**NANOMATERIAL CHARACTERIZATION TECHNIQUES**

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**ABSTRACT**

What nanotechnology is going to do? It is predicted that nanotechnology will alter how we live as it has already influenced the modern technology. Nanomaterials have sparked significant interest across many scientific and industrial areas because of their distinctive features and applications. Accurate characterization of nanoparticles is essential to comprehending their structure, properties and behavior as nanotechnology develops. Any technology that works with materials at the nanoscale and has different biological, chemical, and physical properties from those of the bulk is referred to as nanotechnology. The fundamentals of instrumentation and their uses in the characterization of nanomaterials will be covered in this chapter. Among the various nanotechnologies, nanomaterials are characterized with instrumentations including dynamic light scattering (DLS), X-ray photoelectron spectroscopy (XPS), scanning electron microscope (SEM), secondary mass ion spectrometry (SIMS), transmission electron microscope (TEM), high resolution transmission electron microscopy (HRTEM), atomic force microscope (AFM), X-ray Diffraction (XRD) and Raman micro spectroscopy (Micro-Raman). In addition to imaging techniques, spectroscopic methods are crucial for understanding nanomaterial properties at the atomic and molecular levels. The spectroscopic techniques provide valuable insights into the chemical composition, crystal structure, and functional groups present in nanomaterials. The chapter concludes by highlighting the complementary nature of various nanomaterial characterization techniques and the importance of employing multiple methods for comprehensive material analysis. As nanotechnology continues to evolve, advancing these characterization techniques is vital for realizing the full potential of nanomaterials in diverse fields, including medicine, electronics, energy, and environmental applications.

**Keywords**—nanomaterials; nanomaterial testing; dynamic light scattering (DLS); x-ray photoelectron spectroscopy (XPS); secondary ion mass spectrometry (SIMS); raman micro spectroscopy; characterization of nanoparticles.

1. **INTRODUCTION**

Nanomaterials, with their unique size-dependent properties, have opened up a plethora of possibilities for revolutionizing various scientific, industrial, and technological fields [1]. However, as materials scale down to the nanoscale, they exhibit novel characteristics that can be vastly different from their bulk counterparts. Consequently, accurate and comprehensive characterization techniques are crucial for understanding and harnessing the potential of nanomaterials [2-3]. Nanomaterial characterization techniques encompass a diverse array of analytical tools that aim to probe and quantify the structural, chemical, physical, and optical properties of nanoscale materials [4-5]. These techniques not only enable researchers to gain deeper insights into the intricate details of nanomaterials but also facilitate the design and optimization of novel nanoscale structures for specific applications. The characterization of nanomaterials presents unique challenges owing to their small dimensions, often ranging from a few nanometers to a few hundred nanometers. Traditional characterization methods designed for bulk materials are often inadequate at the nanoscale due to limitations in sensitivity and resolution [6]. Therefore, researchers have developed a specialized suite of techniques tailored to handle nanomaterials and unveil their inner workings. The following sections of this review will delve into various nanomaterial characterization techniques, exploring their principles, capabilities, and limitations. From high-resolution imaging to spectroscopic analysis and dynamic measurement, these techniques play complementary roles in unraveling the mysteries of nanomaterials. As nanotechnology continues to advance, the ongoing development and refinement of nanomaterial characterization techniques will undoubtedly drive innovation and further unlock the vast potential of nanomaterials in shaping the future of science and technology.

1. **EVOLUTION OF NANOTECHNOLOGY**

The primary processes used in nanotechnology involve the consolidation, separation, and deformation of materials by a single molecule or atom. In a speech titled "There's Plenty of Room at the Bottom" from 1959, Nobel Prize-winning American physicist Richard Feynman proposed the concept of nanotechnology [7]. The researcher Norio Taniguchi first used the word "nanotechnology" in his 1974 study on the use of synthesis technology to produce things and characteristics with nanometer-scale dimensions [8]. For the advancement of nanotechnology as a field, the respective discoveries of scanning tunneling and atomic force microscopes in the 1980s are seen as turning points. These microscopes made it possible to image materials at the atomic level, which is essential for altering matter at the atomic and molecular levels. Parallel developments in computer technology made it feasible for supercomputers to simulate and analyze materials on a massive scale, giving rise new insights into both the structure and characteristics of the materials [9]. The simultaneous modeling, visualizing, and manipulating activities had a significant impact on research studies in the 20th century.

1. **CHARACTERIZATION OF NANOMATERIALS**

Nanomaterial characterization refers to the process of analyzing and understanding the properties, structure, and behavior of nanomaterials at the nanoscale. This crucial step involves using various techniques to gather data and obtain insights into the nanomaterial's composition, size, shape, surface characteristics, crystal structure, and other relevant parameters. Effective nanomaterial characterization is essential for designing, optimizing, and tailoring nanomaterials for specific applications in fields such as nanomedicine, nanoelectronics, energy, catalysis, and more [10]. Nanomaterial characterization techniques are diverse and often require specialized instruments and expertise. Some common techniques used in nanomaterial characterization includes:

1. **Imaging Techniques**
* Scanning Electron Microscopy (SEM): Offers surface imaging and morphology analysis of nanomaterials.
* Transmission Electron Microscopy (TEM): The interior structure of nanomaterials can be seen in high-resolution photographs which reveals atomic-level features.
* Atomic Force Microscopy (AFM): Creates surface topographical images and measures forces at the nanoscale.
1. **Spectroscopic Techniques**
* X-ray Diffraction (XRD): Determines the crystal structure and phase composition of nanomaterials.
* Fourier-Transform Infrared Spectroscopy (FTIR): Identifies chemical bonds and functional groups in nanomaterials.
* Raman Spectroscopy: Provides information about molecular vibrations and composition.
1. **Surface Analysis Techniques**
* X-ray Photoelectron Spectroscopy (XPS): Examines the oxidation states and chemical makeup of nanomaterials' surfaces.
* Secondary Ion Mass Spectrometry (SIMS): Provides surface elemental and isotopic information.
1. **Dynamic Measurement Techniques**
* Dynamic Light Scattering (DLS): Measures the size distribution of nanoparticles in a liquid medium.
* Zeta Potential Measurement: Assesses the surface charge and stability of nanomaterials.
1. **Mechanical Characterization Techniques**
* Nanomechanical Testing: Determines the mechanical properties of nanomaterials, such as hardness, elasticity, and tensile strength.
1. **Thermal Analysis Techniques**
* Nanoscale Thermal Analysis: Measures the thermal properties of nanomaterials, including thermal conductivity and heat capacity.

These techniques, among others, play a crucial role in characterizing nanomaterials and understanding their unique properties at the nanoscale. The information obtained from nanomaterial characterization is valuable for optimizing nanomaterial synthesis processes, evaluating their performance in specific applications, and ensuring their safe use in various industries [11]. As nanotechnology continues to advance, the development of more sophisticated and precise characterization techniques is essential to unleash the full potential of nanomaterials in shaping the future of science and technology.

1. **CHARACTERIZATION OF NANOMATERIALS BY IMAGING TECHNIQUES**

Characterization of nanomaterials by imaging techniques is essential for gaining insights into their morphology, structure, and size distribution. These techniques provide visual information about the physical features of nanoparticles and nanoscale structures, enabling researchers to understand their properties and behavior. Several imaging techniques are commonly employed for nanomaterial characterization:

**ELECTRON MICROSCOPE**

Electron Microscopes are scientific devices that use a beam of extremely energetic electrons to study objects on a precise scale. This kind of examination can provide information regarding the topography, morphology, composition, and crystallography of the object [12]. Due to physical limitations, light microscopes could only magnify objects by 500x or 1000x and had a resolution of 0.2 micrometers; as a result, electron microscopes were developed [13]. With the difference that a focused stream of electrons is used to "see through" the object, the Transmission Electron Microscope (TEM), the first type of electron microscope to be invented, is based directly on the Light Transmission Microscope [14]. In Germany in 1931, Max Knoll and Ernst Ruska invented the gadget. The first scanning electron microscope (SEM) was created in 1942, but the first piece of equipment that was made available for purchase was not until the middle of the 1960s [14].

The brief of description of the two-electron microscope is given below:

 **A**. **Scanning Electron Microscope**

The SEM scheme was first put forth by H. Stintzing in a 1927 German patent application. Because the material was exposed to light, X-rays, and corpuscles in a collimated beam, his suggested method was unable to produce a magnified image. Then, in 1935, a German electrical engineer by the name of M. Knoll made a contribution to the SEM paradigm where a specimen was scanned with an electron beam to produce a picture. By introducing DE magnifying lenses to the scanning transmission electron microscope, which was intended to scan thin samples, Von Ardenne modified the SEM slightly in 1938 [15]. Zworykin improved SEM for scanning large samples in 1942, making only a few other changes. SEM was eventually commercialized in 1965 after several changes were made in the Oatley Lab's research and development department [16].

**Principle**: SEM is a type of electron microscope that uses razor-sharp electron beams to scan things' surfaces in order to capture images of them. In this method, the atoms of an object interact with electrons to produce signals that show the object's topography and composition. The configuration of the constituent atoms is examined utilizing 2D beam scanning on the sample surface and picture collecting from acquired secondary electrons. The position of the electron beam with relation to the signal being detected and the scan pattern it produces are combined to form an image [17].

**Instrumentation:** The basic components used in electron optical microscope are:

**Table 1: Components and Infrastructure Requirements of SEM.**

|  |  |  |
| --- | --- | --- |
| **Sr. No.** | **Components** | **Infrastructure Requirement** |
| 1. | A source of electron | Power supply |
| 2. | Lenses | Vacuum system |
| 3. | Scanning coils | Cooling system |
| 4. | Detectors to collect signals | Vibration free color |
| 5. | Sample stage | Room free of ambient electric and magnetic field |
| 6. | Display/Data output stages |  |

SEM scans, however, can show the nanomaterials in three dimensions since they show surface features rather than core structure. The maximum resolution of images taken by SEM is typically 5 nm [18].

**Recent Application in Research**

* In a study conducted by Jonas & Matthias, they discussed that convolutional neural networks (CNNs) were used as the foundation of a deep learning (artificial intelligence) method that automates the examination of nanoparticles that have been imaged by scanning electron microscopy. Even overlapping or contacting particles were frequently separable when nanoparticle SEM images were segmented into coherent foreground areas (particles) and background. Both two-dimensional scanning transmission electron micrographs (STEM) and pseudo-three-dimensional secondary electron micrographs (SE) can be used to provide quantitative data on particle size distributions and shape. The particles were removed from the image and categorized by form (such as sphere or cube) after being separated from the backdrop (segmentation) [19].
* Ghada Dahy in her study put up an intelligent optimization approach to classify several nanoparticle kinds found in SEM images, including lines, junctions, networks, ellipses, and circles. The proposed approach can be divided into four independent phases: preprocessing, feature extraction, feature selection, and classification. All specific results show that the suggested intelligent optimization model may create a high-performance classifier for various types of nanoparticles in SEM images [20].
* Hayat in her work studied the surface structure of the produced AgNPs using scanning electron microscopy. The micrographs indicated agglomeration as well as the spherical shape of the produced NPs. In order to attain stability, a typical phenomenon known as agglomeration is seen in AgNPs. Spherically shaped particles range in size from about 150 nm to 200 nm on average, but larger particles have also been discovered. A wide range of particle sizes, in addition to instability and agglomeration, is a significant problem. When working with NPs, the surface of the particles seemed rough, with irregular borders and an asymmetric texture. The average size of the nanoparticles, which was determined to be between 55 and 65 nm by the XRD, EDX, and SEM peaks, showed that the nanoparticles naturally possess an FCC (Face- Centered Cubic) structure. The zone of inhibition tests and their efficacy against a variety of bacterial strains have all shown that AgNP stability has increased over time [21].
* Saleh & Hassanstudied novel nanomaterials with applications in electrochemical devices, such as MXenes, covalent organic frameworks (COFs) and metal-organic frameworks (MOFs),), and same were synthesized. SEM was used to examine the interaction between biosensors and nanomaterials, both of which are crucial components of electrochemical devices. The SEM results demonstrated the importance of nanomaterials in the development of low-cost electrochemical systems for energy conversion and storage [22].
* Stavila and other members in their study have utilized SEM to examine the interactions of nanoparticles with biological cells. This research is crucial for understanding the potential toxicity and biocompatibility of nanoparticles in nanomedicine and bioimaging applications[23].

**B. Transmission Electron Microscopy**

In electron microscopy, which has sub-nanometer resolution potential, electron beams serve as the light source. Nanomaterials' incredibly minute details are magnified as a result. A high voltage electron beam that is produced by a cathode and is focused by a lens is used in transmission electron microscopy (TEM). The sample is placed in a vacuum at first. The high voltage electron beam first partially transmits through the sample before focusing and amplifying the transmitted electrons. The beam's impact on a phosphor screen, photographic plate, or other light-sensitive sensor produces an image. The only output of TEM is a pair of two-dimensional images of the sample; however, these images and diffraction patterns provide information on the interior structure of the materials [24].

**Principle:** In this procedure, an electron beam is sent through a sample that is so thin that it interacts with the material, creating an image as a result. To be recognized, this produced image can then be magnified and focused on a sensor like a CCD camera, a layer of photographic film, or a fluorescent screen [25].

**Instrumentation:** The basic components used in Transmission electron microscope are:

**Table 2: Components of TEM.**

|  |  |
| --- | --- |
| **Sr. No.** | **Components** |
| 1. | E- Source  |
| 2. | Gun (Thermionic Emission) |
| 3. | Electron Beam |
| 4. | Lenses |
| 5. | Vacuumed Chamber |
| 6. | 2 condenser lenses, objective and intermediate lens |
| 7. | Holder/Stage for Sample |
| 8. | Phosphor or fluorescent screen (Imaging Device) |
| 9. | Computer |

**Recent Application in Research**

* Shimojima along with two others highlighted how combining scanning transmission electron microscopy with the ultrafast optical pump-probe approach boosted the time resolution for differential phase contrast and convergent-beam electron diffraction imaging by a factor of ~1012. These methods provide ultrafast nanoscale images of crystal lattice deformation, magnetization vector, and electric field, among other physical properties of nanomaterials. These methods were used to illustrate it in observations of the photo-induced acoustic phonon propagation with precisions of 4 ps and 8 nm and the ultrafast demagnetization under zero magnetic field with resolutions of 10 ns and 4 ps [26].
* In a study of Zhefei Sun along with nine membersshowed how the most recent significant advancements in the use of in situ TEM for gaining a fundamental understanding of the materials and interface difficulties in All-solid-state lithium batteries (ASSLBs) are routinely carried out, with a focus on real-time observations of reaction and degradation occurring in solid electrolytes, electrodes, and their interfaces. The important real-time data and scientific breakthroughs enabled by in situ TEM techniques were stressed. Determining the ASSLBs' degradation processes using in situ TEM poses both considerable challenges and opportunities [27].
* Saied and others in their **work** noted that nanoparticles implanted in nanocomposite materials are studied using physiochemical analysis via TEM, which reveals details about their distribution and interactions with the matrix [28].
* Sun,J and teamdemonstrated that metal cluster intermediates combine to generate HEA (High entropy alloy) nanoparticles using the techniques of mass spectrometry (MS), (LPTEM) liquid phase transmission electron microscopy (LPTEM) and systematic synthesis. AuAgCuPtPd HEA nanoparticles are produced by the aqueous co-reduction of metal salts with sodium borohydride in the presence of thiolated polymer ligands. The presence of stable single metal atoms and sub-nanometer clusters in the final HEA nanoparticle solution is intriguing since it shows nucleation and growth may not be the main mechanism [29].
* Carone along with four more members, in their studydemonstrated the use of inter-particle plasmonic interaction, which allows for the modification of optical properties, was described. Plasmonic nanoparticle spatial organization is of particular interest. For bottom-up approaches that employ controlled self-assembly to produce increasingly complex structures by destabilizing colloidal particles, colloidal nanoparticles are intriguing building blocks. The behavior of the particles may be explained by displaying stability diagrams of colloidal gold nanostructures and taking into account elements like shape, size, and CTAB/AuNP concentration. They found that the overall stability of the nanoparticles was affected by their morphology, with sharp ends being the primary source of instability. Transmission electron microscopy was utilized to investigate the behavior of the system in each of the zones using a number of methods [30].

**C. Atomic Force Microscopy**

Binning invented the atomic force microscope (AFM) in 1986. AFM is currently used to map and examine the three-dimensional surface topography of a variety of materials, including metals, semiconductors, soft biological samples, as well as conductive and non-conductive materials, in a variety of conditions, including air, liquid, and vacuum. With angstrom scale precession, AFM can provide images with atomic resolution and height resolution. AFM has recently been employed as a method for manipulating nano-objects and in nano-imprinting technology, which uses molecular ink to create features at the molecular level [31]. A subset of AFM is high-resolution scanning probe microscopy (SPM). It is a method for researching nanoparticles that is incredibly effective. The core component of an AFM system is a microcantilever that scans the sample surface with a very sharp probe (tip) at one end. Most frequently, silicon or silicon nitride is used to create the cantilever. The tip radius of a cantilever can be coated with a thin layer of gold or other metals, depending on its intended application. AFM images are built on the deflection of forces at the point of contact between the cantilever tip and sample surface. Wan der Waals force, chemical bonding, capillary force, electrostatic force, salvation force, magnetic force, and other forces can all be used to create mechanical contact [32]. In accordance with Hooke's Law, a laser mounted on top of the cantilever detects the deflection, which is then reflected into a photodiode array. Both AFM and SEM can provide 3-D images, however AFM has a few advantages over SEM. AFM can produce images with a vertical resolution of 0.02nm and a lateral resolution of 0.1 nm, whereas SEM can only achieve a resolution of about 5 nm. AFM can be used to study nanomaterials without the requirement for further sample preparations that could damage the sample. The majority of AFM performs well in a liquid or natural air environment. As a result, it is no longer difficult to investigate biological macromolecules and living entities at the nanoscale [33-34].

**Principle:** The fundamental idea is to measure the force that interacts between the sample surface and a molecularly scaled interacting probe mounted on a flexible cantilever. Different operation modes could be developed based on the interaction process. Surface sensing, cantilever deflection detection, and image processing are the three crucial steps in AFM that work together to produce the three-dimensional topography of the sample's surface at atomic resolution [35].

**Instrumentation:** The basic components of AFM are as follows:

**Table 3: Basic Components of Atomic Force Microscopy.**

|  |  |
| --- | --- |
| **Sr. No.** | **Components** |
| 1. | Piezo Ceramic Scanner |
| 2. | Controller |
| 3. | Laser Diode |
| 4. | Sample Surface |
| 5. | Quadrant photodiode |

**Recent Application in Research**

* Group pf researchers in their study have talked about how, among other things, the shape and size of (mRNA-LNPs) mRNA-lipid nanoparticles affect their efficacy. An entirely new atomic force microscopy approach for characterizing mRNA-LNP in an aqueous medium was reported in a recent study. This technique uses an anti-polyethylene glycol antibody to immobilize mRNA-LNPs on a glass substrate without contaminating them, in contrast to conventional methods that use solid substrates like mica and glass. Spherical and bleb-like structures were visible in the mRNA-LNPs as a result of the AFM imaging, which was consistent with earlier results using cryo-transmission electron microscopy. AFM analysis also revealed the dominance of nanoparticles 60 nm or smaller, which had previously eluded detection by dynamic light scattering and nanoparticle tracking methods. Because mRNA-LNPs are often polydisperse rather than monodisperse, the AFM technique can provide useful additional information regarding these molecules in the development and quality assessment of these molecules [36].
* Farokh and Passian suggested that although it is now possible to probe material properties at surfaces down to the level of individual atoms and molecules, high-resolution subsurface imaging is still a nanometrology difficulty because of electromagnetic and acoustic diffraction. These restrictions have been broken at surfaces by the atomically sharp probe employed in scanning probe microscopy (SPM). Under specific physical, chemical, electrical, and thermal gradients existing in the material, subsurface imaging is feasible. AFM has offered particularly interesting possibilities for nondestructive and label-free measurements among all SPM techniques [37].
* Akhatova and team mates discussed concerns about microplastics and nano plastic contamination led them to discuss studies on the absorption and biodistribution of these emerging pollutants in vitro. In a study, nanomechanical Peak Force Tapping was used to examine the absorption and dispersion of polystyrene spherical microplastics in human skin fibroblasts. Surface-attached versus internalized plastics can be distinguished according to nanomechanical characterization. In whole fixed cells, particles as tiny as 500 nm were seen. This finding offers fresh opportunities for the study of microplastic toxicity [38].
* Joshi and two others have made this suggestion since AFM is currently a widely used tool for examining hydrogels and biopolymers. Even though the phase imaging mode, which is built into modern AFMs if the tapping mode is employed and provides the opportunity to sharpen edges and provide qualitative information about the material composition in the sample, it was surprisingly only occasionally detected. This is true even though it focuses mostly on surface topography and roughness. Several attempts have been made in the literature to evaluate mechanical properties in addition to topography, either by employing PeakForce QNM or nanoindentation. Rarely do biopolymers and hydrogels make use of every electrical mode. Researchers should use simpler phase imaging modes more frequently when working with hydrogels, biopolymers, and other biological samples, the study suggests, as they frequently provide significantly more information than pure topography images and don't necessitate the use of specialized cantilevers or other advanced sample preparation methods. The work's objective was to increase the variety of AFM modes available for use with future samples of this kind [39].
1. **SPECTROSCOPIC TECHNIQUES FOR CHARACTERIZATION OF NANOMATERIALS**

Spectroscopic techniques for nanomaterial’s characterization plays a critical role in understanding their chemical composition, molecular structure, electronic properties, and interactions. These techniques provide valuable insights into the behavior of nanomaterials at the atomic and molecular levels. Here are some key spectroscopic techniques commonly used for nanomaterial characterization:

1. **X-ray Diffraction**

The electromagnetic spectrum's X-ray sector encompasses wavelengths between about 0.1 and 100 Ao. The most useful region is between 0.7 and 2.0 Ao for analytical purposes. High-velocity electrons striking a metal object produce X-rays. Bohr's theory of atomic structure can be used to visualize the creation of X-rays. An effective analytical method for examining the atomic and molecular structure of crystalline materials is X-ray Diffraction (XRD). It provides valuable information about the arrangement of atoms in a solid, revealing details about crystal symmetry, lattice parameters, and the presence of different phases within a sample. XRD plays a crucial role in various scientific and industrial fields, including materials science, chemistry, geology, and nanotechnology. A crystal is considered to have perfect 3-Dimensional order in XRD. Since the structure repeats in every direction due to perfect 3-D order, the complete structure can be uniquely represented by describing it locally (in a repeating 3-d unit). However, the majority of crystalline materials include some degree of disorder that can be seen by XRD [40].

**Principle:** The basis of XRD is Bragg's law of diffraction, which stipulates that the lattice planes in a crystal lattice will diffract X-rays when they impact the lattice at a particular angle. The constructive interference of the diffracted X-rays results in a detectable and interpretable diffraction pattern. Information on the distances between lattice planes and the angles at which diffraction takes place can be gleaned from the resulting diffraction pattern [41].

**Instrumentation:** The basic components of XRD are as follows:

**Table 4: Basic Components of X-ray Diffraction.**

|  |  |
| --- | --- |
| **Sr. No.** | **Components** |
| 1. |  Source (X-Ray) |
| 2. | Sample Holder |
| 3. | Goniometer |
| 4. | Detector |
| 5. | Computer |

**Recent Application in Research**

* Muthukhatija and other researchers used XRD to examine the produced ZnO nanoparticles. By recording their X-ray diffraction pattern, the phase and crystallinity of the ZnO nanoparticles are confirmed. Diffraction peaks that are distinct and narrow imply that the product's particles have a clearly defined crystalline structure. The strong peak intensity indicates the manufactured ZnO NPs are extremely crystalline. P. Alba seeds are excellent reducing agents for produced ZnO NPs, according to this XRD investigation. The Scherrer formula was used to compute the sizes of the particles, which were found to be in the 48 nm range [42].
* After 24 hours of incubation, the created sample's color changes from pale yellow to brown, offering the first palpable sign of AgNP generation, according to their research. The color change is indicative of the presence of AgNP in the sample. The characterisation process was then used to analyze the created AgNP for additional confirmation. Due to their distinct characteristics, silver nanoparticles (AgNP) are a novel material that is electrical, superconducting, antibacterial, and biosensing. According to studies, these nanoparticles are safe for use on people and most effective against bacteria, viruses, and other eukaryotic microorganisms at low dosages without causing any negative side effects [43].
* Sambathkumardiscussed that distinct metal sulfide nanoparticles (Ni9S8, Bi2S3, Co9S8, CuS, and CdS) are produced by solvothermal process at low temperature by using (M[DTC]2) as a single source precursor and long chain amine (HDA) as a capping and shape-directing agent. The created nanomaterials are subjected to several characterizations, such as XRD, TGA, Raman, FT-IR, SEM, EDX, HR-TEM and electrochemical testing. Analysis helped to make sense of the outcomes and characteristics, including spectroscopic, structural, and morphological behaviors [44].
1. **Fourier- Transform Infrared Spectroscopy**

Fourier-Transform A potent analytical method used to investigate the vibrational modes of molecules in a variety of materials is infrared spectroscopy (FTIR). It offers useful details on a material's chemical make-up, molecular structure, and functional groups. FTIR spectroscopy is a versatile instrument used in a wide range of scientific and industrial applications because it functions based on the interaction between infrared light and molecular vibrations [45]. Characterizing nanomaterials in their different forms, such as nanocomposites, nanoparticles, nano coatings, and nanotubes, is a specialty of FTIR [46].

**Principle:** In FTIR spectroscopy, infrared light passed through a material, and the spectrum produced is identified by determining whether light at various frequencies is absorbed or transmitted [47]. When molecules vibrate in certain ways, for as by stretching or bending chemical bonds, they absorb infrared light at specific wavelengths that correspond to those modes. The functional groups and chemical bonds contained in the sample can be determined using the resulting FTIR spectrum, which is a plot of absorbance (or transmission) vs wavenumber (the reciprocal of wavelength) [48].

**Instrumentation:** The basic components of FTIR are as follows:

**Table 5: Basic Components of Fourier Transform Infrared Spectroscopy.**

|  |  |
| --- | --- |
| **Sr. No.** | **Components** |
| 1. | Infrared Source |
| 2. | Interferometer |
| 3. |  Compartment for Sample |
| 4. | Detector |
| 5. | Computer |

**Recent Application in Research:**

* Baraton & Merharihave talked about how Fourier transform infrared spectroscopy is a very adaptable tool for surface characterization of nanoparticle surfaces as long as a certain configuration is connected to the spectrometer. Under appropriate circumstances, it is possible to ascertain the chemical make-up of the nanoparticles' surfaces and to pinpoint the reactive areas of those surfaces that are in charge of the reactivity. The chemical processes taking place at the nanoparticles' surface can also be seen in action in real time as a function of a variety of variables, such as temperature and the gaseous environment. In a study, the two facets of FTIR spectroscopy are combined to follow changes in semiconductor nanoparticle electrical conductivity and surface reactions as a function of environmental variables. The development of chemical gas sensors directly benefits from the FTIR spectroscopy's unique specificity. Since tin oxide is one of the most often used materials for making semiconductor chemical sensors, it was chosen as a case study [49].
* Eid, M.proposed that the researchers can learn about the cellular composition and distribution of significant biological substances such as lipids, proteins, and nucleic acids in microscopic sections of the sample thanks to the great spatial resolution of FTIR micro-spectroscopy, which can go down to single cells. FTIR micro spectroscopy differs from other spectroscopic techniques due to its sensitivity to biomolecule conformation. FTIR imaging is a useful tool for examining the biochemistry of biological materials because of these characteristics. Numerous studies have focused on the potential of nanomaterials (NMs) as biological tools for tissue engineering, therapy, and diagnosis. ATR-FTIR has also been used to look into the toxicity of a number of nanoparticles, such as ZnO, quantum dots, and carbon nanomaterial. ATR-FTIR was used by Wang et al. to explain the nanowire photocatalysis in E. coli. The enhanced cell permeability in treated cells altered the structure of their membranes, based on ATR-FTIR measurements. It was discussed which sizes of quantum dot nanoparticles were harmful to E. coli. [50].
* Pandian and others suggested that numerous nanomaterials, such as nanoparticles and nanocomposites, have been successfully used as bioremediators because they have special physiochemical characteristics, such as dimension, shape, configuration, purity, surface qualities, etc. To adapt nanomaterials for bioremediation applications, all of these factors are crucial. Standardizing, improving, and developing characterization methods for nanomaterials is essential for enabling researchers to properly include nanotechnology applications over bioremediation resolves. In this study, FTIR was used to characterize the nanoparticles [51].
1. **Raman Micro spectroscopy**

Raman micro spectroscopy is a powerful analytical technique that combines Raman spectroscopy with microscopy, enabling researchers to obtain detailed chemical and structural information from small regions within a sample. By providing both spatial and spectral information, Raman micro spectroscopy offers insights into the composition, molecular vibrations, and interactions of materials at the microscale [52]. This technique has widespread applications in various scientific fields, including materials science, biology, geology, and nanotechnology.

**Principle:** The Raman scattering phenomenon, which happens when monochromatic light—typically from a laser—interacts with a sample—is the foundation of Raman micro spectroscopy. Due to molecular vibrations, the dispersed light experiences a shift in frequency, which reveals the material's vibrational modes and chemical connections [53]. Researchers can determine the chemical components of a sample and learn more about its molecular structure by evaluating the Raman shifts [54]. In Raman micro spectroscopy, the laser beam is focused onto a pinpoint area of the material using a microscope objective. To examine the frequency shifts, the Raman-scattered light is collected and routed via a spectrometer. The resulting Raman spectrum offers details on the chemical composition and molecular vibrations of the examined area [55].

**Instrumentation:** The basic components of Raman Micro Spectroscopy are as follows:

**Table 6: Basic Components of Raman Micro Spectroscopy.**

|  |  |
| --- | --- |
| **Sr. No.** | **Components** |
| 1. | Microscope System |
| 2. | Laser Source |
| 3. | Beam Expander & Optics |
| 4. | Raman Spectrometer |
| 5. | Detector |
| 6. | Spectral Calibration |
| 7. | Data Acquisition & Analysis Software |
| 8. | Polarization Control Module/ Temperature Controlled Stage/ Sample Holder |

**Recent Application in Research:**

* Casteneda discussed that several methods were used to characterize oxidized nanomaterials. Different concentrations of NaClO solutions were used to oxidize bismuth nanoparticles (NPs) colloids that had been produced using the laser ablation of solids in liquids (LASL) technique in deionized water. Using Raman micro spectroscopy, the crystalline phases of the bismuth complex were identified [56].
* Ragusadiscussed about a term “Plasticenta”. In a different study, Raman Micro Spectroscopy was used to identify numerous microplastic pieces in human placenta samples for the first time. Using Raman Micro spectroscopy, six human placentas that had been given by consented women who had healthy pregnancies were examined in this study to determine the presence of microplastics. Every microplastics particle's shape and chemical composition were investigated. The remaining nine could only be distinguished by their pigments and were all used in paints, synthetic coatings, adhesives, finger-paints, plasters, polymers, beauty products, and personal care products. Of the twelve, three could be identified as thermoplastic polymers made of stained polypropylene [57].
* Ngyendiscussed about Nanoparticle Tracking: Raman micro spectroscopy offers dynamic insights into the behavior of nanoparticles by allowing for the real-time tracking of individual nanoparticle motion and interactions [58].
* Gupta, P**.** suggested that many nanoparticles have coatings to make them more stable or useful. The coatings are better characterized, their thickness is examined, and their uniformity is evaluated using Raman micro spectroscopy. The development of noninvasive, extremely sensitive, and trustworthy tools for diagnosis and visualization in the realm of nanomedicine has been the focus of research in nanoimaging. Raman micro spectroscopy is regarded as an essential method for studying materials and has contributed to a number of discoveries. The characterization of manufactured NPs and their biological usage in treatments and diagnostics have made great use of microscopic methods [59].
1. **CHARACTERIZATION OF NANOMATERIALS BY SURFACE ANALYSIS TECHNIQUES**

Characterization of nanoparticles using surface analysis techniques is essential for understanding the properties and behavior of nanoparticles at the atomic and molecular levels. These techniques provide insights into surface composition, structure, morphology, and chemical interactions [60]. Here are some key surface analysis techniques commonly used for nanoparticle characterization:

1. **X-Ray Photoelectron Spectroscopy**

X-ray Electron Spectroscopy for Chemical Analysis (ESCA), commonly referred to as Photoelectron Spectroscopy (XPS), is a potent surface analysis method used for the evaluation of nanomaterials. The electrical structure, chemical states, and elemental composition of the topmost atomic layers of nanomaterials can all be learned by XPS. Studying the surface characteristics and interactions of nanoparticles is one of its main applications [61]. Here's how XPS is utilized for nanomaterials characterization:

**Principle:** The photoelectric effect is the basis for XPS operation. Core-level electrons can be ejected from atoms in the top few nanometers of a sample when X-ray photons are focused onto its surface. The energy of these expelled electrons reflects their binding energy within the sample and is characteristic of the element from which they originate. In order to determine the elemental make-up and chemical states of the sample's surface, XPS measures intensity of the electrons and the kinetic energy that are released [62].

**Instrumentation:** The basic components of X-Ray Photoelectron Spectroscopy are as follows:

**Table 7: Basic Components of XPS.**

|  |  |
| --- | --- |
| **Sr. No.** | **Components** |
| 1. | Source of X-ray |
| 2. | Sample’s Chamber |
| 3. | Stage |
| 4. | Analyzer (Energy) |
| 5. | e- Lens  |
| 6. | Detector |
| 7. | Monochromator |
| 8. | Ion Gun |
| 9. | Calibration System |
| 10. | Computer |
| 11. | Vacuum System |
| 12. | Neutralization System for Charge |

**Recent Application in Research**

* Ischenko A. discussed in their paper that when used for chemical analysis, XPS or electron spectroscopy can reveal details about the samples' qualitative and quantitative composition, the valence states of the elements, the chemical makeup of the surface, and the interfaces that shape their properties as nanoparticles and nanostructured materials. The review covers the function of several techniques for the characterization of nanomaterials, highlights their benefits and drawbacks, and discusses potential synergies. There is a description of XPS's primary features. Examples of its application for the investigation of nanoparticles and nanomaterials are provided, along with other techniques to gather further data on the subject under research. XPS records the charging/discharging behavior of nanomaterials while also revealing their chemical composition and dielectric characteristics. The thickness of nanoparticle coatings can be estimated using chemical information from the surface of nanoparticles that have undergone XPS analysis. Since the resolution of the approach allows for the differentiation of a distinctive set of lines in the photoelectron spectra at kinetic energies dictated by the photon energy and the matching binding energies in elements, XPS has a high degree of selectivity. The lines' thickness varies depending on how much of each element is present. A collection of complimentary instrumental methods of analysis must be used in order to get a sufficiently thorough picture of the properties of nanomaterials [61].
* In their study, Garza and colleagues examined the shell thickness of core-shell nanoparticles using Shard's methodology, a simple and non-iterative method that enables the conversion of x-ray photoelectron spectroscopy's peak intensities into shell thickness. They were able to learn more about the morphology and composition of the CSNP by simulating the XPS spectra to extract the parameters (Shard's methodology) and confirming the core-shell ratio with experimental spectra to quantify the shell thickness [63].
1. **Secondary Ion Mass Spectrometry**

A potent surface analysis method used for the evaluation of nanomaterials and other materials is secondary ion mass spectrometry (SIMS). It offers thorough details with high sensitivity and spatial resolution on the elemental and molecular composition of a sample's surface. SIMS is a useful tool for learning about the depth profiling, isotopic composition, and surface chemistry of nanoparticles [64].

**Principle:** The sample's surface is illuminated by a main ion beam that has been focused. Secondary ions, neutral particles, and electrons are ejected from the sample as a result of the impact of these main ions. The surface composition of the sample can then be determined by extracting, mass-analyzing, and detecting these secondary ions [65].

**Instrumentation:** The basic components of Secondary Ion Mass Spectrometry are as follows:

**Table 8: Basic Components of SIMS.**

|  |  |
| --- | --- |
| **Sr. No.** | **Components** |
| 1. | Source of Ion |
| 2. | Stage |
| 3. | Ion Optics (Primary) |
| 4. | Ion Extraction (Secondary) |
| 5. | Detector |
| 6. | Mass Spectrometer |
| 7. | Ion Optics (Analyzer) |
| 8. | Computer |
| 9. | Vacuum System |

**Recent Application in Research**

* The blood brain barrier has historically restricted systemic drug delivery to the central nervous system (CNS), rendering many treatments useless for any cancer cells present in the brain, as mentioned in the study by Muresan and colleagues. Regional drug delivery systems are being developed to fill this gap. Here, they suggested polymeric microneedle (MN) patches that can be surgically removed tumors, like glioblastoma (GBM) with isothiocyanate dehydrogenase wild type, and then attached within a resection cavity site. These MN patches were loaded with polymer coated nanoparticles (NPs) containing cannabidiol (CBD) or olaparib (OLA) to test drug release and penetration depth. They then used on rat brain tissue taken from an ex vivo experiment and a brain model created in a lab. Secondary ion mass spectrometry analysis after its application to a rat brain hemisphere confirmed the presence of OLA and the MN patch up to 6 mm from the insertion site [66].
1. **CHARACTERIZATION OF NANOMATERIALS BY DYNAMIC MEASUREMENT TECHNIQUES**

Characterization of nanoparticles using dynamic measurement techniques focuses on understanding their behavior and properties in response to dynamic processes, such as motion, flow, and interactions with other particles or substances. These techniques provide insights into various aspects of nanoparticles' behavior, including size distribution, stability, surface charge, and interactions [67]. Here are some dynamic measurement techniques commonly used for nanoparticle characterization:

**A. Dynamic Light Scattering**

Researchers examine the flow and diffusive characteristics of scattering particles in suspension or polymers in solution using a technique known as (DLS) dynamic light scattering, also known as photon correlation spectroscopy or quasi-elastic light scattering. DLS is a method for determining the dimensions of particles and biomolecules, usually in the sub-micron range. The main goal of DLS is usually to quantify the size of particles suspended in a liquid media [68]. A potent and popular method in nanotechnology for identifying nanoparticles and comprehending their behavior in solutions is dynamic light scattering (DLS). It offers insights into the stability and interactions of nanoparticles by providing useful information regarding the aggregation, the size distribution, and hydrodynamic characteristics of those particles. DLS uses the concepts of Brownian motion and light scattering to examine nanoparticles suspended in a fluid media [69].

**Principle:** The core idea behind DLS is the detection of changes in scattered light intensity brought on by Brownian motion, or the random movement of nanoparticles driven by thermal energy. A laser beam is focused upon a suspension of nanoparticles, which scatter light in various directions. To determine the particle's size and velocity, these dispersed light patterns are identified and examined. By taking into consideration the Brownian motion of the macro-molecules in the suspension, which is correlated with particle size, DLS calculates the rate of particle diffusion. Brownian motion (BM) describes the random thermal motion of particles induced by the bombardment of solvent molecules. Because smaller particles get knocked out further by the solvent molecules and move more quickly than larger ones, the BM slows down as particle size increases. The fluctuating intensity of the scattering light affects the particle diffusion rate. Static light scattering measures scattered light intensity as a function of angle, whereas DLS tracks fluctuations in scattered light intensity over time at a constant scattering angle (typically 90 degrees). The translational diffusion coefficient, which is usually denoted by the letter D, regulates the Brownian motion's speed [70].

 The Stokes-Einstein equation can be used to measure particle size:

$$dH=kT÷3πηD$$

where,

dH= hydrodynamic diameter

k= Boltzmann Constant

.$η$= Solvent viscosity ( kg/m-s)

T= Temperature

D= Diffusion coefficient

**Instrumentation:** The basic components of DLS are as follows:

**Table 9: Basic Components of Dynamic Light Scattering.**

|  |  |
| --- | --- |
| **Sr. No.** | **Components** |
| 1. | Laser |
| 2. | Beam Spitter |
| 3. | Mirror |
| 4. | Lens |
| 5. | Sample |
| 6. | Photon Detector |
| 7. | Cross- correlator |
| 8. | Computer |

**Recent Application in Research**

* Bhairam along with researchers have discussed in their study that in drug delivery and therapeutics, DLS ensures uniform nanoparticle formulations, assesses stability, and examines interactions with biological fluids [71].
* Shukla studied that designing nano-systems with the necessary solubility and suspension in fluids or electrolytes They noted that it takes a lot of work to do certain types of biomedical research. To fully understand the properties of macro/nano molecules in a solution, dynamic light scattering examines their diffusion behavior. Another use for this approach is calculating the diffusion coefficient and hydrodynamic size. The pharmaceutical industry recommends using DLS as a technology for studying the homogeneity of nucleic acids, proteins, complexes of protein-nucleic or protein-protein acid preparations, as well as to examine protein-small molecule interactions [72].
* Mangala K.discussed in her paper characterization of nutraceuticals with the help of dynamic light scattering technique. Nutraceuticals contains the words pharmaceutical and nutrition. The phrase is typically used to describe any type of feed or its constituent parts that will improve health and aid in the treatment or prevention of diseases. The molecular and physiochemical criteria for various nutraceutical substances, such as size, stability, and surface characteristics, vary. Therefore, in order to address these, each of them requires distribution mechanisms that are appropriately unique. Nanoliposomes, nano-emulsions, lipid nanocarriers, micelles, and biopolymeric nanoparticles are examples of the nanoparticulate delivery systems utilized to enhance nutraceuticals [73].
* Wang. B.suggested about PIDLS, an innovative technique for polarization imaging dynamic light scattering, is suggested for the examination of the particle size and shape of nanoparticles. With this novel method, the morphologies of seven different types of nanoparticles are measured. It was discovered that nanoparticles' degree of linear polarization (DoLP) can serve as a gauge for determining their shape. The assessment of five different types of industrial titanium dioxide confirmed the viability and precision of the suggested method for morphology measurement, and they showed that the PIDLS method can offer new standards for the evaluation of nanoparticle quality. PIDLS is a technology that has great promise for improving nanoparticle production, quality control, and analysis of physical-chemical behavior, such as in drug delivery [74].

**B. Zeta Potential Measurement**

 One method for figuring out the electrostatic charge on the surface of nanoparticles or colloidal particles in a liquid media is zeta potential measurement [75]. It offers important details regarding the stability, behavior, and potential interactions of nanoparticles with other particles and surfaces when they are dispersed. In colloidal science, zeta potential is a crucial variable that is important for comprehending how stable nanomaterial suspensions are [76].

**Principle:** Zeta potential is the name given to the electric potential at the shear plane where a particle's surface meets a liquid medium. It measures how strongly particles are attracted to or repelled by electrostatic forces. Zeta potential is affected by many factors, including surface chemistry, particle charge, ionic strength and solution pH. Zeta potential measurements are frequently made utilizing methods like laser Doppler velocimetry or electrophoretic mobility. In electrophoretic mobility, charged particles in a dispersion of nanoparticles move as a result of the application of an electric field. The zeta potential is determined by measuring the particle's rate of motion. In laser Doppler velocimetry, the zeta potential is determined after the Brownian motion of the particles is examined using laser light scattering to estimate their velocity [77].

**Instrumentation:** The basic components of Zeta Potential Measurement are as follows:

**Table 10: Basic Components of ZPM.**

|  |  |
| --- | --- |
| **Sr. No.** | **Components** |
| 1. | Zeta Potential Analyzer |
| 2. | Dispersion System |
| 3. | Electrode Solution |
| 4. | Electrolyte Solution |
| 5. | Laser Light Source |
| 6. | Detection System |
| 7. | Microscope, Temperature Control System, pH Adjustment System (Optional) |
| 8. | Sample Cuvette |
| 9. | Computer |

**Recent Application in Research**

* Alzubaididiscussed that the creation of nanomaterials is thought to benefit greatly from the abundance of bioactive plant compounds. In the current study, an efficient reducing agent termed ethanolic flaxseed extract was used to produce silver nanoparticles (AgNPs). The synthesis of AgNPs was shown by monitoring how the color of the mixture of silver nitrate (AgNO3) changed from yellow to a reddish solution after the inclusion of the extract and by examining it by UV-visible inspection. Zeta potential and DLS measurements of nanoparticles are suitable for characterizing nanoparticles and identifying the particle size and surface charge of the generated AgNPs. Zeta potential measurements were used to investigate the AgNPs' colloidal stability. It is believed that the zeta potential measurements taken over 30 mV demonstrate a generally stable colloidal stability of the nanoparticle sample [78].
* Bhatt and group of researchers discussed intheir research aims to use an aqueous extract from Pyrostegia venusta leaves to naturally manufacture silver nanoparticles. Zeta potential measurement was one of many methods used to characterize the produced nanoparticles. At 31.7 mv, the zeta potential displayed a strong peak. Additionally, cell apoptosis was used to evaluate the anticancer activity. The results showed that AgNPs had the ability to kill COS-7 cells by employing differential labeling to distinguish between live and dead cells [79].
1. **CHARACTERIZATION OF NANOMATERIALS BY MECHANICAL CHARACTERIZATION TECHNIQUES**

Analyzing the mechanical characteristics and behaviors of nanoparticles is a step in mechanical nanoparticle characterization. Mechanical characterization helps to understand the strength, elasticity, hardness, deformation, and other mechanical properties of nanoparticles by revealing how they react to stresses and pressures from the outside world [80].

1. **Nanomechanical Testing**

To characterize nanomaterials, such as nanoparticles and nanostructured materials, and to comprehend their mechanical characteristics at the nanoscale, a specific approach known as nanomechanical testing is used. The strength, hardness, elasticity, and deformation behavior of nanoparticles are revealed by this method [81].

**Principle:** The basic idea behind nanomechanical testing is to use a nanoscale indenter to apply controlled forces or displacements to the surface of a nanomaterial while monitoring the subsequent mechanical response. This method offers perceptions into the mechanical characteristics of the material at the nanoscale, such as hardness, elastic modulus, deformation behavior, and more [82]. Atomic force microscopy (AFM)-based techniques and nanoindentation are the main techniques for nanomechanical testing.

**Nanoindentation:** A common method for determining the elastic modulus and hardness of nanoparticles and thin films is nanoindentation. The surface of the nanoparticle is indented with a sharp object, and the resulting indentation depth and load are recorded. This information about the material's stiffness and resistance to deformation is utilized to compute the elastic modulus and hardness [82].

**Atomic Force Microscopy:** AFM can be utilized for mechanical characterization in addition to imaging applications. Using AFM-based methods like force spectroscopy or indentation mode, scientists may apply precise forces to nanoparticles and observe how they respond mechanically. This can reveal details regarding the adhesion, stiffness, and deformation characteristics of the nanoparticles [83].

**Significance:** Nanomechanical testing offers insightful information on a material's nanoscale mechanical behavior, which is important for a variety of applications, such as:

* Evaluating the mechanical characteristics of thin films, nanostructures, and nanoparticles.
* Understanding mechanical behavior that is size-dependent and nanoscale phenomena.
* The examination of the results of surface alterations, coatings, and functionalization.
* Designing and improving nanomaterials for particular uses.
* Analyzing the mechanical characteristics of coatings, nanocomposites, and biomaterials [84].

Nanomechanical testing enables researchers to assess the mechanical properties of nanomaterials and deepen their understanding of their behavior at the nanoscale by applying controlled loads or displacements and evaluating the subsequent mechanical response.

**Instrumentation:** The basic components used in nanomechanical testing are:

**Table 11: Components of Nanomechanical testing**

|  |  |  |
| --- | --- | --- |
| **Sr. No.** | **Nanoindentation** | **AFM Based Techniques** |
| 1. | Indenter | AFM System |
| 2. | Application Load System | Force Measurement |
| 3. | Depth Measurement  | Piezoelectric Scanner |
| 4. | Computer | Computer |
| 5. | Environmental Control |  |

1. **DISCUSSION**

Understanding the characteristics, behavior, and uses of nanoparticles and nanostructured materials depends heavily on nanomaterial characterization techniques. Researchers can learn more about the chemical, physical, mechanical, and structural properties of nanomaterials using the approaches covered in this chapter. Each characterization method has advantages and disadvantages that make it appropriate for particular kinds of analysis. A thorough understanding of nanomaterials can be obtained by combining several methodologies, allowing researchers to modify their properties for use in nanotechnology, materials science, medicine, energy, electronics, and other fields.

1. **FUTURE ASPECTS**

The future importance of nanomaterial characterization is expected to be crucial in improving a number of disciplines, technologies, and sectors. For nanomaterials to reach their full potential, precise and thorough characterization procedures will be crucial as nanotechnology develops further and their use in commonplace applications increases [85-86]. The following significant factors underline the future importance of nanomaterial characterization:

* **Customized Material Design:** With the help of advanced nanomaterial characterization, researchers will be able to precisely regulate the characteristics and behavior of nanomaterials, enabling them to create materials with the desired functions. The development of innovative materials for use in electronics, energy storage, catalysis, medicine, and other fields depends on this.
* **Quality Control & Standardization:** As nanoparticles are included into commercial goods, strict characterization will be necessary to guarantee constant quality, safety, and performance. The creation of benchmarks and regulations for product development and regulatory compliance will be made easier with the standardized use of nanomaterial characterization techniques.
* **Drug Delivery & Nanomedicine:** Characterization approaches will be crucial in improving the field of nanomedicine, which uses nanoparticles for targeted drug administration, imaging, and diagnostics. The effectiveness and safety of nanoparticles in medical applications will be improved by precise characterization of their size, surface characteristics, and interactions with biological systems.
* **Environmental and safety considerations**: Thorough characterization will assist in determining the effect nanomaterials will have on the environment and any potential risks. In order to limit possible risks, prudent design and application of nanomaterials will benefit from an understanding of how they behave in diverse environmental situations.
* **Optoelectronics and Nanoelectronics:** Advances in nanoelectronics, photonics, and quantum technologies will be fueled by the characterization of tiny electrical and optoelectronic materials. The creation of quicker, more compact, and more effective devices will benefit from accurate evaluation of materials' electrical and optical properties.
* **Storage and Energy Conversion:** Nanomaterials are essential for improvements in energy conversion (such as solar cells) and storage (such as batteries and supercapacitors). Characterization methods will help with material optimization for improved effectiveness, stability, and capacity.
* **Advanced Functional Materials:** To fully comprehend the distinctive characteristics and functions of emerging domains like metamaterials, nanocomposites, and 2D materials, expert characterization is necessary. The incorporation of these materials into cutting-edge applications will be made possible by characterization.
* **Quantum Nanomaterials:** Characterization methods will be crucial for exploring and utilizing quantum effects at the nanoscale as the science of quantum materials develops, paving the way for innovations in quantum computing, communication, and sensing.
* **Nanotechnology in Agriculture and Environment:** Applications for nanomaterials in water purification, agriculture, and environmental monitoring are being investigated. The usage of nanoparticles in these fields will be safe and productive thanks to thorough characterization.
* **Characterization and Machine Learning Integration:** Characterization of nanomaterials will probably be combined with machine learning techniques in the future, enabling automated data processing, pattern identification, and quicker materials discovery.
1. **CONCLUSION**

Nanomaterial characterization techniques play a pivotal role in unraveling the unique properties, behaviors, and potential applications of nanoscale materials. These techniques provide valuable insights into the composition, structure, morphology, surface properties, and interactions of nanoparticles and nanomaterials. By employing a diverse array of analytical tools, researchers can comprehensively explore and manipulate nanomaterials to drive advancements in various scientific, industrial, and technological fields [87]. The evolution of nanotechnology has spurred the development of an extensive toolkit of characterization methods, each offering specific advantages and insights. From imaging techniques like Scanning Electron Microscopy (SEM), Scanning Tunneling Microscopy (STM), Fluorescence Microscopy, Confocal Microscopy, Optical Microscopy and Transmission Electron Microscopy (TEM) that reveal intricate details of nanomaterial morphology, to spectroscopic methods such as UV-Visible Spectroscopy, Fourier-Transform Infrared (FTIR), X-ray Photoelectron Spectroscopy, and Raman spectroscopy that provide chemical and vibrational information, these techniques collectively enable a holistic understanding of nanomaterials. Nanomaterial characterization techniques are not only vital for fundamental research but also have far-reaching practical implications. They enable precise control and optimization of nanomaterial synthesis, facilitate the design of innovative nanodevices and materials, and ensure the quality and safety of nanotechnology-enabled products. Moreover, these techniques are instrumental in bridging the gap between laboratory-scale discoveries and real-world applications, paving the way for breakthroughs in areas such as medicine, electronics, energy, forensic science and environmental science. As nanotechnology continues to advance, the ongoing refinement and integration of characterization techniques will be paramount. The synergistic use of multiple techniques, combined with computational modeling and data analysis, will lead to deeper insights into nanomaterial behavior and properties [88]. This, in turn, will accelerate the translation of nanomaterials from the realm of scientific exploration to practical solutions that address complex challenges and drive innovation across diverse domains. In essence, the intricate world of nanomaterials demands a multidimensional understanding, and nanomaterial characterization techniques provide the lenses through which we gain access to this miniature realm, unlocking its immense potential for the betterment of society and the advancement of science and technology.

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