IJNRD.ORG ISSN : 2456-4184
INTERNATIONAL JOURNAL OF NOVEL RESEARCH AND DEVELOPMENT (IJNRD) | IJNRD.ORG
An International Open Access, Peer-reviewed, Refereed Journal

TECHNIQUES FOR NANOPARTICLES CHARACTERIZATION

[1]Sanjay Singh*, [2]Preeti Maurya, [3]Khushaboo Soni
[1]Associate Professor, [2]Research Scholar, [3]Research Scholar
Department of Botany,
CMP Degree College, University of Allahabad, Prayagraj 211002 Uttar Pradesh, INDIA

Abstract:

Different characterization approaches are classed based on the technique's concept/group, the information they can supply, or the materials for which they are intended. The distinctive features of nanoparticles, such as those determined largely by size, content, and structure as well as their self-organized film structures, are of tremendous interest for a wide range of applications in the realm of information, energy, environmental, and medical technologies. The selection of the best appropriate method is complicated by the varying advantages and disadvantages of each methodology, and frequently a combinatorial characterization approach is required. A very effective tool for examining the diffusion behaviour of macromolecules in solution is dynamic light scattering (DLS), often referred to as photon correlation spectroscopy (PCS). The current review is focused on the characterization techniques of nanoparticles.

Keywords: Characterization techniques, Scattering, Spectroscopy, DLS, Realm

Introduction:

Due to their distinct physical, chemical, and mechanical characteristics that set them apart from bulk solids and molecules, nanomaterials have garnered interest. When quantum phenomena are present, nanomaterials exhibit different size-dependent characteristics in the 1-100 nm range. For advancements in several technologies, the synthesis and characterisation of new and unique nanomaterials with carefully regulated structures, crystalline phases, forms, sizes, and porosities are crucial (Ikhmayies 2013). Nanotechnology is known as the field of research. So, nanotechnology was presented by Nobel laureate Richard P. Feynman during his 1959 lecture "*There's Plenty of Room at the Bottom*" (Khan, I. et al., 2017; Singh et al 2023). A large variety of MNPs with well-defined architectures and sizes that can be easily matched with the interest of specified applications can be synthesised. An external magnetic force can be used to modify the magnetic nanoparticles (MNP) themselves (Lim et al 2013). To confirm that the manufactured particles are at the nanoscale, characterization is also crucial. Characterization in material science refers to the general and comprehensive techniques used to investigate the properties and structure of the material. For the subject to be understood scientifically, this fundamental procedure is necessary. Any process that deals with material analysis, such as mechanical testing, thermal analysis, and density computation, falls under the category of characterization because it involves tools necessary to examine material properties and microscopic structures. Characterization approaches that have been used for millennia are continuously being joined by newer, more sophisticated procedures. Characterization enables us to evaluate the effectiveness of the procedure as well as the content and structure of the materials. While some methods are quantitative, others are qualitative (Titus et al 2019). Because of their substantial electrical and optical capabilities, nanosized semiconductors are in high demand for the fabrication of nanoscaled optoelectronic and electronic devices with multifunctionality (Tokumoto, M. S. et al 2003; Kumar P. et al 2010; Thomus G. 1997; Talam S. et al 2012). Over the last 80 years, electron microscopy has been a groundbreaking imaging method for scientists

and engineers, opening up the realm of nanoscale materials and permitting analysis of their unique features. The ability of electron microscopes to view submicron-sized objects, even down to single atomic locations, has resulted in the invention of whole new nanotechnologies, as well as astonishing advances through nanoscale engineering of micro-sized components. Many daily things, such as mobile phones, plasma screen televisions, and materials for automobiles and aeroplanes, have necessitated considerable use of electron microscopy to help development. (Inkson, B. J. 2016).

Type of Characterization Techniques:

The characterization techniques based on different methods like microscopy, spectroscopy, X-Ray, Zeta potential Dynamic Light Scattering etc. are used as nanoparticles characterization.

UV-Vis spectroscopy:

For the ultraviolet and visible regions of the spectrum, a deuterium or tungsten lamp, sample and reference beams, a detector, and a monochromator are the components of this technique. The sample will produce the UV spectrum when exposed to UV light. Cuvettes are used to hold samples and are stored inside the instrument so samples can be introduced to the light path. As cuvettes, you can use glass, plastic, silica, or quartz cells. It is impossible to conduct absorbance experiments below 310 nm using plastic and glass cuvettes because they absorb those wavelengths. Due to their transparency to wavelengths above 180 nm, quartz cuvettes are utilised for absorption measurements in the UV range (Taha M et al 2013).

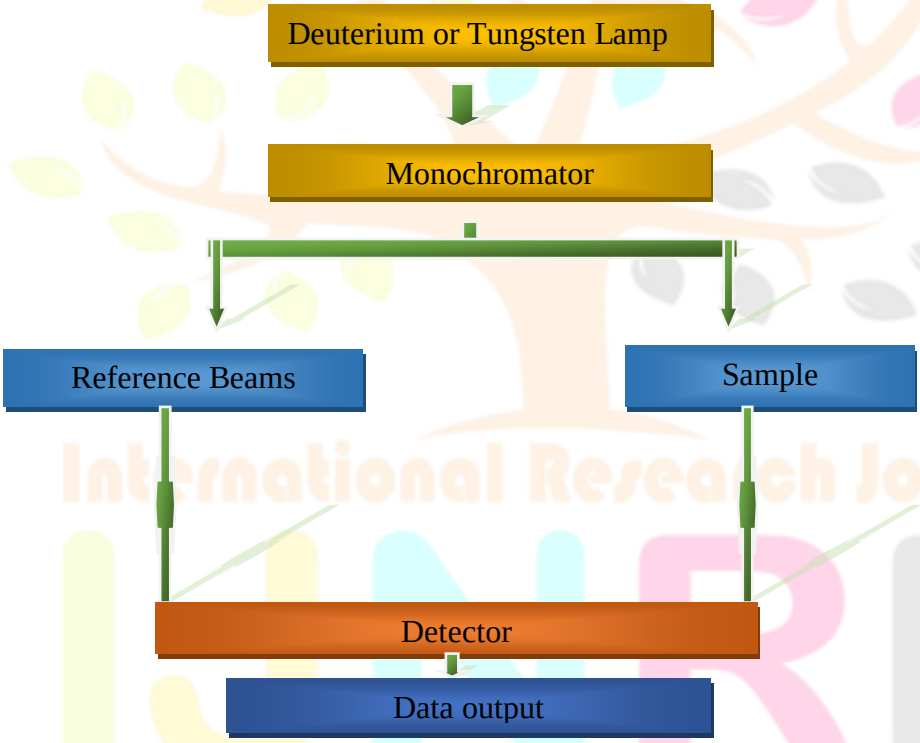

Figure 1: Diagrammatic representation of UV – Vis Spectroscopy (Taha M et al 2013)

The optical characteristics of a solution are determined via absorbance spectroscopy. A light is passed through the sample, and the amount of light absorbed is measured. When varying the wavelength and measuring the absorbance at each wavelength. Beer-Law Lambert's can be used to the absorbance to determine a solution's concentration. When treated with the *Nerium obander* plant extract and an aqueous 1mM Silver nitrate solution, the optical measurement of a UV-visible spectrophotometer shows a distinct absorbance peak, such as 410nm. By indicating a suitable surface, *Azadirachta indica* is produced with iron nanoparticles. UV-visible spectroscopy revealed a Plasmon resonance with high band intensities and peaks in the 216-265nm range. (Heera, P. et al 2015).

SEM (Scanning Electron Microscopy):

SEM is the preferred technique for analyzing surface areas (Stokes, 2008; Reimer, 1998; Egerton, 2011; Goldstein et al., 2007; Kuo, 2014; Pawley and Schatten, 2014). Figure 2 depicts a typical SEM layout, which includes the electron gun (electron source and accelerating anode), electromagnetic lenses to focus

the electrons, a vacuum chamber containing the specimen stage, and a variety of detectors to gather the signals emitted by the specimen (Inkson, B. J. 2016). SEM was used to determine the surface morphology of produced SNPs using a Thermo Scientific Quattro ESEM with an accelerating voltage of 1 kV or 5 kV. To capture photos, each sample was placed on a stub glued with carbon tape and sputtered with gold. The SEM was outfitted with EDX, which was used to investigate the qualitative chemical composition of the SNPs produced in this investigation (Satish, K et al., 2023).

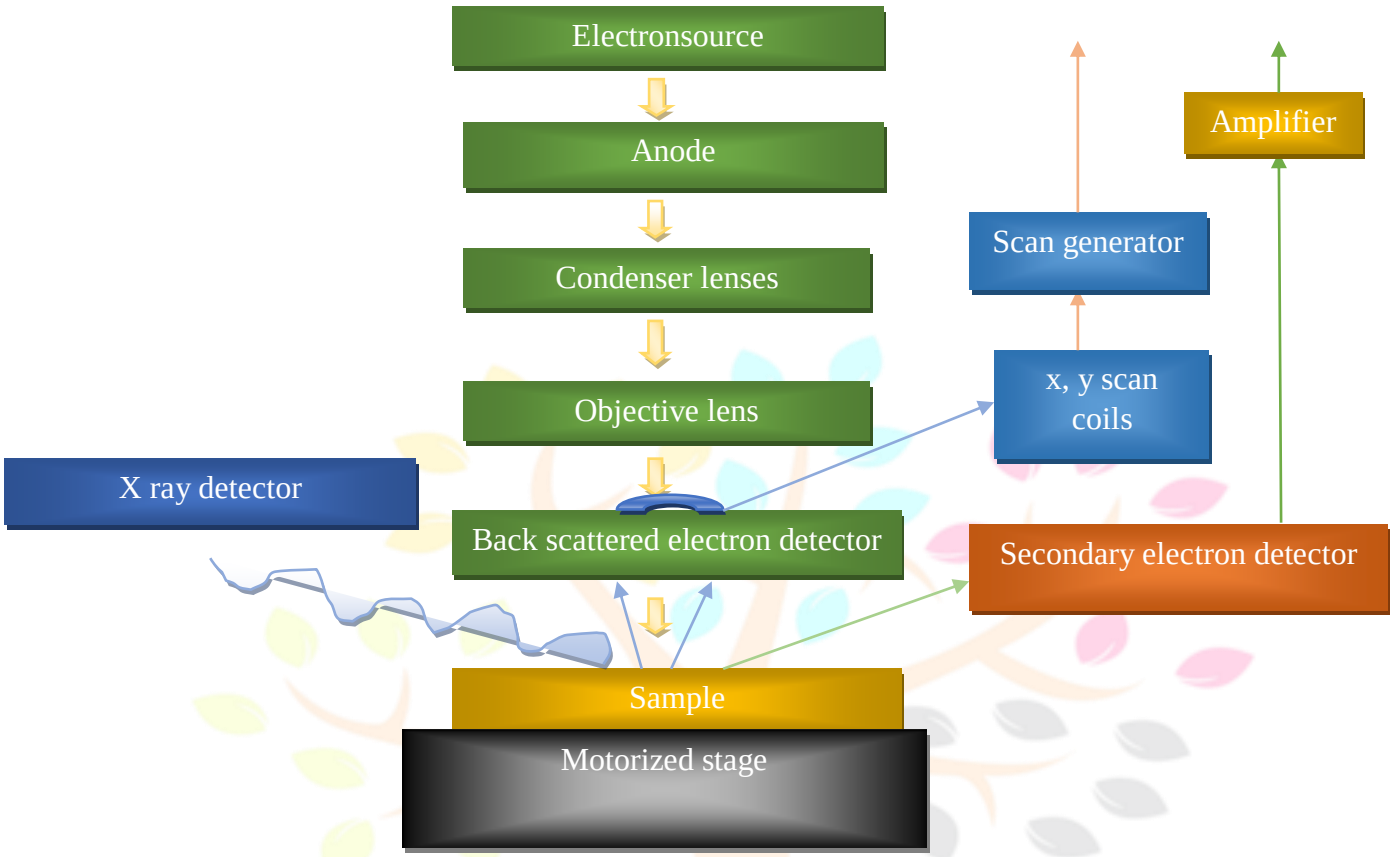

Figure 2: Ray diagram Scanning Electron Microscope (Inkson, B. J. 2016)

TEM (Transmission Electron Microscopy):

The contrast of TEM images differs from that of light microscopic images. Diffraction occurs instead of absorption when the electron beam interacts with the sample. The diffraction
strength varies depending on the plane's orientation of the electron beam. At some angles, the electron beam is substantially diffracted from the axis, whereas at others, it is transmitted. Holders are fitted to the specimen so that it can be tilted to achieve a certain diffraction condition. The light field is obtained by blocking the electrons deflected by inserting the aperture and allowing the unscattered electrons to pass through it. The deflected electrons can also be employed to generate an image known as a dark field (Titus et al 2019).

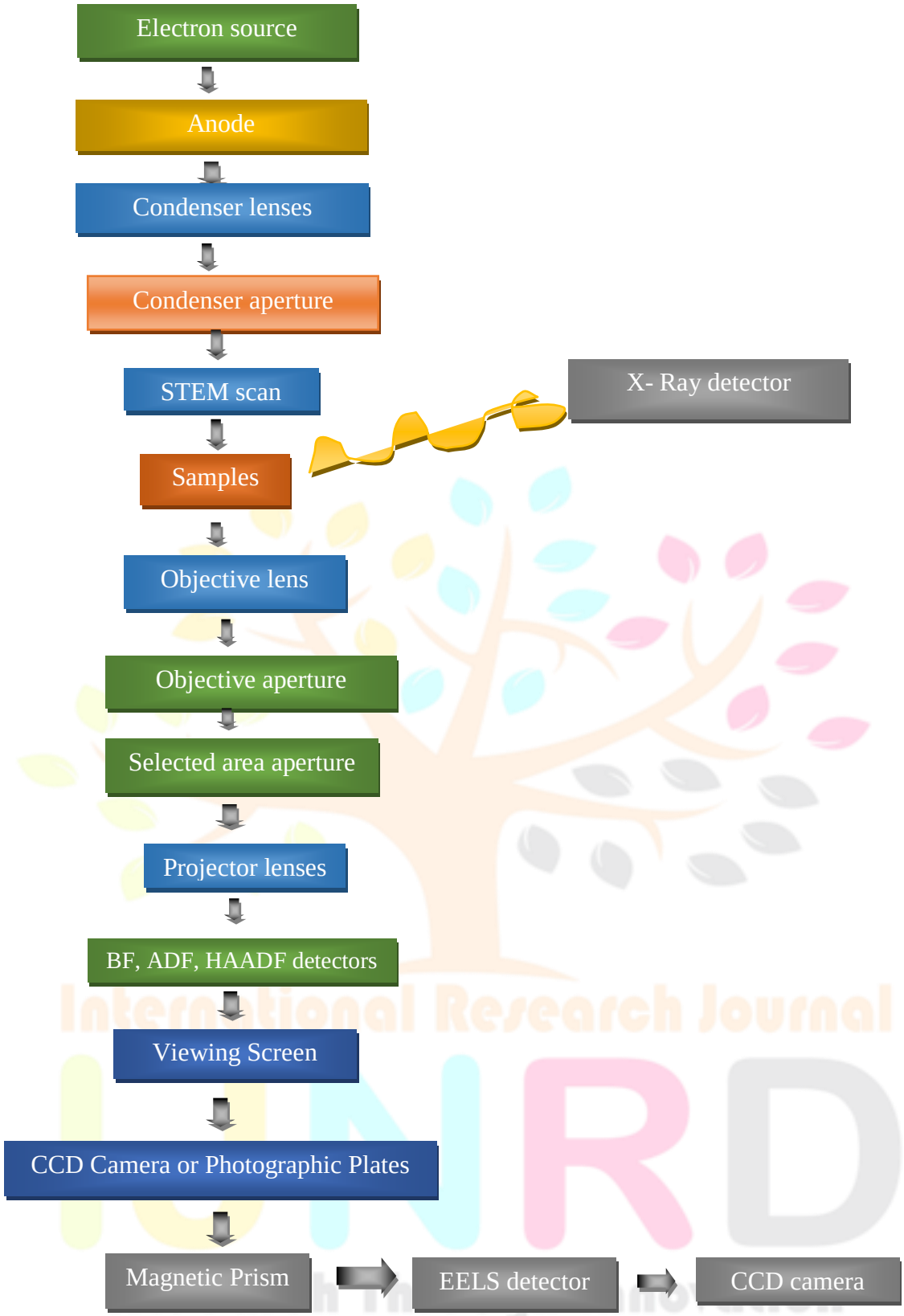

Figure 3: Schematic diagram of Transmission Electron Microscope (Inkson, B. J. 2016)

DLS (Dynamic Light Scattering):

The technique of dynamic light scattering (DLS), also known as photon correlation spectroscopy (PCS) or quasi-elastic light scattering, is used to estimate the size of nanoparticles in a colloidal suspension polymeric solution. DLS examines the modulation of the intensity of scattered light as a function of time to determine the hydrodynamic size of particles using the mechanism of light scattering from a laser passing through a colloidal solution (Lim et al., 2013; Raval, N., et al 2019). The DLS equipment has been used to determine

the particle size of dispersing colloidal samples, investigate formulation stability, and detect aggregation or agglomeration. Particles' Brownian motion is proportional to their hydrodynamic diameter. Smaller particles disperse more quickly than larger ones (Raval, N., et al 2019). Typically, a monochromatic beam of light is used to scatter light from a sample, and a suitable detector picks up the signal. John Tyndall described one of the early light-scattering experiments, describing the Tyndall effect when particles bigger than the wavelength of the input light scatter light from colloidal solutions (Tyndall J. 1868; Stetefeld, J., et al 2016). DLS is the ideal instrument for determining and measuring nanoparticle aggregation. An analytical method for determining the particle size distribution of formulations in the oligomer and submicron size ranges is density laser scattering (DLS). A helium-neon laser is used as the light source for the DLS experiments, which employ scattering angles of 90 or 173 degrees (Raval, N., et al 2019).

Zeta Potential:

Surface charges will be present in suspension for nanoparticles or colloidal particles. The interaction of the applied electric field with the charged particle causes a particle to begin moving as soon as it is done so. The electric field, charge, and suspending medium all influence the motion's speed and direction. The Doppler shift in the scattered light is used to determine a particle's velocity; the velocity proportional to the electrical potential of the particle at the shear plane is known as the zeta potential. Thus, the optical measurement of the particle's motion in an electric field can be utilised to calculate the zeta potential (Xu R. 2008; Delgado AV et al 2007). Particle motion in an electric field is referred to as electrophoresis. In a known refractive index solution, sample particles are suspended. Zeta potential quantifies the charge stability of colloidal particles by calculating the effective electric charge on the surface of a nanoparticle. An enhanced concentration of ions with opposing charges is present close to the surface of a nanoparticle when it possesses a net surface charge. An electrical double layer, consisting of the layer of surface charge and the oppositely charged ions, is generated as the ion layer advances across the nanoparticle. The zeta potential is produced by the differential between the fluid layers containing oppositely charged ions and the bulk fluid in which the particle is suspended. Positively charged surfaces will attract negatively charged particles and vice versa. The size of the zeta potential reveals details on the stability of the particle. Due to stronger electrostatic repulsion, the larger magnitude denotes increased stability.

- In the 0 to 5 mV range, particles frequently agglomerate.
- The voltage ranges for less stable particles are 5–20 mV, moderately stable particles are 20–40 mV, and very stable particles are 40+ mV.

The pH of the solution is yet another important variable that can have an impact on the amount of charge on the nanoparticle surface. The isoelectric point, which is where the surface charge can be reduced to zero, is at a specific pH. (Titus et al 2019).

FTIR (Fourier Transform Infrared) Spectroscopy:

FTIR analysis uses infrared light to scan the samples, identifying organic, inorganic, and polymeric components. A change in the material composition is clearly shown by changes to the typical pattern of absorption bands. FTIR can be used to identify and characterise unknown materials, find additives, detect impurities in a material, and determine decomposition and oxidation. A source, sample cell, detector, amplifier, A/D convertor, and computer are typical components of an FTIR spectrometer. After passing through the interferometer, radiation from the sources reaches the detector. The A/D converter and amplifier amplify the signal, convert it to a digital signal, and then transfer it to the computer to perform the Fourier transform. About 10,000–100 cm^{-1} of infrared radiation is delivered through the sample, with some of it being absorbed and some of it flowing through. The sample transforms the radiation's absorbed energy into vibrational or rotational energy. The final signal produced at the detector has a spectrum that typically ranges from 4000 to 400 cm^{-1}, representing the samples' molecular signature. Because each molecule has a distinct fingerprint, FTIR is a crucial tool for chemical identification (Taha M et al 2013).

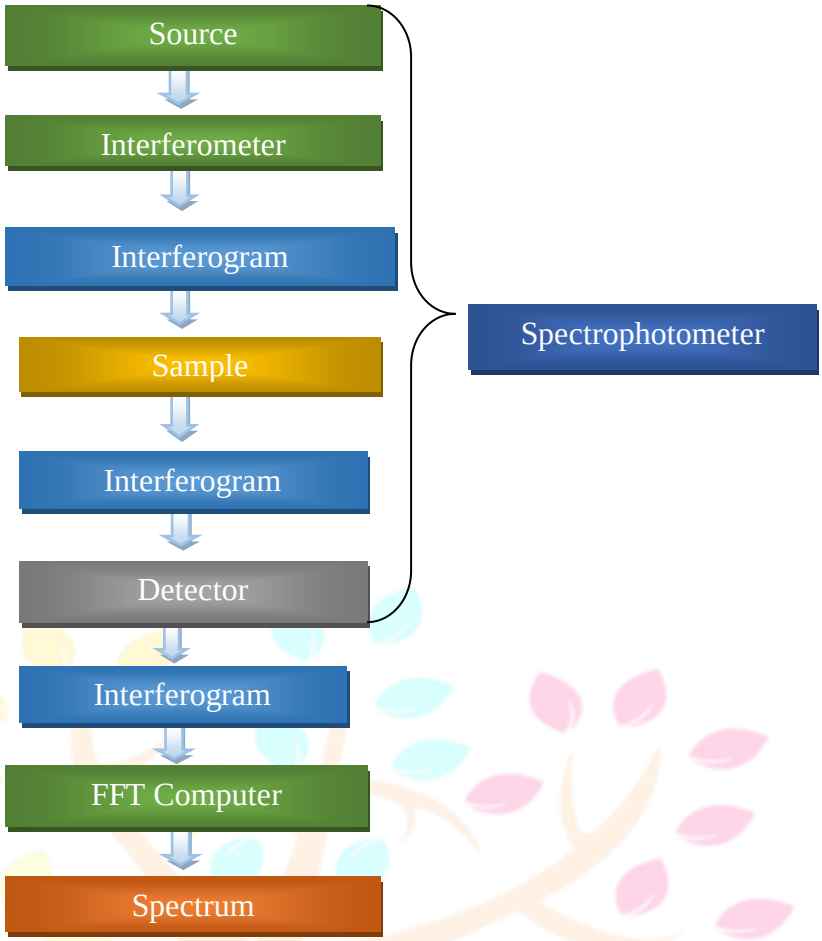

Figure 2: Diagrammatic representation of FTIR Spectroscopy (© 2001 Thermo Nicolet CorporationAll rights reserved, worldwide.)

According to the Laws of Lambert and Beer, the radiant power of an electromagnetic radiation beam—typically, ordinary light—relates to the concentration of the absorbing species and the length of the beam's journey through an absorbing medium, respectively. The laws are typically integrated into the connection (Swinehart, D. F., 1962).

$$A = - \log_{10} P/P_0 = abc$$

Where,

A = absorbance formerly called the optical density
P = radiant power formerly called the intensity
a = absorptivity formerly called the extinction coefficient
b = length of the beam in the absorbing medium
e = concentration of the absorbing species

X-Ray Diffraction

One of the most widely used techniques for characterizing NPs is X-ray(10 pm-10 nm) diffraction (XRD). XRD mainly offers information on the crystalline structure, phase nature, lattice parameters, and crystalline grain size. The latter parameter is calculated using Scherrer's equation and the widening of the most intense peak from an XRD analysis of a specific sample. The XRD techniques, which are often used on powder samples after drying their respective colloidal solutions, have the benefit of producing statistically representative, volume-averaged values. The particle composition can be established by comparing the position and strength of the peaks to the reference patterns provided in the International Centre for Diffraction Data (ICDD, formerly known as the Joint Committee on Powder Diffraction Standards, JCPDS) database. It is not ideal for amorphous materials, and the XRD peaks are too broad for particles smaller than 3 nm (Mourdikoudis, S et al 2018).

Conclusion:

Nanotechnology is known as the field of research. When quantum phenomena are present, nanomaterials exhibit different size-dependent characteristics in the 1-100 nm range. A large variety of MNPs with well-defined architectures and sizes that can be easily matched with the interest of specified applications can be synthesized. Characterization enables us to evaluate the effectiveness of the procedure as well as the content and structure of the materials. Because of their substantial electrical and optical capabilities, nanosized semiconductors are in high demand for the fabrication of nanoscaled optoelectronic and electronic devices. The creation of more cutting-edge and cutting-edge technologies depends heavily on nano nanoparticles. Understanding characterization is crucial for regulated nanoparticle manufacturing and application. Nanotechnology offers a great deal of potential for its futuristic approach, which is influenced by things like the creation of quicker, easier, more effective, and brand-new methodologies for materials characterization.

References:

Khan, I., Saeed, K., & Khan, I. (2019). Nanoparticles: Properties, applications and toxicities. *Arabian journal of chemistry*, *12*(7), 908-931. ,https://doi.org/10.1016/j.arabjc.2017.05.011.

Ikhmayies, S. J. (2013). Characterization of Nanomaterials. JOM, 66(1), 28–29. https://doi.org/10.1007/s11837-013-0826-6.

Lim, J., Yeap, S. P., Che, H. X., & Low, S. C. (2013). Characterization of the magnetic nanoparticle by dynamic light scattering. Nanoscale Research Letters, 8(1), 381. https://doi.org/10.1186/1556-276x-8-381.

Titus, D., James Jebaseelan Samuel, E., & Roopan, S. M. (2019). Nanoparticle characterization techniques. Green Synthesis, Characterization and Applications of Nanoparticles, 303–319. https://doi.org/10.1016/b978-0-08-102579-6.00012-5.

Taha, M., Hassan, M., Essa, S., &Tartor, Y. (2013). Use of Fourier transform infrared spectroscopy (FTIR) spectroscopy for rapid and accurate identification of Yeasts isolated from human and animals. *International Journal of veterinary science and medicine*, *1*(1), 15-20.

Xu, R. (2008). Progress in nanoparticles characterization: Sizing and zeta potential measurement. *Particuology*, *6*(2), 112-115.

Delgado Á. V., González-Caballero, F., Hunter, R. J., Koopal, L. K., &Lyklema, J. (2007). Measurement and interpretation of electrokinetic phenomena. *Journal of colloid and interface science*, *309*(2), 194-224.

Mourdikoudis, S., Pallares, R. M., & Thanh, N. T. (2018). Characterization techniques for nanoparticles: comparison and complementarity upon studying nanoparticle properties. *Nanoscale*, *10*(27), 12871-12934.

Talam, S., Karumuri, S. R., &Gunnam, N. (2012). Synthesis, Characterization, and Spectroscopic Properties of ZnO Nanoparticles. ISRN Nanotechnology, 2012, 1–6. https://doi.org/10.5402/2012/372505.

Argast, A., & Tennis III, C. F. (2004). A web resource for the study of alkali feldspars and perthitic textures using light microscopy, scanning electron microscopy and energy-dispersive X-ray spectroscopy. *Journal of geoscience education*, *52*(3), 213-217.

Tokumoto, M. S., Briois, V., Santilli, C. V., &Pulcinelli, S. H. (2003). Preparation of ZnO nanoparticles: structural study of the molecular precursor. *Journal of Sol-Gel Science and Technology*, *26*(1-3), 547-551.

Kumar, P., Panchakarla, L. S., Bhat, S. V., Maitra, U., Subrahmanyam, K. S., & Rao, C. N. R. (2010). Photoluminescence, white light emitting properties and related aspects of ZnO nanoparticles admixed with graphene and GaN. *Nanotechnology*, *21*(38), 385701.

Thomas G. (1997), “Invisible circuits,” Nature, vol. 389, no. 6654, pp.907-908.

Inkson, B. J. (2016). *Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) for materials characterization. Materials Characterization Using Nondestructive Evaluation (NDE) Methods, 17–43.* https://doi.org/10.1016/b978-0-08-100040-3.00002-x.

Stokes, D. (2008). *Principles and practice of variable pressure/environmental scanning electron microscopy (VP-ESEM)*. John Wiley & Sons.

Reimer, L., 1998. Scanning Electron Microscopy, second ed. Springer, Heidelberg. ISBN: 3-540-63976-4.
Kuo, J. (Ed.). (2008). *Electron microscopy: methods and protocols* (Vol. 369). Springer Science & Business Media.

Pawley, J., Schatten, H. (Eds.), 2014. Biological Low-voltage Scanning Electron Microscopy. Springer. ISBN: 13: 978-1489995841.

Goldstein, J., Newbury, D.E., Joy, D.C., Lyman, C.E., Echlin, P., Lifshin, E., et al., 2007. Scanning Electron Microscopy and X-ray Microanalysis, third ed. Springer.

Egerton, R.F., 2011. Physical Principles of Electron Microscopy: An Introduction to TEM, SEM, and AEM, second ed. Springer.

Satish, K., Bhat, K. S., Ravikumar, Y. S., & Harish, M. N. K. (2023). Silver nanoparticles decorated natural products doped polyaniline hybrid materials for biomedical applications. *Journal of Applied Biology and Biotechnology*, *11*(2), 165-177.https://doi.org/10.7324/JABB.2023.110217.

Raval, N., Maheshwari, R., Kalyane, D., Youngren-Ortiz, S. R., Chougule, M. B., &Tekade, R. K. (2019). Importance of physicochemical characterization of nanoparticles in pharmaceutical product development. In *Basic fundamentals of drug delivery* (pp. 369-400). Academic Press.https://doi.org/10.1016/B978-0-12-817909-3.00010-8.

Tyndall J (1868). On the Blue Colour of the Sky, the Polarization of Skylight, and on the Polarization of Light by Cloudy Matter Generally. Proceedings of the Royal Society of London 17:223– 233 https://doi.org/10.1098/rspl.1868.0033.

Stetefeld, J., McKenna, S. A., & Patel, T. R. (2016). Dynamic light scattering: a practical guide and applications in biomedical sciences. *Biophysical reviews*, *8*, 409-427.Https://doi.org/10.1007/s12551-016-0218-6.

Heera, P., & Shanmugam, S. (2015). Nanoparticle characterization and application: an overview. *Int J CurrMicrobiol App Sci*, *4*(8), 379-386.

Singh, S., Maurya, P., Soni, K., (2023). Nanoparticles: Their Classification, Types and Properties. *International Journal of Innovative Research in Technology*, *9*(8), 2349-6002.

Swinehart, D. F. (1962). *The Beer-Lambert Law. Journal of Chemical Education, 39(7), 333–* . https://doi.org/10.1021/ed039p333.

*****