Ultrasonic Assisted Synthesis, Characterization and Photocatalytic Activity of Nano Particles

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CERTIFICATION

This is to certify that the project entitled literature review on **"Ultrasonic Assisted Synthesis, Characterization and Photocatalytic Activity of Nano Particles"** submitted to the department of Applied Chemistry and Chemical Engineering of Noakhali Science and Technology University for partial fulfillment for the degree B.Sc. (Honours) in Applied Chemistry and Chemical Engineering was carried out by **Munsat Mahbuba Chowdhury, Roll: BFH1804010F, Session: 2017-18** under my supervision and complete guidance. I do thereby approve the style and contents of this paper.

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ABSTRACT

Nanotechnology is used in sectors of science and technology such as energy, medicine and drugs, nanobiotechnology, nanodevices, optical engineering, cosmetics, bioengineering, nano fabrics, and in the defense sector because of its large surface-area-to-volume ratio. Nanomaterials are classified based upon their origin and structure. The classifications which depend upon the origin are natural nanomaterials, artificial nanomaterials, zero-dimensional, one-dimensional, two-dimensional, and three dimensional. Structural classification nanomaterials are classified into four types: carbon-based, metal-based, dendrimers, and composites. Size distribution is the most important information in dealing with nanomaterials. With decrease in size there is increase in portion of surface atoms. Ultrasound causes high-energy chemistry that is accomplished through the process of acoustic cavitation. Ultrasound is used as an important source for the initiation or enhancement of catalytic reactions, in both homogeneous and heterogeneous systems. Ultrasound technology is already into the research field providing nanomaterials or being implicated in decoration procedures of catalyst supports for energy applications and material production. Ultrasound (US) is largely used to accelerate chemical reactions (especially organic syntheses) and in degradation and hydrolysis reactions. US has been only sparingly used to assist analytical reactions despite the proven high potential of this form of energy for their acceleration. Nanomaterials have large surface-area-to-volume ratio that differs in orders of magnitude greater from the macroscopic materials. The size and structure of the nanomaterials depend upon factors such as surfactant additives, reactant concentrations, temperature, solvent conditions on the time of synthesis, and salt. In order to develop reproducible synthesis of nanomaterials, characterization of nanomaterials is found to be important. Characterization refers to the study of composition, structure, and other properties such as physical, chemical, electrical, and magnetic. Many techniques are available for the characterization of nanomaterials, but a degree of uncertainty is seen in each technique. photocatalytic process is environmental friendly, inexpensive and save process for air disinfection, nanomaterial as catalyst under the suitable wavelength of light in photocatalytic process produce reactive oxygen species which react with air pollutant and decomposed them.

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CHAPTER ONE
GENERAL INTRODUCTION

The term nano particle is commonly referred for individual molecules as well as inorganic materials. The definition of nanoparticles and ultrafine particles is always similar since during the 1970s and 1980s, the first fundamental studies with nano particles under the United States and Japan were called as ultrafine particles since they can exhibit size related properties significantly different from those either fine particles or bulk materials. Nano particles acts as a bridge between bulk materials and atomic or molecular structure and are of great interests. The nano size particles provide size dependent properties while constant physical properties regardless of its size is observed in case of bulk materials. The unexpected and interesting properties of nano particles are attributed to the large surface area which is dominated by the contributions made by small bulk of the materials. Textile fibers are also attached to nano particles in order to create smart and functional clothing. It is possible to control the fundamental properties of materials by creating nanometer scale structures such that melting temperature, magnetic properties, charge capacity and even their color without changing the chemical composition. Nano particles can be synthesized by several methods including coprecipitation, hydrothermal synthesis, inert gas condensation, ion sputtering scattering, microemulsion, microwave, pulse laser ablation, sol gel sono chemical, spark discharge, template synthesis, biological and chemical synthesis. [1]
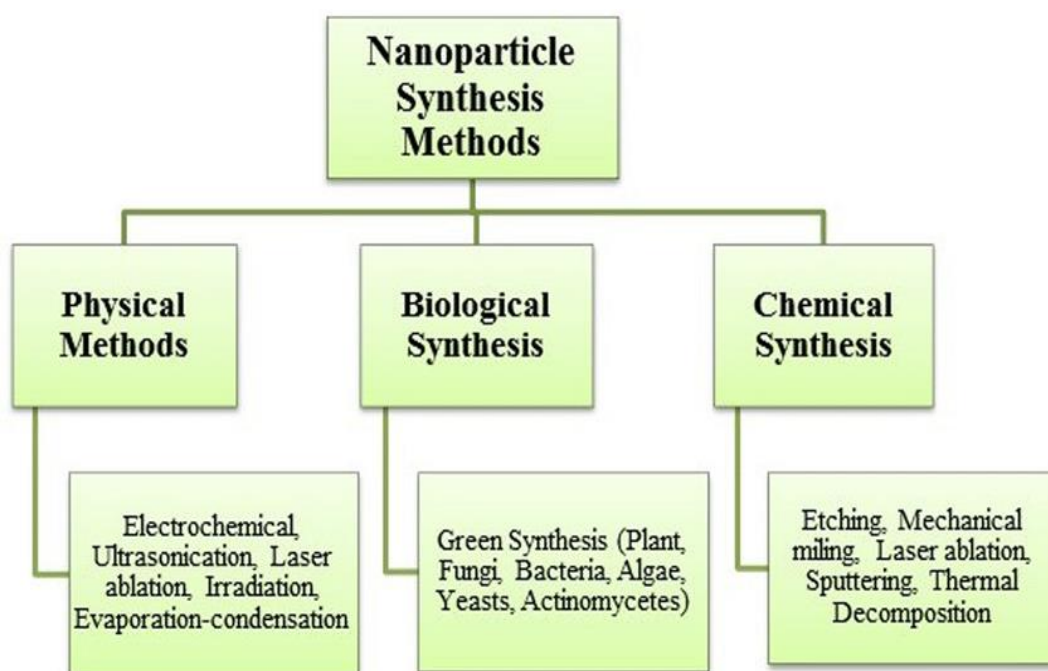
Fig 1 Methods of Nano Particle Synthesis

In chemical synthesis, a novel approach called ultrasonic irradiation has been extensively employed, which has become an important tool for the synthesis of nano particles. Ultrasonic cavitation is an effect that occurs when an ultrasonic wave travels through a liquid medium. It involves a high number of microbubbles that form, grow, and collapse in a matter of a few microseconds. Ultrasonic cavitation will form when liquids are irradiated with ultrasonic irradiation that produces a variety of physical and chemical effects such as high temperature, pressure and cooling rate providing a unique environment for chemical reactions under extreme conditions. Ultrasound is preferable for the preparation of nanoparticles with controllable morphologies. Ultrasound is required as an important source for the initiation and enhancement of catalytic reactions, in both cases of homogeneous and heterogeneous reactions. [2]

In the past three decades, the utilization of ultrasound irradiation in organic and chemical synthesis has increased significantly. The interplay of matter and energy, which encourages chemical and physical changes, is approached in a relatively new way. Ultrasound irradiation has enhanced the conventional reactions that employ strongly acidic conditions, chemicals, high temperatures, lingering reaction times, inconsistency with other functional groups, and subpar yields. [3]

The unconventional energy source with significant promise for a range of applications, from laboratory to industrial sizes, is ultrasound irradiation. Compared to the typical pathways used during conventional heating, ultrasound may result in better reaction kinetics and unanticipated response mechanisms. Sonochemistry hence has the potential to alter and/or improve product selectivity. In addition, this technology represents a less energy-intensive approach with minimal byproduct formation.

The method of ultrasonication is an important component of efforts in order to develop the green processes. In the area of polymer science, ultrasound acts as an initiator by the destruction of chemical bonds of molecules and thus enhance the rate of polymerization and found as one of the most efficient methods for the preparation of stable monomer droplets without any aggregation during the emulsion polymerization process. [4-8]

In this review report, we cover the comprehensive explanation on the basic mechanism of ultrasonic assisted synthesis of nano particles including the parameters affect the synthesis process as well as their applications, advantages, disadvantages along with different characterization methods of nano particles and their photocatalytic activity.

CHAPTER TWO
LITERATURE REVIEW

❖ *Basic Mechanism of Ultrasonic Assisted Synthesis of Nanoparticles:*

The most important and commercially adaptable protocol in order to get a wide range of nano architectures, ultrasonic assisted synthesis is suitable as it is cost effective and able to produce large scale product in a short time with sufficient control of particle dispersion and size. The method doesn't require using of any capping agent and can be combined effectively with other synthetic method such that electrochemical, solvothermal, sol gel methods etc. in order to get more efficient result. [9-11] The pressure during the reaction inside the reaction vessel is created due to sonochemical effect and collapsing bubbles is produced through the mixture because of acoustic cavitation that results in moderate pressure with high temperature. [10,12]. This pressure is responsible for the dissociation of water into two reactive primary ions, H^+ and OH^-, which are responsible for the reaction being initiated. The size and morphology of nano composite by varying exposure time can be controlled by useful probe sonication method. It is observed that the time of ultrasonication is inversely proportional to the particle size. Again, polyhedral plate like of 200-300 nm range was achieved for three times of ultrasonic exposure and also circular shape morphology of particle size 80-150 nm is obtained by increasing the interval to five minutes. [13]

The sonochemical degradation can be determined by the nature of organic compound in terms of volatility, hydrophobicity and surface activity. [14] Thermal degradation of non-volatile organic compounds inside the cavitation bubble is found as negligible during acoustic cavitation since NVOCs are highly surface-active compounds and readily undergo thermal decomposition at the bubble surface. [15]. Moreover, concentration of OH^- radical is found high around the bubble surface as compared to the bulk of the solution. The OH^- group is responsible to contribute the degradation conveniently by attacking the functional groups in NVOCs. Again, hydrogen atoms are converted into HOO^- radical in oxygen rich solution in order to get effective and fast decomposition of organic materials. In general, the slow recombination of H^+ and OH^- takes place in presence of air that increase the formation of HOO^- radicals together with OH^- radical from bulk of the solution. Both ions are powerful oxidants that can breakdown the organic pollutants. Hence, the oxidation process such as ozonation uses ultrasound irradiation. [16,17]

$$H+ + O_2 = HOO^-$$
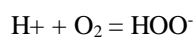
In general, reduction of metal oxide is induced in two different pathways by sonolysis. In the first reaction, solute that doesn't diffuse into gas bubbles will undergo H^+ and OH^- radicals attack produced from homolysis of water. The H^+ radical doesn't directly reduce the metal oxides and rapidly combine with water molecules within the bubbles as they are the primary active species responsible for the reduction process and have lesser chance of interaction with the substrate in the bulk solution. It is found that 80% of H^+ and OH^- radical combines. [18,19]. Adding surfactant like PEG or alcohol will act as radical scavenger consequently. As a result, a significant number of radicals escape from recombination and react with surfactants or organic molecules in the bulk solution that induce various redox reactions. [20,21]. The probability of radicals escaping before recombination is higher as the collapse of cavitation bubbles occurs rapidly at higher frequencies. Typically, H^+ radicals abstract a H from $-CH_2$ of $-OCH_2$ chain, in this case from PEG, forming in the ratio of 1:4 to OH^- radicals. This is due to the process of recombination of H^+ to H_2 gas and its reaction with water molecules, where OH^- form H_2O_2. [22,23]

Besides, the primary radicals and the hydrocarbon radicals are also formed from thermal degradation of alcohol that enter the cavitation bubbles during ultrasonic treatment. [20]

➢ $H_2O = H^+ + OH^-$
➢ $H^+ + H_2O = H_2 + OH^-$
➢ $H^+ + H^+ = H_2$
➢ $R\text{-}(OCH_2CH_2O)_n\, CH_2OH + OH^-/H^+ = R\text{-}(OCH_2C^+HO)_n + CH_2OH + H_2O/\, H_2$

In the second reaction, volatile organic compounds undergo vaporization and destruction instead of homolysis of water during the collapse of microbubbles via direct pyrolysis and combustion reaction. Higher temperatures are conducive to effective formation of radicals. The formed organic molecules consequently initiate the reduction process to take place. [20,24] Sparging gas with high specific ratio is preferable that allows the creation of higher temperature upon collapse of microbubbles since higher temperature are conducive to effective formation of micro bubbles. The generation of radicals due to the generation of higher temperature are favorable at higher ultrasound frequencies. [24]
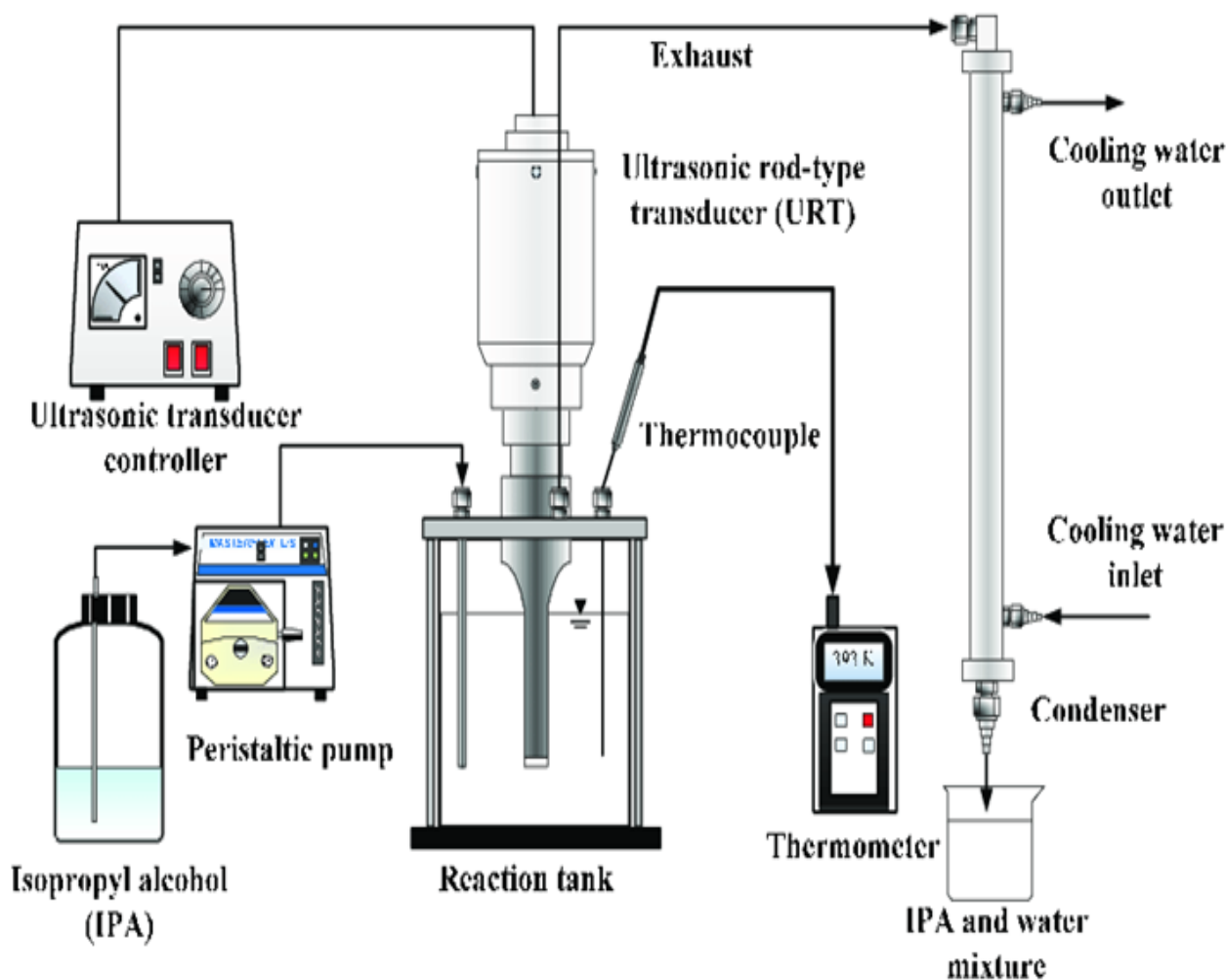
Fig 2 Instruments Ultilized in Ultrasonic Assisted Synthesis of Nano Particles.

A. The Parameters Involve in the Ultrasonication Technique is Given Below:

The selection of starting material in order to determine the size, thickness, as well as the number of layers formed due to various lateral dimensions and crystallinity of the precursor. For example, to produce graphene sheets, there is a choice of either exfoliating graphite or graphite oxide particle. For instance, artificial graphite having small dimension and low crystallinity will produce graphene sheets in single layers predominantly. [25,26]

The optimum sonication time and its intensity based on size is important parameter to form a homogeneous suspension that will remain stable for months. For example, it is suitable to obtain long graphene nanoribbons, that will require shorter sonication time compared to the long hours which will cause more breakage of the sheets and it is observed that uniform size of graphene is produced when the sonication time is extended without increasing the power. [26]

The power intensity of the sonication plays vital role for an ultrasonic assisted catalysis reaction. For example, although the successful amidation reaction was observed only at 360 and 480 W with similar conversion rates, but the amidation reaction between chitosan and graphene oxide were not suitable to induce any reaction at 12o and 240 w due to the requirement of optimum intensity of cavitation in order to activate the carboxylic acid to initiate the reaction. [27]

Table 1 Application of Ultrasonic Assisted Synthesis of Nano Particles for Energy and Environment

Different Applications of Ultrasound	Comments	Reference
Removal of organic contaminants from soil and water.	Complete mineralization of organic contaminants in aqueous medium is achieved using ultrasound under diffused sunlight.	28
Agglomeration of aerosols.	Ultrasound is used for the sedimentation of superfine aerosol.	29
Combined with bioremediation.	Effect of low frequency ultrasound in enzyme catalysis, biosensors and bio sludge processing.	30
Isolation of potentially bioactive components from non-utilized by product streams.	Ultrasound can accelerate the internal diffusion, giving rise to an increased mass transfer, and allow a greater penetration of solvent into the sample matrix and thereby increase the extraction of bioactive compounds from plants and seeds.	31
Preparation of activated metals by reduction of metal ions.	Ultrasound is proven to be effective for the reduction of metal ions without any external reducing agents which is highly active in energy application.	32
Impregnation of metals, metal oxides or metal halides on supports.	Ultrasound is effectively utilized for the impregnation of metal oxide nanocomposites on graphene oxide support.	32
Reaction involving metals via in situ generated organoelement species.	Ultrasound is used for the generation of organoelement species.	33
Reactions involving nonmetallic solids.	Use of high-power ultrasound in reactions involving nonmetallic solids is reviewed.	33
Crystallization and precipitation of metals, alloys, zeolites and other solids.	Ultrasound is efficiently used to improve the metal sulfate precipitation from wastewater.	35
Modification of surface morphology and particle size by high velocity inter particle collisions.	Flowerlike, rodlike morphologies can be achieved by simply altering the ultrasonication power and time without use of any external chemicals.	36
Formation of amorphous nanostructured materials, including high surface area transition metals, alloys, carbides, oxides and colloids.		
Agglomeration of crystals		
Smoothing and removal of passivating oxide coating.		
Micromanipulation of small particles.		
Dispersion of solids.	Ultrasound generates shear forces in the liquids, useful in the dispersion of solids.	33
Intercalation of guest molecules into host inorganic layered solids.	A sonochemical approach is developed for the synthesis of $Fe_3O_4@SiO_2$ core shell NPs with tunable properties.	37
Sonochemistry of polymers	Sonochemical synthesis of nanosized metal organic lead (ii) polymer is reported which is used as precursor for the preparation of nanostructured lead (ii) iodide and lead (ii) oxide.	38
Degradation and modification of polymers.		
Synthesis of polymers.		
Synthesis of organic pollutants in water	Sonolysis of water molecules generates highly reactive OH^- radicals that are efficiently used for the complete degradation of organic pollutants in aqueous medium.	39

B. Advantages of Ultrasonic Assisted Synthesis:

➢ *The creation of nanoparticles takes place at room temperature.*
➢ *Ultrasound technology produces reactions at a surprisingly high rate. Thus, producing nanoparticles takes less time.*
➢ *The method is quick and simple*
➢ *Nanobelts, nano rings, core-in-shell, and cluster-in-cluster bimetallic nanoparticles are only a few examples of the many shapes and combinations of nanoparticles that can be created.*

C. Disadvantages of Ultrasonic Assisted Synthesis:

➢ *Acoustic cavitation can damage heat-sensitive materials.*
➢ *The process is energy intensive.*
➢ *Scaleup is difficult.*

Despite producing nanoparticles in ambient temperature circumstances, sonochemical synthesis requires a large amount of energy, which results in a low yield per unit of energy supplied. [1].

❖ *Characterization of Nano Particles:*

The factors affect the size and structure of the nano materials in order to improve reproducible synthesis of nano materials and characterization of them are surfactant additives, reactant concentrations, temperature, solvent conditions on the time of synthesis and salt. Characterization refers to the study of composition, structure and other properties including physical, chemical, electrical and magnetic. [40,41]

A. Characterization Technique Based Upon Instruments:

➢ *Scanning Electron Microscope (SEM):*

With a wide range of applications, scanning electron microscopy (SEM) is regarded as a flexible technology for analyzing micro- and nanostructures. The SEM method provides details about composition and the fundamental idea behind the topography of surfaces. Magnifications that can be changed range in the benefits of SEM from using a magnifying glass to increase clarity and imposing structure viewing. It gleans knowledge from signals emanating from certain encounters to the compositions of the materials. SEM fits into a variety of categories. Surface-imaging technique in which an electron beam sweeps across the sample surface. It depicts the atomic structure and topographic detail by signal generation via sample interactions. Topographic, morphological, compositional, and crystallographic details are among the characteristics that derived from SEM. Scanning techniques are mainly six types:

- Near Field Scanning Electron Microscopy (NSOM)
- Transmission Electron Microscopy (TEM)
- Energy Dispersive X-ray Microanalysis
- Environmental Scanning Electron Microscopy (E-SEM)
- Cryo Scanning Microscopy
- Atomic Force Microscopy
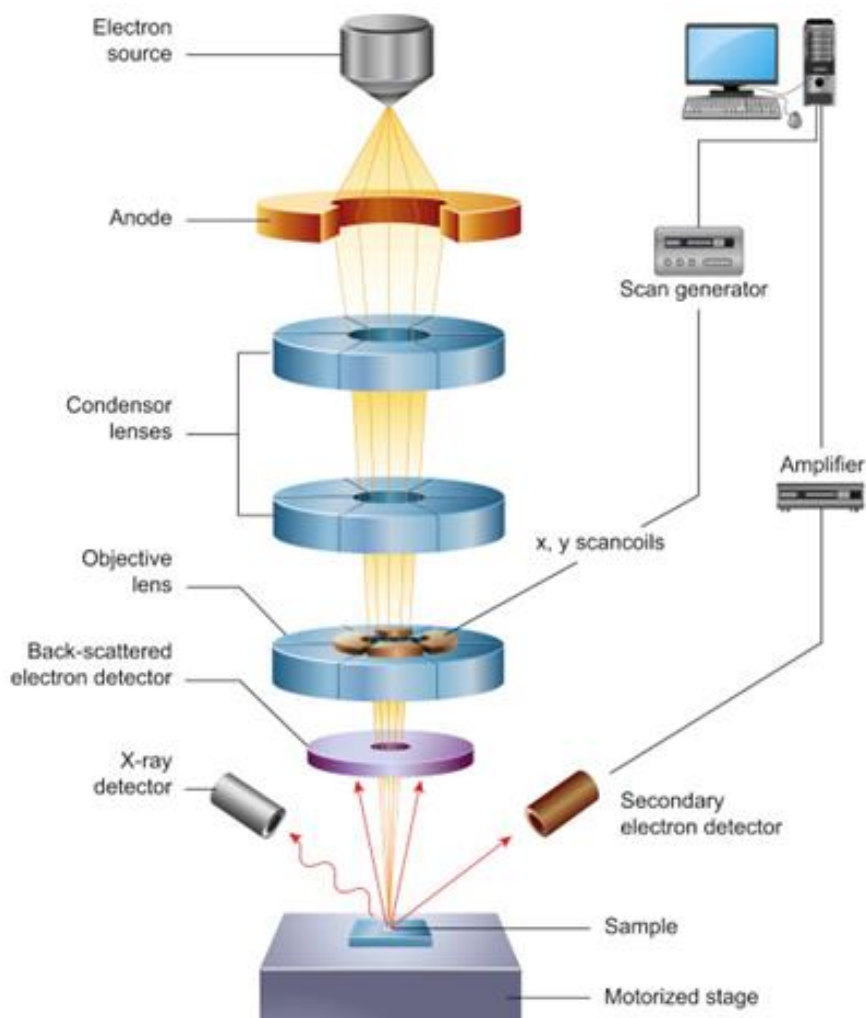
Fig 3 Working Principle of SEM

- *Near Field Scanning Electron Microscopy (NSOM):*

This technique is limited to the diffraction phenomenon of light in which the idea of surface probe microscopy and optical microscopy to exceed far field resolution limits are observed. The laser light is adjusted to emit optical fiber to the tip of the aperture which closer in proximity to the object instead of equipping objective lenses. Though it includes distribution of single molecules on the surfaces of the cells as well as associations in proteins nut the nano particles conjugated at nanoscale spatial determinations that facilities the characterization techniques. The major limitation of this technique is the output of high-resolution images, excitation of weak fluorescent molecules and imaging of surface features. [42,43]
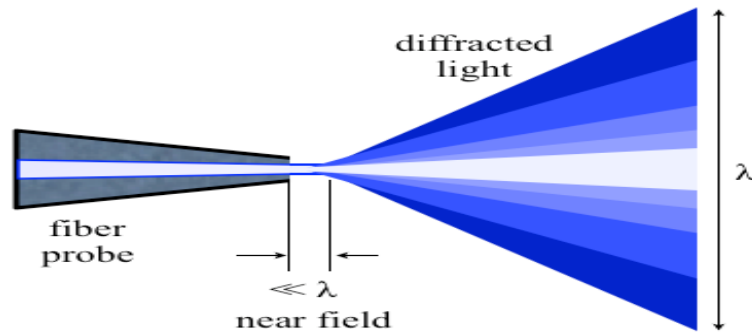
Fig 4 Working Principle of NSOM

- *Transmission Electron Microscopy* (TEM):

The most widely used method for characterizing nanomaterials in electron microscopy is thought to be transmission electron microscopy (TEM). Using TEM, pictures of nanomaterials are produced with a spatial resolution equivalent to that of atomic dimensions. When the electron beam meets the foil specimen, it changes it into elastically or inelastically scattered electrons. a measure of the separation between the specimen, the picture plane, and the objective lens are regarded as having been enlarged by the lens. TEM and SEM both display the size, level of aggregation, dispersion, and heterogeneity of the nonmaterial. TEM is advantageous in providing analytical measurements and spatial resolution and precise particle size of bright field images as well as dark field images.
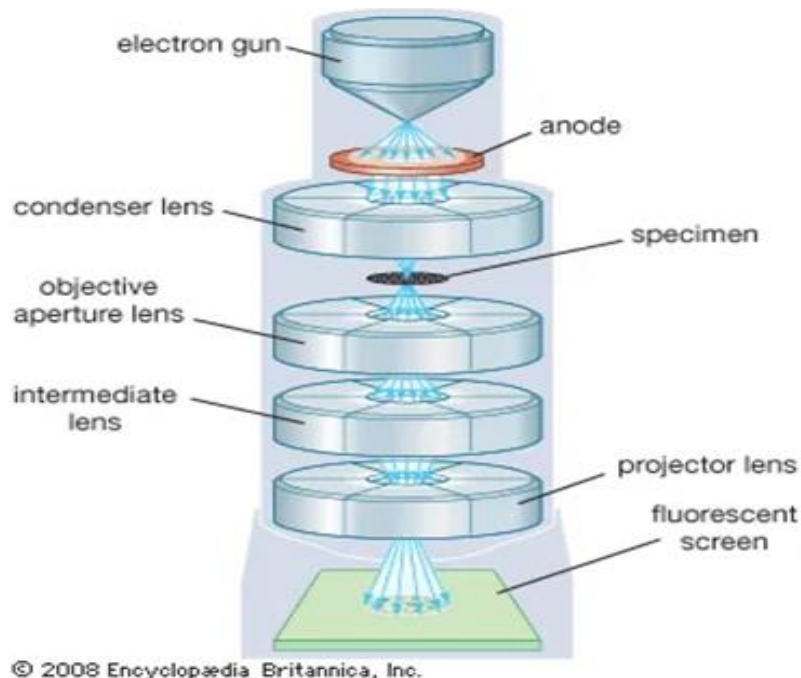
Fig 5 Working Principle of TEM

Also, details information of nanoparticles can be obtained as it utilizes energetic electrons in order to provide morphologic, compositional and crystallographic information. Although TEM performs under the principal of optical microscopy in which electrons are replaced by photons while the glass lenses by electromagnetic lenses to view images in a screen rather than eyepiece, but TEM has benefits in powerful magnification and the provision of information regarding compound and element structures. [44]

- *Energy Dispersive X-ray Microanalysis:*

Energy-dispersive X-ray (EDX) uses a sample matrix and electron beam energy to achieve interaction volume and resolution that are on the order of a few cubic micrometers, and it is used to quantify micrometer dimensions. Based on the specimen of interest, these elements are typically attached using SEM or TEM. Elemental mapping and sample image analysis are both available through EDX. Its use is primarily found in forensic science investigations and in the analysis of tainted samples. Nondestructive analysis of the sample can be performed without any prior sample preparation. Some benefits of employing EDX include quick contaminant and source detection, environmental factor control, increased production yield, and problem source identification. [45]
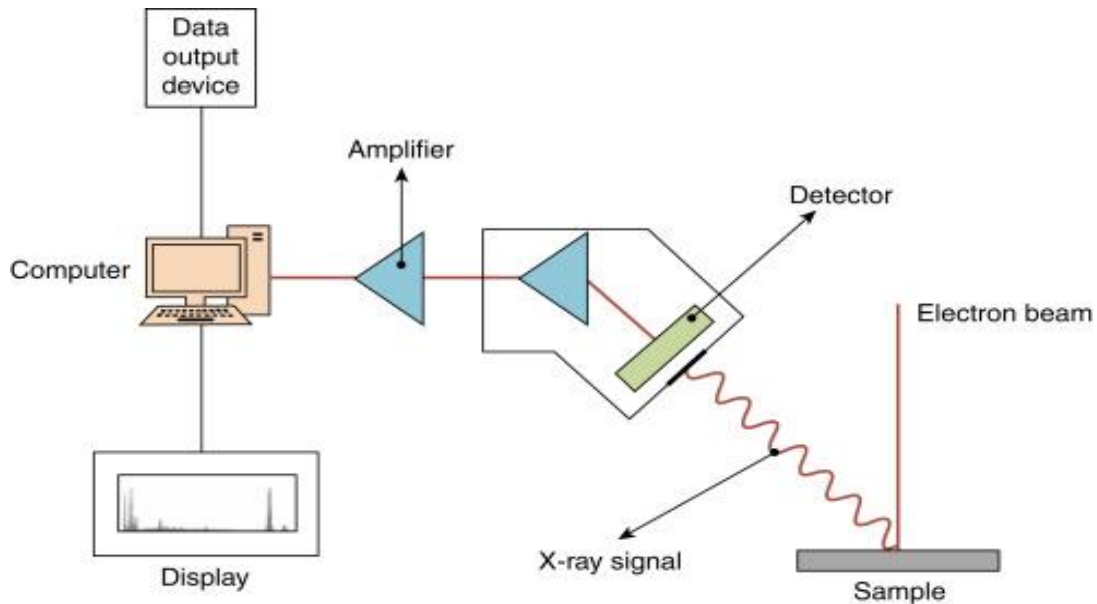
Fig 6 Working Principle of ED Xray Microanalysis

- *Environmental Scanning Electron Microscopy (E-SEM):*

While preserving the ultrahigh vacuum necessary for beam production in the column, this kind of device enables varying pressures inside the microscope sample chamber. The name "variable-pressure SEM" or "low-vacuum SEM" for environmental scanning electron microscopy (E-SEM) only depends on the instrument's maker and the range of pressure adjustments. When a sensitive sample is placed in the microscope chamber and an ultrahigh vacuum is needed in the column, E-SEM is used to separate the two differing pressure levels. Used to analyze topography and morphology at a resolution of 1.2 nm. E-SEM offers the capacity to carry out the measurement in a humid environment. The employment of a field emission gyroscope significantly improves signal-to-noise ratio and spatial resolution. [45,46]
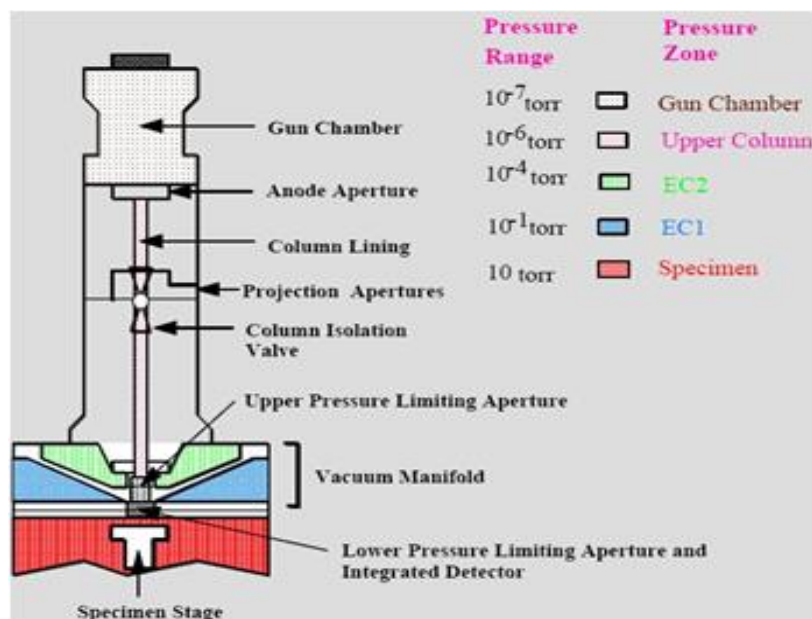
Fig 7 Working Principle of E-SEM

- *Cryo Scanning Microscopy:*

Some samples must be cooled before SEM analysis. In such cases, the sample must be prepared under cryo-conditions and transported to the microscope while still cool. The sample can be prepared using one of two methods under cryogenic conditions:

✓ Making use of the apparatus as a plunger, cryo-ultramicrotome, or freeze dryer. In the cold sample, prepped and moved into a cryo-stage for the SEM chamber.
✓ Inside the cryo-SEM, samples are prepared.

The sample containing moisture needs to be frozen using liquid nitrogen before loading into the cryo-SEM. The frozen sample is loaded into the specimen processing stage with the aid of an airlock system. The sample is sliced with a cold knife to inspect the internal structure. The sample must be coated with Au for imaging, and controlled etching must be performed on the split surface to remove the ice. [47,48]
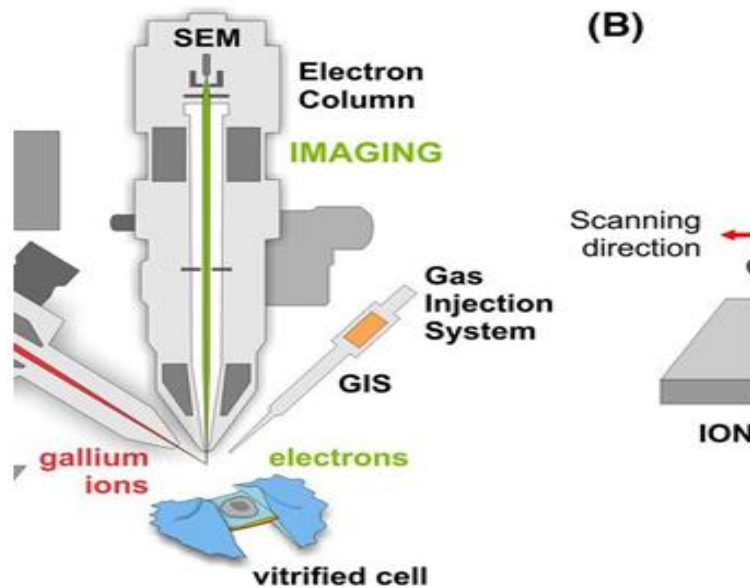
Fig 8 Working Principle of Cryo SEM

- *Atomic Force Microscopy:*

According to Hook's law, which states that the probe should move close to the specimen's surface, atomic force microscopy (AFM) operates. AFM is used to measure local parameters such as height, friction, and magnetism with a probe. An example of scanning probe microscopy is AFM (SPM). The probe measures the sample's local property while concurrently scanning across a small portion of the sample. AFM controls the force while measuring it on the sample and enables picture capture at low forces. The feedback loop, which regulates the tip height, the cantilever, and the optical lever, exhibits tube scanners. [49]
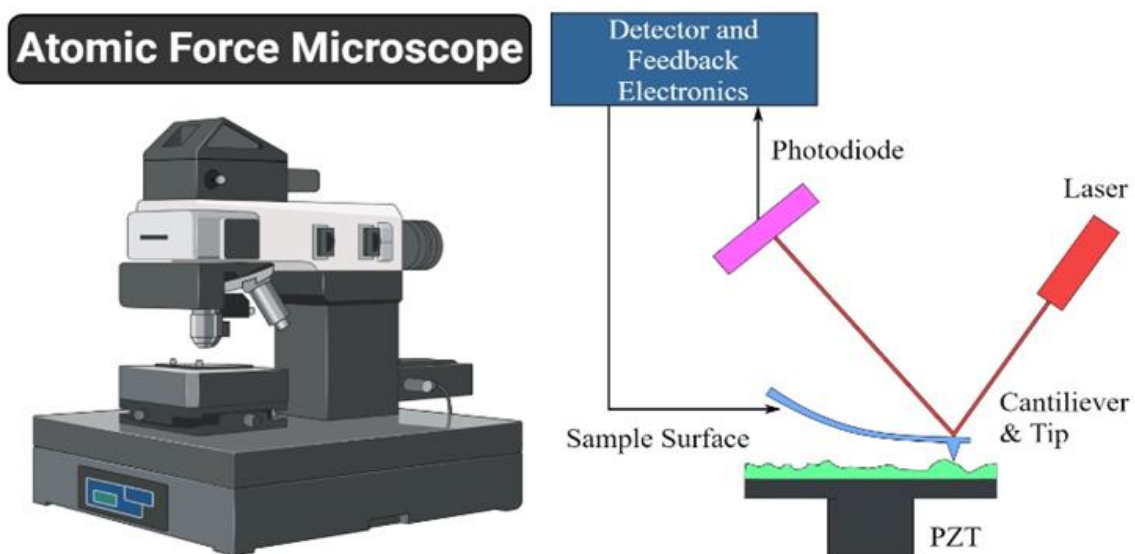
Fig 9 Working Principle of AFM

➢ *Spectroscopic Techniques:* Mainly Three types
- UV/Visible spectroscopy
- Infrared Spectroscopy
- Surface-enhanced Raman Scattering (SERS)

- *UV/Visible Spectroscopy:*
      This method is used to characterize various kinds of organic, inorganic, and biological materials. This technique counts the amount of light that enters a sample. It is a tool for locating, describing, and researching nanoparticles. UV spectroscopy is used to investigate the connection between the absorbance spectra and particle size distributions of quantum-sized nanocrystals. UV spectroscopy was discovered to be one of the best methods for assessing the size, shape, and surface characteristics of metal nanoparticles in the form of colloidal dispersion systems. Additionally, UV spectroscopy can measure the quantity of metal ions employed as a precursor during the production of metal nanoparticles. A UV visible spectrometer is employed in the fields of medicine, research, forensic investigation, etc. because of its two benefits: easy use and speedy substance analysis. A double-beam UV visible spectrometer's performance suffers, and its replacement costs are double the initial capital expenditure when its mirrors are covered in dirt or filth. The noise produced by the sample source or the instruments' electronic components reduces the device's sensitivity and measurement accuracy. [50,51]
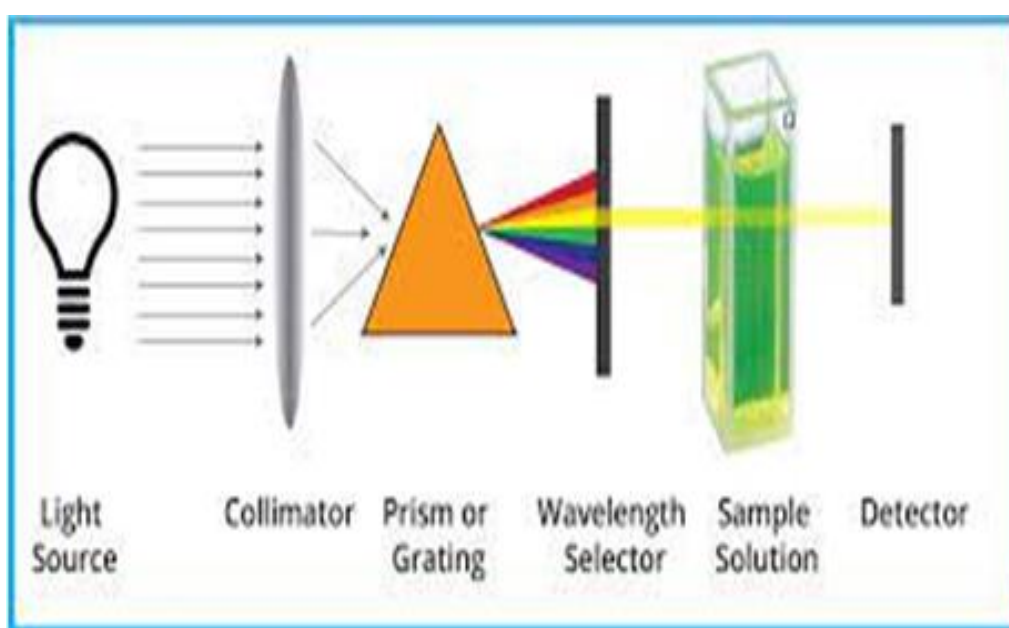
Fig 10 Working Principle of UV visible Spectroscopy

- *Infrared Spectroscopy*:
      FT-IR spectroscopy examines the existence of functional groups by assessing the vibrational frequencies of the chemical bonds. When the vibrational excitation energy of molecules is in the range of 10^{13}-10^{14} Hz, it corresponds to IR radiation and the vibrational transitions of the functional groups on the nanoparticle surface are observed by IR spectroscopy, both qualitatively and quantitatively. Thermal treatment is typically utilized because it polishes the sample's surface, weak species that have been physisorbed and chemically sorbed, and it is purely according to the temperature. An electron acceptor, an electron donor, a proton donor, and a proton acceptor are all corresponding to Lewis acids and basic sites as well as Bronsted acids and basic sites. Although it is a non-invasive and non-destructive method, the signal-to-noise ratio is high. IR is used to determine the nanoparticle's size. Attenuated total reflection infrared spectroscopy, or IRATR, is a method for detecting agent-induced changes within a cell. Multivariate methods like principle component analysis and linear discriminant analysis help to distinguish between inferred IR spectra and the results of combining ATR and other techniques. The disadvantages include the fact that the substance's molecular weight isthe purity of the substance cannot be assessed using the IR approach while dealing with an unknown chemical, the relative positions of distinct functional groups in a molecule are unknown, and these factors all apply. [52-54]
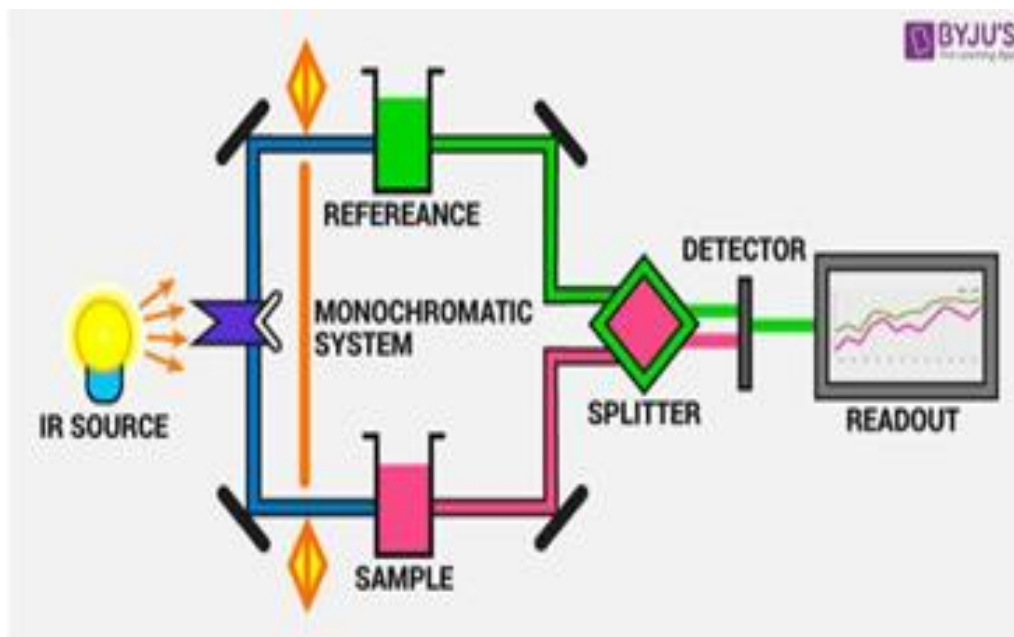
Fig 11 Working Principle of IR

- *Surface-enhanced Raman Scattering (SERS):*
  RS is regarded as a simple, effective, and non-destructive method even though it is frequently used to characterize nanoparticles. Low Sensitivity is seen as a drawback in conventional Raman spectroscopy. Nanoparticle characterization is done using this method. It is regarded as a strong technique to improve RS. Surface Plasmon resonance and resonance enhancement are the two mechanisms that provide the enhancement effect. When the pieces of technology are coupled with a Raman spectrometer, the result is known as "nano-Raman," which combines atomic force and scanning tunneling microscopes.
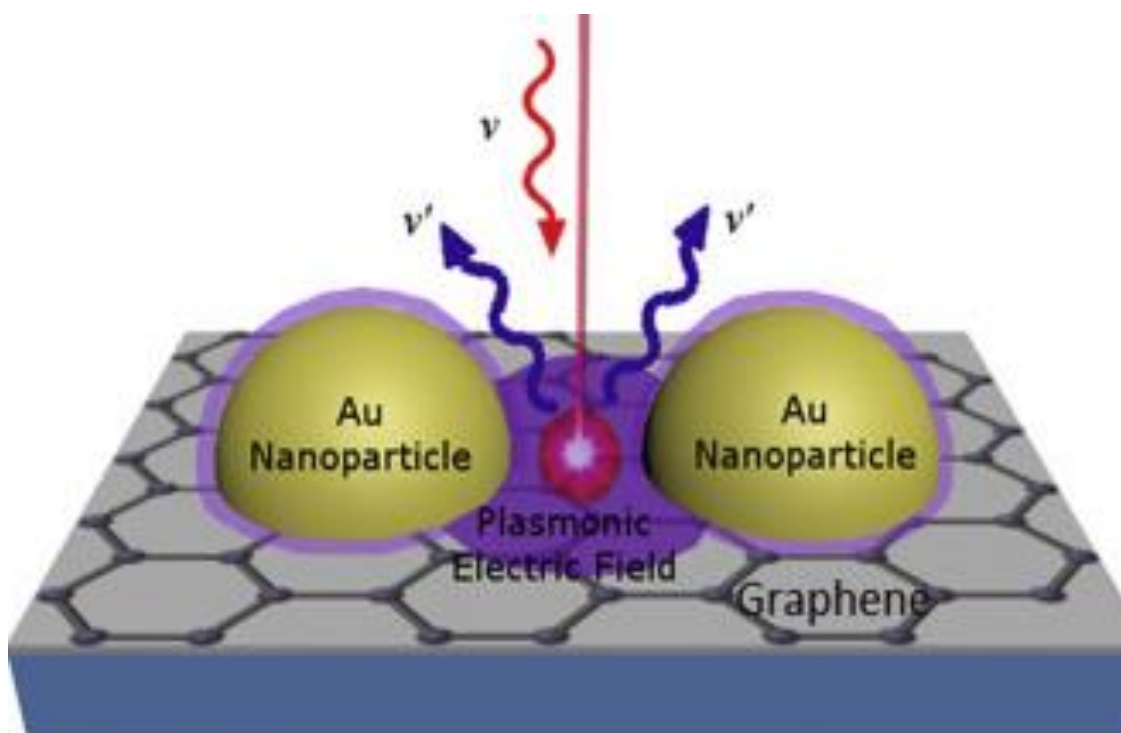
Fig 12 Surface Enhance Raman Scattering's Principle

➢ *Probe Characterization Technique:* Mainly three Types:
• Electron probe characterization
• Photon probe characterization
• Ion particle probe characterization
• *Electron Probe Characterization:* Three types mainly.
✓ Scanning probe electron microscopy (SPM)
✓ Electron probe microanalysis (EPMA)
✓ Scanning Transmission Electron Microscopy:

✓ *Scanning Probe Electron Microscopy (SPM):*
 SPM is an image-creating technique for nanoscale surfaces and structures. It monitors the strength of the interaction between the surface and the tip and uses light waves for imaging. Three-dimensional topographic pictures are acquired by scanning the surface of the material with a sharp probe that is retained at a few angstroms or nanometers due to interactions between the sharp probe and the surface of the nanomaterial. There are two scanning modes: touch and noncontact. This technology includes AFM, magnetic force microscopy, and scanning tunneling microscopes. The ability to monitor slight variations in object height and the use of specialized probes to quickly and effectively photograph specimens are two benefits of SPM. Due to the smaller maximum image size, it cannot be used for surfaces that are solid, solid, or liquid. SPM is used to gain an understanding of the topographical, electrical, and magnetic properties of the nanomaterials. Information can also be conveyed to samples via SPM.
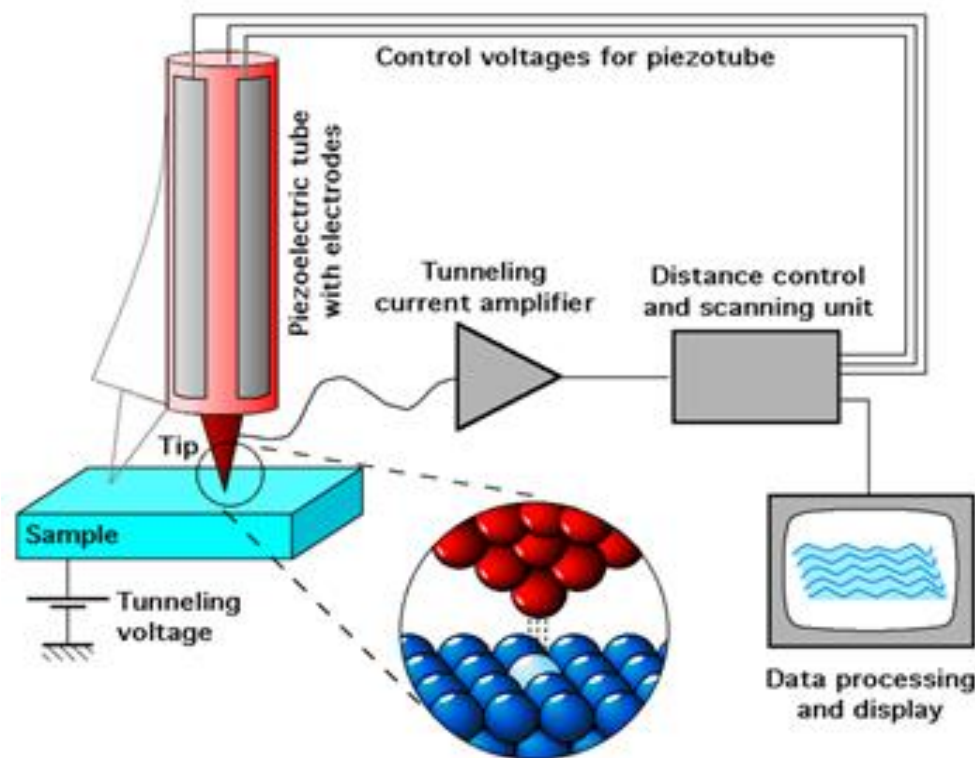
Fig 13 Working Principle of SPM

✓ *Electron Probe Micro Analysis (EPMA):*
 Materials in multiphase can have their local chemical composition identified using electron probe microanalysis (EPMA). It provides a nondestructive in situ chemical analysis that produces a detailed image of the material. The X-rays generated by the specimen are the basis for this approach. It is based on the idea of bombarding a solid material with an accelerated and concentrated electron beam. The incident electron beam has the power to release matter and energy from the sample. Heat, derivative electrons, and X-rays are released during the electron-sample interaction. These X-rays are used to examine the material's surface and typical composition. Using EPMA, the geological materials are examined. With EPMA, the lightest elements cannot be detected, and different valence states cannot be detected. EPMA is used to analyze distinct stages. In some circumstances, it is possible to determine a mineral's age without using isotopic ratios.
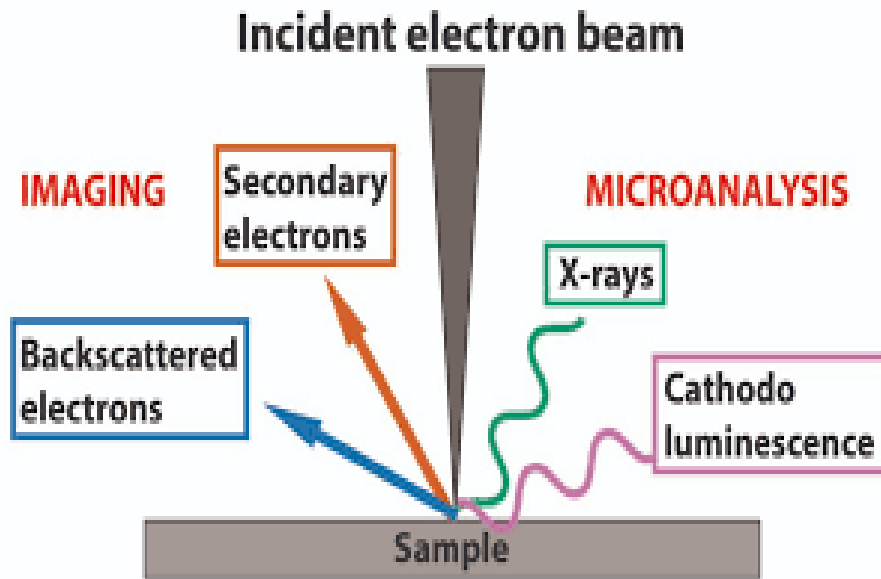
Fig 14 Working Principle of EPMA

✓ *Scanning Transmission Electron Microscopy (STEM):*

Analysis can be done on any of the two instruments in scanning TEM, which combines the TEM and SEM principles. Various electron, electromagnetic, and other signals can be produced when the electron probe interacts with the specimen inside the scanning transmission electron microscope. It is possible to create photographs of the specimen using this signal. It offers information on spectroscopic data, diffraction data, and imaging data. [55,56]
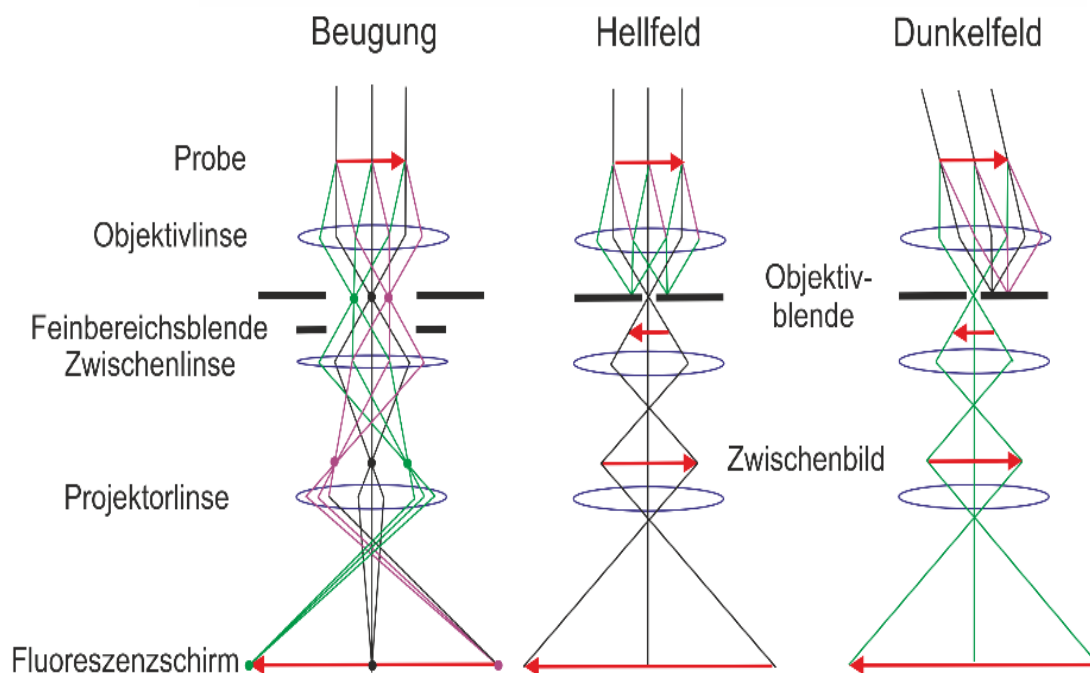
Fig 15 Working Mechanism of STEM

● *Photon probe Characterization:* Five types mainly
✓ Photo electron Spectroscopy (PES)
✓ Ultraviolet Visible Spectroscopy
✓ Atomic Absorption Spectroscopy
✓ Inductively Coupled Plasma (ICP) Spectroscopy
✓ Fluorescence spectroscopy:

✓ *Photo electron Spectroscopy (PES):*

By calculating the binding energies of the electrons present in the substance, one can calculate the number of electrons that are emitted from solids, gases, or liquids as a result of the photoelectric effect. High vacuum and sizable regions are needed for analysis in this pricey technique. There are two forms of photoelectron spectroscopy (PES):

▪ *UV PES:*

Photons with energies between 10 and 50 eV are utilized to analyze chemical bonding and valence energy levels, notably the bonding nature of molecular orbitals.

Fig 16 Principle of UV PES

▪ *X-ray PES (XPS):*

Also referred to as a surface-sensitive spectroscopic method, it measures in the parts per thousand range. By exposing the target material to an X-ray beam while both counting the number of expelled electrons and measuring the kinetic energy of the target material. XPS, also referred to as electron microscopy, is employed in chemical analysis.
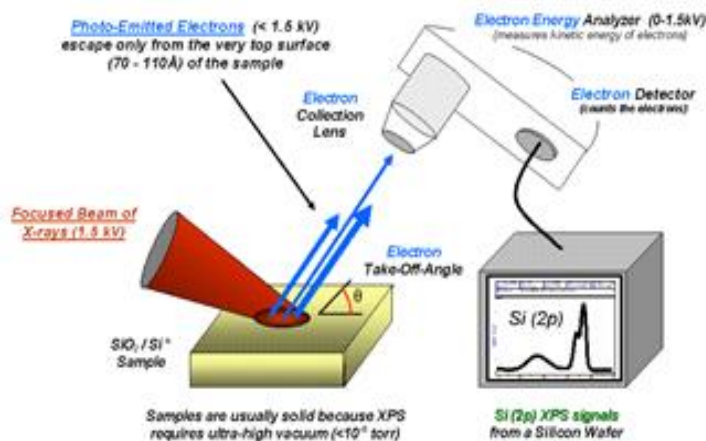
Fig 17 Principle of X-Ray PES

✓ *Ultraviolet Visible Spectroscopy:*

The molecule is capable of absorbing UV radiation. The electrons are excited by UV radiation, moving them from their ground state to a higher energy state. The amount of UV light that is absorbed is equal to the difference between the ground state and the higher state. Between 190 and 800 nm, the electromagnetic spectrum is separated into the visible (400–800 nm) and UV (190–400 nm) areas. UV spectroscopy makes use of the Beer-Lambert equation. Beer-law Lambert's follows the axiom that the extent of light absorption increases with the number of molecules capable of absorbing light of a given wavelength. UV visible spectroscopy is used to determine analyses quantitatively. The device performance degrades if there is dust or grime in the mirror, and the noise generated by the sample source as well as from the electronic components also degrades the measurement accuracy.
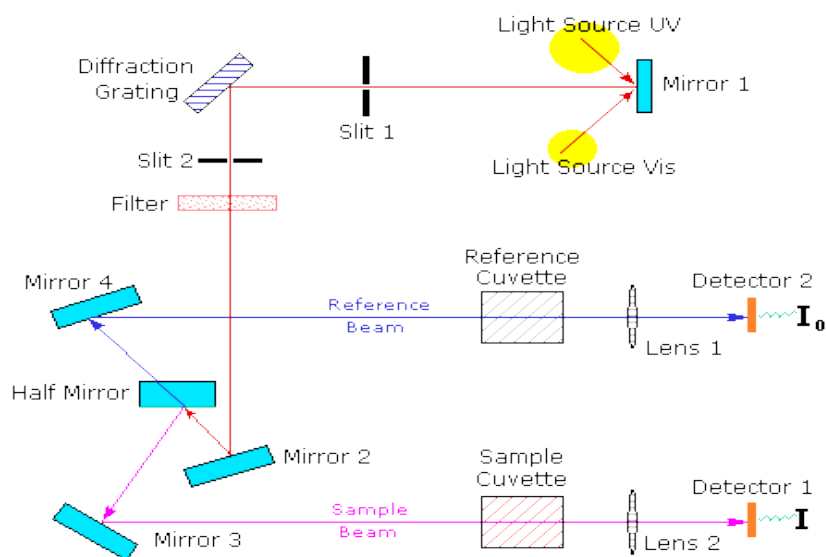
Fig 18 Principle of UV Visible Spectroscopy

✓ *Atomic Absorption Spectroscopy (AAS):*

Atomic ingestion with the aid of free atoms in a gaseous state, spectroscopy uses optical radiation absorption to identify chemical elements. It is based on the Beer-Lambert law, which establishes the relationship between the sample concentration required for analysis and the standards obtained from previous analyses. Parts per billion are measured via this delicate device. Atomic absorption spectroscopy has some drawbacks, including the ability to evaluate only solutions, the need for a sizable sample, and issues with refractory materials.
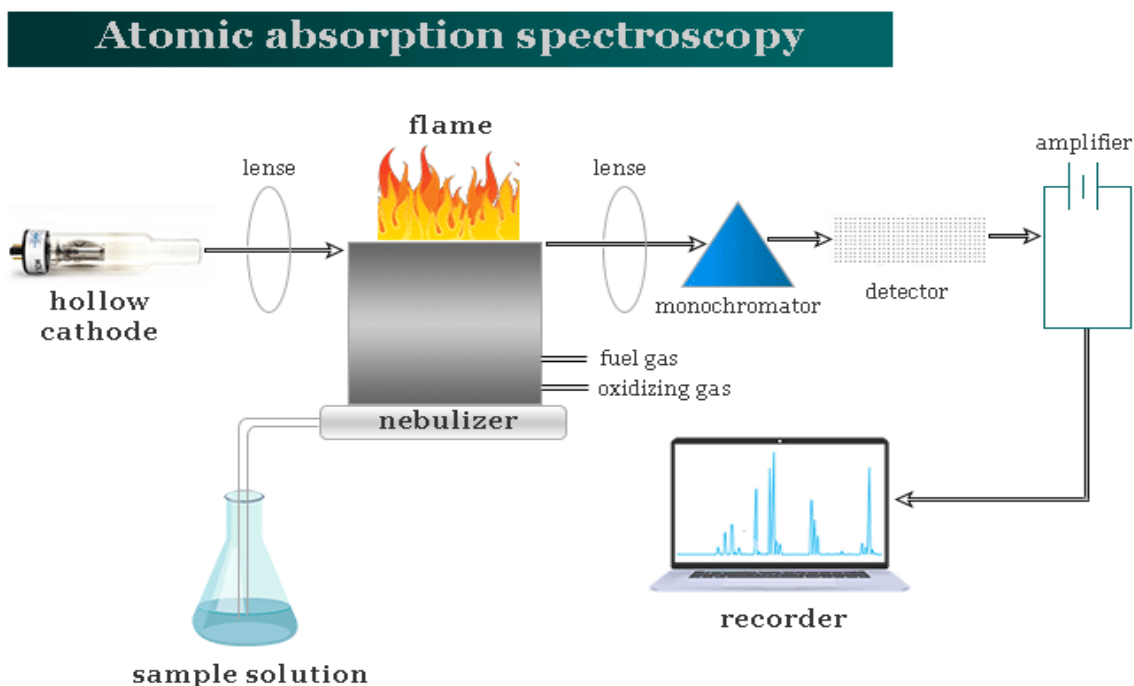
Fig 19 Principle of AAS

✓ *Inductively Coupled Plasma (ICP) Spectroscopy:*

Metals and nonmetals can be detected by ICP spectroscopy, a form of mass spectroscopy, at concentrations as low as 1 part in 10^{15}. ICP is used to ionize the materials, and a mass spectrometer is used for separation and quantification. ICP outperforms atomic absorption spectroscopy in terms of sensitivity, speed, and precision. This kind of device is utilized for measuring isotope ratios rather than for typical multi-element analysis.
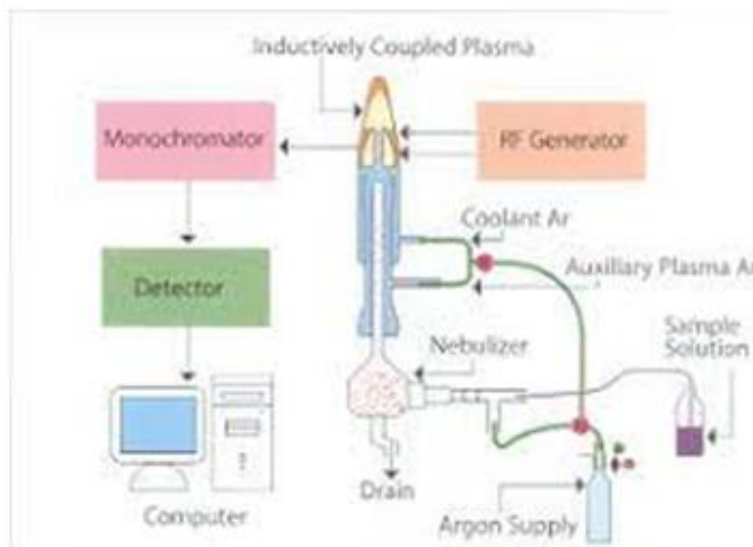
Fig 20 Principle of ICP Spectroscopy

✓ *Fluorescence spectroscopy:*

An electromagnetic spectroscopy technique called fluorescence spectroscopy examines the sample's fluorescence. Fluorescence and phosphorescence, two-photon emission processes, take place as a result of molecular relaxation from an electronic stimulated state. In the photonic process, polyatomic fluorescent molecules change from their vibrational to their electronic states. Excitation takes place, and after excitation the molecules are relaxed to the excited state's vibrational level. The excitation, which takes place at femto- to picosecond time intervals, is employed in biotechnology, DNA sequencing, forensics, and genetic analysis. Using this method, the components with fluorescence capability will be located. A contaminated fluorescence sample could result in an incorrect readout. [57,58]
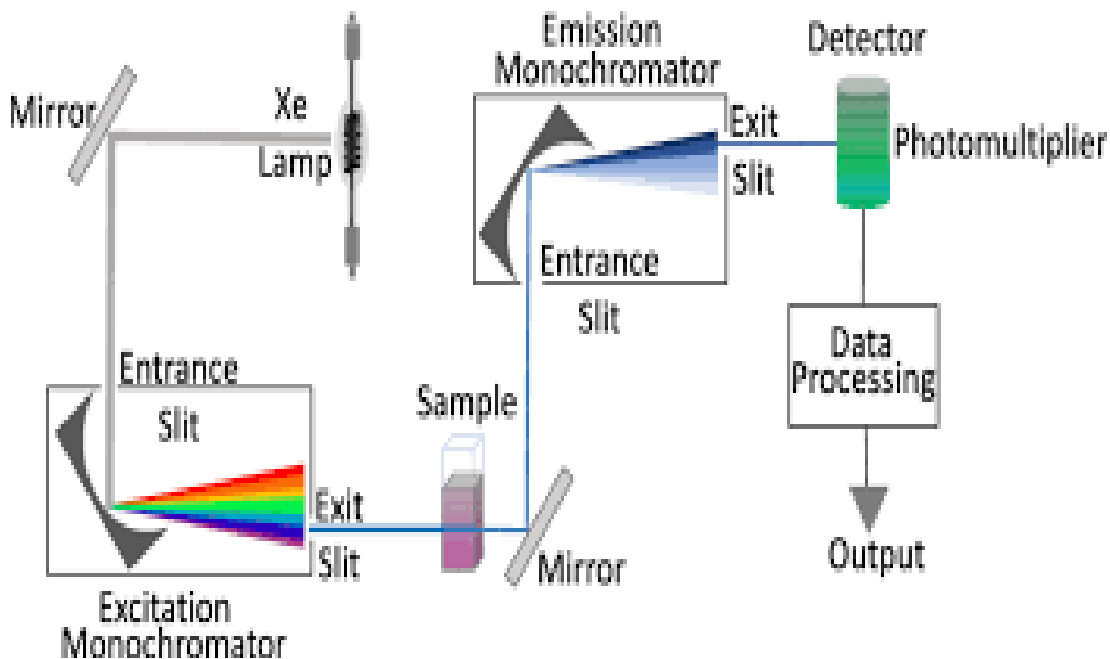
Fig 21 Fluorescence Spectroscopy

• *Ion Particle Probe Characterization:* Mainly five types.
✓ Rutherford Backscattering (RBS)
✓ Small Angle Scattering
✓ Small Angle Neutron Scattering (SANS)
✓ Small Angle X-ray Scattering (SAXS)
✓ Cathodoluminescence

✓ *Rutherford Backscattering (RBS):*

Without employing reference standards, Rutherford backscattering spectrometry (RBS) is an ion scattering method used for thin-film analysis. A backscattered yield at a specific angle is measured while high energy ions are transmitted onto the sample and the energy distribution. The thickness density of the silicide layer can be calculated using this method, which also analyzes the level of contamination. For the periodic table's lowest elements, RBS offers less accuracy. [59]

✓ *Small Angle Scattering:*

This method is used for large-scale structures ranging in size from a few tens of thousands of angstroms to several tens of thousands of angstroms, and it provides information about the shape, size, and orientation of structures. This approach is used to examine a disordered system's internal structure.

✓ *Small Angle Neutron Scattering (SANS):*

Neutron scattering methods are frequently employed for materials with lengths between a few nanometers and a millimeter. This method is used to study the size and shape of the particle dispersed in a homogeneous medium. The scattering of a monochromatic neutron beam and the measurement of the scattered neutron intensity as a function of scattering angle form the basis of small-angle neutron scattering (SANS). The main drawback of SANS is that thin films and substrates are incompatible with it.

✓ *Small Angle X-ray Scattering (SAXS):*

Small-angle X-ray scattering (SAXS) provides structural analysis and physical information for a sample between 1 and 100 nm by describing the average particle size and shapes. This method is used to determine details like pore size, surface-to-volume ratio, lattice type, and dimensions. While SAXS offers various properties for crystalline or amorphous materials, XRD is restricted to crystalline materials. Part of the incident X-rays are elastically scattered in this method. A 2D X-ray detector picks up a scattering pattern from the sample that is perpendicular to the direction of the incident X-ray beam. A variety of nanomaterials and polymers can be studied using scattered X-ray data that has been gathered inside the scattering angle to learn more about their sizes, shapes, orientations, and structures. It uses a nondestructive way to deliver comprehensive information on the structure and information about a nonrepeating structure. The method's drawback is that it produces low-resolution photos. [60]

✓ *Cathodoluminescence:*

An amalgam of optical and electromagnetic phenomena is cathodoluminescence. It is the opposite of the photoelectric effect, in which exposure to photons results in the emission of electrons. Cathodoluminescence is regarded as a method for getting the material's composition, optical, and electrical properties, and comparing them to the microstructure, composition, and morphology in a SEM. The given sample is fully described, along with correlations. The disadvantage of this method is that it can only evaluate samples that have a luminescence property. [61,62]

B. Characterization Based Upon Nano Material Properties:

➤ *Optical Characterization Techniques: Mainly five types.*
- Confocal Laser Scanning Microscopy
- Scanning Near Field Optical Microscopy
- Two photon Fluorescence Microscopy
- Dynamic Light Scattering (DLS)
- Brewster Angle Microscopy

- *Confocal Laser Scanning Microscopy:*

Confocal microscopy's capacity to capture blur-free images from dense specimens at various depths is its key selling point. Instead of projecting into an eyepiece, point-to-point information about images is gathered and rebuilt with a computer. The advantages of laser scanning confocal microscopy over optical microscopy include depth of field, the elimination of picture deterioration when images are out of focus, and the collecting of images from thick specimens. When the specimen is thicker than the plane of focus, spatial filtering has the advantage of eliminating out-of-focus light. There are various imaging modalities visible, including single, double, triple, and multiple illumination modes. Confocal microscopy scans the specimen point by point using raster scanning. In comparison to lateral resolution, the detection procedure and axial resolution were shown to be slower. Confocal microscopy exposes the entire column rather than just the optical portion, which results in photodamage. [63]
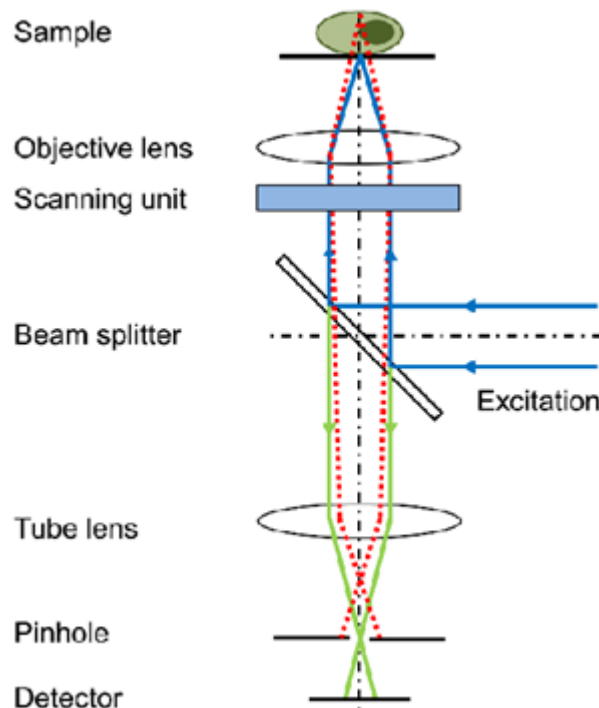
Fig 22 Principle of Confocal Laser Scanning Microscopy

- *Scanning Near Field Optical Microscopy (SNOM):*

With the aid of near-field optical microscopy, the topography and optical characteristics of nanomaterials can be assessed. This approach also offers information on the nanofeatures' surface and their optical and electrical characteristics. The near-field scanner is lighted at the back side from a constant distance and has an arbitrarily narrow aperture. The optical resolution of transmitted or reflected light is constrained by the aperture's diameter when the samples are scanned at a close range below the aperture. The optical pictures are acquired by scanning the sample surface point by point and line by line, and the optical resolution is typically 60–100 nm. High resolution pictures up to 25 nm, analysis of several characteristics, and use for various sample types are only a few benefits of scanning near-field optical microscopy (SNOM). The limitations on working distance and depth of field, lack of information on nonconductive soft materials, and lengthy scan periods for large sample regions are only a few of the drawbacks of this sort of scanning. The fields of nanotechnology, nano photonics, nanooptics, biological sciences, and material research are where this kind of microscopy is most frequently used. Using SNOM, it is feasible to determine dynamic characteristics at the subwavelength scale and single-molecule detection. [64]
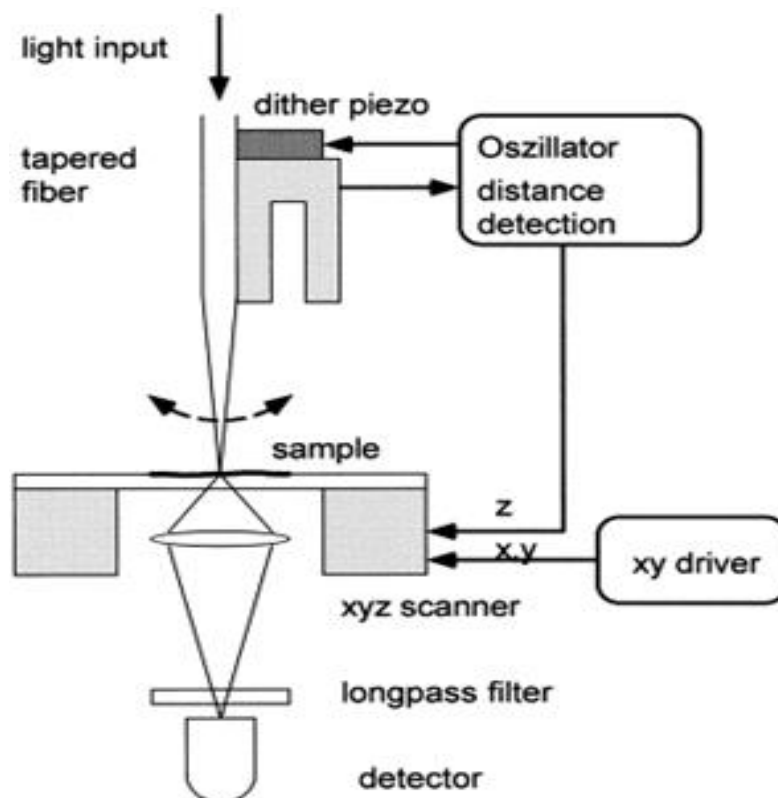
Fig 23 Principle of SNOM

- *Two photon Fluorescence Microscopy:*

Two-photon fluorescence Microscopy is used for studying biological systems. It is a nonlinear process that entails the absorption of two photons whose total energy exceeds the energy difference between the excited states and the ground state of the molecule. In a chemical transition, this energy gap is sufficient to create an excited electronic state. A fluorescent molecule's two-photon absorptions follow a quadratic function of the excitation radiance. Lower resolution is the fundamental disadvantage of two-photon fluorescence microscopy, whereas other benefits include deeper tissue penetration and less photodamage. Major applications for this kind of scanning include physiology, neurobiology, embryology, and tissue engineering. Noninvasive optical biopsy is a specific use for two photon fluorescence microscopies. [65,66]

- *Dynamic Light Scattering (DLS):*

For determining particle size and quantifying molecules in suspension, dynamic light scattering (DLS) is utilized. Photon correlation spectroscopy and quasielastic light scattering are other names for DLS. Scattering methods may be used to determine the hydrodynamic size, shape, structure, aggregation state, and biomolecular confirmation of an object. A monochromatic light source is used to determine the size distribution of polymer scattering modes from submicron to nanoscale scales. Using DLS, the temporal variations are seen. This method measures Brownian-moving particles, and the speed depends on the particle size, sample viscosity, and temperature. The particle diffuses through the medium due to Brownian motion. This device tracks the dispersed intensity with time at a constant scattering angle. The static light scattering gauges the intensity scattered in relation to angle. Using DLS, one may determine the temporal and light intensity fluctuations' timescales, as well as the average size, size distribution, and polydispersity of molecules and particles in solution. The length of experiments is determined to be brief. Many of the advantages of DLS include accuracy in hydrodynamic size measurement, measuring diluted samples, analyzing a wide range of concentrations, and providing repeatable readings. Additionally, lower prices are discovered. DLS has certain drawbacks, such as its susceptibility to mechanical disturbances, lack of selectivity, weak signal, analysis of heterogeneous size distributions, unsuitability for measuring asymmetric nanomaterials, and application only to transparent samples. DLS is used to measure the particles since it primarily focuses on the size of the particle, which includes proteins, polymers, micelles, polysaccharides, and nanoparticles. The measurement only depends on the size of the core particle, the size of the surface structure, the concentration of the particle, and the sorts of ions in the medium if the system is not dispersing in size. [63,67]

- *Brewster Angle Microscopy:*

The Brewster angle microscope is used to view thin coatings on liquid surfaces. Based on the idea that, when using p-polarized light, no light is reflected from the air–water interface under Brewster's angle of incidence, but that this idea changes when the angle of incidence is constant, causing a monolayer to develop on the water's surface. The Brewster angle condition is altered by this monolayer. The imaging of distributed or adsorbed monolayers is done using this form of microscopy. Phase

transitions, domain microstructure characterization, phase separation in mixed monolayers, and changes brought on by complex formation are all studied using Brewster angle microscopy. With this method, monolayer optical anisotropy is the main emphasis. Additionally, a refractive index other than that of water can also be utilized. [69,70]

➢ *Physiochemical Characterization: Mainly six types:*
- Fluorescence Correlation Spectroscopy (FCS)
- Raman Scattering (RS)
- Nuclear Magnetic Resonance Spectroscopy (NMR)
- Mass Spectroscopy (MS)
- Zeta Potential Analysis
- X-Ray Diffraction (XRD)

- *Fluorescence Correlation Spectroscopy (FCS):*
　　Spontaneous intensity fluctuations, such as those that occur when fluorescence correlation spectroscopy is used to get quantitative data such as hydrodynamic radii, average concentrations, chemical reaction rate, and diffusion coefficients (FCS). The binding kinetics between the donor and acceptor are examined using FCS and its derivatives. FCS has an advantage over DLS in that it just needs a minimal quantity of fluorescent particles to monitor probe particles. FCS also stops the medium from making disruptive contributions. The benefits of FCS are due to the careful selection of fluorophores with low photobleaching, high quantum yield, and low extinction coefficient. The lack of models, which restricts FCS applications, is one of the drawbacks.
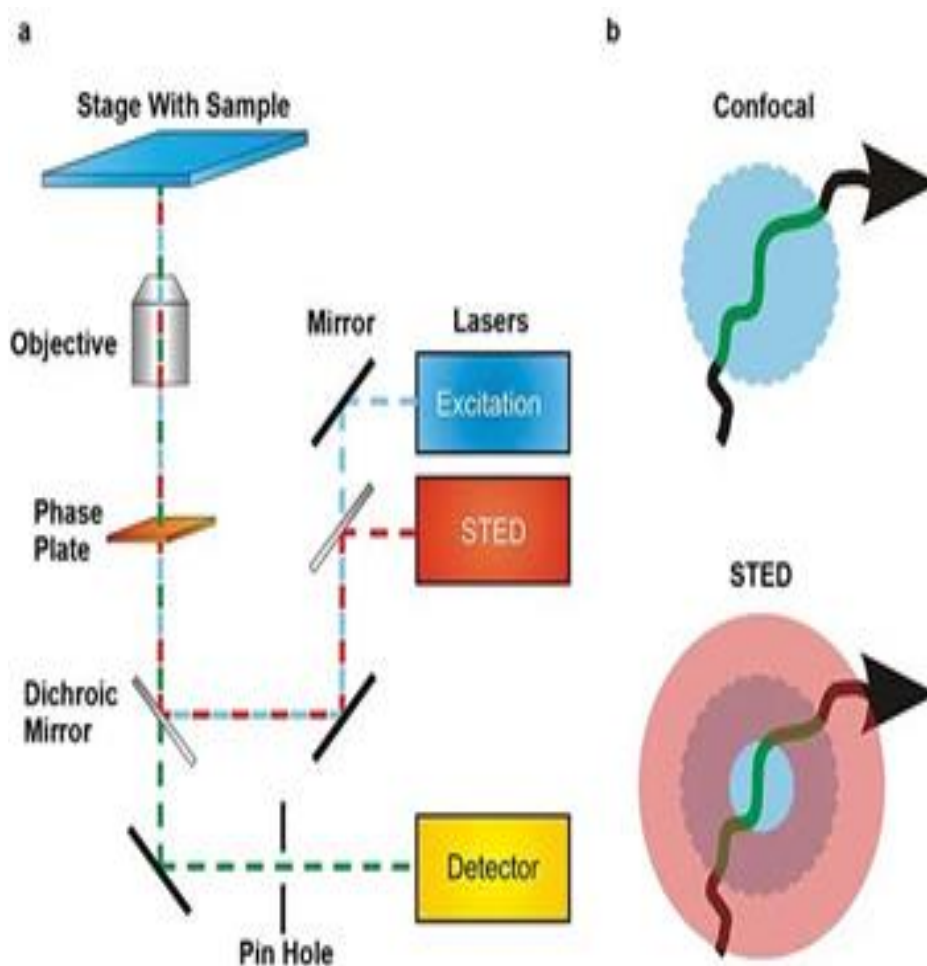
Fig 24 Principle of FCS

- *Raman Scattering (RS):*
　　Structural characterization of nanomaterials is done using Raman scattering (RS). The fundamental idea behind RS is to measure the inelastic scattering of photons of various frequencies emitted by incoming light after they have interacted with the electric dipoles of the molecule. RS is discovered to be advantageous to IR spectroscopy. Further tissue abnormalities can be detected with RS since water molecules employed for investigating biological materials in aqueous solutions are discovered to be weak in Raman caterers.
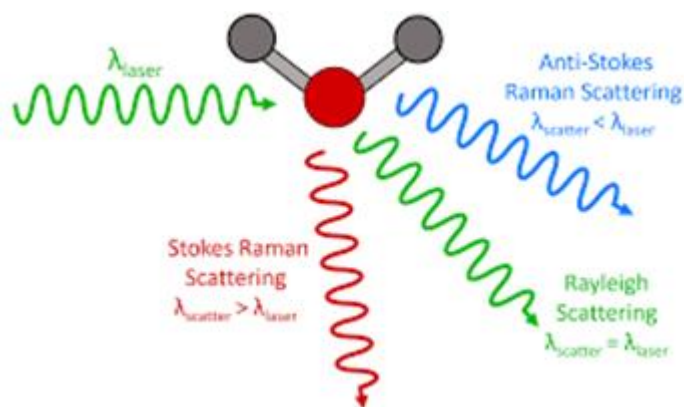
Fig 25 Principle of Raman Scattering

Analyzing average size and size dispersion is one of its benefits. Smaller cross sections, the required for intense laser stimulation, the lack of spatial precision in characterizing nanotechnology applications, and the need for several samples to provide enough RS signals are drawbacks of RS. Surface-enhanced RS (SERS) deployment has a significant impact on RS signals in addition to RS. Tip enhanced Raman spectroscopy (TERS) employs an aperture with a less metallic tip than that of the optical fiber in order to achieve surface enhancement in Raman signals. Comparatively speaking to RS, SERS, and TERS, RS gives details on topological data in addition to the structural, chemistry, and electrical characteristics of the nanomaterials. [71,72]

- *Nuclear Magnetic Resonance Spectroscopy (NMR):*
  Nuclear magnetic resonance is used to work with atomic nuclei that have magnetic moments and angular momentum when exposed to an external magnetic field (NMR). NMR is a method for transformative molecular characterization and offers a wealth of information about the chemical surroundings of atomic nuclei. NMR is used to study chemical structure, reactions, and even dynamics. The distinct spectral lines of nuclei in various habitats are seen using NMR, which is incredibly sensitive to the magnetic environment of nuclei. NMR deals with atomic nuclei with magnetic moments and angular momentum that are exposed to a magnetic field and magnetically arranges materials. The atoms' nuclei, which have magnetic characteristics that may be used to reveal chemical information, are what cause the NMR phenomena. Dendrimers and fullerene derivatives' physiochemical properties, such as their structure, purity, and functionalities, are examined. Recent research has used pulsed field gradient NMR to measure the diffusivity of nanomaterials while also examining size and species interactions. Some of the drawbacks include smaller sample preparation volumes, poor detection sensitivity, and time requirements. [73-76]
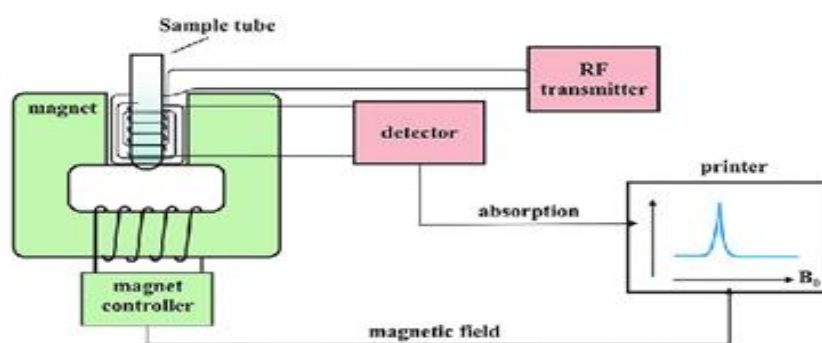
Fig 26 Principle of NMR

- *Mass Spectroscopy (MS):*
  Mass spectrometry is an analytical method used to determine the mass, elemental content, and chemical makeup of the molecules. Numerous physiochemical properties of nanomaterials, including mass, composition, and structure, may be identified using different MS methods. The foundation of MS is how it uses the mass-to-charge ratio to discriminate between charged particles of various masses. It provides excellent detection accuracy and sensitivity. MS is now employed for characterization and quantification in the domains of nanotechnology, environmental research, and toxicological investigations. The elements are vaporized, atomized, and ionized in inductively coupled plasma (ICP) using MS, which also offers elemental chemical analysis. Combining these methods provide accurate measurements of the number of metallic nanoparticles and the elemental composition of their impurities in nonmetallic nanoparticles. High accuracy and precise measurement are provided. Some drawbacks have been highlighted, including the price of the equipment and the absence of databases for identifying species. [61,77]

Fig 27 Principle of Mass Spectroscopy

- *Zeta Potential Analysis:*

In an ionic solution, charged particles and dipolar ions form a thin layer known as the "stern" layer, with a diffusive layer at the outer region that contains loosely connected ions. Electric double layers are caused by these two layers. The movement of a charged particle shears ions, causing them to migrate in the diffused layer rather than remaining in the bulk dispersion on the outer layer. Zeta potent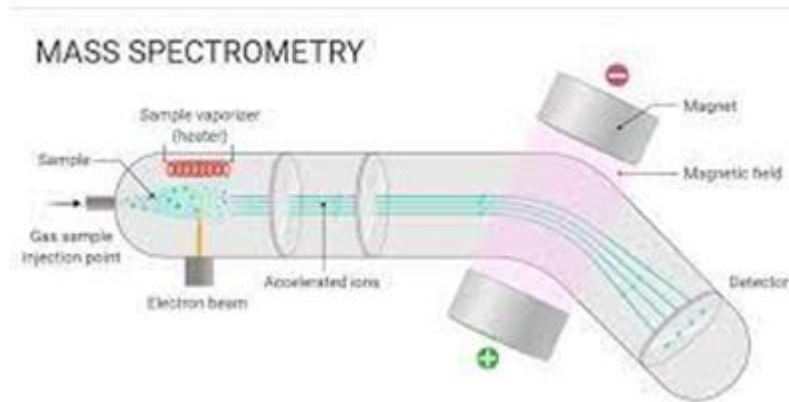ial, often referred to as shear surface electric potential, is typically calculated by the speed of charged species moving toward the electrode in a sample solution with an external electric field. It is necessary to maintain a 30-mV zeta potential. The effects of being below 30 mV include aggregation, instability, and coagulation. Zeta potential is measured by electrophoretic light scattering. This gauges a charged particle's liquid-state velocity. The main issue with this approach is an electroosmotic effect, which decreases accuracy. Zeta potential measurement has the drawback of being affected by environmental changes, such as pH, ionic strength. [78-80]
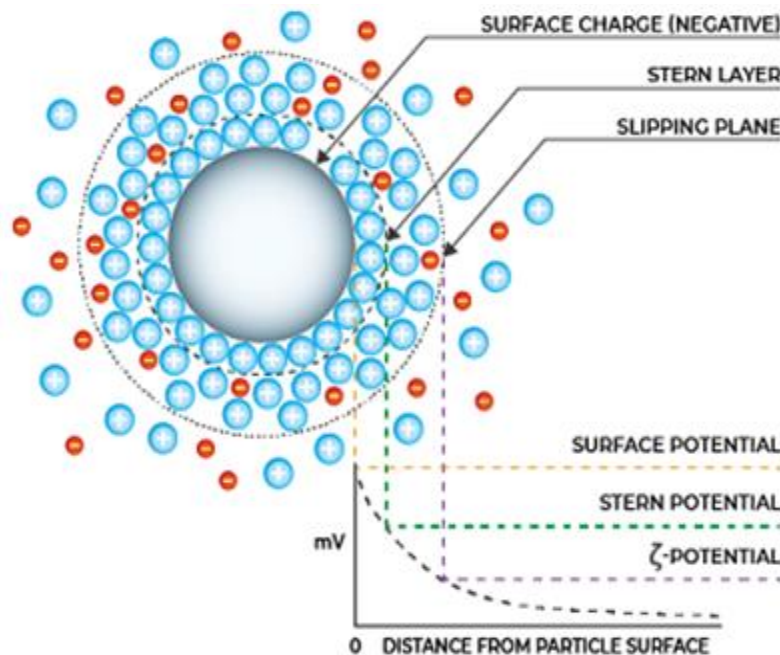
Fig 28 Zeta Potential Measurement

- *X-Ray Diffraction (XRD):*

Diffraction methods are used to determine the particle and structure's average size. Variations in crystal structure, phase quantification and identification, crystallite size and shape, distortion of the lattice, size and periodicity of noncrystalline, orientation, and other such information are examples of this kind. The goniometer is positioned on the crystal that will be measured, and it is rotated gradually while being blasted with X-rays. The two-dimensional pictures are transformed into three-dimensional images using the Fourier transform (FT). If the crystals are too tiny, errors or poor resolution might result. X-rays are electromagnetic radiation, and crystals are thought to be arrays of atoms. A caterer array creates spherical waves of caterer arrays. Bragg's law states that the waves are constructively added directions.

2d sin θ = nλ

where n is an integer, is the incidence angle, d is the spacing between diffracting angles, and d is the wavelength of the beam.

A well-known method, X-ray diffraction (XRD), produces high-resolution pictures at the atomic scale. The drawbacks have been observed, such as the sample's accessibility in just one confirmation condition and its low intensity as compared to electron diffraction. [77, 81]

Fig 29 XRD Analysis

❖ *Photo Catalytic Activity of Nano Materials:*

Photocatalysis is a photo-activated chemical reaction occurring when free radical mechanisms are initiated as contact is made between the compound and photons that have sufficiently high energy levels. Redox reactio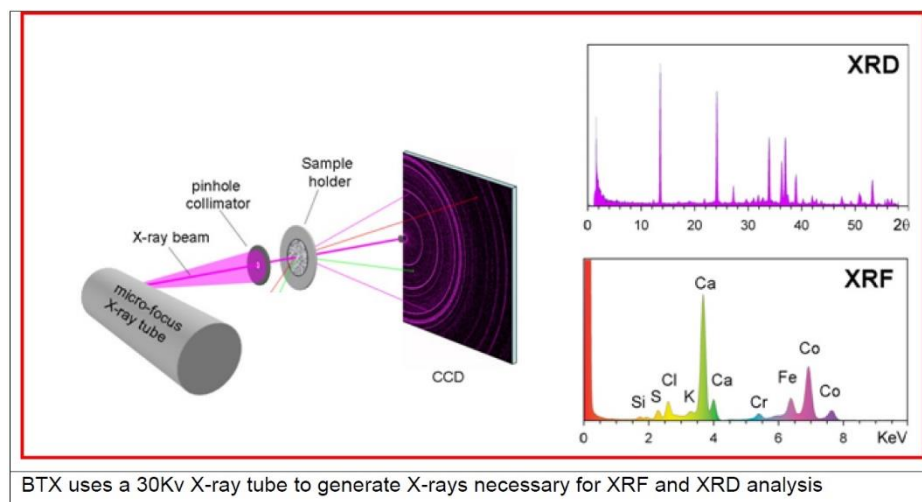ns that occur at the surface of a semiconductor material when exposed to UV or visible light are the basis for the phenomena known as photocatalysis. The catalyst's capacity to produce electron-hole pairs, which subsequently take part in a redox reaction to produce hydroxyl and superoxide radicals, which can then undertake secondary reactions, is what determines how photo catalytically active it is. [82]
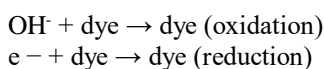
A. Mechanism of Photocatalysis:

The energy difference between valence band (HOMO) and conduction band (LUMO) is known as band gap. Semiconductors function as photocatalysts because they can conduct electricity even at ambient temperature in the presence of light. The energy of photons is absorbed by an electron (e) in the valence band of a photocatalyst when it is exposed to light of the desired wavelength (enough energy). This electron is then stimulated to the conduction band. In this process, the valence band develops a hole (h+). This process creates the photo-excitation state and produces the e+ and h+ pair. This excited electron is employed to reduce an acceptor, in which a hole is employed to oxidize molecules of the donor. The significance of photocatalysis resides in the fact that a photocatalyst simultaneously creates an oxidation and a reduction environment. [83]

When the incoming light strikes the target semiconductor (with energy higher than or equal to bandgap), an electron and hole pair is produced, and they go in the direction of the photocatalyst's surface, where redox reactions take place with the substances bonded to its surface. The water molecules are oxidized by the holes to create hydroxyl radicals, which are subsequently reduced by the electrons to make O2 from the dissolved oxygen in the water.

$Photocatalyst + h\,\gamma \rightarrow e - + h^+$
$H_2O + h^+ \rightarrow OH- + H^+$
$O_2 + e - \rightarrow O_2^-$
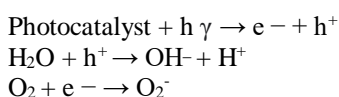
The redox processes of the dye molecules were triggered by the hydroxyl and O2 ions, which led to the formation of smaller compounds and dye decolorization. When H^+ ions are present, the superoxide anion radicals that are created react with them to form additional OH^- radicals.

$OH^- + dye \rightarrow dye$ (oxidation)
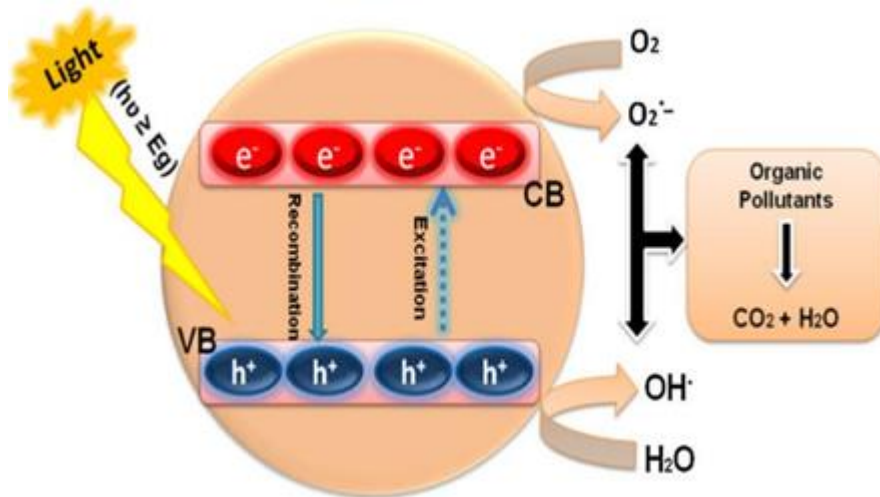$e - + dye \rightarrow dye$ (reduction)

Fig 30 Basic Mechanism of Photocatalysis

Without dissolved oxygen and H_2O molecules, photocatalysis of dye compounds is not feasible since they result in the generation of OH^- radicals. Numerous semiconductors with photosensitive characteristics, including ZnS, CdS, ZnO, and TiO_2, have also been investigated for use in photocatalysis. However, the main issue is that most semiconductors have broad bandgaps, which only absorb UV light energy, which makes only 4% of sunlight. Another issue is the rapid recombination of holes and electrons, which reduces the effectiveness of the photocatalyst. A novel family of materials with a perovskite structure of formula ABX_3 that was introduced for photocatalysis is currently gaining relevance in which A and B are the different size cations where A is generally greater than B in size. These cations make it easier to comprehend how the crystals behave. The X-anion may be an oxide or a halide. The ideal ABX_3 perovskite material has cubic symmetry and belongs to the space group Pm3m. Its B and A cations are each sixfold coordinated and is surrounded by an octahedron of X-anions. The lattice distortions in the ABO_3 perovskite materials cause the transfer of many crystal phases, including the monoclinic, tetragonal, triclinic, and orthogonal phases. Varying optical and electrical characteristics are produced by the different degrees of orientation. Due to its wide range bandgap, which can be adjusted, and their photophysical characteristics with respect to A and B cations, ABO_3 materials are superior to other photosensitive semiconductors for photocatalysis. [84]

B. Types of Photocatalytic Reactions:
Based on how the reactants appear physically, two types of photocatalytic reactions may be distinguished.

➢ *Homogeneous Photocatalysis:* This type of photocatalytic reaction occurs when the semiconductor and reactant are both in the same phase, such as a gas, solid, or liquid.
➢ *Heterogeneous Photocatalysis:* These photocatalytic processes are referred to as heterogeneous photocatalysis when the semiconductor and reactant. [83]

C. Determination of Photocatalytic Activity of $SiO_2@TiO_2$ core shell nanocomposite:
The variation of intensity of absorption may noticed by UV visible spectroscopy that observed by the photocatalytic degradation of methylene blue acts as model pollutant. The result provide only 10 percent degradation of photo catalyst in which core shell $SiO_2@TiO_2$ gives efficient photocatalytic activity that rises with increasing percentage of titania but uncoated SiO_2 in unable to act as photocatalyst. The following figure resembles the photocatalytic activity of TiO_2 photocatalyst. [85]
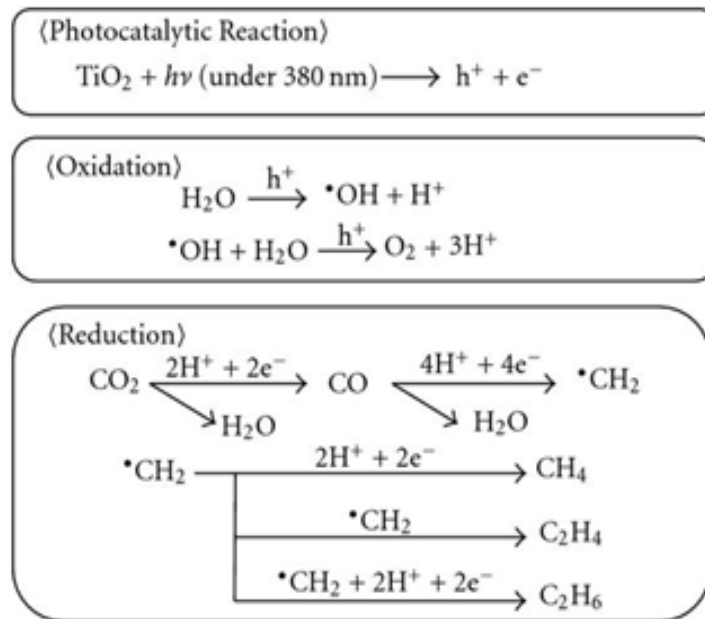
Fig 31 Reaction Mechanism

D. Photocatalytic Activity of CdS Nano particle:

The prepared nano particle resembles photo catalytic activity by the photodegradation of methylene blue, carried in a photochemical reactor at 25^0 C with water supplying system in which 1 c is required through the experiment, followed by magnetic stirring and aeration through the pump in order to provide sufficient oxygen and completeness of the reaction. The result shows that the photodegradation of MB by CdS obtained in presence of RTIL is greater than that of prepared sample in water and as a result 2degradation is completed within 220 minutes in pure RTIL when the composition of aqueous solution of RTIL and pure RTIL remains 1:1. Since there are some factors on which the photocatalytic activity depends including surface area, crystal structure, particle size, band gap energy etc. UV light can be absorbed efficiently in small sized materials with large surface area. Again, efficiency of photocatalysis can be controlled by limiting factor, indicates the rapid recombination of photo generated electrons and holes in semiconductor particles. Recombination rate of CdS in presence of RTIL will be lower than the prepared sample due to its reduced size as well as the photocatalytic activity of CdS decreases as compared to the sample in water. [86]

Fig 32 Photocatalytic Degradation of CdS

➢ *Reaction Mechanism Involve During the Photocatalytic Degradation:*

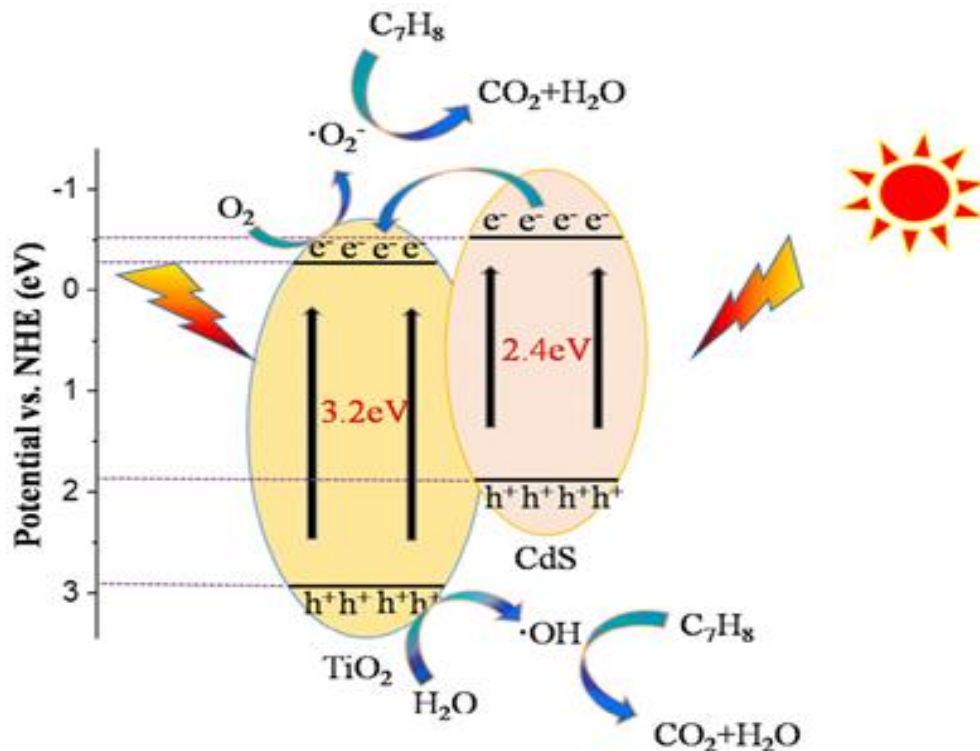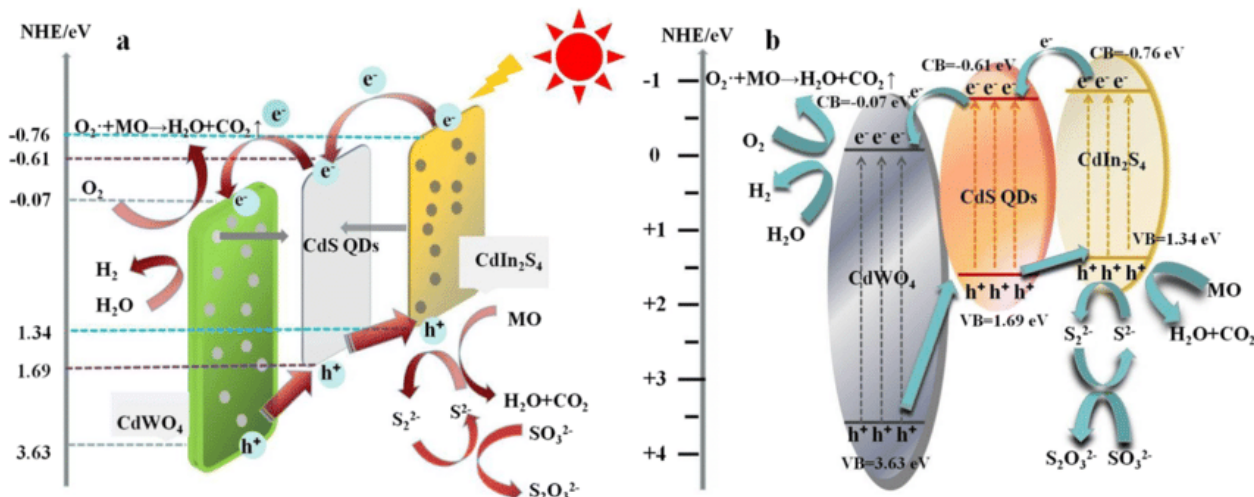Fig 33 Reaction Mechanism During Photocatalytic Degradation of Cds

CHAPTER THREE
CONCLUSION

Nanomaterials draw attention due to their unique physical, chemical, and mechanical properties from bulk solids and molecules. Nanoparticles make a connection between bulk materials and atomic structures. Small size, high surface area, easy blend with liquids, deep access to cells, strength, and ductility are some of the advantages found in nanomaterials. Some challenges, such as safety-related issues in exposure to engineered nano materials mainly in air and in water, loss of jobs in manufacturing and farming and easy availability of automated weapons are current concerns in the field of nanomaterials. Ultrasound is a unique technique related to cavitation (extreme high local temperatures (around 5000 K) and pressures (over 1000 atmospheres) generated in a liquid phase) which is nowadays a well-regarded eco-environmental technology in organic synthesis. Increase in demand and requirements has led to development in the field of nanomaterials. Characterization of nanomaterials is necessary to analyze the properties of nanomaterials. Photocatalysis is a photo-activated chemical reaction occurring when free radical mechanisms are initiated as contact is made between the compound and photons that have sufficiently high energy levels. A brief discussion about ultrasonication method for the synthesis of nano particles including its mechanism, applications, advantages, disadvantages is provided in this chapter. Overall, the chapter also includes several characterization techniques of synthesized nano particles in details along with their photocatalytic activity.

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