules and the mechanisms of the toxicological effects of nanoparticles in living systems [86]. Synchrotron radiation circular dichroism (SRCD) is useful for performing conformational analyses of biomolecules based on the higher energy transitions of chromophores such as $n \rightarrow \pi^*$ transitions, $\pi \rightarrow \pi^*$, $n \rightarrow \sigma^*$, etc. [9]. Additionally, the enhanced brilliance of modern synchrotron facilities and advances in focusing optics allow for high spatial resolution in the 0.5 1 μm range for hard X-rays (>3 keV) and of around 30 nm for soft X-rays (-1KeV). All of these properties—high chemical sensitivity, high spatial resolution and low detection limits—of synchrotron-based X ray microprobe techniques are of particular interest in nanotoxicological studies. In addition, synchrotron-based techniques such as X-ray diffraction, X-ray small angle scattering, and X-ray photo electron spectroscopy have become important nano-characterization tools [87].

2.6.9. X-ray Tomography

X-ray tomography is a non-destructive material testing in which reconstruction of the 2D and 3D images is done. The production and use of X-ray was well known before and now more and more advancements are taking place for increasing its field of utilization [88]. Ultrasonic testing being the most reliable and established method for material characterization is unable to extract the 3D data for the calculation of internal voids/ porosity. With the repeatability of measurements, it has been found that x-ray computed tomography emerged as a very useful technique in determining the porosity in a given material with good accuracy in case of large voids and calculation of shape, size and distribution of internal voids with little less accuracy. The high resolution in X-ray computed tomography (XCT) is needed for the accurate and precise measurement of small pores [89]. To get the good surface characteristics and for deeply understanding of image noise, the modulation transfer functions can be used. Accuracy and repeatability of measurement, results are significantly influenced by the methods used in determining the starting threshold. When default settings will be used in commercial CT evaluation software, the errors in the measurement are genuine [90].

Table 1 Overview of Advanced Characterization Techniques for Organic and Inorganic Materials

S/N	Technique	Applicable Material Type	Application
1	UV–Vis Spectroscopy	organic	Electronic transitions, conjugation, and bandgap analysis
2	FT-IR Spectroscopy	Organic & Inorganic	Molecular vibrations, functional group identification
3	Raman Spectroscopy	Organic & Inorganic	Molecular structure, crystallinity, and stress analysis
4	NMR Spectroscopy	Organic	Molecular structure, dynamics, and functional groups
5	X-ray Photoelectron Spectroscopy (XPS)	Inorganic & Surface-modified Organic	Surface elemental composition and oxidation states
6	X-ray Diffraction (XRD)	Inorganic	Crystalline structure, phase identification, and lattice parameters
7	Scanning Electron Microscopy (SEM)	Organic/Inorganic	Morphology, surface structure, and particle size
8	Transmission Electron Microscopy (TEM)	Organic/Inorganic	Nanoscale imaging, crystallography, and defects
9	Atomic Force Microscopy (AFM)	Organic/Inorganic	Topography, surface roughness, and mechanical properties
10	Differential Scanning Calorimetry (DSC)	Organic	Thermal transitions (Tg, Tm), crystallinity, and purity
11	Thermogravimetric Analysis (TGA)	Organic/Inorganic	Thermal stability, decomposition patterns
12	Inductively Coupled Plasma Mass Spectrometry (ICP-MS)	Inorganic	Trace elemental analysis
14	Scanning Tunneling Microscopy (STM)	Inorganic & Conductive Organic	Electronic states, atomic resolution surface mapping
17	Nano-Computed Tomography (Nano-CT)	Inorganic & Composite	3D internal structure, porosity, and defect analysis
18	Kelvin Probe Force Microscopy (KPFM)	Organic/Inorganic	Surface potential mapping and electronic properties
20.	In Situ/Operando Techniques (multi-modal)	Organic/Inorganic	Real-time structural, thermal, or chemical changes under operational conditions

3. Emerging Trends and Innovation in Material Characterization

Material characterization has undergone a paradigm shift in recent years, driven by the urgent need to develop high-performance materials for applications ranging from energy storage and catalysis to biomedical devices and environmental remediation. Traditional characterization techniques, while foundational, often provide limited insight into the real-time behavior of materials under operational conditions. This has catalyzed a wave of innovation, giving rise to a suite of advanced methodologies that enable deeper, more holistic understanding of material structure, dynamics, and functionality. Among these, in-situ and operando methods, multimodal characterization strategies, and high-throughput automated techniques are at the forefront of emerging trends reshaping the landscape of materials science [91].

3.1. Advances in In-situ and Operando Techniques

In-situ and operando characterization techniques have revolutionized the way materials are studied by allowing scientists to observe changes in real-time under realistic working environments. Unlike ex-situ methods which examine materials after processing or reaction has occurred in-situ techniques capture the dynamic evolution of material properties as a function of stimuli such as temperature, pressure, electric field, or chemical environment. Operando techniques take this further by combining structural characterization with simultaneous performance evaluation, providing direct correlation between material structure and functional behavior during actual operation [92].

These techniques are particularly transformative in fields like catalysis, battery research, and phase-change materials, where structural or compositional changes dictate performance. For instance, in battery materials, operando X-ray diffraction (XRD) and X-ray absorption spectroscopy (XAS) have been used to monitor phase transitions and oxidation state changes during charge-discharge cycles, thereby revealing degradation mechanisms that affect long-term stability and efficiency. Similarly, in catalytic systems, operando infrared (IR) and Raman spectroscopy have enabled real-time monitoring of surface intermediates and active site evolution under reaction conditions [93].

The implementation of synchrotron-based in-situ methods has further enhanced spatial and temporal resolution, allowing the study of transient phenomena and reaction kinetics on the sub-second timescale. Such advances are instrumental in designing materials with tailored properties and in accelerating the development of next-generation technologies [94].

3.2. Multimodal Characterization Approaches

The complexity of modern materials especially hybrid systems that integrate organic and inorganic components necessitates characterization techniques that can probe multiple aspects of structure and function simultaneously. Multimodal characterization involves the integrated use of two or more complementary techniques to provide a comprehensive picture of material behavior across different length and time scales [95].

By combining microscopy, spectroscopy, and diffraction methods, multimodal approaches capture both surface and bulk information, as well as chemical and physical attributes. For example, correlative imaging that combines scanning electron microscopy (SEM) with energy-dispersive X-ray spectroscopy (EDS) enables spatial mapping of elemental composition alongside morphological analysis. Similarly, coupling atomic force microscopy (AFM) with infrared spectroscopy (AFM-IR) allows for high-resolution chemical imaging of nanostructured materials and biological specimens [96].

Recent developments in data fusion and artificial intelligence (AI) have further enhanced the power of multimodal characterization. Machine learning algorithms can now integrate data from disparate sources—such as X-ray tomography, electron microscopy, and Raman maps—to uncover hidden patterns and correlations that are not evident from any single technique alone. These integrative approaches are proving invaluable in fields such as biomaterials, photovoltaics, and nanocomposites, where understanding structure-property-function relationships is essential for performance optimization [97].

3.3. High-throughput and Automated Techniques

High-throughput and automated techniques have emerged as powerful solutions to this challenge, enabling rapid screening and analysis of large material libraries with minimal human intervention. It integrates robotics, microfabrication, and real-time data acquisition to evaluate numerous samples in parallel. Techniques such as automated X-ray diffraction (HT-XRD), combinatorial sputtering, and high-speed spectroscopy are commonly employed in materials discovery platforms to screen for optimal compositions and processing conditions. For example, in the development of perovskite solar cells, high-throughput photoluminescence and absorption measurements have accelerated the identification of stable and efficient materials by orders of magnitude compared to conventional trial-and-error methods [98].

Automation is further facilitated by advances in software and machine learning, which enable intelligent data analysis, anomaly detection, and decision-making in real-time. Tools such as autonomous scanning probe microscopy can adaptively choose regions of interest based on prior measurements, thereby increasing efficiency and reducing operator bias. The integration of high-throughput screening with cloud computing and digital twins is pushing the frontier even further, enabling virtual experiments and predictive modeling that complement physical characterization. These developments are expected to play a central role in accelerating the design and deployment of advanced materials in a sustainable and cost-effective manner [99].

3.4. Integration of Machine Learning and AI in Characterization

AI is a transformative technology that enables machines to emulate human cognition and behavior, facilitating intelligent decision-making, task execution, and problem-solving. By leveraging data-driven AI techniques, these systems can surpass human capabilities in speed, efficiency, and scalability, making them invaluable tools across various fields, including materials science. The integration of AI and ML into the processing of characterization data such as spectroscopy and microscopy has revolutionized the way complex data sets are interpreted in materials science. AI algorithms, particularly deep learning models, are increasingly employed to automate baseline correction, denoising, peak identification, and feature extraction in spectroscopic techniques like FTIR, NMR, and Raman spectroscopy [100]. Similarly, in microscopy (e.g., SEM, TEM, AFM), convolutional neural networks and other vision-based algorithms enable rapid phase classification, defect detection, and morphological analysis with high accuracy and reproducibility. These approaches not only reduce manual effort and bias but also enhance the speed and depth of data interpretation, allowing researchers to uncover hidden patterns and correlations that are otherwise difficult to detect using traditional methods. Moreover, AI is being leveraged in autonomous or “self-driving” laboratories to guide experimental design based on real-time feedback from characterization data. As AI continues to evolve, its role in processing and interpreting spectroscopy and microscopy data will be central to accelerating materials discovery and development.

Among the most impactful branches of AI is ML, which utilizes data-driven algorithms to extract patterns, make predictions, and optimize decision-making without explicit programming. In the realm of materials science, ML plays a pivotal role in accelerating the discovery, design, and optimization of advanced materials. Traditional methods of material development, which rely heavily on empirical experimentation and computational simulations, are often time-consuming, resource-intensive, and limited in scope. ML circumvents these challenges by analyzing vast data sets, identifying intricate relationships between structural and functional properties, and generating predictive models that guide material synthesis and characterization. Through descriptor generation, model construction, material property prediction, and experimental validation, ML enhances the efficiency of materials research, enabling the rapid screening of candidates with desirable characteristics. Moreover, by integrating AI with established computational methods such as first-principles calculations and MD, researchers can significantly reduce computational costs while expanding the predictive accuracy and applicability of simulations. This synergy between AI and ML has revolutionized materials science, paving the way for data-driven approaches that not only expedite the innovation cycle but also unlock new possibilities for designing next-generation materials with tailored properties [101].

4. Multidisciplinary Applications of Advanced Characterization Techniques

Advanced material characterization techniques are the backbone of interdisciplinary innovation, empowering researchers to tailor materials for applications spanning electronics, energy, environment, healthcare, and quantum computing. The convergence of novel characterization tools with machine learning, in situ analytics, and high-resolution imaging has significantly accelerated the pace of discovery, especially in areas where structural, compositional, and functional precision are paramount. This section dives into the multidisciplinary applications of advanced characterization techniques

4.1. Organic Electronics and Photovoltaics

The field of electronics and photonics has rapidly grown and evolved over the last few decades, driven by the demand for smaller, faster, and more efficient devices. The miniaturization of electronic components and the development of new materials have led to significant advancements in information technology, telecommunications, energy generation, and sensing applications. Since the discovery of the semiconducting nature of poly-thiophene, organic electronics have experienced tremendous development [102]. In particular, organic field-effect transistors (OFETs), light-emitting diodes (OLEDs), and photovoltaic cells (OPVs) are key candidates for next generation of electronics and optoelectronics, thus revolutionizing the information, photonics, and energy sciences [103]. Recently, the electrical and optoelectrical performance of OFETs, OLEDs, and OPVs have improved impressively, due to new molecular design strategies, improved process engineering, effective interface optimization, and advanced device architecture. However, despite these remarkable advances, as a new research topic, organic devices still face some key challenges, as follows: (i) the important aspects of packing motifs and growth strategies of organic semiconducting films for high-performance applications are still difficult; (ii) the relationship between charge transport physics and the molecular structure properties is not fully understood; and (iii) an in-depth understanding of electrode, dielectric, and interface properties under various device structures and operating circumstances is still lacking. To solve these issues, it is important to utilize more advanced characterization techniques [104].

In-situ/operando techniques for studying organic semiconductors have been developed as powerful tools to achieve unprecedented insights into complex film growth, electronic states, and structure-property relationships under conditions relevant to device operation or device manipulation, which cannot be revealed by common ex-situ measurements. Hence, in situ/operando techniques can contribute to our understanding of the nature of molecular assembly mechanisms and intrinsic electronic properties, which can further improve molecular design and device performance [105].

Recently, Lee et al. [106] used in-situ optical microscopy to ob serve the crystallization process of TIPS-pentacene thin films using a continuous-flow microfluidic-channel-based meniscus-guided coating (CoMiC), which could precisely manipulate the flow behavior via microfluidic channels (Figures 13(a) and 13(b)**)**. Based on the in-situ characterization and numerical simulation, the relationship between flow pattern, thin-film crystallization, and electrical performance of OFETs is comprehensively analyzed and reveals that chaotic advection leads to device-to-device uniformity. This work provides effective strategies to tune solution-based crystallization properties for performance optimization of OFETs, solar cells, and displays.

Furthermore, top-view and side-view in-situ high-speed optical microscopies were used to obtain three-dimensional (3D) meniscus geometry under multiple experimental conditions during solution shearing (Figures 13(c)) [107]. The top-, side-, and 3D view microscopies for the visualization of the contact line/crystallization process and cross-sectional meniscus shape are shown in Figure 13 (d), contributing to the mathematical model for mass and momentum transport within the meniscus geometry. Therefore, in-situ high-speed optical microscopy enables the analysis and prediction of the crystallization process of organic films under multiple experimental parameters.

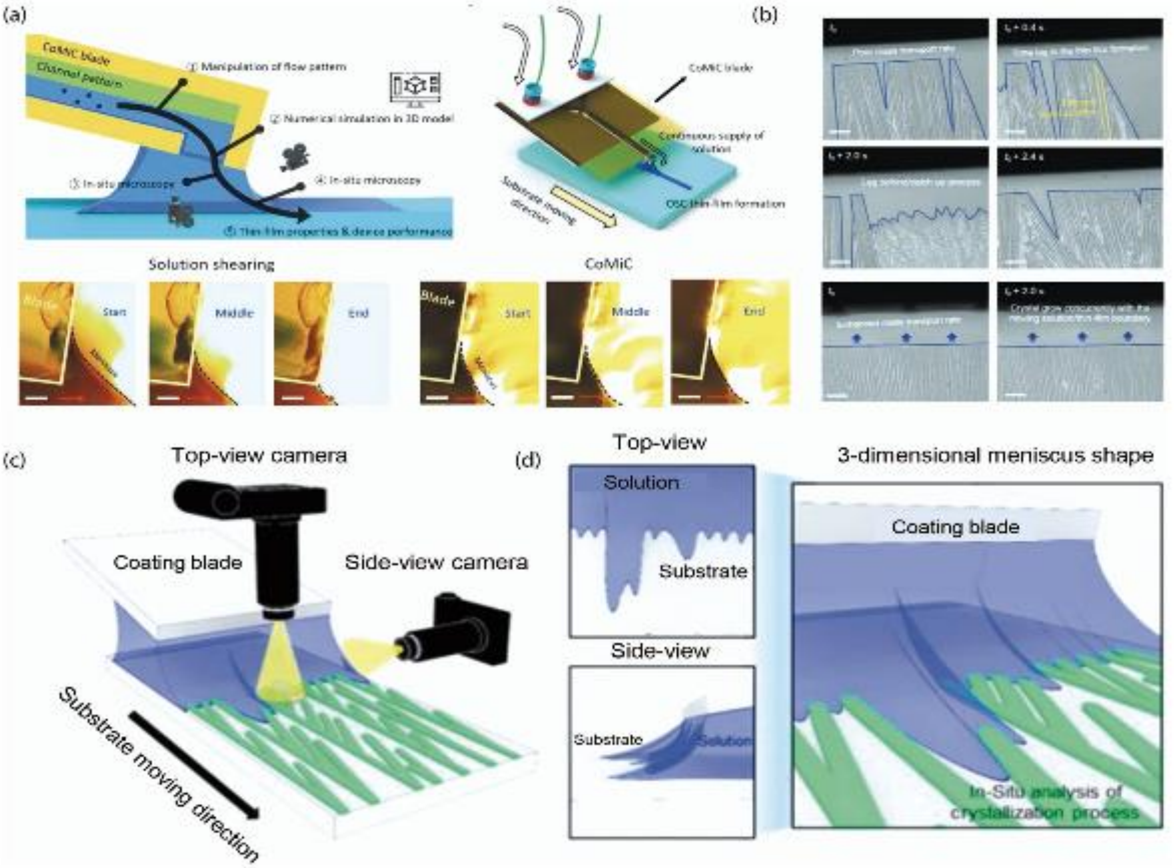

Figure 13 In-situ optical microscopy of organic films using CoMiC: (a) Flow path and meniscus shape during coating. (b) Crystallization and film boundary evolution of doped TIPS-pentacene via FM-CoMiC and SHM-CoMiC [106] c) Top and side-view in-situ microscopy showing how 3D meniscus geometry relates to crystallization during solution shearing. (d) Multi-angle microscopy visualizing contact line dynamics, crystallization, and meniscus cross-section [107]

4.2. Nanomaterials, Nanocomposites and Hybrid Materials

The emergent significance of two-dimensional (2D) nanomaterials are determined by the continuous innovation of new properties such as optical, electrical, and mechanical, which further strengthen their utility toward electronic, energy storage, and biomedical domain [108]. The graphene is one of the most 2D nanomaterials due to their physicochemical properties and high energetic capability, and also, transition metal dichalcogenides (TMDCs) having good fundamental properties involve practical applications. The specific determination of the properties of the nanomaterials is the most important section in nanotechnology and which can be executed by advanced techniques that are responsive to nanoscale dimensions (nm). In recent years, nanomaterials' advanced characterization techniques involve determination of character of nanosized materials [109].

Raman spectroscopy is one of the most standard characterization methods of carbon-based nanomaterials such as graphene and graphite; the characteristic bands are located at the 1580cm^{-1} and the 2D band at 2700cm^{-1}. A third attribute, the D band, around 1350cm^{-1} is not Raman spectra, its defects of the pristine graphene band value. The D band also called as "defect induced band." The G band at 1586cm^{-1} results from in-plane vibration sp2 carbon atoms and is the majority significant feature of graphite materials. The 2D band involved in double resonance (e.g. second-order overtone) process indicates scattering of two photons with the opposite moment with K point of symmetry (stacking order) due to the "Brillouin zone of graphene." Raman spectroscopy is also used in the investigation of CNTs, metal oxides, nano-belts, quantum dots, and nanorods [110]. However, in certain metal nanoparticles, they may additionally improve the Raman signals known as surface-enhanced Raman spectroscopy (SERS), which is used in the determination of plasmonic and chemical enhancement effects signal ranging from 108 to 1011, which is able to characterize such nanomaterials in innovative details [111].

Nilchi et al [112] chemically synthesized and evaluated a hydrous manganese dioxide–polyacrylonitrile (MnO2–PAN) as an organic–inorganic composite material. The physico-chemical characterization was carried out by Fourier transform infrared spectroscopy (FT-IR), X-ray powder diffraction (XRD), CHN elemental analysis, scanning electron microscopy (SEM), nitrogen adsorption–desorption studies and thermogravimetry-differential scanning calorimetry (TGA-DSC). Figure 14 (a) depicts XRD patterns of the synthesized materials and polyacrylonitrile. The analysis of X-ray patterns revealed that the XRD data corresponding to synthetic manganese dioxide agreed well with the Powder Diffraction Files Search Manual (card no.12 716, 1986). It was also observed that the 2y values at the peak points of MnO_2–PAN are the same as those in MnO2, and hence their crystalline structure is very similar. Furthermore, comparing the XRD patterns indicates that manganese dioxide has been loaded on polyacrylonitrile. The IR spectrum of MnO_2 – PAN composite is recorded in Figure 14 (b). The broad band in the region of 3200–3650cm^{-1} is due to interstitial water and hydroxyl groups and the sharp peak at 1620 cm^{-1} corresponds to the bending vibration of water molecules. Strong absorption peak at 2140 cm^{-1} can be assigned to cyanide stretching vibration. The spectrum of the sample shows the characteristic band for–CH_2 at 1453 cm^{-1}. The band at 1080–1100 cm^{-1} is assigned to the C–O stretching vibration. The bands in the 400–550 cm^{-1} region are due to the Mn–O stretching. Investigation of infrared spectra of ion exchange material prior to and after irradiation (Figure 14 (b)) showed that there is no significant difference. Therefore, these ion exchangers were resistant to gamma irradiation of up to 200 kGy.

The nitrogen adsorption–desorption studies showed that the BET surface area of manganese dioxide–polyacrylonitrile composite is 53.03 m2/g. Scanning electron microscopic photograph of the prepared MnO2–PAN composite bead is shown in Figure 14 (c). The results revealed that the particles were not homogeneous. The pore size of the inner part of the particles was larger than that near the surface. The kinetic of sorption on these adsorber beads must be very fast, since the MnO2 powder, which is the active material, is found to be dispersed throughout the binding matrix. The TGA-DSC thermal analysis of MnO_2–PAN composite is shown in Figure 14(d), which shows decomposition steps.

Batch experiments were carried out as a function of contact time, interference of the coexisting ions and initial pH of adsorptive solution applying a radiotracer technique. The effect of temperature on the distribution coefficient of cesium has been utilized in order to evaluate the changes in the standard thermodynamic parameters. The results indicated that Csþ ions could be efficiently removed using MnO2–PAN composite in the pH range of 4–9 from aqueous solutions and the uptake of cesium is affected to varying degrees by the presence of some diverse co-ions. The equilibrium isotherms have been determined and the sorption data were successfully modeled using Freundlich model.

Organic-inorganic hybrid materials are a class of advanced materials that combine the advantageous properties of both organic and inorganic components. These materials are designed to leverage the flexibility and functional diversity of organic compounds with the robustness and thermal stability of inorganic materials. This synergy opens new avenues for applications across various fields such as electronics, photonics, catalysis and biomedicine. Organic-inorganic hybrid materials are at the forefront of materials science due to their ability to combine the best properties of both organic and inorganic components [113].

Organic/inorganic material hybrids are not simply physical mixtures. They can be defined as nanocomposites at the molecular scale, having at least one component, either the organic (or biological) component or the inorganic component, with a characteristic length scale on the nanometer size (a few Å to several tens of nanometers). The properties of hybrid materials do not simply result from the sum of the individual contributions of their com ponents, but also from the strong synergy created by an extensive hybrid interface. Indeed, the mineral/organic interface, including the types of interactions present, the surface energy, and the existence of labile bonds, plays a strong role in modulating of a number of properties (optical, mechanical, separation capacity, catalysis, and chemical and thermal stability) [114]. Today, the potential of hybrid materials is reflected by the fact that many of them are entering a variety of markets. New materials and systems produced by man in the future must aim toward higher levels of sophistication and miniaturization, be recyclable and respect the environment, be reliable and consume less energy or help save energy [115].

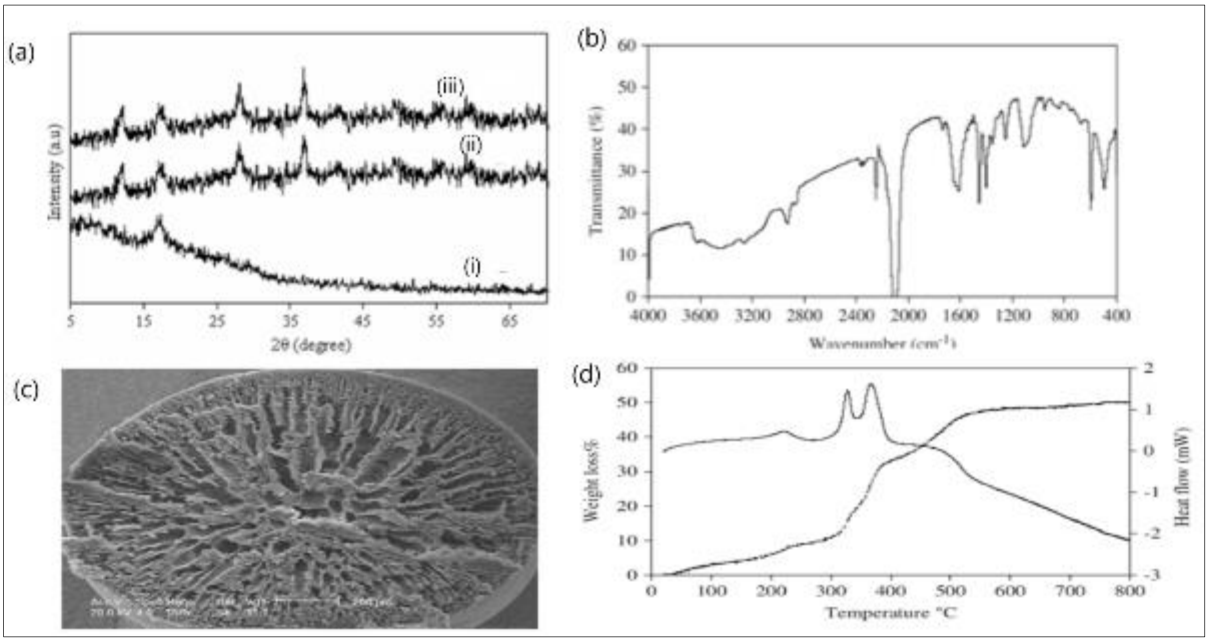

Figure 14 (a) XRD of PAN, MnO_2, and MnO_2–PAN composite; (b) IR spectrum of MnO_2–PAN; (c) SEM cross-section of composite bead; (d) TGA/DSC curves of the composite [112]

4.3. Catalysis

Catalysis represents one of the most characterization-intensive domains due to their reliance on surface phenomena and dynamic chemical processes. XPS, Auger electron spectroscopy (AES), and Fourier-transform infrared spectroscopy (FTIR) provide surface composition, oxidation states, and functional group information. Chemisorption techniques and temperature-programmed desorption (TPD) further quantify surface area and active site density. In situ Raman and infrared (IR) spectroscopy, coupled with mass spectrometry or X-ray absorption spectroscopy (XAS), enable real-time observation of catalytic mechanisms under working conditions. These tools are pivotal for revealing transient intermediates and reaction kinetics.

According to Joshi et al., MoS_2 catalysts were created and placed on carbon fiber (CF) materials to generate solar hydrogen via membrane-less electrochemical water splitting. Three separate peaks at around 33°, 44°, and 65° were identified in the XRD patterns of the CF–MoS_2 sample. These peaks were corresponding to the (100), (104), and (200) hexagonal MoS_2 (2H-MoS_2) reflections, respectively. This confirms what has been reported before. As a result of less stacking, the MoS_2 nanosheets grown on CF exhibited a weak and broad (200) diffraction peak. Diffraction peaks for the (002), (100), (104), and (200) reflections were used to describe the nanostructured 2H-MoS_2 CF–MoS_2. In the XRD pattern of the (100), (104), and (200) planes of the hexagonal MoS_2 phase were identified by four distinct diffraction peaks at 33.1°, 44°, and 68.7°, respectively. The XRD pattern showed a notable peak at 25.6°, which is the diffraction pattern often seen in CF-generated amorphous carbon (JCPDS Card no. 37–1492) [116].

In situ DRIFTS could be a promising technique to clarify the reaction mechanism of catalyst since spectroscopy could continuously and directly observe the change in the specimen. For example, in situ DRIFTS was carried out on Au/SiO_2 containing functional SiO_2 -X (X =–Schiff, –NH_2) catalysts -X support and Au-NPs active center [117]. As shown in Figure 15 (a), Au/SiO_2 -NH_2 exhibited distinct CO adsorption and the formation of ionic carbamate species. In contrast, a new adsorption peak was observed in Au/SiO_2 -Schiff besides the peaks of Au/SiO indicating a new adsorption pattern for CO_2 -NH_2, (i.e., the surface-bonded carbamate zwitterion intermediates). Interestingly, after the CO evacuation, the quick disappearance of this new band suggested its instability, and the vulnerability to be easier removed than ionic carbamate species (Figure 15 (b)). Following this, the further in situ DRIFTS carried under the H_2 atmosphere showed that the formate product could only be found with the presence of surface-bonded carbamate zwitterion instead of the ionic carbamate (Figure 15 c & d)). These results indicated that the micro- environment building by support could influence or even decide the reaction process. However, the advantage of the FTIR technique could also be the shortage, i.e., organic impurities also induce the interference signal due to the high sensitivity of FTIR [118].

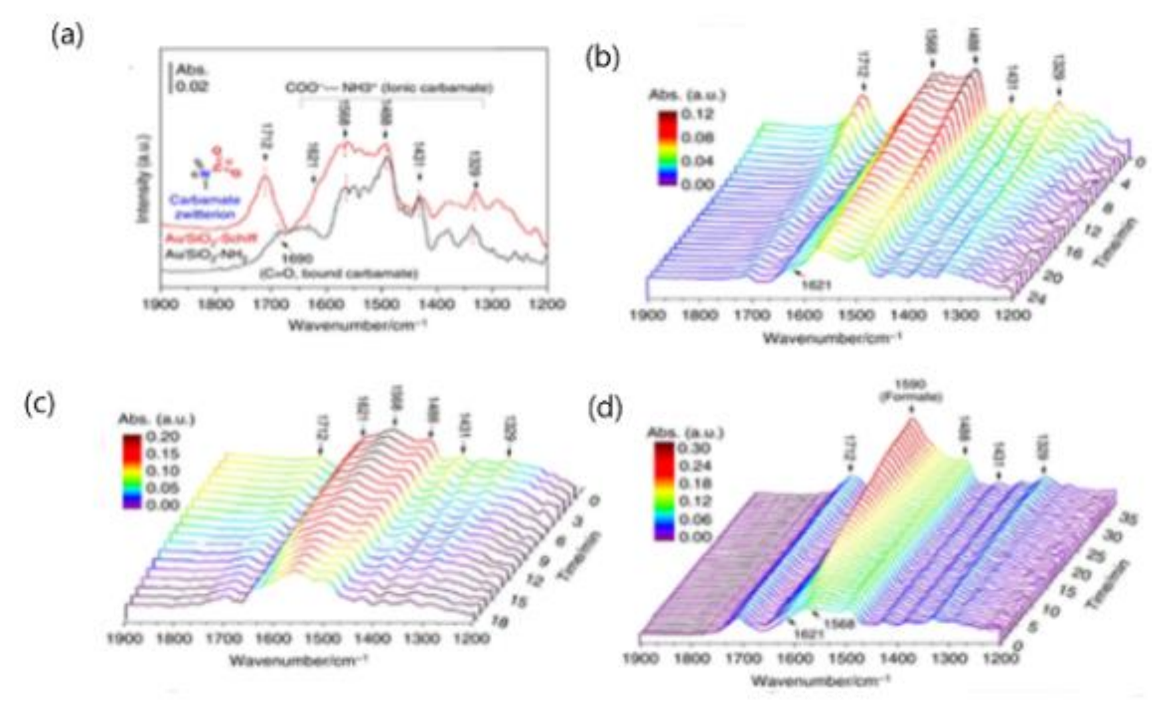

Figure 15 (a) FTIR of Au/SiO_2 and Au/SiO_2–Schiff under humid CO_2; (b) Time-resolved DRIFTS post wet-CO_2-NH_2 evacuation; (c,d) In situ DRIFTS of CO_2 hydrogenation on Au/SiO_2–Schiff without (c) and with (d) carbamate zwitterion [117]

4.4. Energy Storage Systems

Inorganic multifunctional nanomaterials play vital part in energy storage, energy generation, energy saving, energy conversion as well as in energy transmission applications owing to their distinctive properties, like chemical stability, higher surface area, outstanding thermal and electrical conductivity. Lower toxicity, lower cost, more functions and higher performance are the path being developed for future energy related applications using inorganic multifunctional nanomaterials. Despite they have terrific potential possibility, the scientific research community yet wants to make huge efforts to address their execution as well as achievement in extensive implementations. Inorganic multi-functional nanomaterials, mechanism of working is currently remains fuzzy. Therefore, to get more knowledge of the properties-structure relations of inorganic multifunctional nanomaterials, in depth and comprehensive characterization employing cutting-edge tools and techniques coupled with scientific research interpretation models ought to be altered. The features of materials have a direct impact on how devices work [119].

Excitingly, in the last several years, the rapid development of various material characterization and electrochemical analysis techniques makes it possible and easy to obtain an in-depth mechanism understanding of Li–S battery systems. Generally, the former was used to probe the structure or components of the electrode/electrolyte under in situ or ex situ conditions, while the latter mainly focuses on the overall electrochemical reaction and device performance. Usually, material characterization techniques work together with electrochemical analysis methods, and provide the complementary information to each other, especially for the in situ and in operando characterization experiments. Usually, in situ means in position, while in operando means a special case of in situ, where the battery is in operation [120].

X-ray absorption spectroscopy (XAS), as a typical example of advanced characterization techniques, has played a critical role in promoting the mechanism understanding of Li–S battery systems [121]. Specifically, ex situ/in situ XAS experiments not only accelerate the understanding of chemical/electrochemical reaction mechanism in Li–S batteries, but also contribute to the identification of Li poly sulfides and radical anions upon cycling. In addition, other advanced characterization techniques, such as X-ray diffraction (XRD), UV–visible spectroscopy, Raman spectroscopy, Fourier transform infrared spectroscopy (FTIR), nuclear magnetic resonance (NMR), electron paramagnetic resonance (EPR), X-ray photoelectron spectroscopy (XPS), transmission electron micros copy (TEM), transmission X-ray microscopy (TXM), etc., combined with ex situ or in situ experimental methods also provided complementary information to each other in understanding the structure changes, morphology evolutions, as well as the behavior of sulfur and polysulfides in Li–S batteries [122].

Trocoli et al. [123] created rechargeable micro batteries that use $LiMn_4$ (LMO) and zinc, which have high specific power. Figure 16a-d shows surface and cross-sectional SEM images of the films. Grain structures in the samples are stick-shaped and randomly orientated; the thicker the LMO1000 layer, the greater the grain dimensions. Faceted crystals seem associated with grain elongation, suggesting a preferred development direction. The samples LMO400 and LMO1000, which range from 400 to 1000 nm, respectively, have similar thicknesses as evaluated by ellipsometry. An LMO/Li_2SO_4 /Zn complete battery was built using these electrodes, with the help of an aqueous electrolyte and a zinc metallic foil that was 1 µm thick.

Schwieters et al. [124] used ICP techniques to study graphite electrodes from old lithium-ion batteries to determine how much lithium was lost in the SEI. The methods used were laser ablation, inductively coupled plasma mass spectrometry (LA ICP-MS), and inductively coupled plasma optical emission spectroscopy (ICP-OES). The main goal was to determine how much lithium was used up during the formation of SEIs, so aged graphite electrodes were analyzed objectively for lithium concentration. It was found that a large amount of lithium was lost during the initial stages of lithiation before graphite intercalation compounds like LiC30 were formed.

X-ray tomography is dealing in a unique field for characterization and that is the characterization of electrochemical devices. Lithium ion-batteries, solid oxide fuel cells and polymer electrolyte fuel cell are some of the main leaders in this category [88]. Due to the volumetric nature of the XCT, it emerges as one of the most effective and efficient technique in determining the complex internal features especially in case of parts manufactured by the additive manufacturing process. Large research is going on to firmly established the X-ray tomography as a measurement tool compared to other methods of dimensional metrology and making it industrial pertinent technology [125].

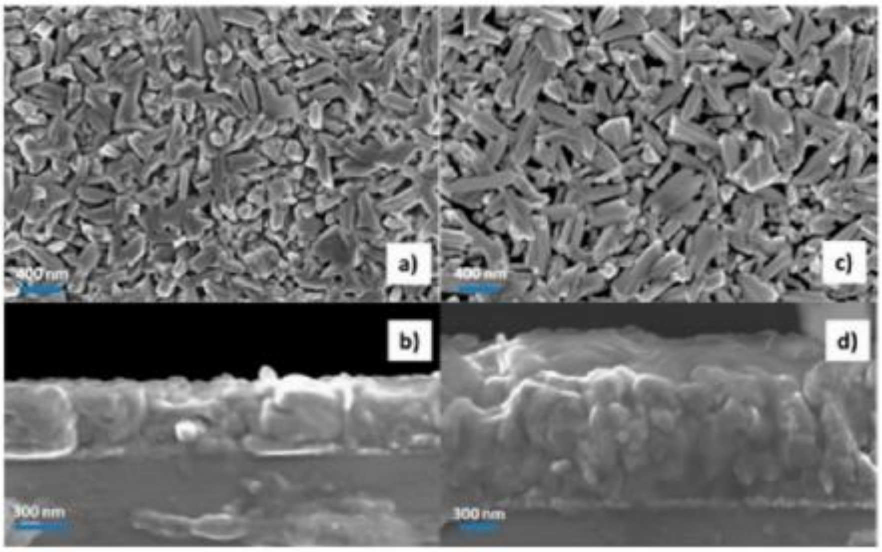

Figure 16 Top and cross-sectional SEM micrographs of the LMO electrode. (a) LMO 400 top. (d) LMO 1000 top. (b) LMO 400 cross section. (c) LMO 1000 cross section. Adapted from [123]

4.5. Environmental Applications

In recent years, the environmental pollution concern has intensified due to high living standard and consumer demand. For example, the disposal of toxic heavy metals, evaluation of toxic greenhouse gases, oil spillage, use of non-biodegradable materials, pesticides spreading, use of fertilizers, sludge and waste of other hazardous materials are posturing serious threats to the environment [126]. Emerging contaminants (ECs) or micro-pollutants are a vast and expanding array of anthropogenic compounds that are commonly present in water, but only recently has been identified as significant water pollutants. These contaminants contain a variety of extensively used synthetic compounds including cosmetics, pesticides, pharmaceuticals, steroid hormones, and personal care products, etc. that are indispensable in modern society around the globe. It is estimated that the worldwide production of these contaminants has been increased from 1 million to 500 million tons per year [127].

Most of the ECs compounds can be dissolved in water because of their chemical structures and are a potential danger for the aquatic life, environments, and humans through water cycle [128]. Recently, there has been a continuous increase in the number of ECs comprising parent and their derivatives in various forms of water such as drinking water, groundwater, wastewater and surface water. The recognition and assessment of these ECs in various waters have turned into a key scientific task, which demands highly sensitive analytical techniques for detection at nano grams per liter(ngL^{-1}) scales. Therefore, the development of fast, responsive analytical methods for efficient monitoring and determination of wide-ranging ECs are important [127].

Different analytical techniques have been developed and used recently for the detection and determination of several types of ECs in groundwater, wastewater and surface water. Major techniques are comprised of gas or liquid chromatography coupled with mass spectroscopy. The mass spectroscopy techniques have shown significant results in the precise determination of ECs present in complex matrix samples of wastewater or surface water in recent times mainly because of sensitivity, specificity, and selectivity [129]. For instance, advanced mass spectroscopic techniques such as ion trap and triple quadrupole are used to determine ECs assessment in the ngL−1 level, whereas more developed techniques such as triple quadrupole, linear ion traps quadrupole, quadrupole-time of flight, triple quadrupole and quadrupole-linear ion trap are used for the structural interpretation of transformed products [128]. Other reported analytical techniques for ECs determination include immunoanalytical techniques, microbiological assays or capillary electrophoresis. Though capillary electrophoresis is considered as less sensitive compared to liquid chromatography technique, whereas immunoanalytical technique depends more upon antibody used and has limitations for various analytes determination simultaneously and microbiological assays are largely sample nature dependent [130]. Recently, the development of advanced analytical techniques has achieved significant improvements, but still, there is a need for more sensitive recognition and assessment of unknown compounds. Developing state-of-the-art identification techniques for unknown ECs as analytes are highly desired which could advocate low cost, accurate and redundant approaches for prompt and enhanced results compared to the expensive, laborious and time-consuming techniques. For precise determination of ECs in samples, pretreatment of the samples needs significant consideration for an analytical approach [131].

4.6. Materials for Quantum Technologies

Quantum dots are quasi zero-dimensional nanomaterial with a size less than 10nm in all three directions. This limits mobility of internal electrons into nano-scale dimensions in each direction and the quantum confinement effect comes into the role [132]. The degree of quantum confinement effect is quantified by the size and exciton Bohr radius of the quantum dots. Quantum dots exhibit composition-dependent and size-dependent optical and electronic properties. Quantum dot nanotechnology entered into the field of electronic, bioimaging, and biomedical industries owing to their increased quantum yield and good optoelectronic properties. For the application of quantum dots, various factors control their properties including stability and efficiency. In order to understand the characteristic of quantum dots, a complete collection of the data is required for which a number of analytical tools exploited for the complete analysis of quantum dots. Every characterization technique has its own importance in the analysis of quantum dots [133].

Quantum dots, single-photon emitters, and superconducting materials require ultra-sensitive techniques for precision. Photoluminescence spectroscopy, EPR, and cryo-TEM provide insights into energy levels, spin dynamics, and nanostructure integrity. Synchrotron-based X-ray techniques and scanning tunneling microscopy (STM) are used for mapping quantum confinement effects and electronic band structures, enabling precise tailoring of quantum materials for next-gen computing and sensing applications [134]. For example, using synchrotron X-ray 2D and 3D elemental imaging, Jackson et al. investigated the biodistribution of cadmium selenide quantum dots capped with zinc sulfide in living Daphnia magna. 2D/3D tomography confirmed that the QDs were localized exclusively in the gut area of the organism, and were not dissolved to any extent, based on the correlation between the Zn and Se fluorescence distributions [135]. The results provided key information for elucidating the translocation route of nanoparticles in vivo and their speciation. However, the study would have been even better if the author had determined the speciation of Zn, Se, and Cd in vivo.

In quantum dots, XRD analysis basically gives the information about the phase purity, crystal structure, and particle size From the XRD pattern, the presence of peaks confirms the presence of phase formed such as in the XRD pattern of the product formed by the hydrothermal reaction to prepare graphene quantum dots using starch shows the broader peak at 24Å confirms the formation of graphene structure [73]. In the XRD pattern of carbon quantum dots prepared by using sugarcane bagasse, apart from the formation of carbon quantum dots face-centered cubic crystal structure of carbon quantum dots has been confirmed [136] (Figure 17 (a)). Additionally, the XRD pattern can be used for the estimation of crystallite size from the quantum dots. To estimate size broadening and strain broadening, the full width at half maximum (FWHM) of the Bragg peaks as a function of the diffraction angle is analyzed [137].

TEM plays an important role during the analysis of quantum dots. TEM has been widely used for the analysis of quantum dots [138]. The absence of TEM measurement makes it difficult to confirm the shape of obtained quantum dots. From TEM measurements synthesis of elliptical, circular, quadrate, hexagonal, and triangular-shaped graphene quantum dots have been confirmed. The presence of crystalline single or few-layered graphene in the graphene quantum dots with their dimensions has been confirmed by Pan et al. for the first time in 2010 [139]. A quasi spherical-shaped carbon quantum dots with the size of less than 20nm and hollow structured is confirmed using TEM studies [140, 141]. The TEM study also gives information about the range of sizes of quantum dots exists in the sample. TEM performed by Jing et al. to study as prepared carbon quantum dots showed the smooth size distribution of quantum dots lies in the range of 1.5 nm–4.0 nm along with the inter-planar space in the high-resolution TEM image corresponding to the facet of graphitic carbon [142] (Figure 17 (b)). The SEM measurement of In_2S_3 quantum dots performed by Li et al. shows the agglomeration of quantum dots forming compact structures explained by the surface energy minimization [143] (figure 17 (c)). The clustering of copper quantum dots has been confirmed along with the absence of any impurity which can be formed during the synthesis by the SEM-EDX measurement [144]. Likewise, dynamic light scattering (DLS) is frequently used technique for the particle size analysis using colloidal solution of quantum dots. DLS is an accurate and rapid measurement performed on the liquid phase of the sample. The diffusion of colloidal particles of a solution leads to dynamic change in the monochromatic light forms the basis of DLS. However, for the determination of core size distribution in quantum dots, it is difficult to access using DLS as ligand shell cannot be neglected. Also, the scattering intensity of particles below the size of 10nm is very small [145]. The detailed DLS measurement was performed by Kalwarczyk et al. to calculate hydrodynamic radius of CdSe/ZnS quantum dots. The results indicated two types of hydrodynamic radius ascribed to the single quantum dots and aggregates of quantum dots [146].

Photoluminescence spectroscopy gives the information about the size distribution of the quantum dots. The study performed by Narayanan et al. change in excitation wave length leads to the insignificant change in PL spectra confirming narrow dispersion on the size of CdS [147]. The luminescence intensity is a very important factor for the optical application of the quantum dots which can be obtained from the PL [148]. Apart from above-mentioned information, PL also give information about the extent of coupling of excitons present in the quantum dots. A study performed on $CsPbBr_3$ quantum dots shows the sensitivity toward temperature and size of the quantum dots suggesting strong coupling of excitons to the vibrational degrees of freedom present in the lattice and the surface of the $CsPbBr_3$ quantum dots [149] (Figure 17 (d)). The detailed PL study of carbon quantum dots has been performed and compared with the graphene quantum dots. The PL study gives the information about the quantum yield of quantum dots helping in determining their application as optoelectrical properties. The quantum yield of carbon quantum dots was found to be less than 10% by different research studies due to emissive surface traps which have been reduced by surface passivation confirmed from the PL study. The presence of layered structure and better crystallinity in graphene quantum dots is responsible for greater quantum yield [150].

Also, Raman spectra help in determining the bonds present in the as-prepared quantum dots and it’s very accurate that small amount of impurity can also be detected. This is best technique to study carbon-related quantum dots specially graphene quantum dots. In graphene, two bands at 1350 cm^1 and 1580 cm^{-1} in the Raman spectra corresponding to defect band (D-band) and sp2 hybridized band (G-band), respectively. These two bands ratio provides the information about the quality of graphene quantum dots. The strong intensity of G-band with respect to D-band indicates low disorder in the graphene quantum dots [148]. Raman experiments performed on GaAs-AlGaAs quantum dots assist in determining the many-particle interactions in the quantum dots along with the identifying the electronic transitions [151]. The study performed by Kumar et al. to synthesize carbon quantum dots confirms the graphitic nature and the purity of the as-prepared carbon quantum dots using Raman spectra [152].

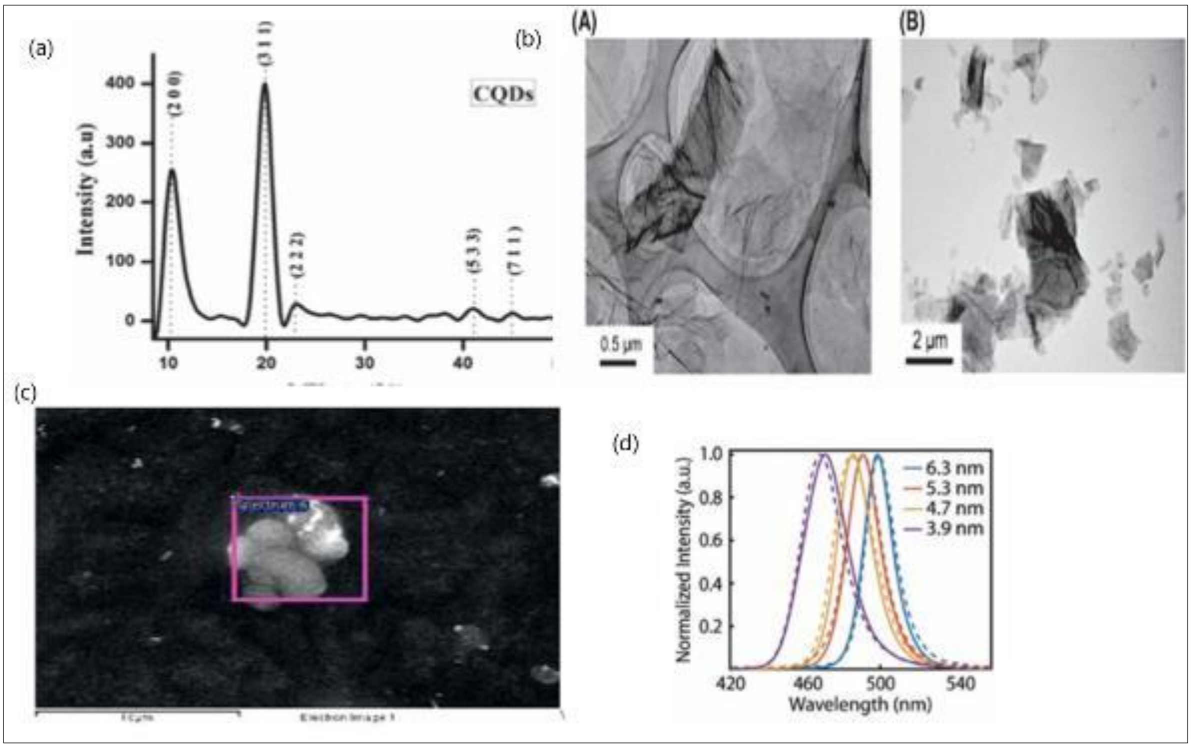

Figure 17 (a) Characterization of Quantum dots a. XRD pattern of the synthesized CQDs [136]; (b) (A,B) TEM images of the pristine and oxidized GSs, respectively [142]; (c) SEM image of metallic copper quantum dots; (d) PL spectra of CsPbBr3 QDs in hexane (solid line) and on Sapphire substrate (dash line) [149]

5. Challenges and Future Directions

Despite the remarkable advancements in material characterization techniques, several challenges persist that limit their widespread application and effectiveness. These challenges are multidimensional, spanning technical, economic, and practical domains, and they critically influence the pace at which new materials can be developed and optimized. One of the foremost challenges is the resolution-sensitivity trade-off that affects many techniques. High-resolution methods, such as transmission electron microscopy (TEM) or scanning tunneling microscopy (STM), often require stringent sample preparation and operate under vacuum or cryogenic conditions, which may not accurately reflect the materials' behavior under ambient or working environments. On the other hand, in-situ and in-operando techniques, while capturing real-time dynamics, frequently suffer from lower resolution or limited spectral ranges, making it difficult to probe atomic-scale changes with sufficient clarity.

Also, Cost and accessibility represent another major barrier. Sophisticated instruments such as synchrotron radiation sources, neutron scattering facilities, or advanced cryo-EM platforms require substantial capital investment and operational expertise. This restricts their usage to a few high-end laboratories or national facilities, limiting access for many researchers, especially those in developing regions. Additionally, the cost of routine operation and maintenance further exacerbates this challenge, discouraging broader adoption even in relatively well-funded institutions. Likewise, integration and compatibility between different characterization platforms remain underdeveloped. Multimodal and correlative techniques often demand that samples be transferred between systems or probed using non-standard geometries, which may introduce inconsistencies or sample degradation. Developing universally compatible platforms and standard operating protocols is still a work in progress, requiring cross-disciplinary collaboration between instrument developers, chemists, physicists, and engineers.

Data overload and interpretation complexity are growing concerns with the advent of high-throughput and automated techniques. The sheer volume of data generated necessitates robust data management systems and advanced analytical tools, such as machine learning (ML) and artificial intelligence (AI), to extract meaningful insights. However, the adoption of AI/ML is hindered by the lack of curated datasets, domain-specific algorithms, and user-friendly interfaces, making it difficult for non-experts to apply these tools effectively. The absence of effective methods for exploring the wide range of potential candidate structures and elemental compositions hampers the discovery of innovative materials. The development of inorganic multifunctional nanomaterials is closely tied to the presently hot scientific research approach, artificial intelligence. Al is being used in the area of materials to screen various element combinations in a predefined framework, which is an innovative way to mimic material features and identify novel materials. Currently, elemental combinations are screened based on human bias, and the hunt for new potential materials is still empirical. Researchers frequently concentrate on the fascinating combinations of numerous elements. Al can effectively sort through every potential combination of components, producing a limited number of potential materials and significantly increasing the effectiveness of searching for new desire materials [119].

Future directions in material characterization aim to address these challenges through several transformative strategies. First, the development of hybrid instruments capable of integrating multiple modalities (e.g., spectroscopy and microscopy) in a single platform will offer a more holistic understanding of materials. These integrated systems will reduce inter-instrument inconsistencies and facilitate simultaneous spatial and compositional analysis. Second, miniaturization and portability of high-performance instruments are expected to democratize access to advanced characterization. For instance, benchtop NMR, portable X-ray analyzers, and compact mass spectrometers are increasingly becoming available, offering reasonable performance for field applications and smaller laboratories. Third, the increased use of automation and robotics in material testing—combined with AI-guided experimental design—can accelerate discovery and reduce human error. These systems will be capable of autonomous operation, from sample preparation to data interpretation, enhancing reproducibility and efficiency. Lastly, interdisciplinary collaboration and standardization will be critical in shaping the future of material characterization. Collaborative databases, open-access repositories, and unified data formats will help streamline knowledge sharing and reproducibility, fostering a more integrated research ecosystem.

6. Conclusion

Material characterization is an indispensable pillar in the development, optimization, and application of both organic and inorganic materials. As the complexity of materials continues to grow in response to emerging technologies in electronics, energy, healthcare, and the environment, the demand for precise, high-resolution, and multifunctional characterization techniques intensifies. This review has highlighted a wide array of traditional and advanced characterization techniques, underscoring their principles, advantages, and applications across different material systems.

Emerging trends, including in-situ and operando methods, multimodal and high-throughput techniques, and the integration of artificial intelligence, are reshaping the landscape of materials research. These innovations offer unprecedented insights into structure-property relationships, enabling more informed design of materials with tailored functionalities. However, the adoption and implementation of these advanced methods are constrained by challenges such as instrument cost, data complexity, technical expertise, and a lack of standardization.

Looking ahead, the future of material characterization lies in the convergence of automation, integration, and intelligence. Innovations in portable instrumentation, real-time analytics, and AI-driven workflows are expected to revolutionize how materials are studied, leading to faster discoveries and more sustainable applications. As the field moves forward, fostering interdisciplinary collaboration, building open data infrastructures, and developing user-friendly tools will be essential for bridging the gap between characterization capability and practical deployment. Ultimately, these efforts will empower researchers to unlock the full potential of next-generation materials for societal benefit.

Compliance with ethical standards

Disclosure of conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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