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College of Sciences

Biology Department

Nanotechnology

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MSc. Nano-biotechnology

Lab. 5: Characterization of Nanoparticles

• Characterization refers to study of materials features such as its: **Composition - Structure** – **Physical** – **Electrical - Magnetic** and etc.

• Nanoparticle properties vary significantly with **size** and **shape**.

• Characterization of NPs will depend on the **type of NP** and **its intended purpose**.

• Physiochemical characterization include (Non-imaging and Imaging)

Tools used in non-imaging physiochemical characterization of NPs

1. UV-Vis Spectrophotometer (e.g. Nanodrop)

• Allows identification, characterization, and analysis of metallic NPs (e.g., silver, gold).

• It can be used to determine size and evaluate the dispersion and local structure of NPs synthesized with metal oxides, selenides (Se^{2-}), and sulfides.

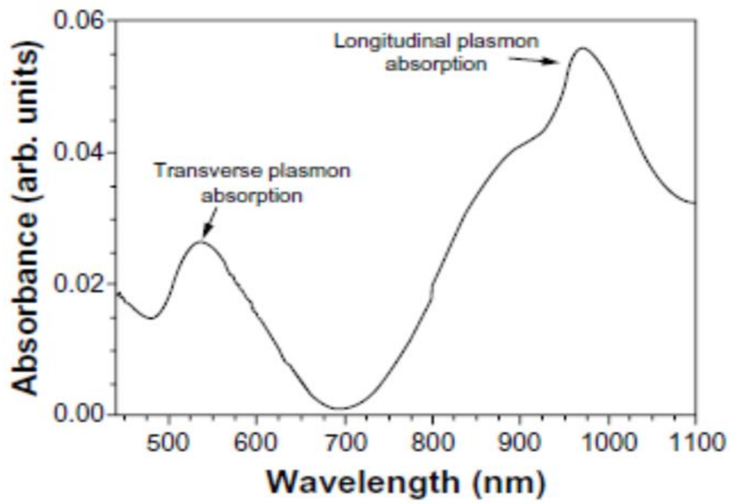
• The NPs (**especially gold NPs**) have very high **extinction coefficients*** when their diameter is > 10 nm, **they have a very high absorbance**.

*__Extinction coefficient__: refers to several different measures of the absorption of light in a medium.

• It is difficult to measure absorbance of highly concentrated samples using a cuvette with 1 cm path length.

In the figure: (للاطلاع)

Spherical Gold NPs has higher absorbance around 540 nm, While Gold nanorods around 970nm.



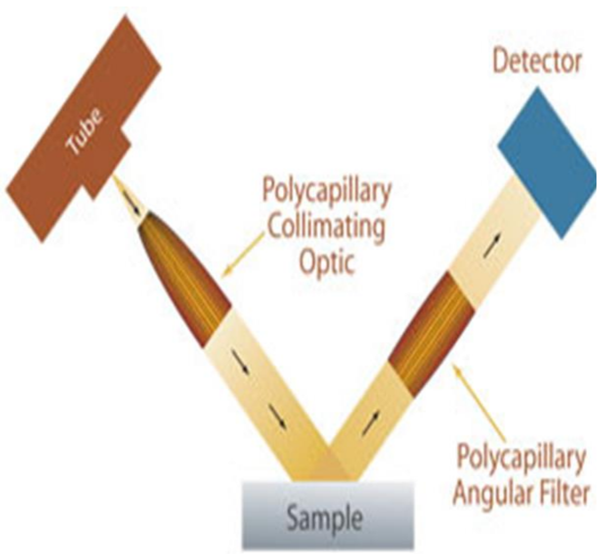
2. X-ray Diffraction (XRD)

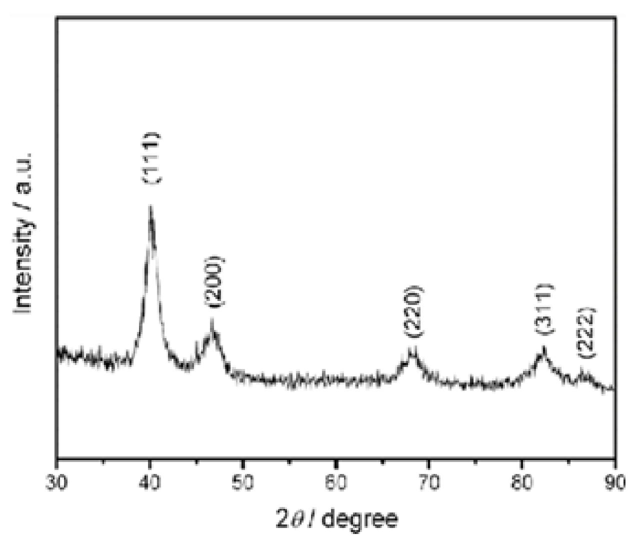
• Used to study the structural identification of nanoparticles, composition and physical properties of materials

1. **Amorphous:** The atoms are arranged in a random pattern.
2. **Crystalline:** The atoms are arranged in a regular pattern.

• When X-rays **interact** with a crystalline substance (Phase), one gets **a diffraction pattern.**

• The X-ray diffraction pattern of a pure substance like a **fingerprint** of the substance.

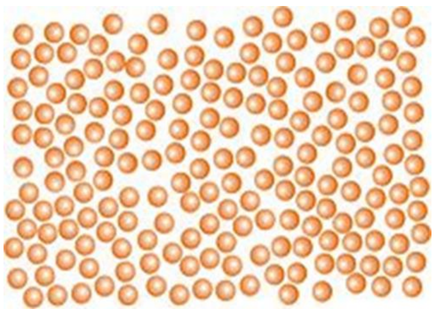




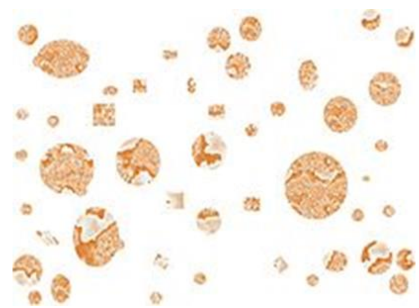
3. Dynamic light scattering (DLS)

Dynamic light scattering measures **variation in scattered intensity with time at a fixed scattering angle (typically 90º)**, while Static light scattering (SLS) measures scattered intensity as a function of **angle**.

DLS is an important tool for characterizing the **size of nanoparticles in solution**.



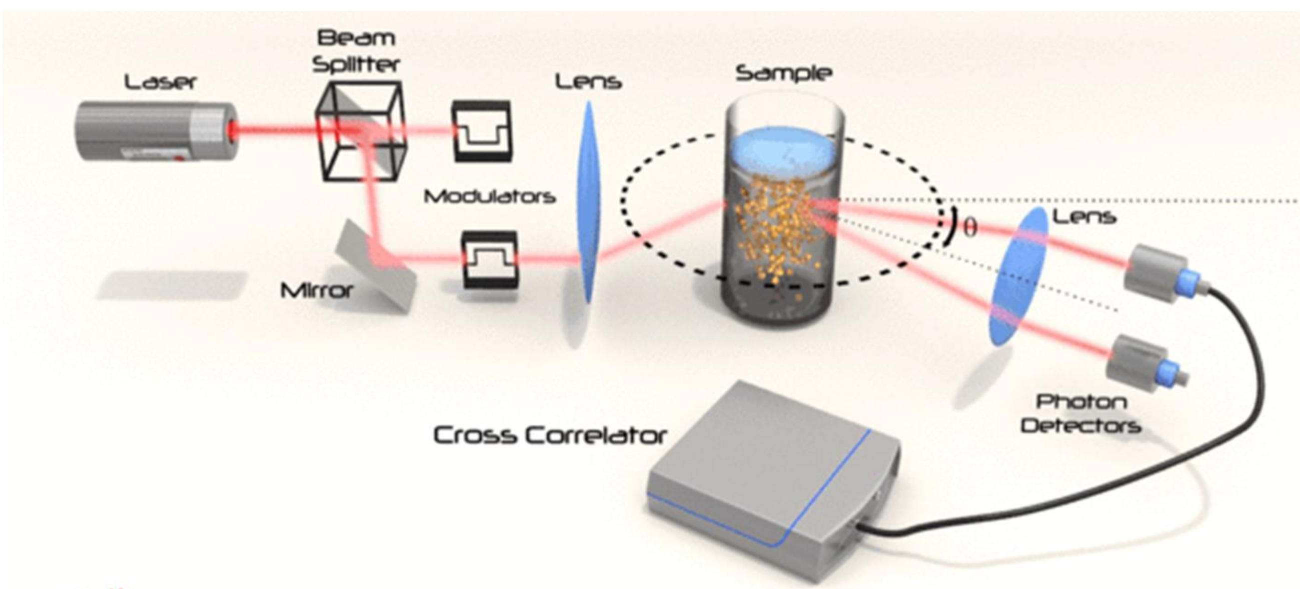
Monodisperse(uniform)



Polydisperse (non-uniform)

►DLS measures hydrodynamic size, size distribution, and polydispersity.

► DLS is a valuable tool for **determining** and measuring the **agglomeration** state of nanoparticles as a function of time or suspending solution.



4. Imaging of Nanomaterials

Electron Microscopy:

- Uses some type of probe that generates an image by physically scanning the sample surface in a horizontal scan pattern

- Depending on the type of microscope, several different surface characteristics can be analyzed.

Some types of electron microscope

1. Scanning Electron Microscope (SEM)
2. Transmission Electron Microscope (TEM)
3. Atomic Force Microscope (AFM)
4. The Scanning Transmission Electron Microscope (STEM)

1. Scanning Electron Microscope (SEM)

• Type of **Electron microscope** that **produces images** of a sample by scanning the surface with a focused beam of electrons.

• The electrons interact with atoms in the sample.

• Producing various **signals** that contain **information** about the surface **topography** and **composition** of the sample

• Generates **photo-like images.**

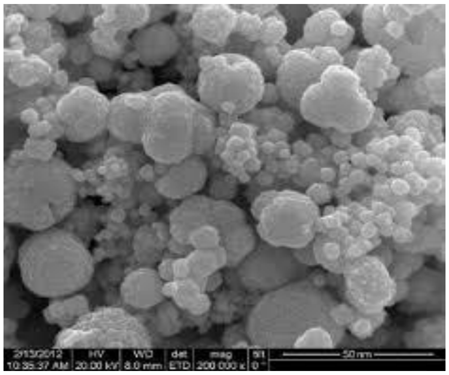
Limitations:

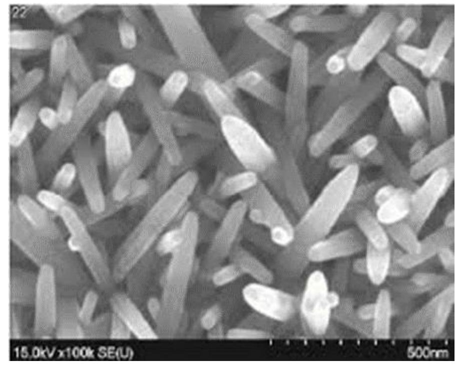
1- Samples must have **surface electrical conductivity**.

2- Non-conductive samples need to be **coated with a conductive layer** (Like

silver paste).

JEOL 6700F Ultra High Resolution Scanning Electron Microscope





SEM-Image for **Spherical nanoparticles** SEM-Image for **Nanorods**

2. Transmission Electron Microscope (TEM)

Image is generated based on the interaction pattern of electrons that transmit through the specimen.

***Advantages*:**

High-resolution , **3-D image** construction possible but aberrant .

Additional analysis techniques like X-ray spectrometry are possible.

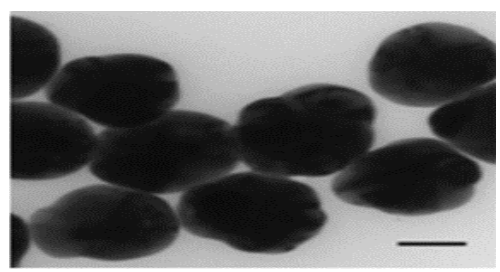
Limitations:

1- Needs high-vacuum chamber

2- sample preparation necessary

3- Mostly used for 2-D images

TEM image of Ag NPs

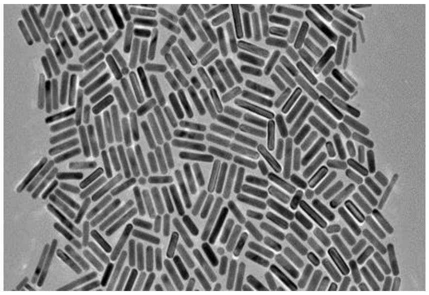


Nanomaterials shapes

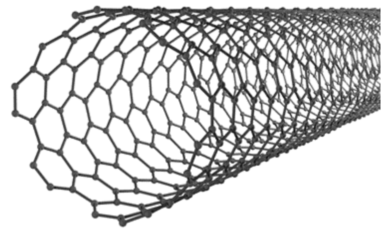
1. Nanofibres

Nanofibre is the generic term describing nano – objects with two external dimensions in the nanoscale.

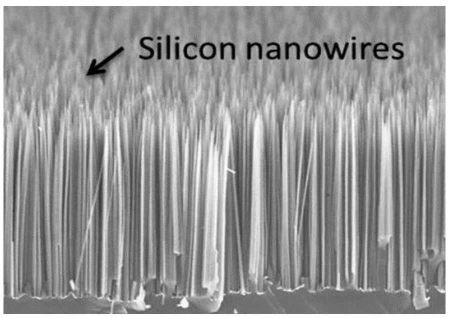
► **Nanorod**: is a rigid nanofibre.



► **Nanotube**: is a hollow nanofibre.

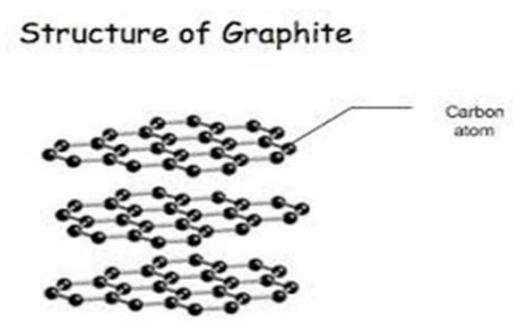


► **Nanowire**: is an electrically conducting nanofibre



2. 3. Graphene – based materials

- Graphene is an allotrope (different structural modification of an element) of carbon in the form of two dimensional, atomic –scale, hexagonal lattice in which one atom forms each vertex.
- It is the basic structural element of other allotropes including graphite, charcoal, carbon nanotubes and fullerenes (carbon in a hollow sphere, tube or other shapes.



a) Graphene:

the graphene lamellae stacked to make bulk graphite were from the ease of their detachment (like writing with graphite on paper) known to be only weakly bound to each other.

b) Carbon nanoparticles:

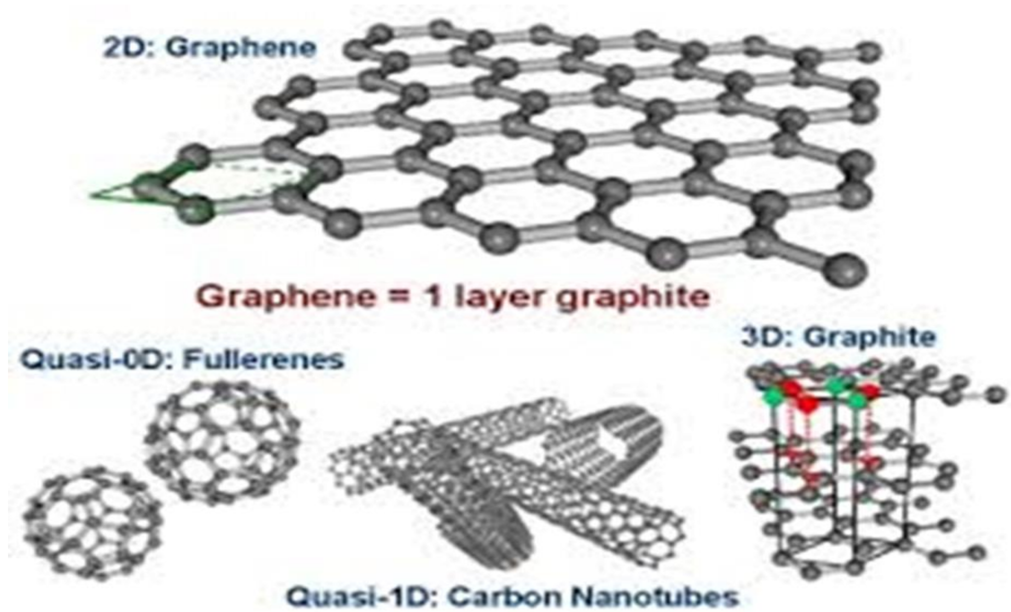
► Fullerenes can be a graphene curled up to form an enclosed spherical shell (exist as C_{60} , C_{70}) .

► They can have made in carbon arc or burning hydrocarbons methods.

c) Carbon nanotubes (CNT):

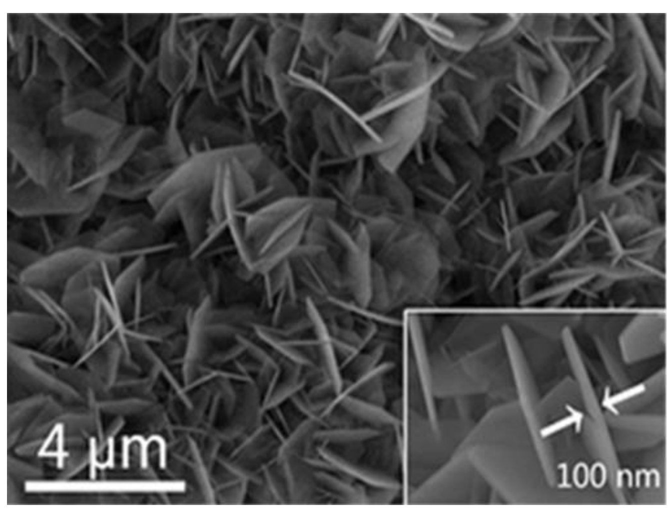
► The carbon nanotube is a seamless tube made by rolling up graphene.

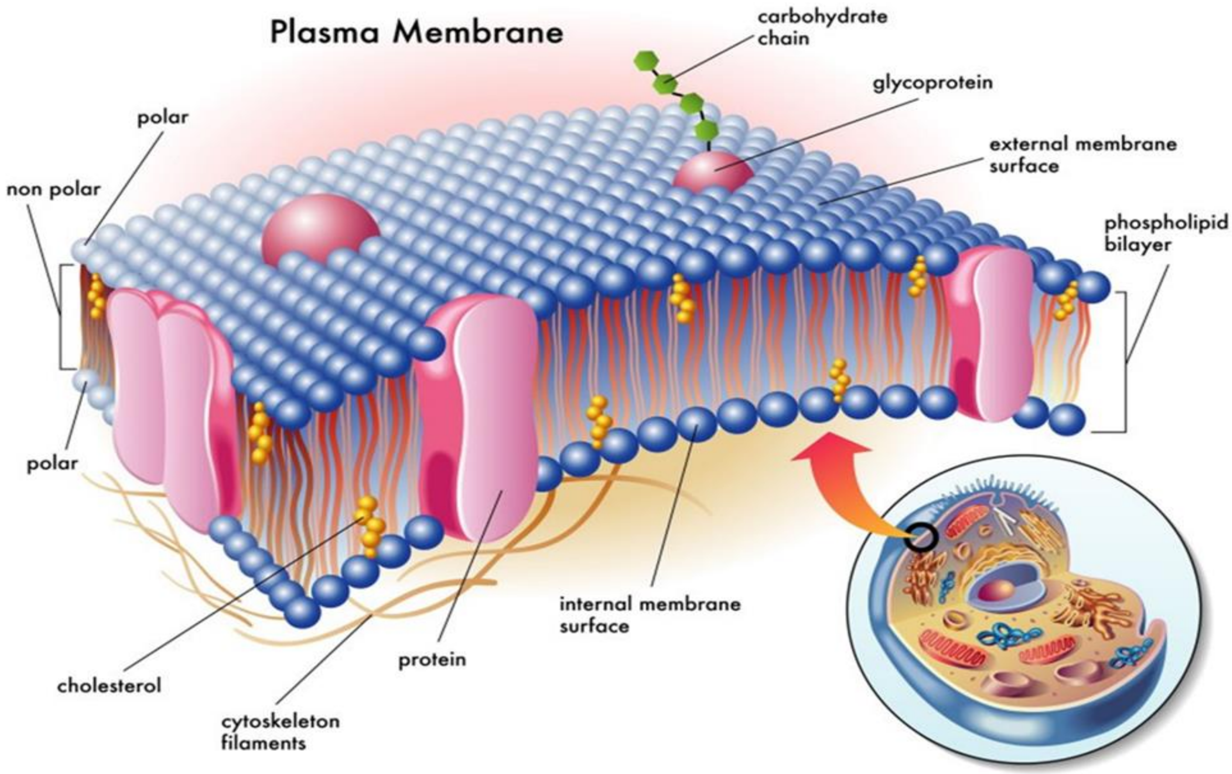
► Carbon filaments are formed by passing hydrocarbons over hot metal surface.



3. Nanoplates

Thin coating on a substratum have not been consider as nano-objects, but simply as a thin film, because typically they have been more than 100 nm thick.





Aggregation and Agglomeration of nanomaterials

► Aggregation: Attraction of nanomaterials by weak forces such as Van der Waals forces.

► Agglomeration: Attraction of nanomaterials by stronger forces like covalent bonds and ionic bonds.

● This concept explained by classical DLVO theory (Derjaguin, Landau, Verwey and Overbeek theory) which assume that the balance between repulsion and attraction potential energies control dispersed particles interaction, particles with the same charge propagation layer and particles-solvent interaction causes repulsive force, the type of interaction between particles defined by the sum of these two forces.