Lecture 4

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Characterization of Nanomaterials

1. Characterization of synthesized nanoparticle is an essential step to understand and attest the successfulness of synthesis protocol. Despite the approach used for synthesis, the nanoparticles are generally characterized for various physico-chemical properties viz. size, shape, surface properties, phase constitution and microcrystal structure. Characterization refers to the study of material's features such as its composition, structure,& various properties like physical, electrical, magnetic etc. Nanoparticle is a microscopic particle whose size is measured in nanometers (nm). ¬ These particles can be spherical, tubular, or irregularly shaped and can exist in fused, aggregated or agglomerated forms.

General techniques used for the characterization of nanoparticles are

- Transmission electron microscopy (TEM)
- Scanning electron microscopy (SEM)
- Atomic force microscopy (AFM)
- Dynamic light scattering (DLS)
- X-ray photoelectron spectroscopy (XPS)
- Powder X-ray diffraction (XRD)
- Energy dispersive X-ray analysis (EDX)
- Fourier transform infrared spectroscopy (FTIR)
- Thermogravimetric analysis (TGA)
- Zeta potentiometer
- Ultraviolet-visible spectroscopy
- and nuclear magnetic resonance (NMR).

Table summarizes various techniques used for the physicochemical characterization of nanoparticles

Parameter of nanoparticles	Technique
Nature	Ultraviolet-visible spectroscopy (UV-vis.)
	X-ray diffraction (XRD)
	Energy dispersive X-ray analysis (EDX)
Particle size	Transmission electron microscopy (TEM)
	Dynamic light scattering (DLS)
	Atomic force microscopy (AFM)
	Laser diffractometry

Table summarizes various techniques used for the physicochemical characterization of nanoparticles

Parameter of nanoparticles	Technique
Charge determination	Zeta potentiometer
Chemical analysis	X-ray photoelectron spectroscopy (XPS)
	Fourier transform infrared spectroscopy (FTIR)
	Scanning electron microscopy (SEM)
	Thermogravimetric analysis (TGA)
	Static secondary ion mass spectrometry

1. The various structural characterization methods that are most widely used in characterizing nanomaterials and nanostructures. Structural Characterization: Characterization of nanomaterials and nanostructures has been largely based on the surface analysis techniques and conventional characterization methods developed for bulk materials.The structural characterization techniques include:X-ray diffraction (XRD), Small angle X-ray scattering (SAXS) ,Scanning electron microscopy (SEM) ,Transmission electron microscopy (TEM) ,Scanning probe microscopy (SPM) ,Gas adsorption

2. Chemical characterization is to determine the surface and interior atoms and compounds as well as their spatial distributions. (Optical spectroscopy ,Absorption and transmission spectroscopy ,Photoluminescence (PL) ,Electron spectroscopy ,Ionic spectrometry ,Colorimetry ,Fluorescence , Mass Spectrometry

General techniques used for the characterization of nanoparticles are

Scanning electron microscopy (SEM) : The SEM is an instrument that produces a largely magnified image by using electrons instead of light to form an image. A beam of electrons is produced at the top of the microscope by an electron gun. The electron beam follows a vertical path through the microscope, which is held within a vacuum. The beam travels through electromagnetic fields . In a typical SEM, the beam passes through pairs of scanning coils or pairs of deflector plates in the electron column to the final lens, which deflect the beam horizontally & vertically. The image displayed is therefore a distribution map of the intensity of the signal being emitted from the scanned area of the specimen.

- Advantages: 1- Bulk-samples can be observed and larger sample area can be viewed, 2- generates photo-like images, 3- very high-resolution images are possible 4- SEM can yield valuable information regarding the purity as well as degree of aggregation
- Disadvantages: 1- Samples must have surface electrical conductivity 2- non- conductive samples need to be coated with a conductive Layer 3- Time consuming & expensive. 4- Sometimes it is not possible to clearly differentiate nanoparticle from the substrate. 5- SEM can’t resolve the internal structure of these domains.

Scanning tunnelling microscope (STM) The STM is a fundamental tool in nanoscience and nanotechnologies. It is used in both industrial and fundamental research to obtain atomic-scale images of metal and semiconducting surfaces (Figure 29). It provides a three-dimensional profile of the surface roughness, allowing the observation of surface defects and the determination of the size and conformation of molecules and aggregates. Another astonishing property of the STM is that it can be used to manipulate (move!) individual atoms, trigger chemical reactions.

- The Atomic Force Microscope (AFM) was developed specifically to overcome the intrinsic limitations of the STM, which is not suitable for imaging surfaces coated with biological entities such as DNA or proteins. The AFM operates in air and not under a vacuum. Some versions of the instrument also allow operation in liquid, which is very advantageous when imaging biological samples that often need buffers to remain biologically active. The AFM measures the interaction force (attractive or repulsive) between the probe and the surface.. The vertical movement of the probe is recorded to create a topographic map of the surface under study.

X-ray diffraction (XRD) is a very important experimental technique that has long been used to address all issues related to the crystal structure of solids, including lattice constants and geometry, identification of unknown materials, orientation of single crystals, preferred orientation of polycrystals, defects, stresses, etc.

Recently, a new technique namely nanoparticle tracking analysis (NTA) has been reported which allows direct tracking of nanoparticles based on their Brownian motion, which allow estimation of individual nanoparticles in solution.

In addition, isoelectric focusing electrophoresis (IEF) has also been reported to analyze the size distribution of gold nanoparticles

Nanomaterials, which are similar to biological moieties in scale, can be used as diagnostic and therapeutic nanomedicines Compared to their bulk material counterparts, the distinct physicochemical properties of the nanomaterials, such as size, surface properties, shape, composition, molecular weight, identity, purity, stability and solubility, are critically relevant to particular physiological interactions These physiological interactions may provide benefits in medical applications, including improvements in efficacy, reduction of side effects, prevention and treatment.In this context, it is important to understand how the different physicochemical characteristics of nanomaterials affect their *in vivo* distribution and behavior.

- The different techniques used for characterization of nanomaterials, based on their different physicochemical properties of the nanomaterials, such as
- 1- Size :In engineered nanomaterials, size is a crucial factor that regulates the circulation and navigation of nanomaterials in the bloodstream, penetration across the physiological drug barriers, site- and cell-specific localization and even induction of cellular responses.

- **2-Surface properties:**

Many characteristics of nanomaterial interfaces are functions of atomic or molecular compositions of the surfaces and the physical surface structures that respond to the interactions of the nanomaterial with surrounding species . From the aspect of nanomedicine, these characteristics are considered the elements of surface properties in the environment of biological fluid.

- **3- Shape:**

In addition to size and surface properties, the shape of nanomaterial can play an important role in drug delivery, degradation, transport, targeting and internalization . Efficiency of drug delivery carriers was highly influenced by controlling the shapes of the carriers. Furthermore, flow and adhesion of drug delivery carriers throughout the circulatory system and the in vivo circulation time of the nanomedicine can be controlled by modulating the shapes of drug-loaded nanomaterials.

- **4. Composition and purity**

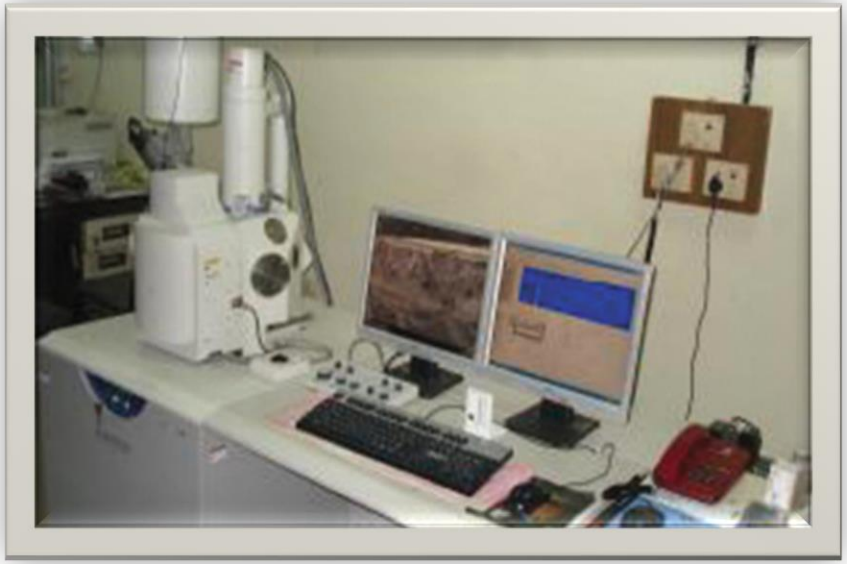
A broad variety of nanomaterials are utilized in the production of approved or potential nanomedicines. These nanomaterials can be categorized by their structural types, such as NP and its derivatives, liposome,, dendrimer/, virosome, emulsion, quantum dot, fullerene, carbon nanotube and hydrogel, and each type may consist of polymers, metals and metal oxides, lipids, proteins, DNA or other organic compounds. Composition of a nanomaterial affects transport, delivery and biodistribution. In biomedical applications of nanomaterials, there may be a need to combine two or more types of nanomaterials to forma complex such as a chelate, a conjugant or a capsule. Consequently chemical composition analysis of the nanomaterial complex is more complicated than that for a single entity.

- **5. Stability**

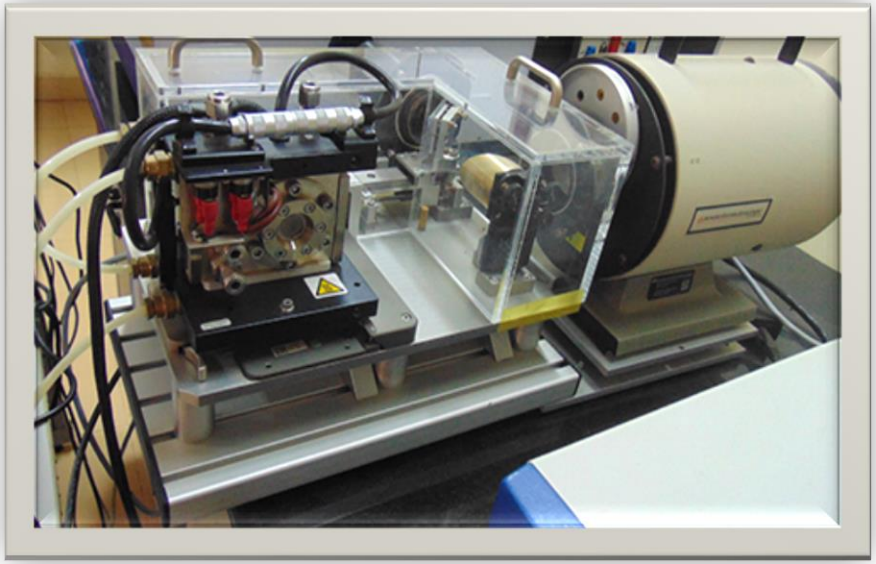
Pharmaceutical stability refers to retaining the same properties for a period of time after the pharmaceutical is manufactured. Similar to conventional single-molecule pharmaceuticals, the stability of nanomedicines may be affected by one or more factors, such as temperature, moisture, solvents, pH, particle/molecular size, exposure to different types of ionizing and non-ionizing radiation, enzymatic degradation and even the presence of other excipients and impurities The stability of nanomaterial may impact its corresponding toxicity; for instance, a number of studies have shown that quantum dot cytotoxicity might be induced during synthesis, storage or even in vivo by oxidative or photolytic degradation of quantum dots

Transmission_Electron_Microscope

X-ray_Photoelectron_spectrometer



Scanning_Electron_Microscope



FTIR_spectromete